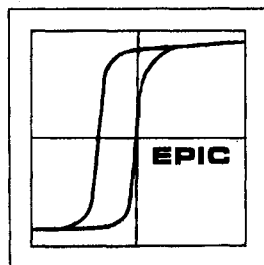


POLYIMIDE PLASTICS: A State-of-the-Art Report,

JOHN T. MILEK

S-8
October 1, 1965



ELECTRONIC
PROPERTIES
INFORMATION
CENTER

HUGHES

HUGHES AIRCRAFT COMPANY
CULVER CITY, CALIFORNIA

PREFACE

✓ This survey report on polyimide plastics has four objectives:

- (a) To obtain a state-of-the-art picture of this newly-developed family of resins with regard to all available commercial materials, as well as those in their developmental stages: their chemistry, property data, and applications.
- (b) To report ~~Hughes Aircraft Laboratory~~ test data on various polyimide materials and compare same with data found in the literature search.
- (c) To comply with an ~~Air Force~~ request to prepare a survey report on this family of plastics.
- (d) To collectively tie together the properties of the various polyimide forms which, heretofore, have been generally treated individually on a tradename basis rather than a family basis.

A literature search was instituted to obtain all available reports, bibliographies, periodical and book information. Acknowledgement is noted here for the able assistance of Mr. Jan Seremak and Harold Wilcox of the library staff in the literature search. Manufacturers were contacted to obtain catalog data, formulations, properties, and application information. Excellent cooperation and major assistance was afforded to the author by the following companies: E.I. DuPont de Nemours Co., Westinghouse Electric Corp., Research Laboratories, and the Amoco Chemical Co. The Radiation Effects Information Center, Battelle Memorial Institute, aided in furnishing irradiation effects data.

Various Materials Technology Laboratory personnel, of Hughes Aircraft Co., were interviewed to obtain laboratory test data on all the polyimide materials evaluated to date.

This report was prepared by Hughes Aircraft Company under Contract No. AF 33 (615)-2460. The contract was initiated under Project No. 7381, Task No. 738103. The work was administered under the direction of the Air Force Materials Laboratory, Research and Technology Division, with Mr. R.F. Klinger acting as Project Engineer.

The typing and artwork preparation of this report were ably accomplished by Sharon Bender and Jean Forest. The author acknowledges their patience and fortitude in an effort to make the report as "up-to-date" as possible by including last minute reference insertions.

It is unfortunate that a considerable body of Westinghouse Electric Company polyimide and polyamide-imide data were not available or releasable to the author due to certain military and government restrictions; hence, this report is lacking perhaps a vital collection of very useful and important information. However, part of it was announced in the latest available Technical Abstract Bulletin (September 1, 1965) number 65-17 released by the Defense Documentation Center. It is regretted that these data could not be incorporated in the body of the report.

SUMMARY

A state-of-the-art report has been prepared on polyimide resins and includes the polyamide-imide as well as the polyester-imide molecular species. The compilation of information is based on a comprehensive search of the literature (periodical, report, and patent) plus major vendor contacts.

The history and major materials developments are noted in the preface and patent sections. The introduction treats the molecular structure, reactant chemistry, polymerization reactions and mechanisms.

The entire property spectrum is covered: chemical, electrical, mechanical, optical, physical, and thermal to provide a comprehensive materials characterization picture of this new class of polymeric resins.

The chemical property section covers data on the effects of chemicals, solvents, fluids, fuels, and moisture on these polyimide resin materials. Included in the electrical property section are data on arc resistance, corona effects, dielectric constant, dielectric strength, dissipation factor, electrical resistivity, loss factor, and power factor of the film, SP-Polymer, laminates, binder solutions, varnishes and enamels.

The following mechanical properties data are encompassed: tensile, compressive, shear, flexural, endurance, friction and wear, plus miscellaneous (hardness, adhesive bond strength, tear strength, cut-through force, impact strength) properties. The optical properties include infrared spectral curves over wide frequency and temperature ranges and emissivity data.

Data on density, viscosity, hygroscopic and weathering behaviour are detailed in the physical properties section. The literature produced a large volume of thermal property data in view of the good characteristic high-temperature resistance of this class of resin (mostly on the H-Film) and included thermal stability, melting point, thermal expansion, thermal conductivity, specific heat, thermal degradation, weight loss, thermal life, heat distortion, flame resistance and heats of ablation. Irradiation effects on the various properties are treated in a separate property section of the report.

The molding and fabrication processes and techniques are covered in some detail and result mostly from vendor catalog instruction sheets and patent claims.

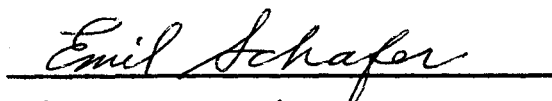
Applications are divided into electrical, mechanical, aircraft, missile and space. Electrical applications are further sub-divided into magnet wire enamels, insulation varnish coatings, electric motors, capacitors, cable and wire, computers, and miscellaneous. Mechanical applications covered include bearings and adhesive bonding.

The prices and availability section gives the current picture on individual polyimide products and their vendor suppliers as well as fabricators of polyimide cable and wire products. The tradenames and patent section contains identification information plus annotations of all U.S. patents and a listing of foreign patents.

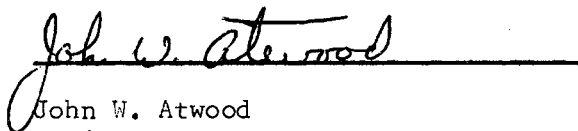
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Specifications drafted on polyimide products are summarized in the section titled "Specifications." Finally, the toxicology or health hazards involved in polyimide fabrication (or processing) and handling are dealt in a separate section.

This report has been reviewed and is approved for publication.

A handwritten signature in cursive script, reading "Emil Schafer", is written over a solid horizontal line.

Emil Schafer, Assistant Head
Electronic Properties Information Center

A handwritten signature in cursive script, reading "John W. Atwood", is written over a solid horizontal line.

John W. Atwood
Project Manager

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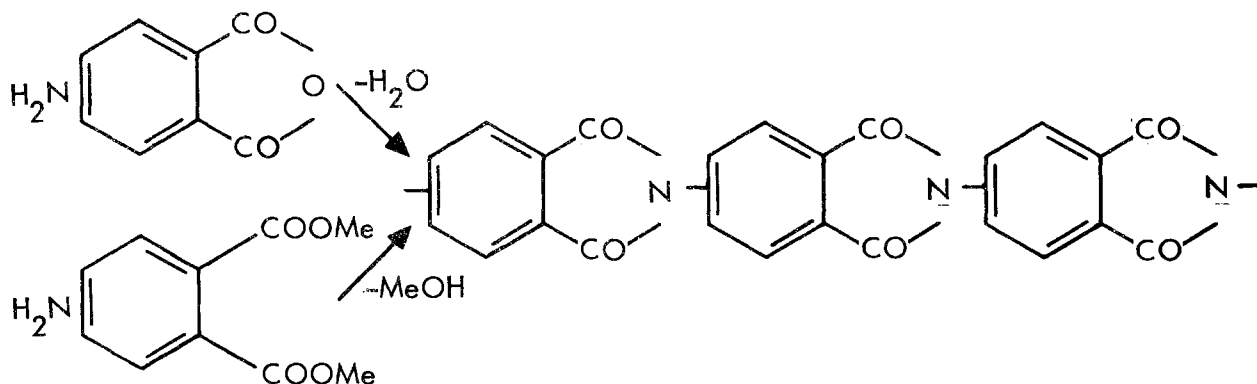
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I. INTRODUCTION AND POLYIMIDE CHEMISTRY

Polymer chemists in the past few years have concentrated on a group of aromatic polymers known as polyimides, polybenzamidazoles, and polyquinoxalines in view of their excellent thermal stability or resistance to thermal degradation which has been ascribed or related to their aromatic character. This aromatic character also gives them very high melting points and tends to make them insoluble.

This survey report is concerned only with the polyimide, aromatic polyamide-imide and polyester-imide group of polymers. The basic method for preparing polyimides consists of reacting pyromellitic dianhydride with an aromatic diamine to yield a polyamic acid. The acid is then dehydrated to yield the high molecular weight polyimide. Many variations are possible by using a variety of amino compounds and other anhydrides.

Jones and co-workers (Ref. 64) in their review of polyimides report that the first recorded synthesis of a polyimide is that of Bogert and Renshaw who observed in 1908 that 4-amino-phthalic anhydride and dimethyl 4-aminophthalate, on heating, eliminate water and methanol yielding a polymolecular imide:



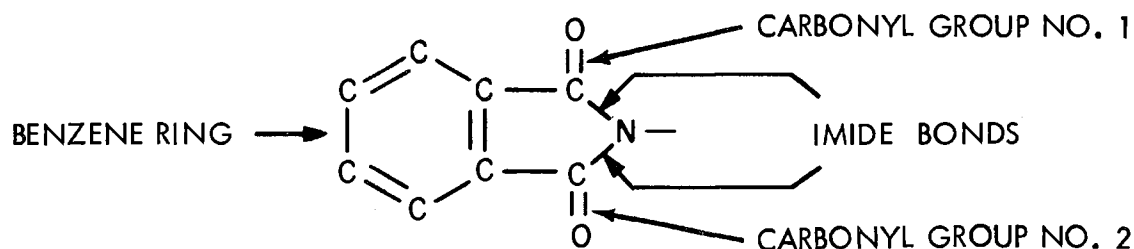
The patent literature contained references to the preparation of polyimides as early as 1945. Around 1959, the DuPont Company introduced its first polyimide polymer to the electrical industry under the tradename of "Pyre-ML" for wire enamel, coated glass fabrics, and insulating varnish applications.

R.B. Seymour in an annual review of plastics (IEC, vol. 57, no. 8, page 71, August 1965) quotes...."The present use of [polyimides] amounts to less than 1/2 million pounds, but it has been predicted its use as fiber and enamel will exceed 10 million pounds in 1970." The current availability and companies supplying the various forms of polyimides are detailed in Section V: Availability and Prices.

In reviewing the polyimide literature (including patents), it was found that a variety of abbreviations, tradenames, and code designations were used to identify or designate polyimide and polyamide-imide resin formulations. There were many causes of confusion and have been resolved by the author to his best ability at the end of the Introduction Section.

Polyimide and polyamide-imide polymers have experienced to date a normal growth (in the author's opinion) much as most plastics, although there are no official market or production statistics to prove it. Some unofficial figures are cited in Section V. of this survey report and the future indications are very bright for the polyimides.

To the non-chemist, the terms imide and amide may be somewhat preplexing. They are both amino (or ammonium) derivatives of organic compounds. The imide is a cyclic structure and characterized by a double bond linkage between one nitrogen atom and two carbonyl groups (in polymers, these latter are usually attached to a benzene ring):

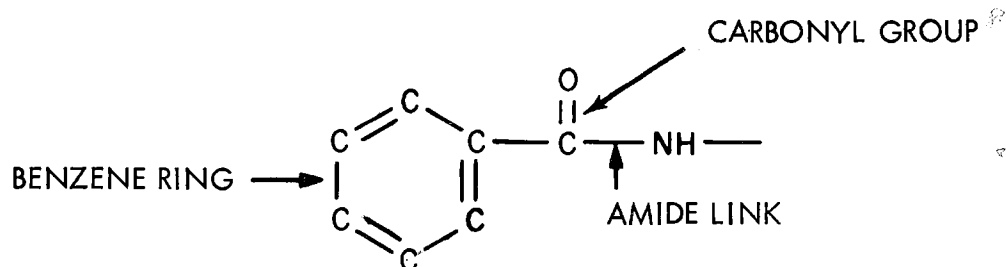


The term "imide" as employed in this report refers to a compound which differs structurally from a carboxylic acid anhydride by having the >O which separates the carboxyl groups in the anhydride replaced by >N-.

A "linear polymeric polyimide" is a linear condensation polymer in which the imide hydrogens of a di-imide are replaced by bivalent radicals which link together the bivalent residues of the di-imide molecules as recurring units of a linear polymeric chain.

The linear polymeric polyimides as a general class of polymers have been known for many years (Br. Pat. 570,858; cf also U.S. 2,149,286; 2,407,896; 2,421,024; 2,502,576).

The amide, on the other hand, has only one nitrogen atom attached to carbonyl which is sometimes attached to a benzene ring:

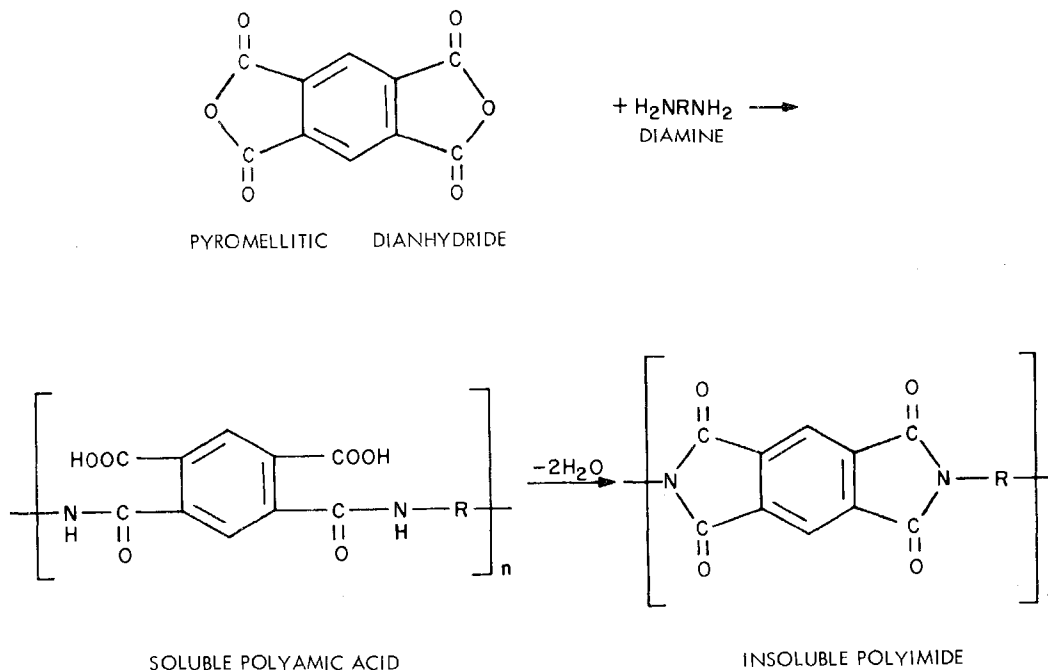


It will be noted below that the imidization process involves the formation of an amide intermediary structure or stage and then further reaction or processing to the imide stage. Subsequent polymer research and development investigations have shown that it is possible to have amide-imide combination structures depending on the utilization of di-, tri-, or tetra functional carbonyl compounds attached to the benzene ring. In the discussion section below, an attempt will be made to present all the important ramifications of polyimide and polyamide-imide chemistry and all pertinent polymerization reaction mechanisms as revealed in the patent and open literature.

The following plan or outline is being used to discuss the chemistry and polymerization of the polyimides and polyamide-imides. The basic reactants and processing steps are treated in the sequence:

- a) Dianhydrides (Reactant)
- b) Diamines (Reactant)
- c) Mixing and Proportion of Reactants
- d) Solvent System
- e) Polyamic Acid Solution (Stage)
- f) Imidization Stage (Dehydration and Thermal Heating)
- g) Polyimide
- h) Amide-Imide Co-Polymerization
- i) Miscellaneous Effects

because the general overall reaction takes the form:



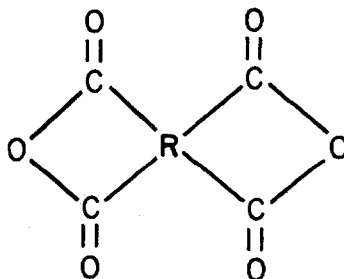
A wide range of organic dianhydride and diamine compounds have been explored in the various laboratories as evidenced in the open literature as well as in the patent literature. These programs aimed at the determination of optimum chemical structure or molecular arrangements to achieve a breed or family of stable high temperature polymers -- over long periods of time. As a result

of this research work, a relative order of thermal stability has been postulated (in order of decreasing relative thermal stability) by Freeman and co-workers at Westinghouse Electric Corporation: (ref. 46): phenyl ring>phenyl-phenyl bond>aromatic ether>aromatic amine>phenoxy silane; bezophenone diphenyl-methylene. In another paper, Frost and Bower (also of Westinghouse) report the following approximate order of thermal stability for the linking groups: imide>ether; sulfide, direct phenyl-phenyl bond>amide, ester,>methylene>isopropylidene.

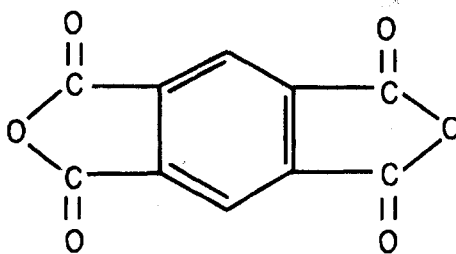
In addition to the above thermal stability requirements were the following: (a) there must be a soluble or formable intermediary or "B" stage to permit application of the polymer to service needs, (b) the material must have a high molecular weight in order to possess the desirable strength, toughness, flexibility and other "plastic" properties commonly associated with useful structural and insulating materials.

Dianhydrides

The aromatic dianhydrides are one of the necessary and prime reactants required to form polyimide polymers and are characterized by the formula:

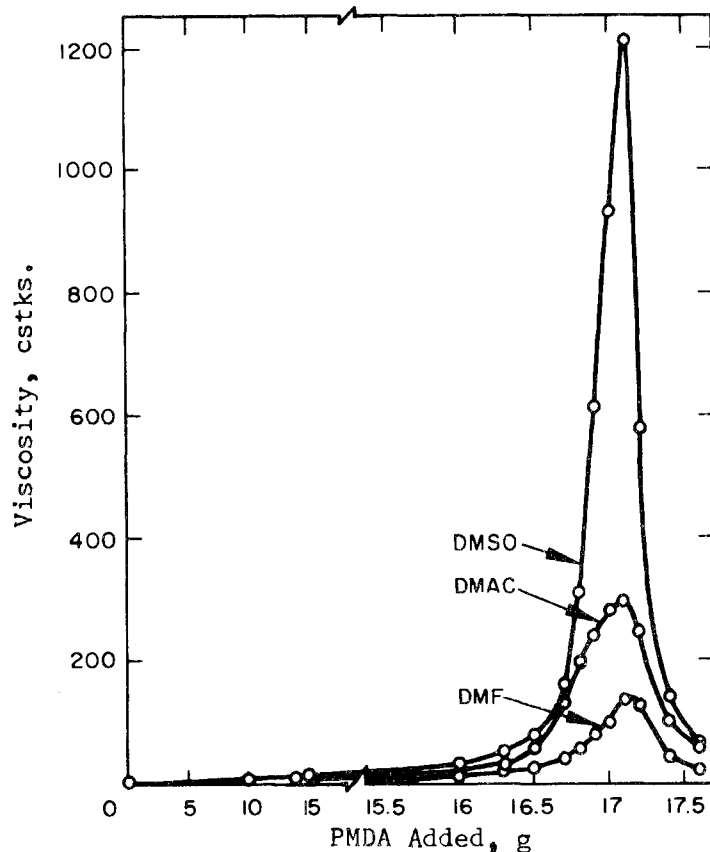


where R is a benzenoid compound. The most popular and useful dianhydride is pyromellitic dianhydride (PMDA):



which is a white powder.

Bower and Frost (Ref. 15) in their studies of the polyimide reactions showed that excess dianhydride has a deleterious effect on the viscosity of the polyamic acid solution:



Reaction of PMDA with MDA in three solvents

[Ref. 15]

Equilibrium generally occurs in the dianhydride-diamine reaction, after 78 mole percent of the anhydride are consumed (e.g. PMDA and m-PDA). Benzo-phenone tetracarboxylic dianhydride (BTDA) resin solutions were found to have similar behavior.

In the patent literature (see Section VI) are cited a wide range of other aromatic dianhydrides which can be used as starting reactant materials:

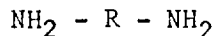
- 2,3,6,7-naphthalene tetracarboxylic dianhydride;
- 3,3', 4,4'-diphenyl tetracarboxylic dianhydride;
- 1,2,5,6-naphthalene tetracarboxylic dianhydride;
- 2,2', 3,3'-diphenyl tetracarboxylic dianhydride;
- 2,2-bis(3,4-dicarboxyphenyl) propane dianhydride;
- bis(3,4-dicarboxyphenyl)sulfone dianhydride;
- 3,4,9,10-perylene tetracarboxylic dianhydride;
- bis(3,4-dicarboxyphenyl) ether dianhydride;
- ethylene tetracarboxylic dianhydride;
- naphthalene-1,2,4,5-tetracarboxylic dianhydride;
- naphthalene-1,4,5,8-tetracarboxylic dianhydride;
- decahydronaphthalene-1,4,5,8-tetracarboxylic dianhydride;

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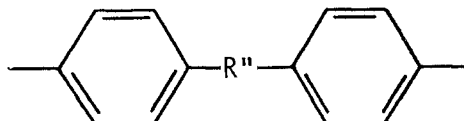
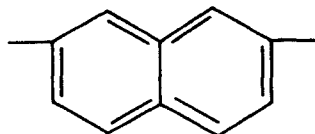
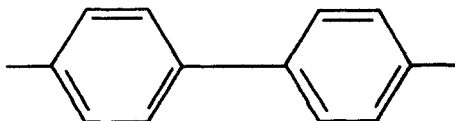
4,8-dimethyl-1,2,3,5,6,7-hexahydronaphthalene-1,2,5,6-tetracarboxylic dianhydride
2,6-dichloronaphthalene-1,4,5,8-tetracarboxylic dianhydride;
2,7-dichloronaphthalene-1,4,5,8-tetracarboxylic dianhydride;
2,3,6,7-tetrachloronaphthalene-1,4,5,8-tetracarboxylic dianhydride;
phenanthrene-1,8,9,10-tetracarboxylic dianhydride;
cyclopentane-1,2,3,4-tetracarboxylic dianhydride;
pyrrolidine-2,3,4,5-tetracarboxylic dianhydride;
pyrazine-2,3,5,6-tetracarboxylic dianhydride;
2,2-bis(2,3-dicarboxyphenyl) propane dianhydride;
1,1-bis(2,3-dicarboxyphenyl) ethane dianhydride;
1,1-bis(3,4-dicarboxyphenyl) ethane dianhydride;
bis(2,3-dicarboxyphenyl) methane dianhydride;
bis(3,4-dicarboxyphenyl) methane dianhydride;
bis(3,4-dicarboxyphenyl) sulfone dianhydride;
benzene-1,2,3,4-tetracarboxylic dianhydride;
1,2,3,4-butane tetracarboxylic dianhydride;
thiophene-2,3,4,5-tetracarboxylic dianhydride;

Diamines

Primary diamines form the second prime reactant in the preparation of polyimide polymers and have the general formula:

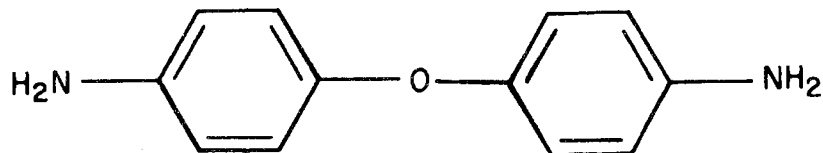


where R is a benzoid compound and can take a wide range of structures:



where R'' can be a carbon, nitrogen, silicon, phosphorous or sulfur atom (or

radical grouping). An often cited diamine is p,p'-oxydianiline:



In the patent literature (see Section VI) are cited numerous diamines and substituted diamine compounds:

meta-phenylene diamine, para-phenylene diamine;
4,4'-diamino-diphenyl propane;
4,4'-diamino-diphenyl methane;
benzidine;
4,4'-diamino-diphenyl sulfide;
4,4'-diamino-diphenyl sulfone;
3,3'-diamino-diphenyl sulfone;
4,4'-diamino-diphenyl ether;
1,5'-diamino-naphthalene, 3,3'-dimethyl-4,4'-biphenyl diamine;
3,3'-dimethoxy benzidine;
2,4-bis (beta-amino-t-butyl)toluene;
bis-(para-beta-amino-t-butyl-phenyl)ether;
bis-(para-beta-methyl-delta-amino-pentyl)benzene;
bis-para-(1,1-dimethyl-5-amino-pentyl)benzene;
1-isopropyl-2,4-metaphenylene diamine;
m-xylylene diamine;
hexamethylene diamine;
heptamethylene diamine;
octamethylene diamine;
nonamethylene diamine;
decamethylene diamine;
diamino-propyl tetramethylene diamine;
3-methylheptamethylene diamine;
4,4-dimethylheptamethylene diamine;
2,11-diamino-dodecane;
1,2-bis-(3-amino-propoxy ethane);
2,2-dimethyl propylene diamine;
3-methoxy-hexamethylene diamine;
2,5-dimethylhexamethylene diamine;
2,5-dimethylheptamethylene diamine;
3-methylheptamethylene diamine;
5-methyl-nonamethylene diamine;
2,17-diamino-eicosadecane;
1,4-diamino-cyclohexane;
1,10-diamino-1,10-dimethyl decane;
1,12-diamino-octadecane;
 $\text{H}_2\text{N}(\text{CH}_2)_3\text{O}(\text{CH}_2)_2\text{O}(\text{CH}_2)_3\text{NH}_2$;
 $\text{H}_2\text{N}(\text{CH}_2)_3\text{S}(\text{CH}_2)_3\text{NH}_2$;
 $\text{H}_2\text{N}(\text{CH}_2)_3\text{N}(\text{CH}_3)(\text{CH}_2)_3\text{NH}_2$;
piperazine;

Scroog and co-workers (Ref. 123) at the E.I. duPont de Nemours Company in their review of polyimide polymerization methods reported on the use of the following diamines:

- a) bis (4-aminophenyl) ether
- b) bis (4-aminophenyl) sulfide
- c) bis (4-aminophenyl) sulfone

The density of pyromellitimide films made from these diamines are shown in the following table:

Polypyromellitimide Films (R = Diamine Component)

R	η_{inh} (a)	Density
m-phenylenediamine	>0.3	1.43
p-phenylenediamine	0.5	1.41
p,p'-biphenylenediamine	>0.3 (b)	1.43
bis(4-aminophenyl)methylene	1.7	1.36
bis(4-aminophenyl) isopropylidene	0.5	1.30
bis(4-aminophenyl) thioether	>0.3 (b)	1.41
bis(4-aminophenyl) ether	1.0 (b)	1.42
bis(4-aminophenyl) sulfone	>0.3	1.43
bis(3-aminophenyl) sulfone	0.5	--

(a) 0.5% in H_2SO_4

(b) fuming HNO_3

Mixing and Proportion of Reactants

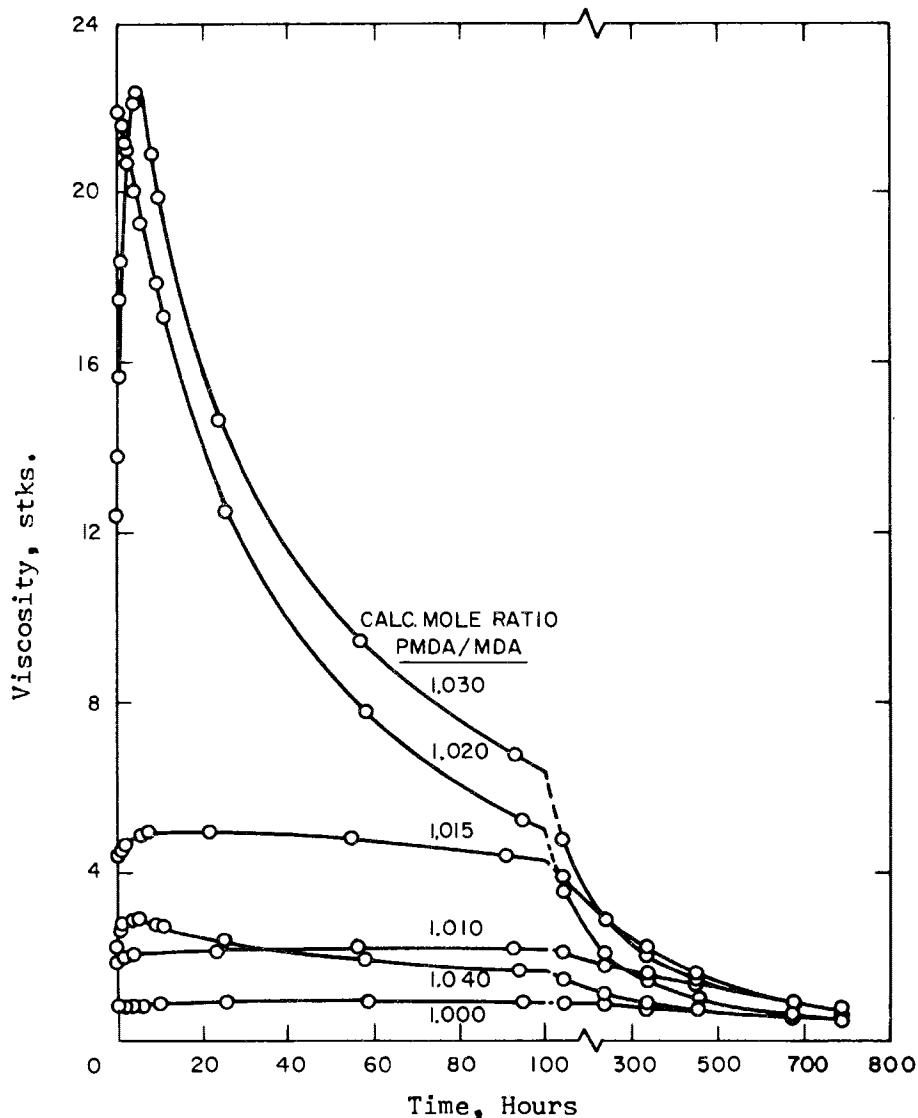
In the above sections it was pointed out that dianhydrides and diamines are the prime reactants required to form polyimide polymers. However, if dianhydride is added to diamine in solution, a high viscosity (equivalent to high molecular weight) is observed at equimolar ratios(1 : 1). When the order is reversed (i.e. diamine to PDMA in solution), a high viscosity is not observed and satisfactory films are not obtained.

In the patent literature (see Section VI) are cited numerous other methods of mixing and proportioning the basic reactants: dianhydrides and diamines.

According to Bower and Frost (Ref. 15), the simplest method of preparation is the slow addition of solid PDMA to a stirred solution of a diamine until maximum viscosity is attained. The reaction, which is somewhat exothermic, has been run successfully at temperatures from -20° to $+70^\circ C$ in suitable solvents (see Solvent Section below) A Waring blender is often cited as the best for mixing and reacting the dianhydride and diamine.

Temperature control during the reaction stages is very important and continually stressed in the patent literature (see Section VI). The preferred temperature is $35^\circ - 40^\circ C$ throughout the run. In the early stages of the run there will be little change in viscosity. When about 95% of the PDMA has been added, a rapid rise in viscosity is observed after each addition, reaching a constant value in 10 - 15 minutes (see Solvent System below).

Frost and Kesse (Ref. 109) present the following graph showing the effect of reactant ratio on the viscosity behavior of polyamic acid:



Effect of reactant ratio on viscosity behavior
of 12% MDA-PMDA in DMAc at 35°C.

[Ref. 109]

The initial reaction between PMDA and a diamine is very rapid, and the reactants are largely converted to polypyromellitic acid in a few minutes. However, as the reaction approaches completion, the concentrations of amine and anhydride groups become very small and the rate of reaction approaches zero. As PMDA and a diamine react, the rate of concentration will be proportional to the product of amine and anhydride concentrations.

From the reaction-viscosity curves, it is evident that the overall result is that the molecular weight of the polymer and the viscosity of the solution rapidly reach maximum values and then decline. The maximum is most sharply defined when the reactants are used in equivalent quantities, since on excess of either one prevents the attainment of a high viscosity.

Monsanto (Ref. 116) research workers found that a very slow rate of addition of distilled and recrystallized anhydride to amine yielded a polymer with a high inherent viscosity (1.1). Rapid addition of recrystallized pyromellitic dianhydride, in contrast, yield polymer solutions of inherent viscosities of 0.41 - 0.78 (20,800 cps, 29.2°C, 20% concentration).

Endrey, in Patent U.S. 3,073,784, noted that the degree of polymerization of the polyamide-acid is subject to deliberate control. The use of equal molar amounts of the reactants under the prescribed conditions provides polyamide-acids of very high molecular weight. The use of either reactant in large excess limits the extent of polymerization. However, up to 5% excess of either the diamine or the dianhydride may be used. More than 5% excess of either reactants results in an undesirably low molecular weight polyamide-acid. It is desirable to use 1 to 3% excess of either reactant, preferably the dianhydride, to control the molecular weight of the polyamide-acid. Besides using an excess of one reactant to limit the molecular weight of the polyamide-acid, a chain terminating agent such as phthalic anhydride may be used to "cap" the end of the polymer chains. Endrey also states..."in the preparation of the polyamide-acid compositions, it is essential that the molecular weight be such that the inherent viscosity of the polymer is at least 0.1, preferably 0.3 - 0.5.

Scroog and co-workers (Ref. 123) in their polyamic acid synthesis studies reported that..."the preparation of polyamic acids is generally carried out by adding pyromellitic dianhydride to a solution or slurry of the diamine in a polar solvent such as dimethylacetamide or dimethylformamide. The polymerization may be conducted heterogeneously by addition of pyromellitic dianhydride as a solid or as a slurry in a relatively inert liquid such as acetone, γ -butyrolactone, or benzonitrile. The rate of reaction appears to be limited by rate of solution of pyromellitic dianhydride; normally 30 - 60 minutes is adequate for laboratory-scale polymerization. Total solutes average 10 - 15 although it has been possible to attain high molecular weight polyamic acid at concentration levels of 35 - 40%. At such high concentrations, the slurry method of addition enables the best mixing, and efficient cooling is required to compensate for the heats of reaction and stirring. When the solid pyromellitic dianhydride is added, a red-orange color is observed at the solid-liquid interface; this rapidly lightens to a lemon-yellow as the pyromellitic dianhydride dissolves and reacts with the diamine.

Best results are obtained from both types of procedure at temperatures of 15 - 75°C; above 75°C a decrease in the molecular weight of polyamic-acid becomes marked. Above 100°C cyclization to imide is appreciable, causing eventual precipitation of polyimide as well as a lowered molecular weight of the polyamic acid. Above 150°C, the cyclization is so rapid that polyimide sometimes precipitates before all of the dianhydride can be added:

Effect of Temperature of Polymerization on Inherent Viscosity of Polyamic Acid
[Reaction of Bis(4-aminophenyl) Ether and Pyromellitic Dianhydride]

Solvent	Bis(4-amino-phenyl) ether		Pyromellitic dianhydride		Solids, %	Temp., °C.	Time at temp., min.	η_{inh}^a
	g.	Moles	g.	Moles				
DMAc	10.00	0.05	10.90	0.05	10.0	25	120	4.05
DMAc	10.00	0.05	10.90	0.05	10.0	65	30	3.47
DMAc	20.00	0.10	21.80	0.10	10.6	85-88	30	2.44
DMAc	20.00	0.10	21.80	0.10	10.7	115-119	15	1.16
DMAc	20.00	0.10	21.80 ^b	0.10	10.3	125-128	15	1.00
DMAc	20.00	0.10	21.80 ^c	0.10	15.7	135-137	15	0.59
N-Me capro-lactam	10.00	0.05	10.90	0.05	14.2	150-160	2	0.51
N-Me capro-lactam	10.00	0.05	10.90	0.05	12.9	175-182	1-2	Only partly soluble
N-Me capro-lactam	20.00	0.10	21.80	0.10	15	200	1	Insol.

a Determined at 0.5% concentration in the particular solvent at 30°C.

b Increment of 0.35 g. pyromellitic dianhydride added before determination of η_{inh} .

c Increment of 0.25 g. pyromellitic dianhydride and then 0.21 g. of bis(4-aminophenyl)ether added before determination of η_{inh} .

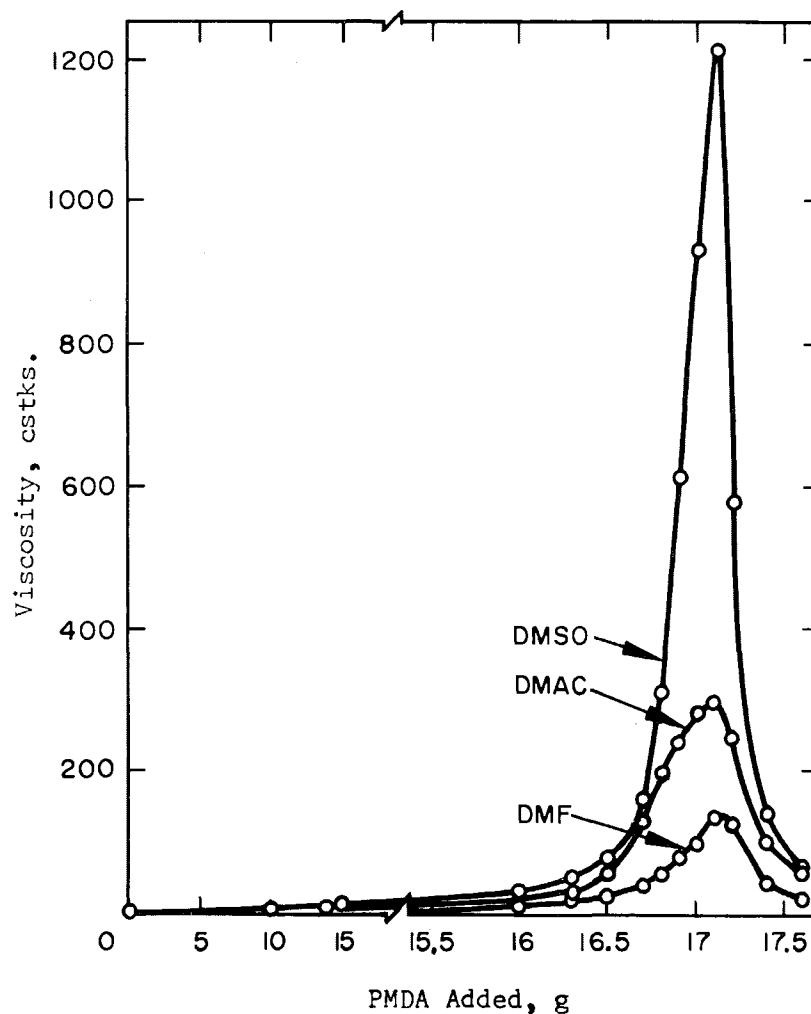
[Ref. 123]

Solvent System

The choice of a solvent system for the condensation of a dianhydride with a diamine is important. In order to build up a polymer of reasonably high molecular weight, it is essential to find a solvent which will retain the prepolymer or polyamide-acid in solution and create conditions favorable for a high degree of condensation. Both dimethyl acetamide (DMA or DMAc) and dimethyl formamide (DMF) serve as excellent solvents in this respect. Another important requirement, if a completely soluble prepolymer is to be obtained, is that the solvent used should be anhydrous and substantially free from pyromellitic acid (gelation complications can arise from the tetra-functionality of this compound). Edwards and Endrey have stressed, in the patent literature (British patent 903271) the importance of highly purified reactants and anhydrous solvent systems for best results.

The interaction of DMAc with various aromatic compounds in solution have been studied by various investigators: Hatton and Richards (1960) and Bower and Frost (1963) who have demonstrated that crystalline 1:1 complexes of DMAc and PMDA can be isolated.

Bower and Frost show the viscosity behaviour between PMDA and MDA in three different solvent systems: DMA, DMSO, and DMF:

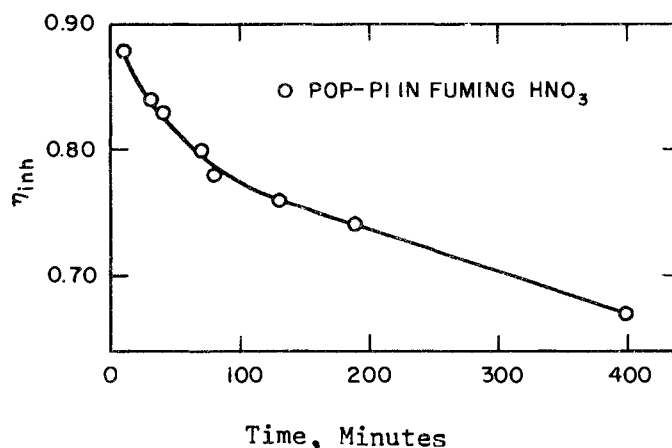


Reaction of PMDA with MDA in three solvents.

[Ref. 15]

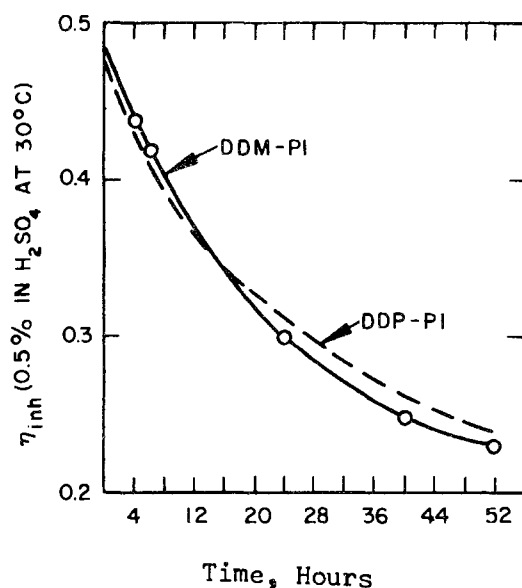
It is evident that excess PMDA causes a decrease in viscosity regardless of solvent used. N-methyl-2pyrrolidone (NMP) is also widely used according to technical bulletins from the E.I. du Pont de Nemours Company. Other literature from companies claim up to about 40% of a diluent such as xylene can be included in the solvent system.

Scroog and co-workers (Ref. 123) at the E.I. du Pont de Nemours Company in their polymerization studies noted that the polyimides are insoluble in conventional solvents. In addition, they found that only fuming nitric acid to be a general solvent for the species suitable for qualitative molecular weight evaluation at low temperature; concentrated sulfuric acid is effective for some polyimides. They summarized the stabilities of solutions of various polyimides in fuming HNO_3 and in concentrated H_2SO_4 in the following two graphs:



Inherent viscosity vs. time for dilute solutions of polyimide;
15°C., 0.5 g. polymer/100 ml. solvent.

[Ref. 123]



Stability of polypyromellitimides in concentrated H_2SO_4 ; 30°C.

[Ref. 123]

Various patent claims hold that highly polar solvents are necessary to dissolve the polyimide polymer because these solvents associate strongly with both the polymer and the reactants. This association accelerates the reaction. The patent literature (see Section VI) also contains a wide range of possible or potential solvents which can be utilized for polyimide polymerization reactions:

N,N-dimethylmethoxy acetamide, N-methyl caprolactam, dimethylsulfoxide, N-methyl-2-pyrrolidone, tetramethylene urea, pyridine, dimethylsulfone, hexamethylphosphoramide, tetramethylene sulfone, formamide, N-methylformamide, butyrolactone and N-acetyl-2-pyrrolidone.

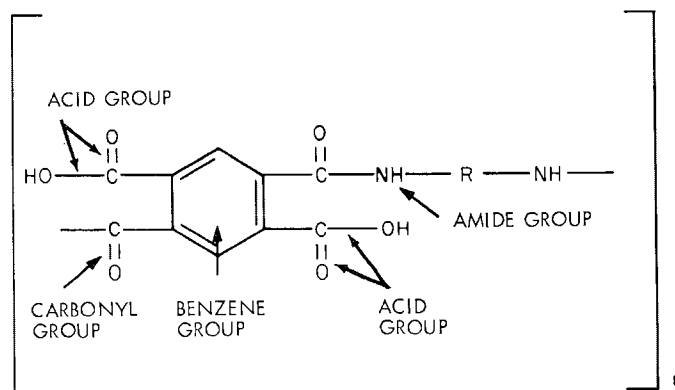
The solvents can be used alone, in combinations of solvents, or in combination with poor solvents such as benzene, benzonitrile, dioxane, xylene, toluene and cyclohexane.

The solvents useful in the solution polymerization process for synthesizing the polyamide-acid compositions are the organic polar solvents having a dipole moment whose functional groups do not react with the diamines or the dianhydrides. The normally liquid organic polar solvents of the N,N-dialkylcarboxylamine class are useful for this purpose. The preferred solvents are the lower molecular weight members of this class, particularly N, N-dimethylformamide (DMF) and N,N-dimethylacetamide (DMA or DMAC). They are easily removed from the polyamide salts by evaporation, displacement or diffusion.

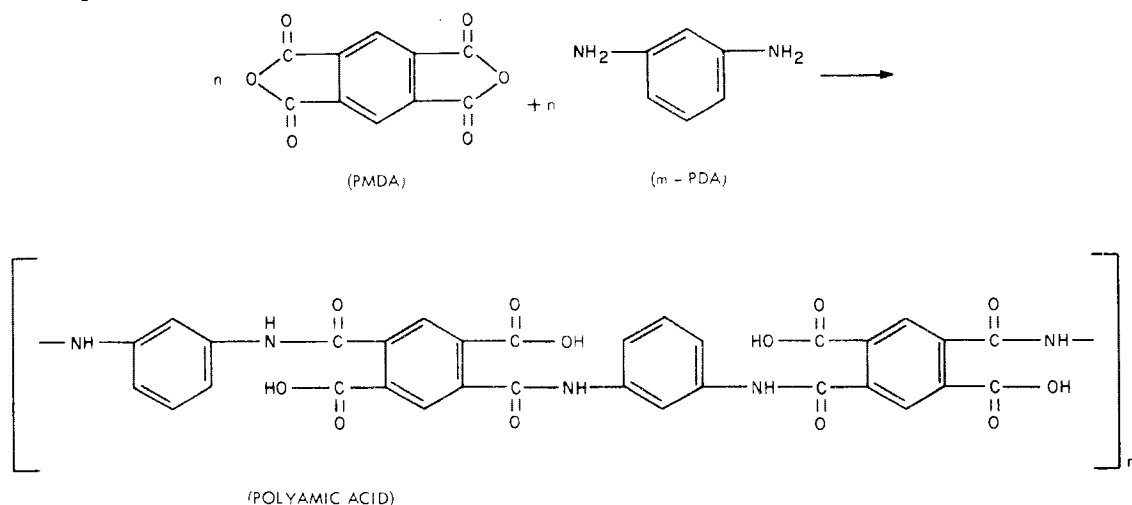
The quantity of organic polar solvent used in the process need only be sufficient to dissolve the diamine and to provide with the ultimate polymeric component dissolved therein, a sufficiently low viscosity for forming the composition into shaped articles. Patent claims note that the most successful results are obtained when the solvent represents at least 60% of the final polymeric solution, that is, the solution should contain 0.05-40% of the polymeric component.

Polyamic Acid Solution (Stage)

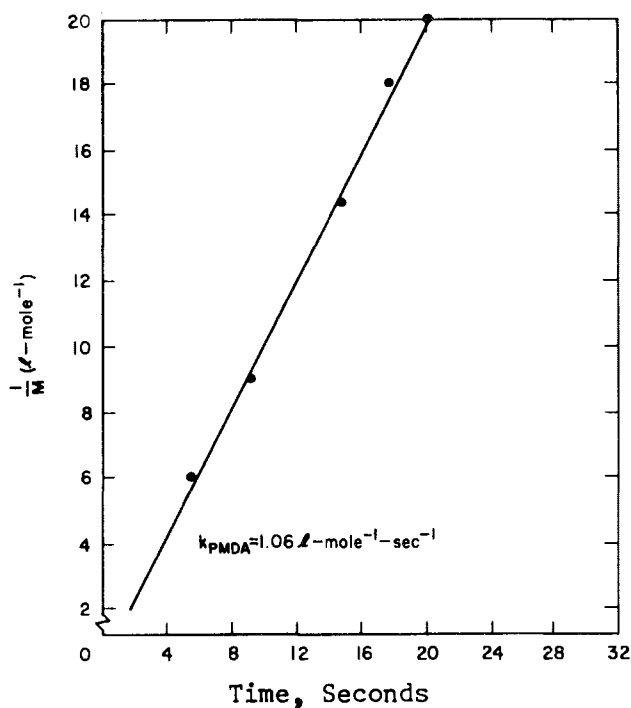
A polyamic acid (also termed polyamide-acid) solution is formed as an intermediary product by reacting the dianhydrides and diamines discussed above and is represented by the formula:



These polyamide-acid solutions can take on a wide range of chemical composition as a result of the different dihydride and diamine starting reactants. The reaction of pyromellitic dianhydride with m-phenylenediamine in anhydrous dimethylacetamide (5% solution) proceeds according to the equation:



Wrasidlo and co-workers at Narmco report the results of their kinetic studies on the above condensation of PMDA and m-PDA:



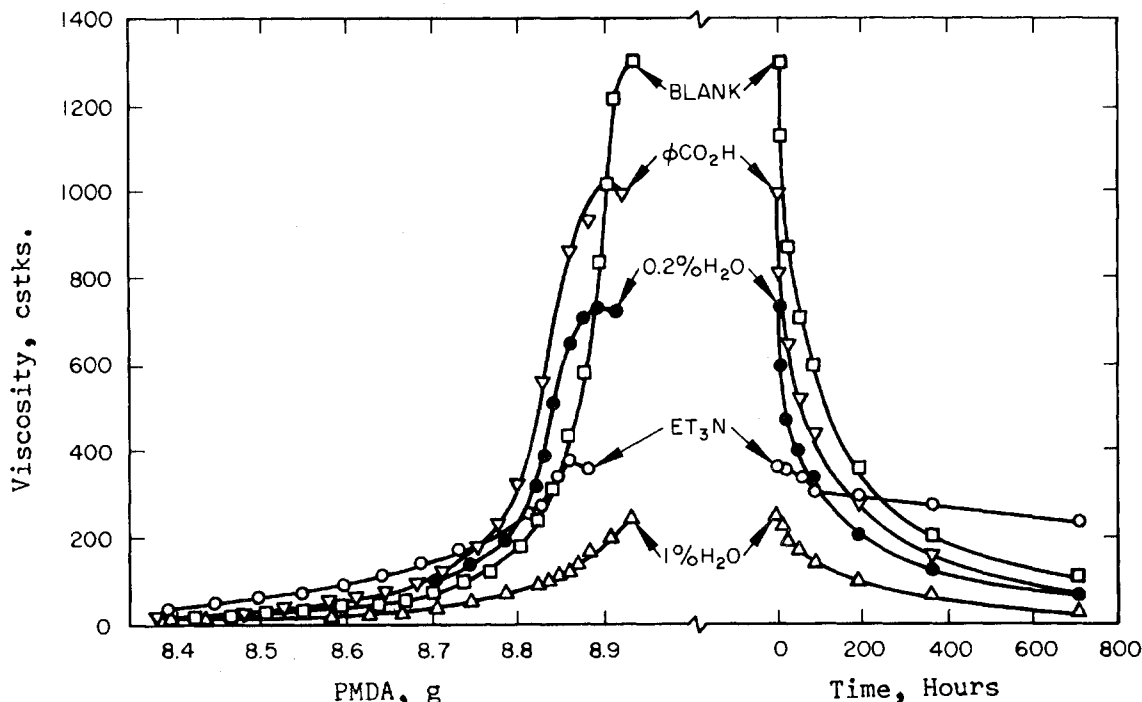
Kinetic Study: Condensation of PMDA and m-PDA

Plot $\frac{1}{M}$ as a function of time for rate constant of aminolysis (linear plot for second order)

[Ref. 119]

and obtained a linear plot of the reciprocal molarity of PMDA as a function of time.

While the molecular weight of the resin solution at the maximum viscosity (see preceding Solvent System section), is estimated to be in the range of $10^5 - 10^6$, it varies with time as shown in the following graph and table:



Viscosity changes of DAPE-PMDA solutions prepared in the presence of various additives.

[Ref. 15]

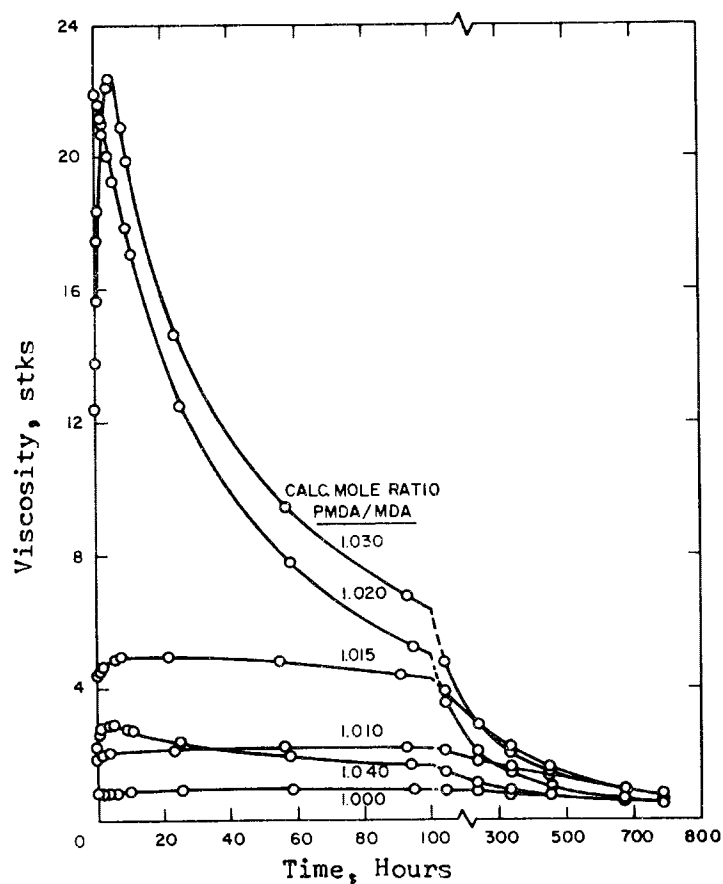
Intrinsic Viscosity Decline of DAPE-PMDA at 25°C

Aging time, hours	$[\eta]$, dl./g.	Approximate MW
1.5	1.25	620,000
48	1.05	560,000
164	1.00	450,000

[Ref. 15]

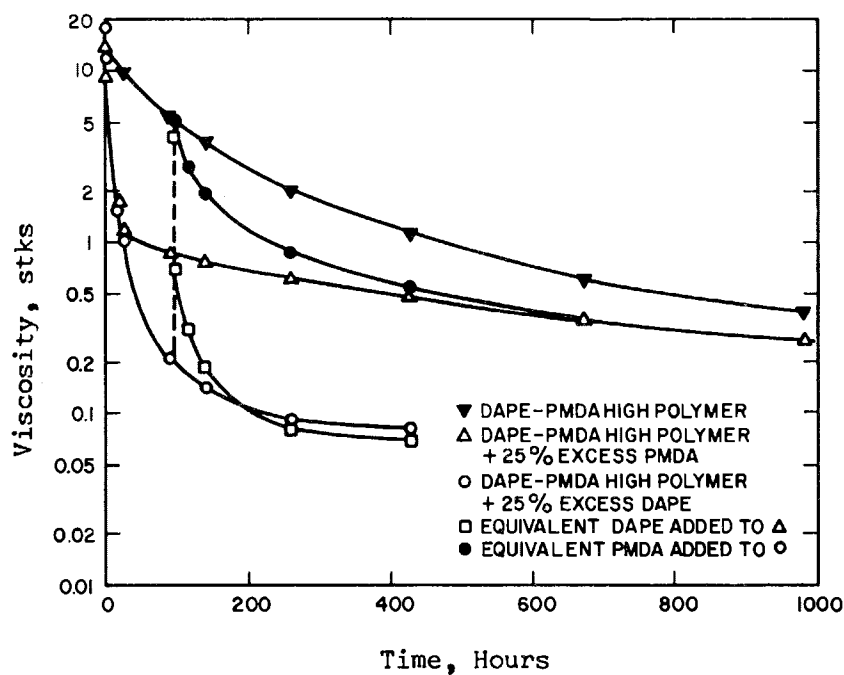
molecular weight values of the order of $200,000 \pm 5\%$ have been observed by Westinghouse Electric researchers.

Frost and Kesse (Ref. 109) showed that the stability of polyamic acid solutions over various time periods was a function of their reactant ratios:



[Ref. 109]

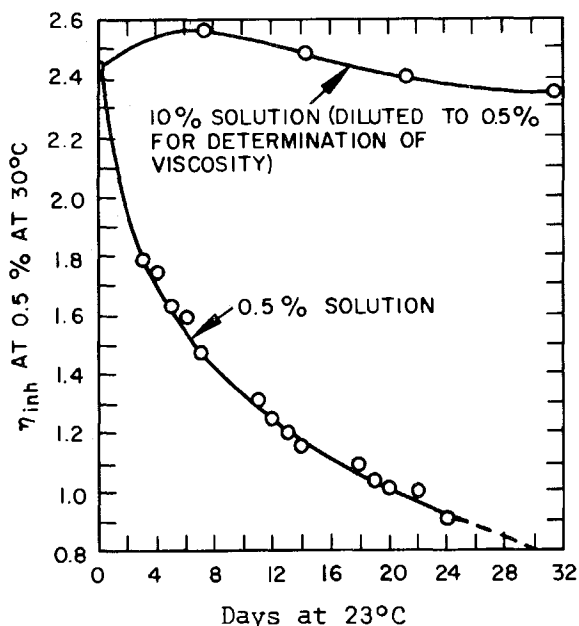
Effect of reactant ratio on viscosity behavior of 12% MDA-PMDA in DMAC at 35°C.



[Ref. 109]

Effect of additional DAPE and PMDA on 10% DAPE-PMDA in DMAC at 35°C

Similarly, Sroog and co-workers at the E.I. du Pont de Nemours Company reported on the effect of concentration on stability of polyamic solutions:



Effect of concentration on stability of POP-PA in DMAc at 23°C.

[Ref. 123]

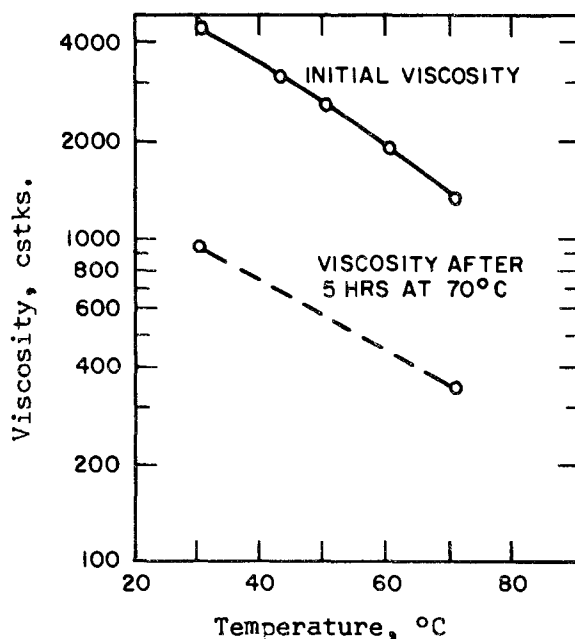
The stability at 23°C of the polyamic acids in solution is dependent upon the polymer concentration, dilute polymer solutions decreasing in viscosity much more rapidly than concentrated solutions. They have also elaborated on the typical preparation of a polyamic acid citing various details of the reaction process and noting that the polymer solutions may be stored in dry, sealed bottles at -15°C until needed.

These same authors reported on a typical method of polyamic acid preparation as follows:

"Typical Preparation of a Polyamic Acid: A 500-ml flask fitted with mercury-seal stirrer, nitrogen inlet, drying tube, and stopper is carefully flamed to remove traces of water on the walls and is allowed to cool under a stream of dry nitrogen in a dry box. In the dry box 10.0 g. (0.05 mole) bis(4-aminophenyl) ether is added to the flask through a dried powder funnel, and residual traces are flushed in with 160 g. of dry dimethylacetamide. There is then added 10.90 g. (0.05 mole) pyromellitic dianhydride to the flask through a second dried powder funnel over a period of 2-3 minutes with vigorous agitation. Residual dianhydride is washed in with 28 g. dry dimethylacetamide. The powder funnel is replaced by the stopper and the mixture is stirred for 1 hour. A small surge in temperature to 40°C occurs as the dianhydride is first added, but the mixture rapidly returns to room temperature.

The above procedure yields a 10% solution which can be stirred without difficulty. In some cases the mixture becomes extremely viscous, and dilution to 5-7% solids may be required for efficient stirring. Polyamic acids so prepared exhibit inherent viscosities of 1.5-3.0 at 0.5% in dimethylacetamide at 30°C."

Temperature also affects the viscosity of the solution as shown in Bower and Frost's experiments:



Effect of temperature on viscosity of 11% DAPE-PMDA in DMAC.

[Ref. 15]

At 0°C, the solution can be stored for several weeks without a noticeable change in viscosity. The viscosity of the solution decreases greatly when it is aged at 35°C. Higher temperatures or the presence of water, excess amine or excess anhydride accelerate the rate of decline.

A rapid early drop in viscosity has been found by Frost and Kesse to be due primarily to hydrolysis by adventitious water and to exchange reactions of amine or anhydride groups with the o-carboxamide linkage. Slow decline over a long period of time results from hydrolysis by water produced in the slow conversion of polypyromellitic acid to polypyromellitimide.

In the patent literature it is often recommended that polyamic acids should have an inherent viscosity of at least 0.1, preferably 0.3 to 5.0. The inherent viscosity is calculated by the following formula:

$$\text{Inherent viscosity} = \frac{\text{Natural log} \left(\frac{(\text{viscosity of solution})}{(\text{viscosity of solvent})} \right)}{C}$$

Where C = concentration expressed in grams of polymer per 100 mil of solution. The inherent viscosity is directly related to the molecular weight of the polymer.

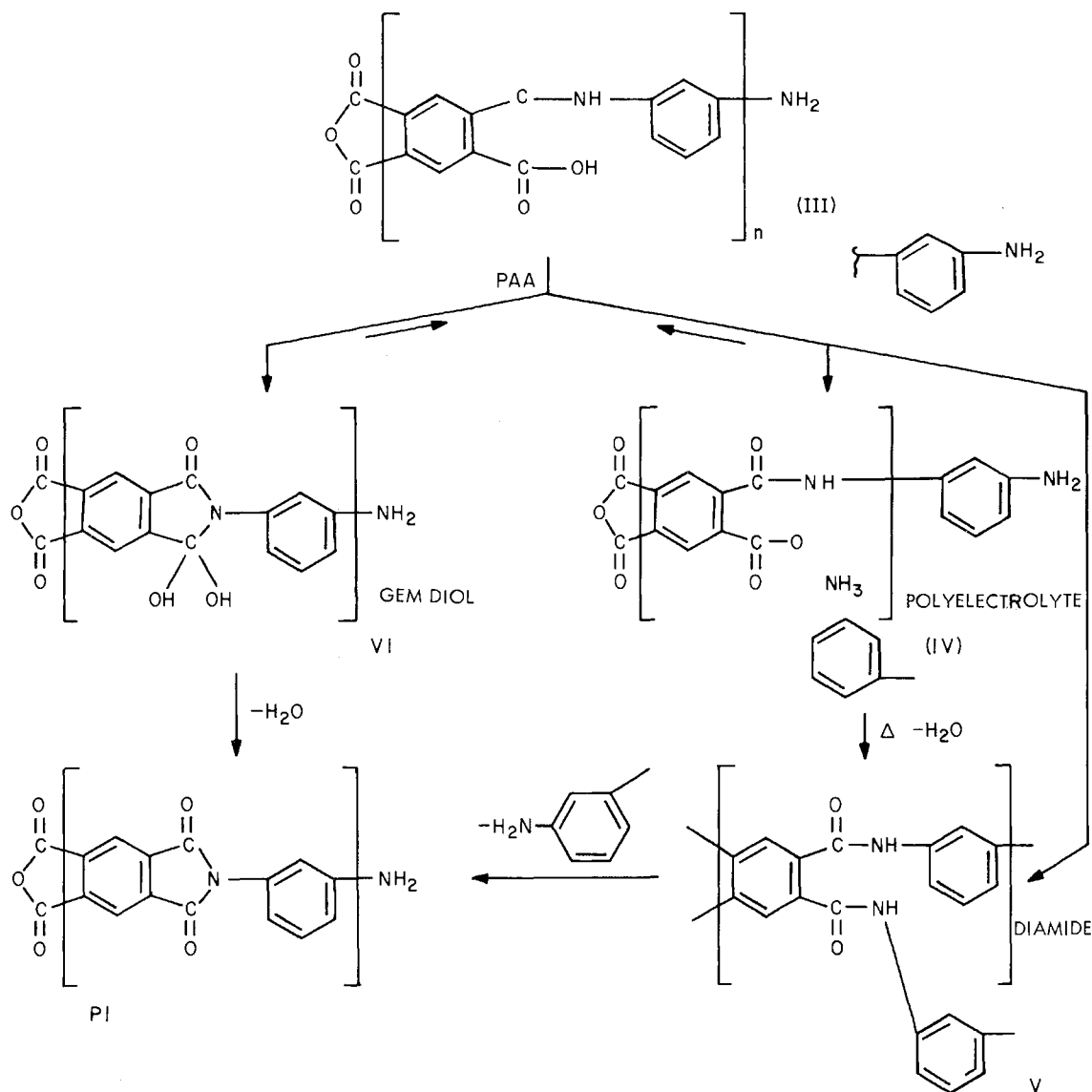
The Narmco Research and Development Division of the Whittaker Corporation has synthesized a number of polyimide resin systems in a development program on high temperature adhesives and resins for the Bureau of Naval Weapons. In one set of experiments they prepared a pyridine solution (2700gm, 35% solids) of polyamic acid by the slow addition of a stoichiometric slurry of 3,3', 4,4'-benzophenone dianhydride to a solution of m-phenylene diamine. Imidization was effected by heating at 400°C in argon under high vacuum for one hour. Weight loss data for this polymer are reported in the Thermal Properties section of this report.

In another experiment, a stoichiometric solution of m-phenylenediamine in DMAC was slowly added to a slurry of 1,4,5,8-naphthalene dianhydride and 3,3',4,4'-benzophenone dianhydride in DMAC (at 35°C) under argon. After the polyamic acid was obtained, it was heated at 400°C in argon under high vacuum for one hour to convert the polyamic acid to imide.

Imidization Stage (Dehydration and Thermal Heating)

In imidization, the controlling step is the removal of water and this seriously limits the dimensions of the final sample. According to Cooper and co-workers at Princeton University, only thin films can be polymerized successfully, because of this diffusional limitation in the elimination of water. An exception to this is DuPont's SP Polymer which has been made almost 1 millimeter thick.

According to Wrasidlo and co-workers at Narmco, imidization of the polyamic acid may occur at least in two ways according to the following equations:

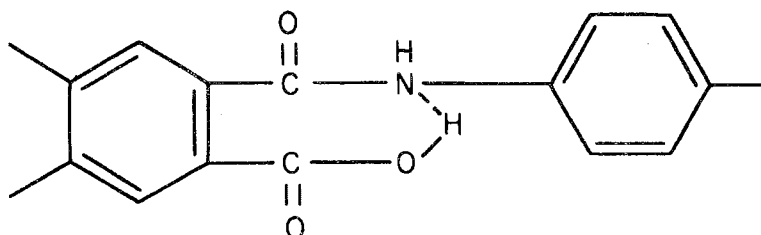


Possible reaction pathways for polyimide formation

[Ref. 119]

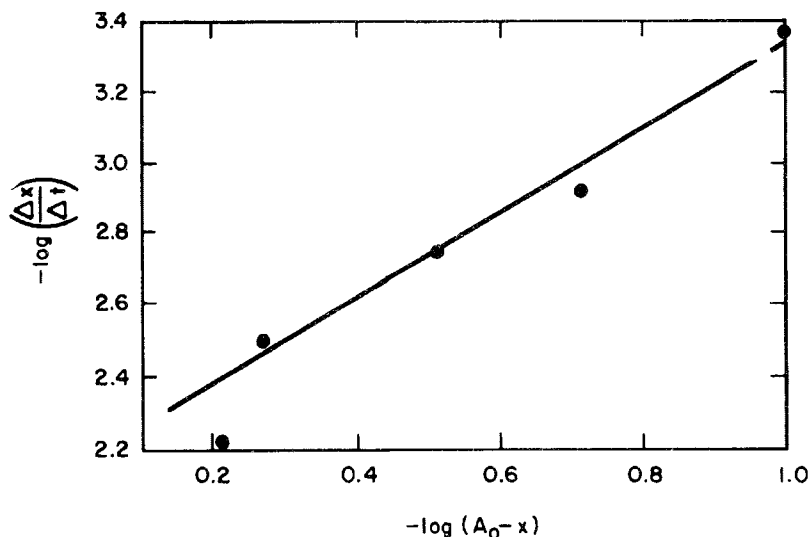
The reaction path involving the formation of a polyelectrolyte (IV) is considered less likely for the following reasons:

1. The aromatic nature of the amine is decreasing the basicity of the amino group.
2. In relatively dilute solutions of a highly polar solvent, the salt (if in existence) would be dissociated to a large extent, which would therefore shift the equilibrium to the left.
3. The proton from the carboxyl group may participate in intramolecular hydrogen bonding through the nitrogen of the adjacent amide group, forming a ring of following structure:



The stability of the ring was estimated to be 2-5 Kcal/mole, which could be sufficient to prevent the transfer to the proton to form the anilinum type ion with the amino group.

The reaction order for the imidization process was obtained from the slope of the line in the following figure.

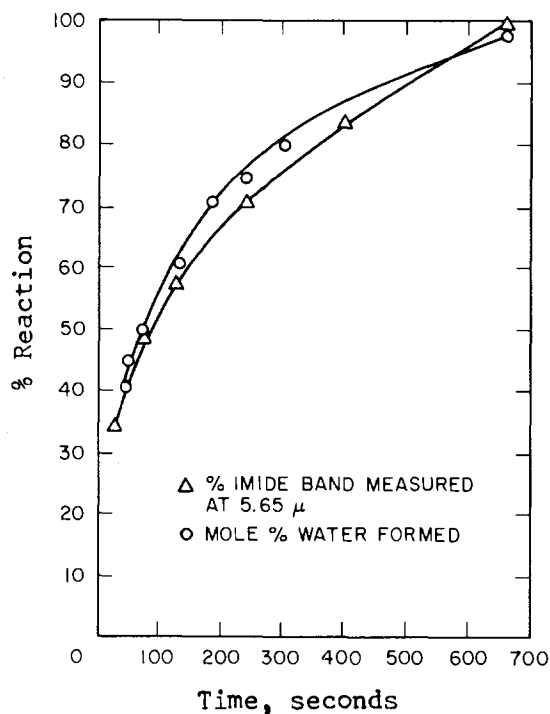


Kinetic Study: Condensation of PMDA and m-PDA

Plot $-\log\left(\frac{\Delta x}{\Delta t}\right)$ as a function of $-\log(A_0 - x)$ for Reaction Order of Imidization

[Ref. 119]

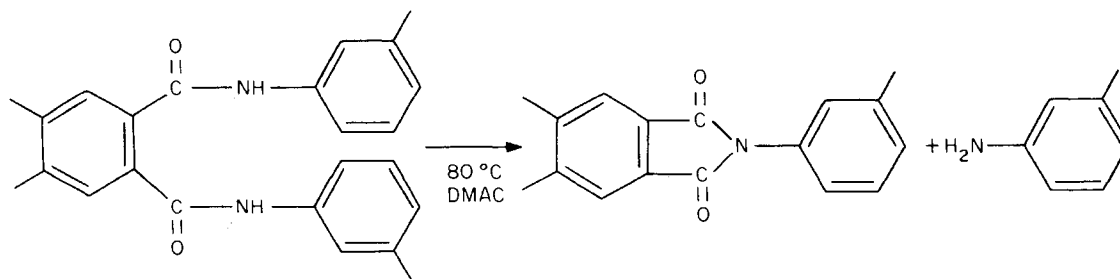
The amount of polyimide formed per unit time was measured by Wrasidlo and co-workers in the infrared by an increase of the imide carbonyl band at 5.65 μ . A comparison of the mole percent of water formed and percent imide conversion was found to be as follows:



Kinetic Study: Condensation PMDA and *m*-PDA Plot of Percent Conversion as a Function of Time

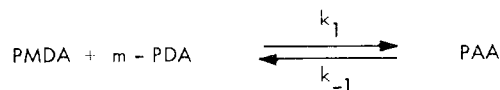
[Ref. 119]

It was apparent to Wrasidlo and co-workers that polyimide formation from the polyamic acid simultaneously involved water formation. These findings suggested that imidization of the polyamic acid cannot occur through the formation of the diamide (V).



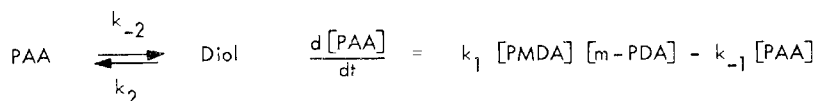
From the previous reaction pathways scheme, it seems plausible that the mechanism for imidization goes through the postulated gem diol intermediate.

Considering the separate rates of the forward and reverse reaction between the diol and the amide, as well as the rate of the succeeding process,

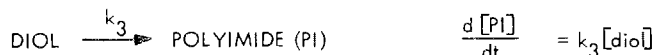


WHERE

$$\frac{k_1}{k_{-1}} = \text{OBSERVED RATE} = 1.06 \text{ l-mole}^{-1} \text{-sec}^{-1}$$



$$\frac{d[\text{diol}]}{dt} = k_2 [\text{PAA}] - k_{-2} [\text{diol}] - k_3 [\text{diol}] = 0$$



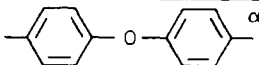
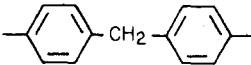
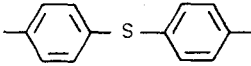
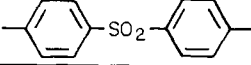
The following expression was obtained by these authors for the overall rate of polymerization:

$$\frac{d([\text{PI}])}{dt} = 3.87 \times 10^{-3} [\text{PAA}]$$

They claim that the importance of the above equation lies in the fact that the exact overall rate of polymerization may be determined from the concentration of the polyamic acid alone.

In their study of polyamic acid films, Scroog and co-workers at E.I. du Pont de Nemours doctored thin layers (10-25 mils) of polyamic acid solutions onto dry glass plates and dried them for 20 minutes in a forced draft oven (with nitrogen bleed) at 80°C. The resulting films were only partly dry and after cooling could be peeled from the plates, clamped into frames, and dried further in vacuo at room temperature. Further, films which had been dried to a solids level of 65-75% were clamped to frames and heated in a forced draft oven to 300°C for an additional hour. These authors observed that films of polyamic acids containing 25-35% of residual solvent can be prepared by careful drying of cast solutions. These films on dissolving exhibit considerably lower inherent viscosities than the original polyamic acids:

DECREASE OF INHERENT VISCOSITY OF POLYPYROMELLITAMIC ACIDS DURING FILM CASTING AND DRYING (R = DIAMINE COMPONENT)

R	SOLVENT	η_{inh} SOLUTION	DRYING CONDITIONS	η_{inh} FILM
	DIMETHYLACETAMIDE	2.1	30 min., 80°C	1.3
		2.1	30 min., 80°C	
			5 days, 23°C	1.3
	DIMETHYLACETAMIDE	1.0	10 min., 120°C	0.4
		1.2	30 min., 80°C	0.33
	BENZONITRILE/DI-METHYLFORMAMIDE (3/2)	1.0	15 hr., 25°C	0.5
	DIMETHYLACETAMIDE	1.3	6 hr., 70°C	0.7
		2.3	20 min., 110°C	0.5
	DIMETHYLACETAMIDE	1.2	30 min., 120°C	0.5
	DIMETHYLACETAMIDE	0.7	30 min., 120°C	0.4

* POP-PA FILM IS POLYAMIC ACID FROM BIS (4-AMINOPHENYL) ETHER AND PYROMELLITIC DIANHYDRIDE

[Ref. 123]

Polyamic acids have no true softening points, since dehydration occurs with heating to form polyimide; this can occur in solution at temperatures as low as 100°C. The polymer, or film cast from solution, will not cyclize extensively until temperatures of 120-150°C are reached according to Scroog and co-workers.

Bower and Frost (Ref. 15) reported that the curing of aromatic polyimide resins presents certain problems. Thin films of excellent clarity and toughness are obtained from many of the polymers simply by spreading the solution on a flat surface and baking it at 150°C for an hour or two. As the thickness is increased, however, the film becomes more brittle and less clear, until the cured resin is obtained in the form of brittle opaque fragments, shiny on the upper surface, powdery beneath. This effect is most noticeable in polymers high in imide. The effect can be minimized by removing most of the solvent at low temperatures, preferably under vacuum, and completing the cure with a slowly rising temperature. Toughness and flexibility of the polymers high in imide groups can often be improved by heating the film to a temperature of about 300°C for a few minutes. These same recommendations are often cited in the polyimide patent literature (see Section VI).

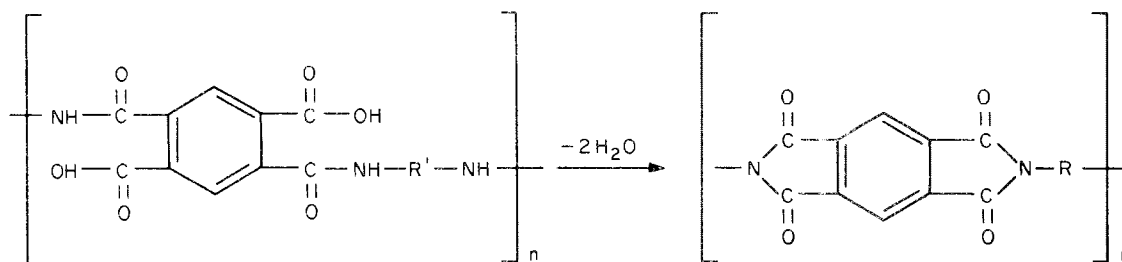
Films of the more stable polyimides, such as those prepared from 4,4'-diaminodiphenyl sulfide or ether (DAPE), or from one of the diaminobenzanilides, remain a clear amber and flexible after several hundred hours of aging at 325°C in air. They also reported on the effects of different curing schedules on cast films prepared from DAPE-PMDA polymer solutions.

Bower and Frost have also pointed out from their experimental work that

the imidization reaction occurs very slowly in solution at moderate temperatures. After 55 hours at 35°C the formation of residual carboxyl groups, nor did the infrared spectrum show any evidence of imide groups. However, when the solution was heated to the boiling point, the insoluble polyimide precipitated in a few minutes.

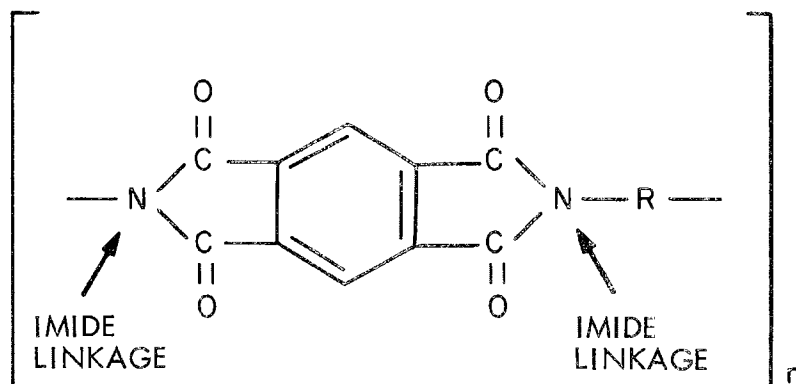
According to Frost and Kesse (Ref. 109), degradation by hydrolysis can occur when polyimide films are cast by heating the solution. The effect can be minimized by casting thin films, from which water can diffuse rapidly, or by using a chemical method, such as treatment with acetic anhydride and pyridine, to remove the water produced in imidization. When a thick layer of solution is heated, however, extensive hydrolysis occurs before the water can escape, and the polymer is obtained as a powder of low molecular weight. Thick pieces can be cured without degradation by treatment with acetic anhydride, since no free water is produced in this method and hydrolysis does not occur.

Removing water from the polyamic acid solutions forms the polyimide:



Polyimide

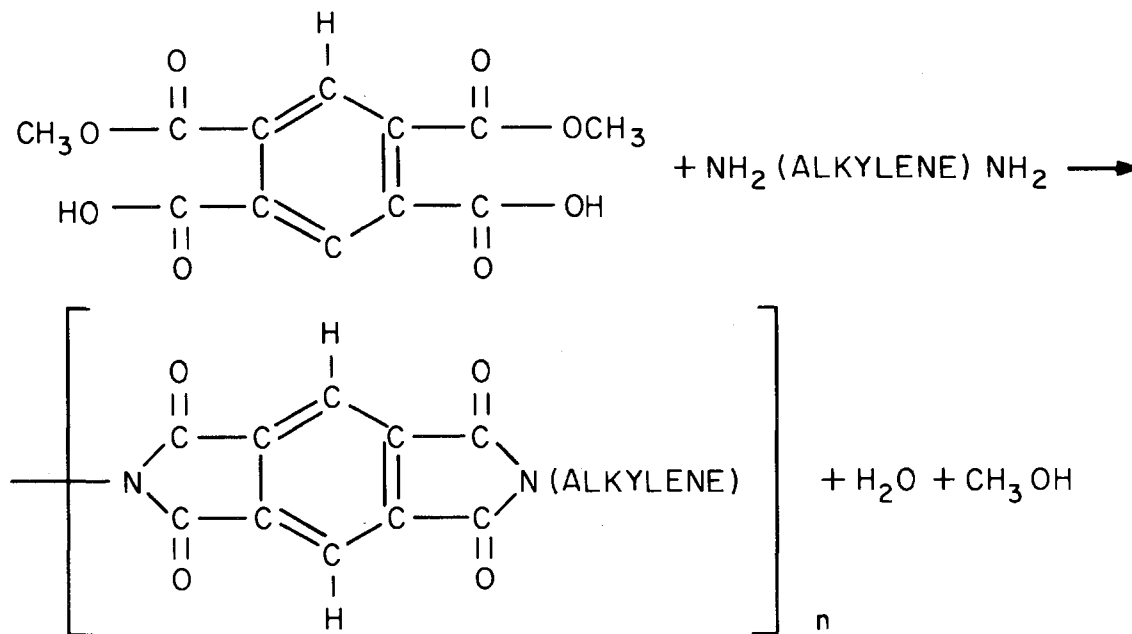
After the dehydration stage whether by heat or chemical conversion agents, imidization or the formation of a solid polyimide is the final product and has the formula;



This structure will vary depending on the combination of dianhydride and diamine used. The - R - group can vary widely in chemistry (see Patent Section VI).

Polyimides derived from aliphatic diamines are generally soluble and thermoplastic, while the aromatic amines are usually infusible and insoluble. For example, when the reactants are heated together, the mixture becomes solid before a high molecular weight is reached. However, when the reaction is carried out at moderate temperatures in a solvent, it is possible to attain a very high molecular weight while the polymer is in the soluble amide form. The polymer can then be applied from solution to form films, laminates, varnish coatings, etc. The imidization reaction occurs during or after solvent removal and yields a linear polymer consisting of a series of benzene rings alternating with various linking groups. In addition to the cyclic imide groups derived from the pyromellitic dianhydride, other connecting groups can be included as part of the diamine reactant.

In the early patent literature (e.g. British Patent 570,858 (1945); U.S. 2,710,853 (1955); U.S. 2,731,447 (1956); U.S. 2,900,364 (1959) polyimides were prepared by polyamide-salt techniques. Thus, pyromellitic dianhydride was reacted with ethanol to form the diester-diacid, which was treated with diamine to precipitate the monomeric diester-diacid salt. The salt was then heated to form a polymerizing melt, which, after dehydration and dealcoholation in the melt, formed the melt. A typical diacid diester or acid ester reaction is the following:

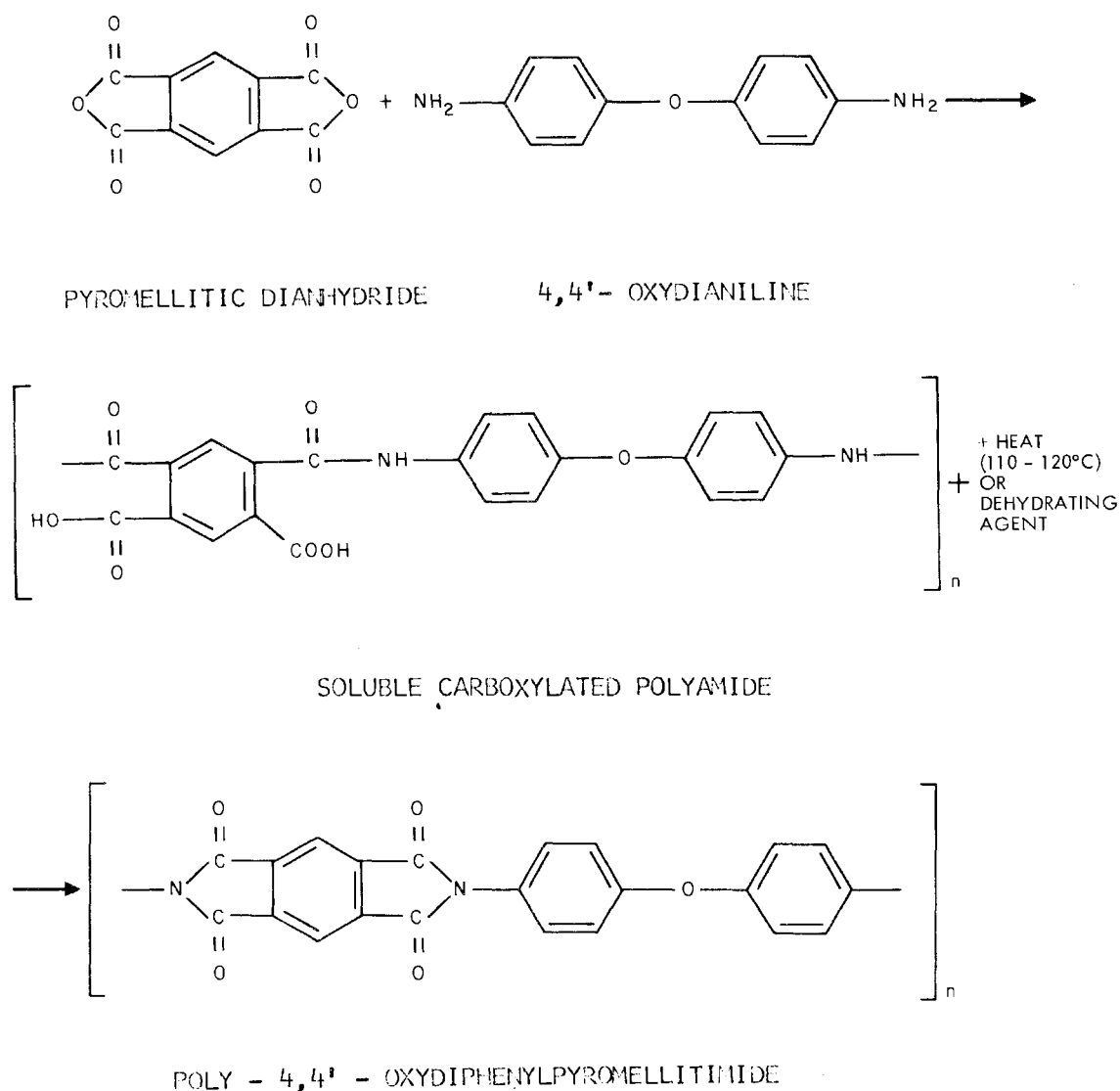


WHERE n = USUALLY ABOVE 100

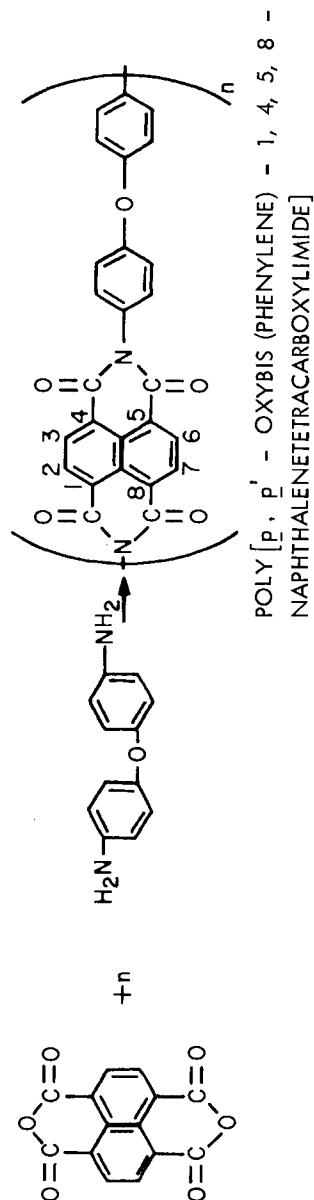
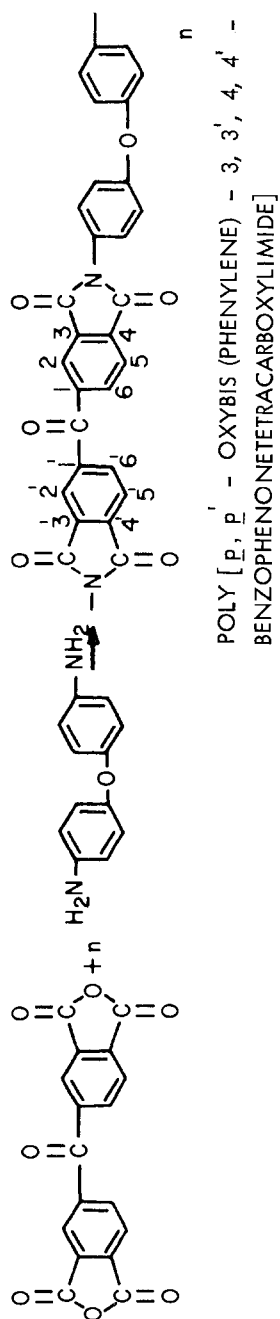
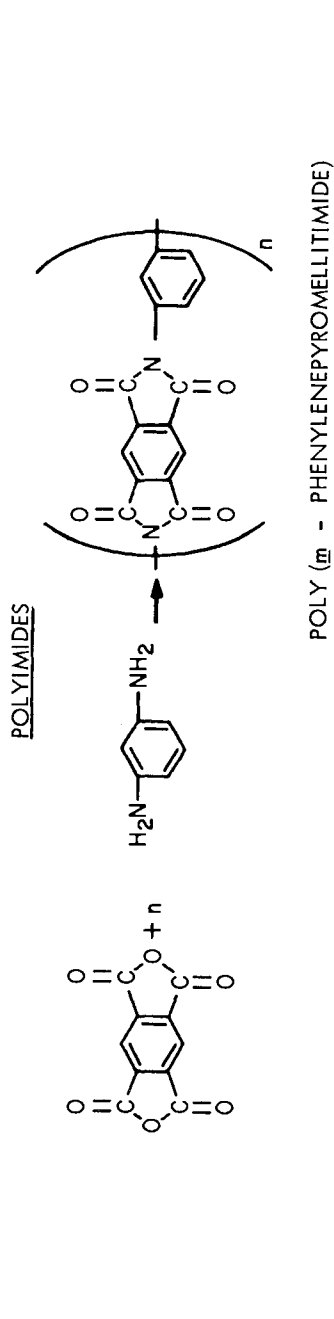
Other approaches to preparing polyimides followed in the subsequent patent disclosures (see Patent Section VI).

Scroog and co-workers recently reviewed the polyimide polymerization literature (see Ref. 123) and detailed the formation of polypyromellitimides by direct polymerization of pyromellitic dianhydride and aromatic diamines in solvents such as dimethylacetamide or dimethylformamide to soluble, high molecular weight polyamic acids (also called polyamide acids). The polyamic acids are then dehydrated to polyimides of high molecular weight. These authors emphasized the relationship of polyimide structure to properties by illustration of specific diamines.

The polyimide polymer, Poly-4,4'-oxydiphenylpyromellitimide (PMI), is prepared by reaction of pyromellitic dianhydride and 4,4'-oxydianiline in dimethylacetamide solvent to form a soluble polyamide acid solution stage and then further dehydration (either by chemical or heat):



The Narmco Research and Development Division of Whittaker Corporation investigated various polyimides for high temperature adhesive and laminate purposes and consisted of the following structures:



and combinations of carboxylic acid derivatives with different diamines:

Polyimides

CARBOXYLIC ACID DERIVATIVE	AMINE	SOLVENT*	PREPOLYMER		FINAL POLYMER		WT LOSS (TGA)	
			MELT TEMP °C	η_{inh}	AFTER	η_{inh}	1000°F (538°C)	1100°F (593°C)
$\text{HO}_2\text{C}-\text{C}_6\text{H}_2(\text{CO}_2\text{CH}_3)_2-\text{CO}_2\text{H}$ $\text{H}_3\text{CO}_2\text{C}-\text{C}_6\text{H}_2(\text{CO}_2\text{H})_2-\text{CO}_2\text{CH}_3$ $\text{H}_3\text{CO}_2\text{C}-\text{C}_6\text{H}_2(\text{CO}_2\text{CH}_3)_2-\text{CO}_2\text{CH}_3$ $\phi\text{O}_2\text{C}-\text{C}_6\text{H}_2(\text{CO}_2\phi)_2-\text{CO}_2\phi$ $\phi\text{O}_2\text{C}-\text{C}_6\text{H}_2(\text{CO}_2\phi)_2-\text{CO}_2\phi$ $\text{OOC}-\text{C}_6\text{H}_2(\text{COO})_2-\text{COO}$	$\text{H}_2\text{N}-\text{C}_6\text{H}_4-\text{NH}_2$ $\text{H}_2\text{N}-\text{C}_6\text{H}_3(\text{NH}_2)-\text{NH}_2$ $\downarrow$ $\text{H}_2\text{N}-\text{C}_6\text{H}_4-\text{NH}_2$ $\text{H}_2\text{N}-\text{C}_6\text{H}_3(\text{NH}_2)-\text{NH}_2$ $\text{H}_2\text{N}-\text{C}_6\text{H}_4-\text{O}-\text{C}_6\text{H}_4-\text{NH}_2$ $\text{NH}_2\cdot\text{HCl}-\text{C}_6\text{H}_4-\text{O}-\text{C}_6\text{H}_4-\text{NH}_2\cdot\text{HCl}$ $(\text{C}_6\text{H}_4\text{S}(\text{NH}_2))_2$ $(\text{C}_6\text{H}_4\text{S}(\text{NH}_2\cdot\text{HCl}))_2$ $\downarrow$ $\text{H}_2\text{N}-\text{C}_5\text{H}_3(\text{NH}_2)-\text{NH}_2$ $\text{H}_2\text{N}-\text{C}_6\text{H}_4-\text{SO}_2-\text{C}_6\text{H}_4-\text{NH}_2$ $\text{H}_2\text{N}-\text{C}_6\text{H}_3(\text{NH}_2)-\text{NH}_2$ $\text{H}_2\text{N}-\text{C}_6\text{H}_4-\text{O}-\text{C}_6\text{H}_4-\text{NH}_2$ $\downarrow$	 DMAC DMAC PYR DMF DMF DMAC PYR DMF DMAC***	NO MELT NO MELT NO MELT NO MELT 58-69 NO MELT NO MELT 65-80 NO MELT 140 92-93 	 0.03 0.50** 0.28 0.07 0.32 0.32 0.05 0.10** 0.11** 0.04 0.03	 1 hr @ 400°C/VACUUM 1 hr @ 400°C/VACUUM 1 hr @ 400°C/VACUUM 1 hr @ 500°C/VACUUM 1 hr @ 400°C/VACUUM 1 hr @ 400°C/VACUUM 2 hr @ 400°C/VACUUM	 INSOLUBLE INSOLUBLE INSOLUBLE INSOLUBLE INSOLUBLE INSOLUBLE INSOLUBLE INSOLUBLE INSOLUBLE 0.36 0.42	 23.0 7.1 1.0 0.33 37.0 19.8 0 15.0 3.0 0	 15.5 6.6 6.5 8.0 6.2

* DMAC - N.N. Dimethylacetamide

PYR - Pyridine

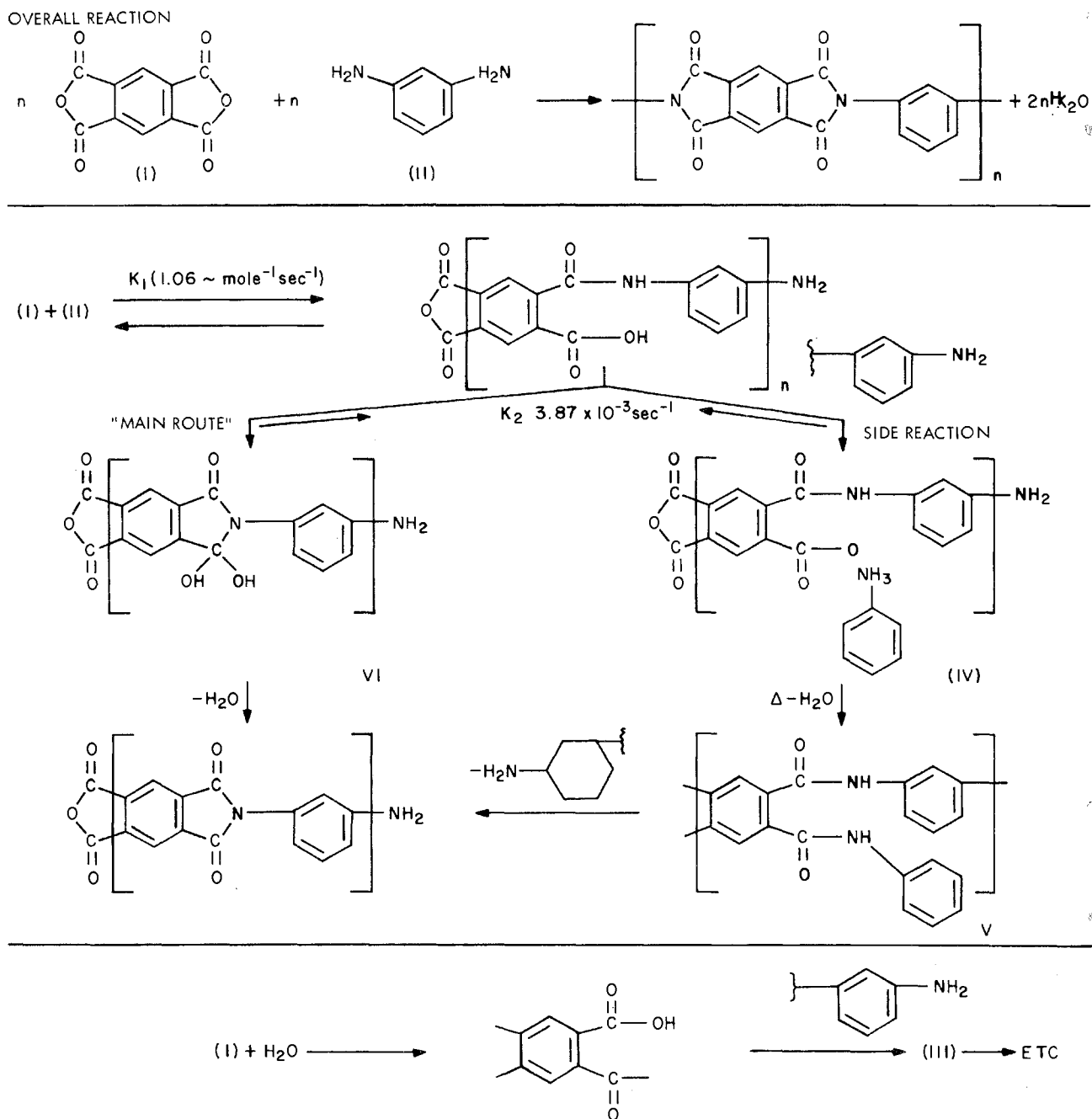
DMF - N.N. Dimethylformamide

** 0.5% solution in dimethyl sulfoxide

*** 1.5 hours at 120°C in DMAC

**** T_g increased to 282°C after exposure to 450°C for 1 hour under argon

They found that the polyimides possessed good thermal stability; the pre-polymers possessed good solubility and fusibility necessary for processing; and the test results of preliminary laminates show that polyimides possess a definite potential as high-temperature binding resins. A kinetic study was also performed of the reaction between pyromellitic dianhydride and m-phenylenediamine and is summarized in the following chart:

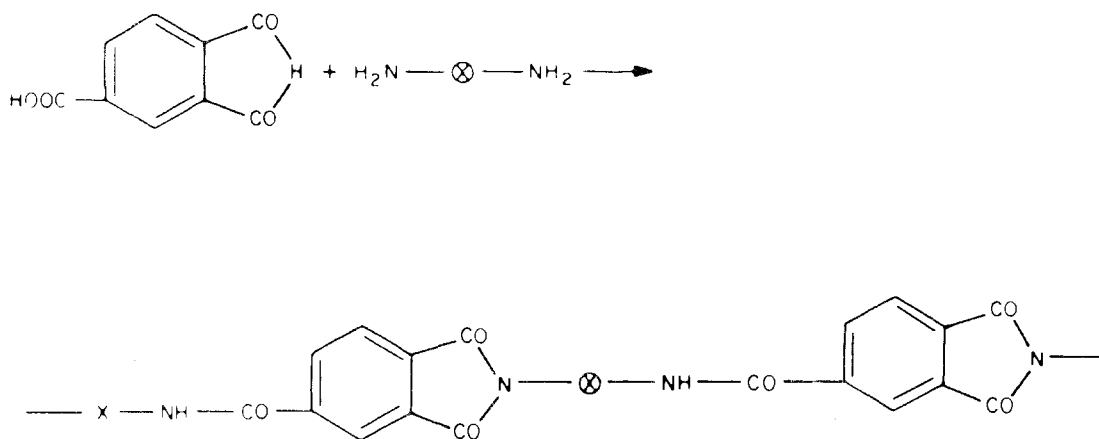


Amide-Imide Copolymerization

Amide-imide copolymers offer a wide range of chemical compositions and properties. Bower and Frost (Ref. 15) reported on the preparation of random amide-imides utilizing the interfacial reaction method. It consisted briefly of taking a solution of m-phenylenediamine (MPD), borax, sodium sulfite in water and stirring in a Waring blender while a solution of isophthaloyl chloride plus terephthaloyl chloride and xylene was added rapidly. The mixture was stirred for 2 minutes and the precipitated polymer was filtered off. It was then washed twice with water, once with acetone, and dried for one hour at 150°C. The product was a gray powder. The powder was dissolved in DMAC solvent and PMDA added in small portions until a maximum viscosity was reached. Films were cast from the resulting solution by baking at 150°C for 2 - 20 hours, followed by 1 - 4 hours at 200°C. These authors reported that clear tough films were obtained which could be stripped and creased repeatedly without cracking. This polymer had an amide/imide ratio of 3 for the quantities reported in the reactions of one set of experiments.

In another set of experiments utilizing a homogeneous reaction, Bower and Frost prepared a solution of MPD, triethylamine and DMAC in a Waring blender and Iso-phthaloyl chloride (IPC) added slowly. After a slight temperature rise and cooling to 30°C, the solution was filtered and diluted with additional DMAC. To the filtrate was added PMDA over a period of several hours until a maximum viscosity of 1150 centistokes was reached. Films were cast by baking for two hours at 150°C and then one hour at 200°C. These authors found that films up to 2 mils thick were clear and moderately flexible. Thicker films were opaque and brittle. This polymer had an amide/imide ratio of 1:1.

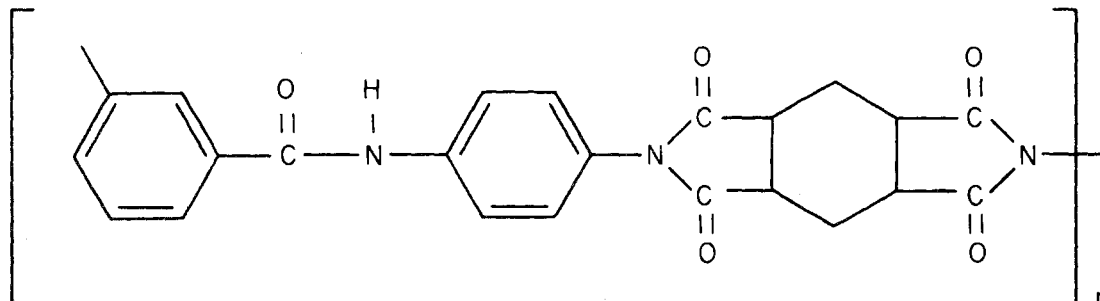
Trimellitic anhydride when reacted with diamines also yields amide-imide polymers and are the basic ingredients for the AMOCO Chemicals Corporation polyamide-imide AI polymer (Type 10):



[Ref. 64]

Jones and co-workers (Ref. 64) state that these polymers are somewhat inferior to the pyromellitimide polymers with respect to thermal and oxidative stability, the amide linkages being more vulnerable than the imide groups.

When 3,4'-diaminobenzanide is reacted with pyromellitic dianhydride in a 1:1 molar proportions, a polyamide-imide polymer results:



The amide group replaces the ether group usually used in imide polymerization.

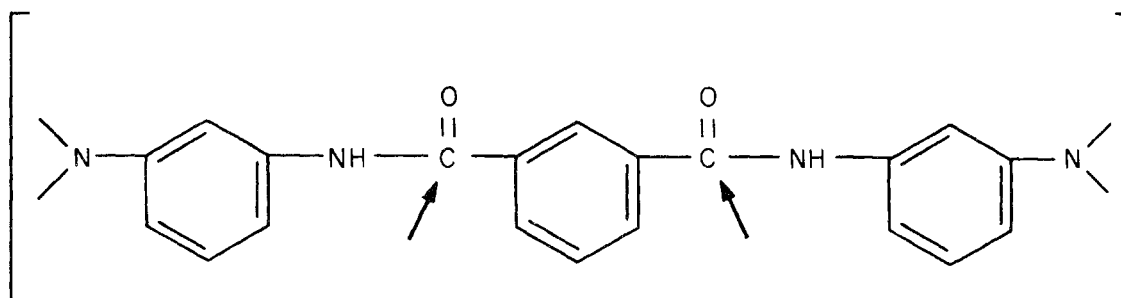
As the ratio of amide to imide is increased, in general, the flexibility of the films after curing at 150°C increases, but the films high in amide tend to degrade and become brittle when heated for any length of time at 300°C.

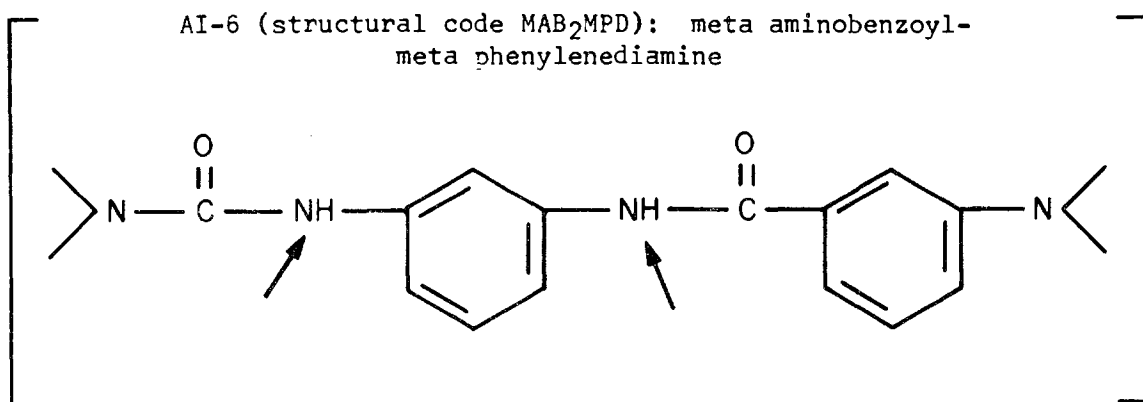
Westinghouse Electric Company investigated a wide range of copolymers having various proportions of amide to imide groups in the polymer chain. Such compounds have advantages of lower T_g (second order transition point), better solvent solubility, better alkali resistance, and improved flow properties in the mold. Their AI-7 copolymer was developed and reported by J.H. Freeman et al. (Westinghouse Research Report 62937-33501, September 30, 1962). Other amide-imide copolymers included the following:

AI-2	Amide/imide ratio = 1.0
AI-6	" " " = 1.0
AI-8	" " " = 0.5
AI-7	" " " = 3.0

Polymers AI-2 and AI-6 are alike in their molecular structure except that the positions of the carbonyl and nitrogen atoms of the amide groupings are reversed:

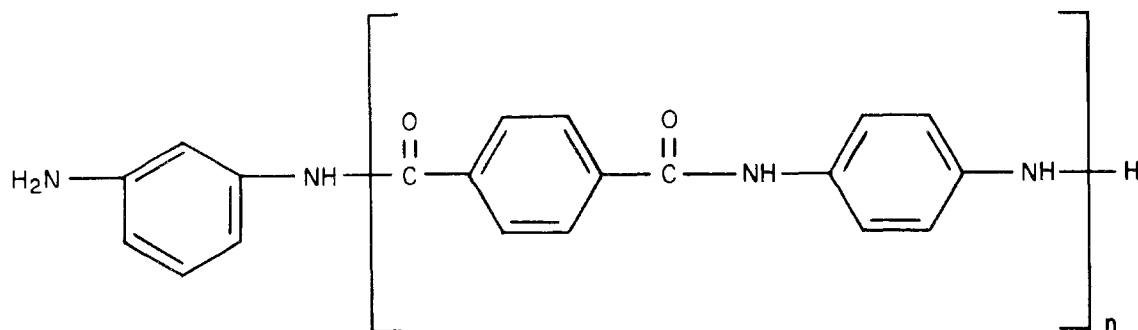
AI-2 (structural code MPD₂IP): meta Phenylenediamine-Isophthalic or N,N'-bis(2-aminophenyl)isophthalamide



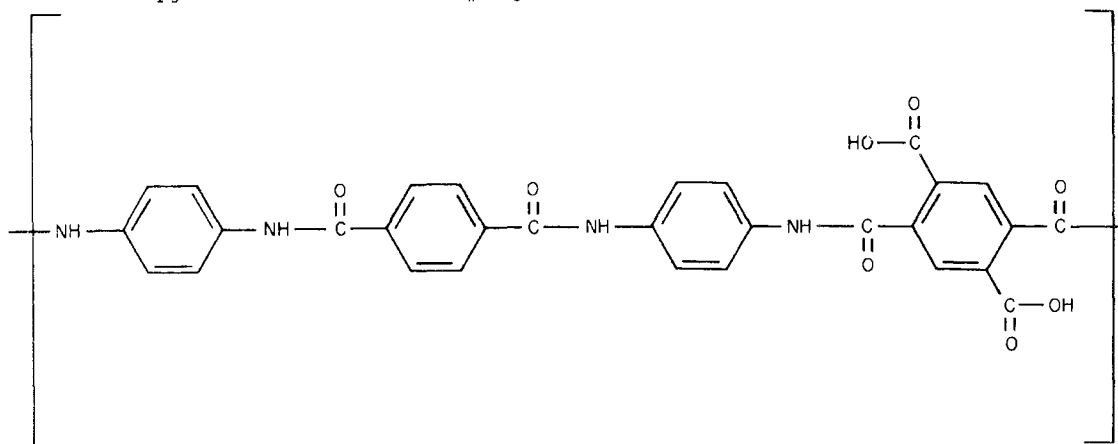


The arrows point to the differences in the two copolymers.

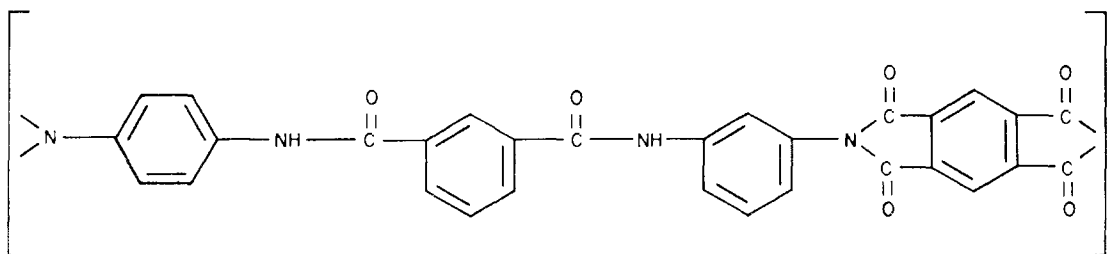
In general, aromatic amide and aromatic imide type polymers are formed by reactions between aromatic diamines and derivatives of aromatic acids such as acyl halides and anhydrides. A typical example of an amine terminated condensation product from the reaction of a diamine and diacid or its derivatives is shown in the following formula:



Such a product may then be reacted with pyromellitic dianhydride to produce a soluble pyromellitic acid polymer:

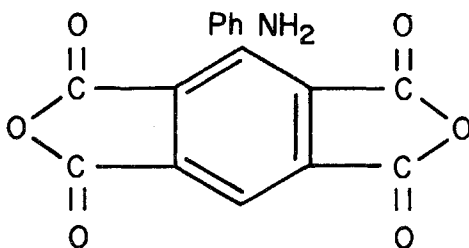


Upon the application of heat and pressure, this "B" stage material is converted to an infusible product:

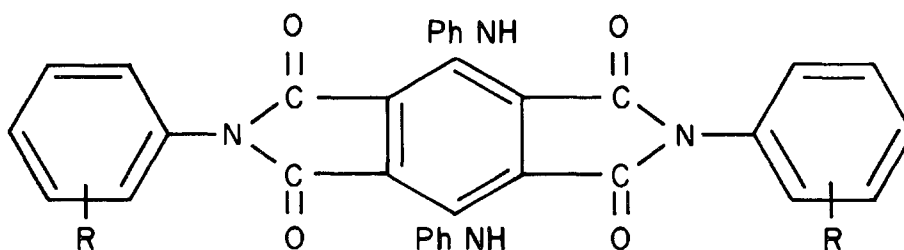


Although the polymer molecules are nominally linear in structure, they become infusible and insoluble under the influence of heat and pressure. However, there is little evidence of cross linking reactions unless additional functional groups are deliberately introduced. Freeman (Ref. 49) in his review of new organic resins and reinforced laminates for service to 300°C noted that the chemistry of polyamide-imide aromatic condensation products is rather complex and conditions of manufacture are rigid and carefully controlled.

Manukian (Ref. 128), at the Technische University Zurich, studied the reaction of the dianhydrides of dihalopyromellitic acids with aromatic amines and NH_3 . For example:

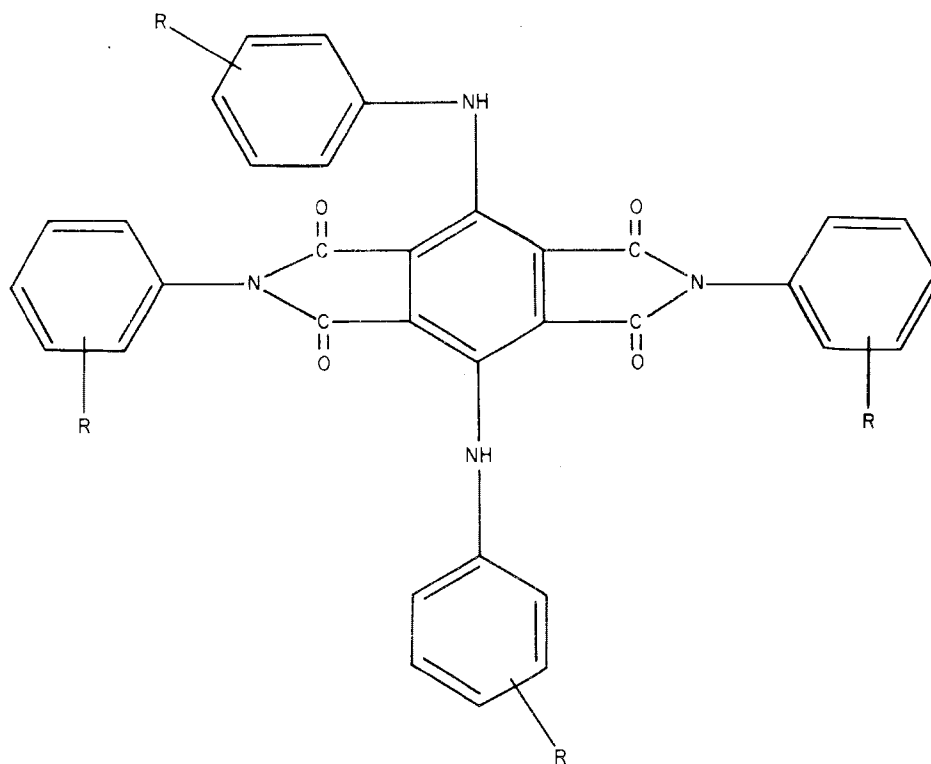


treated with various substituted anilines, yielded the following polyamide-imide structure:



where $R = \text{H}, \text{o-Me}, \text{p-Me}, \text{o-Cl}, \text{m-Cl}, \text{o-MeO}$.

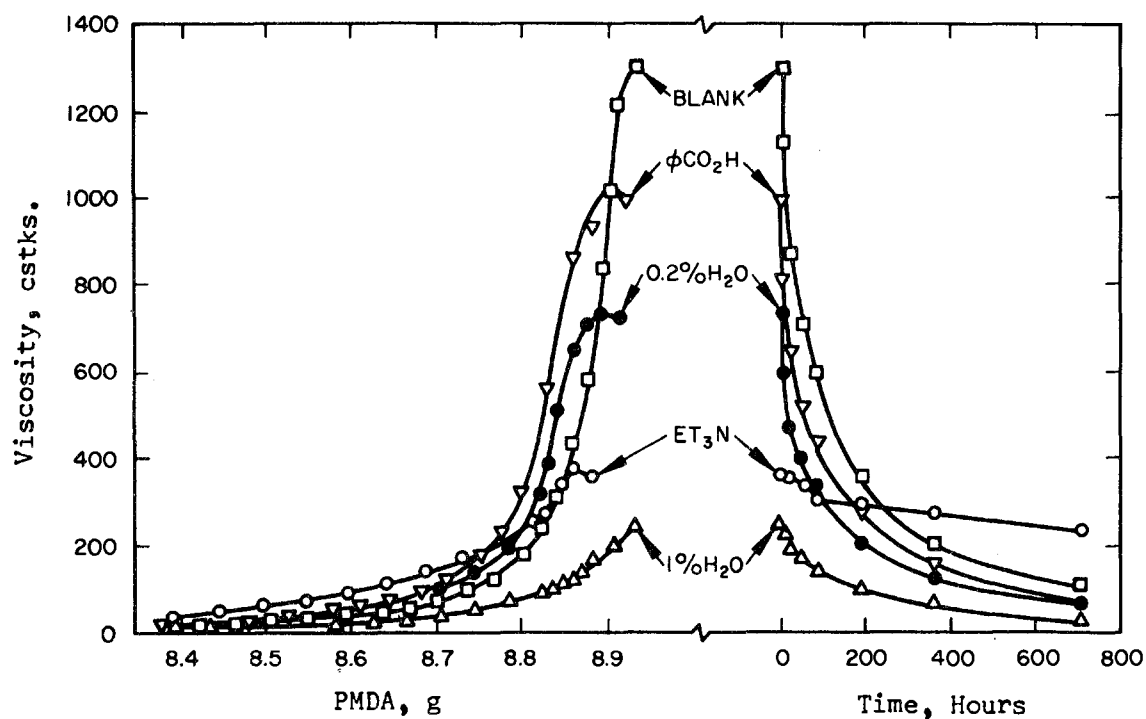
He also described the preparation of diimides which could be used as dyes for plastics and cited a typical diimide structure:



Frost and Bower of the Westinghouse Research Laboratories detailed the preparation of linear polymeric amide-modified polyimides obtained by the reaction of a suitable dianhydride with amide-modified aromatic diamines. They claimed such linear polymers are characterized by extraordinarily high thermal and oxidative stability, good film forming properties, toughness, and other properties which make them particularly suitable for use as wire coatings, molding and laminating resins, films for electrical insulating or mechanical use, varnish components, etc. Their patent, U.S. 3,179,635, details the wide range of amide-modifier groups as well as dianhydride groups usable to make such amide-imide copolymers (see Patent Section VI). Also included in the patent are detailed methods of preparing these solutions, the concentration of reactants, solvents, temperature and viscosity controls.

Miscellaneous Notes

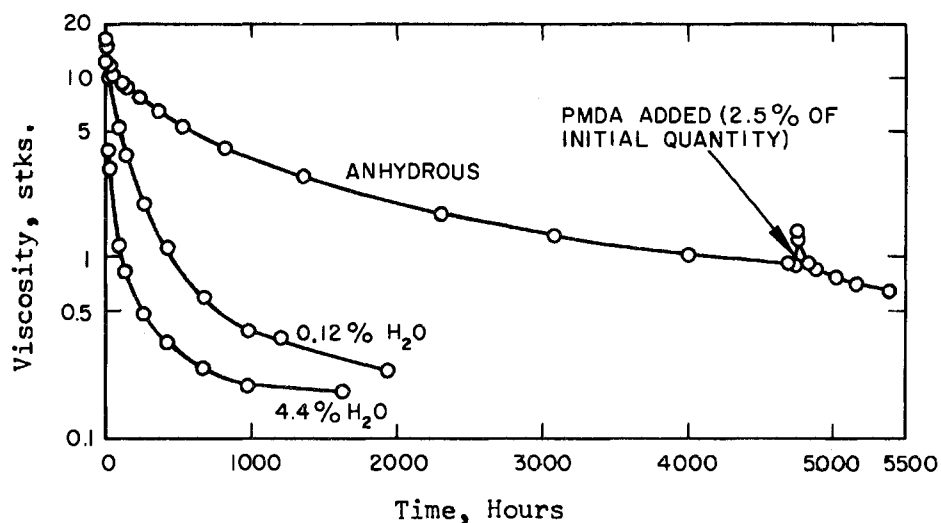
Bower and Frost (Ref. 15) in their study of aromatic polyimides reported that in most cases satisfactory results were obtained by using commercial reagents without further purification. Higher molecular weights were obtained by using purified reagents and anhydrous solvents. (This same claim was voiced in various patents in Section VI). They also investigated the effects of various additives on the dianhydride-diamine reaction and found the following viscosity changes to take place:



Viscosity changes of DAPE-PMDA solutions prepared in the presence of various additives.

[Ref. 15]

Frost and Kesse (Ref. 109) studied the effect of varying water contents on the viscosity of 10% DAPE-PMDA in DMAC and showed it to have a degrading effect:

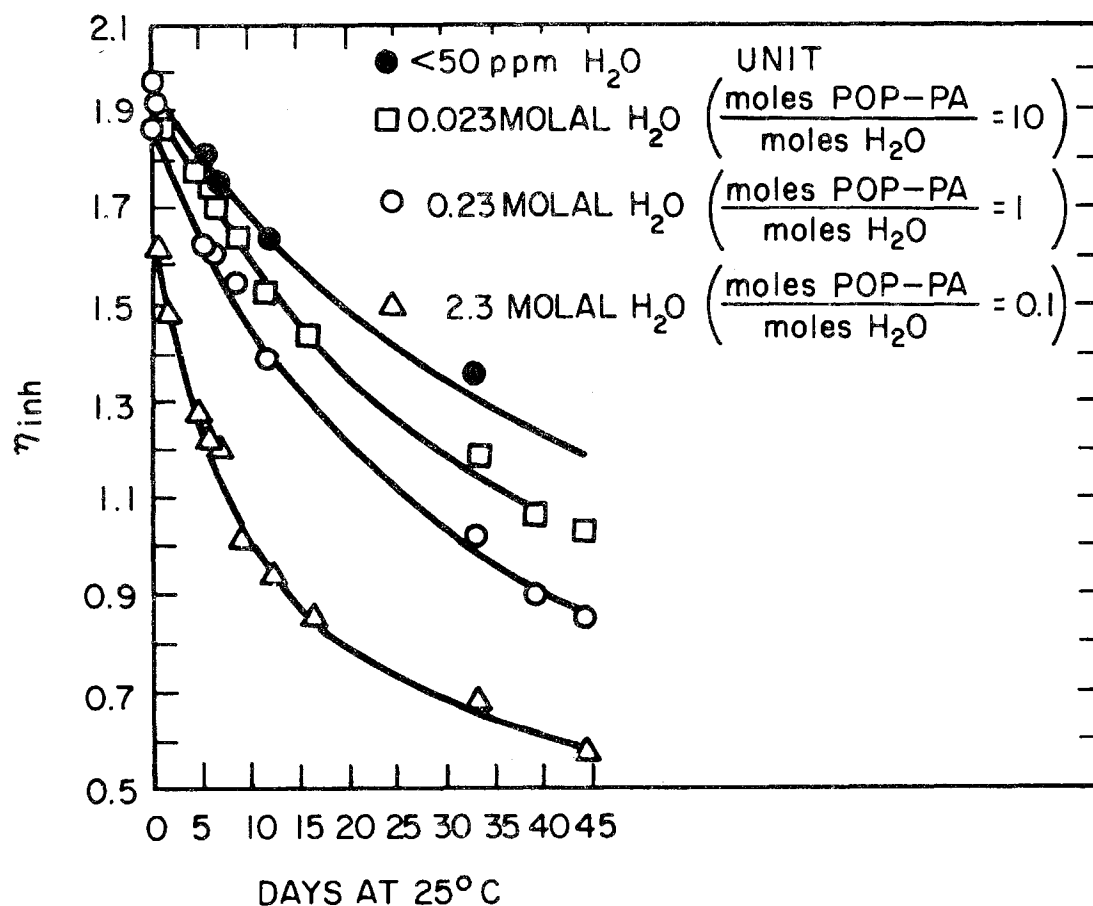


Effect of water on viscosity of 10% DAPE-PMDA in DMAC at 35°C

[Ref. 109]

They concluded that as the degradation by hydrolysis proceeds, the polymer is broken down into shorter chains terminated by amine or carboxyl groups.

Scroog and co-workers at E.I. du Pont de Nemours also investigated the effect of water on polyamic acid solutions and reported the following data:

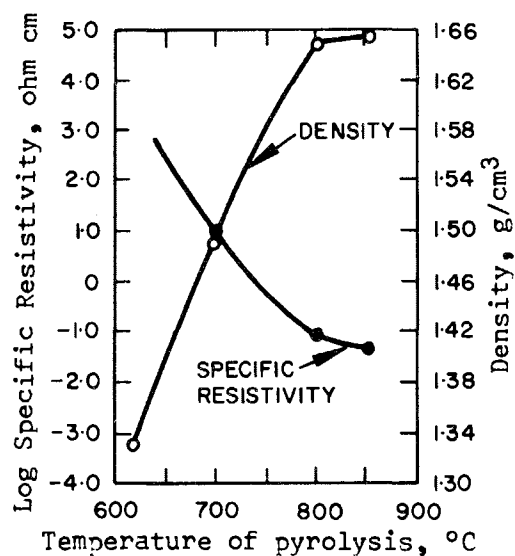


Effect of added water on 0.23 molal POP-PA [polyamic acid from bis(4-amino-phenyl)ether and pyromellitic anhydride] solution in dimethylacetamide (DMAc).

[Ref. 123]

Bruck (at the Applied Physics Laboratory of The Johns Hopkins University), in his studies of the vacuum pyrolysis of H-Film or poly[N,N'-(p,p'-oxydiphenylene) pyromellitimide], found that it would result in a semiconductor product. To prove such was the case, he conducted electron paramagnetic resonance (EPR), absorption (9140 Mc), resistivity, weight loss, density, and composition tests.

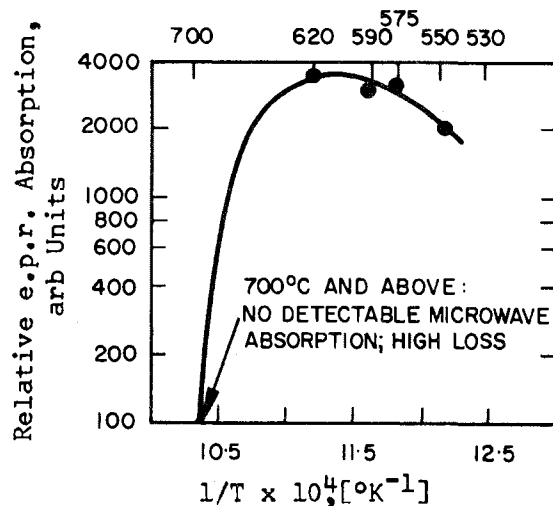
He obtained specific resistivities (at room temperature, 50 Mc) as low as 10 to 0.05 ohm-cm by vacuum (5×10^{-6} torr) pyrolysis between 620° and 850°C as depicted in the following graph:



Specific resistivity and density of poly[N,N'-(p,p'-oxydiphenylene) pyromellitimide] film as a function of temperature of pyrolysis

[Ref. 122]

His E.P.R. absorption data are summarized in the graph below as a function of pyrolysis temperature:

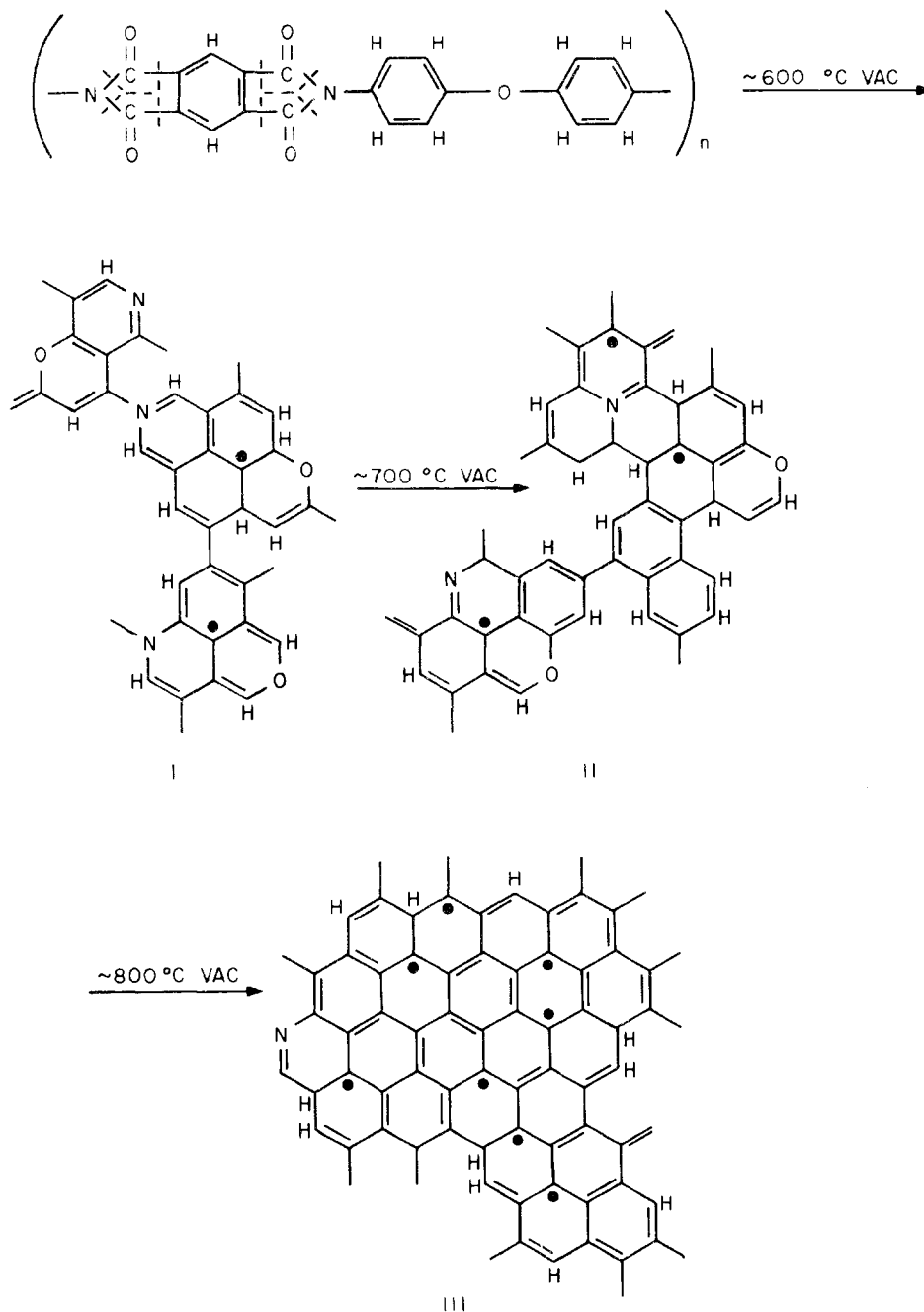


Relative e.p.r. absorption of poly[N,N'-(p,p'-oxydiphenylene) pyromellitimide] as a function of temperature of pyrolysis

[Ref. 122]

The E.P.R. absorption results agree with workers who have examined other semiconductor pyrolysates, coals and carbonaceous solids. The drop in the E.P.R. absorption coincides with a sharp decrease in specific resistivity.

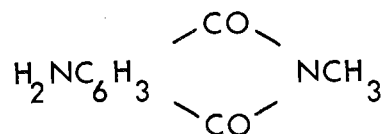
Bruck has provided a schematic diagram based on his experimental evidence to illustrate the pyrolytic conversion of H-Film or poly[N,N'-(p,p'-oxydiphenylene) pyromellitimide] to semiconducting products:



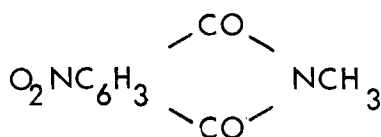
[Ref. 122]

It is of historical interest that polyamic acids and ester-imides were investigated as early as 1908 by Bogert and Renshaw (Ref. 137) during experiments on the preparation of 4-amino-0-phthalic acid and some of its derivatives.

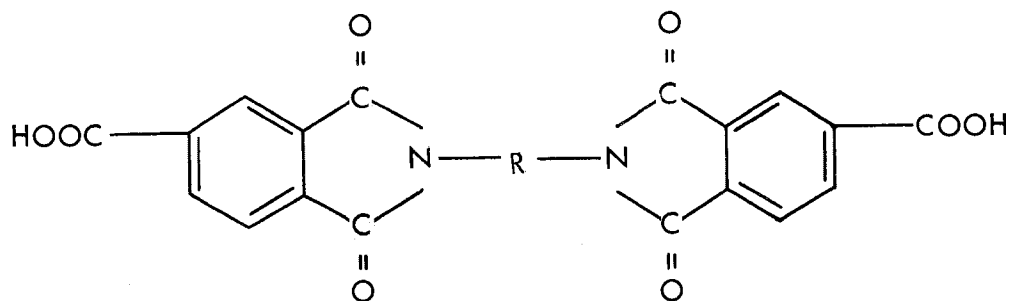
a) 4-aminophthalthemethylimide



b) 4-nitrophthalthemethylimide



Polyester-imide polymers have been investigated in Germany by Schmidt and co-workers (Ref. 88) and utilize trimellitic anhydride as well as terephthalic groups in conjunction with various diamines. These products have been designated as Terebec FN, Terebec FH, Experimental Product 109, and Experimental Product 115 by Dr. Beck and Company, GmbH, Hamburg, Germany. The Terebec compositions vary in nitrogen (from imide groups) from 1.6 to 7.4% and interestingly the heat shock resistance varies directly with the imide content. The authors give the following structural formula for these products:



formed by the reaction of trimellitic anhydride with aromatic diamines and esterifying with polyalcohols. Other advantages of these polyester-imides is their improved thermal aging durability, surface hardness, wear resistance and low dielectric loss characteristics at elevated temperatures.

The Schenectady Chemicals, Incorporated Company announced in September 1965 the availability of its "Isomid" single film magnet wire coating. It is a unique polyester-imide polymer bridging the complete temperature spectrum from Class A through H+ insulation wire coatings. The enamel is self-sufficient in that it does not require a topcoat.

ABBREVIATIONS, TRADENAMES, AND CODE DESIGNATIONS

AI - abbreviation for amide-imide.

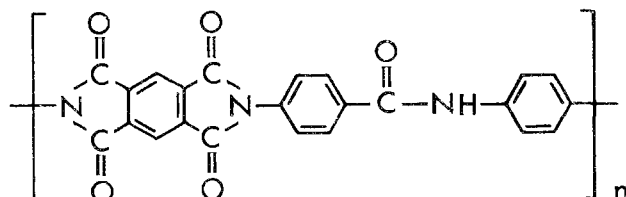
AI-1 - an polyamide-imide evaluated by the Westinghouse Electric Company made by reacting isophthaloyl chloride with two moles of meta-phenylenediamine.

AI-2 - an experimental polyamide-imide evaluated by Westinghouse Electric Company having the composition(MPD₂-IP)-PMDA where MPD₂IP is N,N'-bis 3-aminophenyl isophthalamide and PMDA is pyromellitic dianhydride.

AI-6 - an experimental polyamide-imide evaluated by Westinghouse Electric Company whose composition is given as (MAB₂-MPD)-PMDA where MAB is meta-aminobenzoyl, MPD is metaphenylene diamine, and PMDA is pyromellitic dianhydride.

AI-7 - an experimental polyamide-imide evaluated by Westinghouse Electric Company whose composition is identified as MPD₁₆-IP₉-TP₃-PMDA₄ where MPD is meta-phenylene diamine, IP is isophthalic anhydride, PMDA is pyromellitic dianhydride, TP was not identified.

AI-8 - a polyamide-imide experimental polymer evaluated by Westinghouse Electric Company using 3,4'-diaminobenzanilide and pyromellitic dianhydride. (also designated MAB-PPD-PMDA)



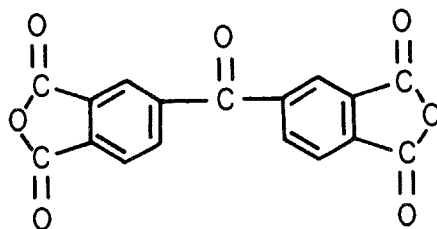
AI-83 - an experimental polyamide-imide material evaluated by Westinghouse Electric Company whose composition is identified as (MAB-PPD)-BTDA where (MAB-PPD) is the abbreviation for 3,4'-diaminobenzanilide and BTDA is benzophenone tetracarboxylic dianhydride.

AI-93 - an experimental polyamide-imide evaluated by Westinghouse Electric Company whose composition is identified by MPD-(MAB-PPD)₂BTDA₃ where MPD is meta-phenylene diamine, (MAB-PPD) is 3,4'-diaminobenzanilide, BTDA is benzophenone tetracarboxylic dianhydride.

AI-94 - an experimental polyamide-imide polymer made by using benzophenone tetracarboxylic dianhydride (BTDA) with MPD₂IP (meta phenylene-diamine-isophthalic) and DMAC (dimethylacetamide). It was evaluated by the Westinghouse Electric Company.

AI-95 - an experimental polyamide-imide evaluated by Westinghouse Electric Company having a composition which is identified as (MAB-PPD)₃.₄-IP₂.₄-PMDA. MAB-PPD is the abbreviation for 3,4'-diaminobenzanilide; IP is the abbreviation for isophthalic acid or anhydride; PMDA is the abbreviation for pyromellitic dianhydride.

- AI-96 - an experimental polyamide-imide evaluated by Westinghouse Electric Company whose composition is identified as DAPE-(MAB-PPD)BTDA₂ where DAPE is 4,4'-diaminophenyl ether, MAB-PPD is 3,4'-diaminobenzanilide, BTDA is benzophenone tetracarboxylic dianhydride.
- AI-107 - an experimental polyamide-imide polymer evaluated by the Westinghouse Electric Company formed by reacting benzophenone tetracarboxylic dianhydride (BTDA) with metaphenylenediamine (MPD) and TABA(?).
- AI-220 - tradename of the Anaconda Wire and Cable Company for their polyamide-imide (AI Polymer) magnet wire enamel. It is based on Trimellitic anhydride.
- AI-220 - tradename designation for an impregnating varnish offered by the Sterling Varnish Company based upon unmodified poly(amide-imide) polymers developed by Amoco Chemical Corporation in conjunction with Anaconda Wire and Cable Company.
- AI Polymer (Type 10) - a single system poly(amide-imide) resin based on Trimellitic anhydride of the Amoco Chemicals Corporation. It is not considered a copolymer.
- AIvar-220 - tradename designation for an impregnating varnish offered by the Sterling Varnish Company based upon modified poly(amide-imide) polymers developed by Amoco Chemical Corporation in conjunction with Anaconda Wire and Cable Company.
- Amoco AI Polymer (Type 10) - a single system poly(amide-imide) resin based on Trimellitic anhydride of the Amoco Chemicals Corporation. It is not considered a copolymer.
- Aramidyl - tradename designation for an aromatic amide-imide experimental polymer of the Westinghouse Electric Company.
- BTCA - abbreviation for benzophenone tetracarboxylic acid (an anhydride reactant material).
- BTDA - abbreviation for 3,3',4,4'-benzophenone tetracarboxylic dianhydride (an anhydride reactant material).



- DAPE - abbreviation for 4,4'-diaminophenyl ether (an amino reactant material).
- DAPS - abbreviation for diaminophenyl sulfide (an amino reactant material).

DDM - abbreviation for 4,4'-diamino-diphenyl methane (a diamine reactant material).

DDP - abbreviation for 4,4'-diamino-diphenyl propane (a diamine reactant material).

DMA - abbreviation for N,N'-dimethylacetamide (a solvent material).

DMAC - abbreviation for dimethyl acetamide (a solvent material).

DMF - abbreviation for N,N'-dimethylformamide (a solvent material).

DMSO - abbreviation for dimethyl sulfoxide (a solvent material).

"Durad" - the American Super-Temperature Wires, Incorporated designation of a modified polyimide wire insulation.

"Durad F" - the American Super-Temperature Wires, Incorporated designation for their FEP-coated wire with a "Durad" polyimide jacket.

"Durad T" - the American Super-Temperature Wires, Incorporated designation of TFE-coated wire with a "Durad" polyimide jacket.

EE-6379 - a pressure-sensitive "Kapton" Type H-Film tape product manufactured by the Permacel Company. It is composed of DuPont "Kapton" Type H-Film 1 mil thick backing with a high temperature pressure-sensitive silicone rubber adhesive.

EI - abbreviation for ester-imide.

EI-2 - a polyimide experimental polymer evaluated by Westinghouse Electric Company and made by taking Resin I-8 (MPD-BTDA) and adding 10% PE (pentaerythritol).

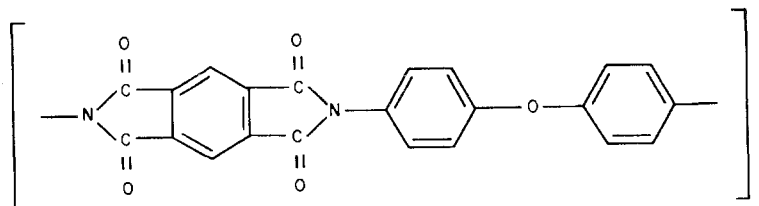
EI-11 - a polyimide experimental polymer evaluated by Westinghouse Electric Company. It is made by taking Resin I-1 (DAPE-PMDA) and adding 10% PE (or pentaerythritol).

FXM-34-B-25 - an experimental polyimide adhesive developed by the American Cyanamide Company (Bloomington Department) for high temperature applications. The carrier is 112 glass cloth with Volan A finish; film weight is 13-14 lbs/sq. ft; volatiles 10-12% after 60 minutes at 500°F.

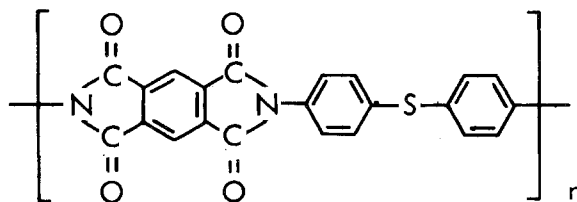
Glass/ML - an experimental insulated (0.0132") prepared by the General Electric Company, Schenectady, New York using DuPont ML polyimide solution on coated double glass fiber serving and evaluated in Reference 72.

H-Film - tradename for a polyimide film material marketed by the E.I. du Pont de Nemours Company now known as "Kapton" Type H-Film.

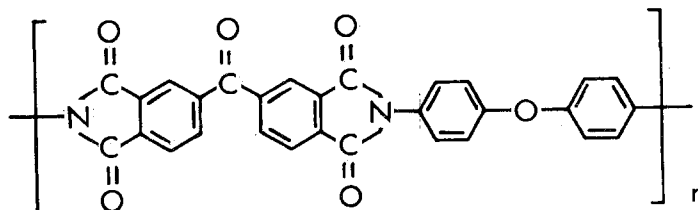
- HML Asbestos - an experimental insulation (0.0052") prepared by the General Electric Company, Lowell, Massachusetts comprising aluminum phosphate impregnated, felted asbestos over HML enamel and evaluated in Reference No. 72.
- HML(FW) - an experimental insulation (0.0010") prepared by the General Electric Company, Fort Wayne, using a heavy aromatic polyimide enamel and evaluated in Reference 72.
- HML(GE) - an experimental insulation (0.0014") made by the General Electric Company, Schenectady using DuPont "Pyre-ML" heavy aromatic polyimide enamel and evaluated in Reference 72.
- HML(PD) - an experimental insulation (0.00105") prepared by the Phelps Dodge Company, Fort Wayne using a heavy aromatic polyimide enamel and evaluated in Reference 72.
- I-1 - a polyimide experimental polymer evaluated by the Westinghouse Electric Company using 4,4'-diaminophenyl ether and pyromellitic dianhydride (also designated DAPE-PMDA).



- I-3 - an experimental polyimide material evaluated by Westinghouse Electric Company made by reacting pyromellitic dianhydride with meta-phenylene diamine. (Also designated MPD-PMDA).
- I-6 - a polyimide experimental polymer evaluated by Westinghouse Electric Company using 4,4'-diaminophenyl sulfide and pyromellitic dianhydride. (Also designated DAPS-PMDA).

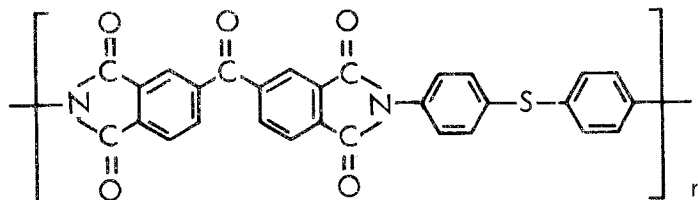


- I-7 Laminate - a Westinghouse Electric Corporation developmental polyimide using 181-A1100 glass cloth and made from Diaminophenyl Ether and Benzophenone Tetracarboxylic Dianhydride (DAPE-BTDA).



I-8 - a polyimide experimental polymer evaluated by Westinghouse Electric Company and made by reacting meta-phenylene diamine with benzophenone tetracarboxylic dianhydride. (Also designated as MPD-BTDA).

I-9 - an experimental polyimide evaluated by the Westinghouse Electric Company having the molecular structure:



also identified as DAPS-BTDA where DAPS is diaminophenyl sulfide and BTDA is benzophenone tetracarboxylic dianhydride.

I-10 - an experimental polyimide evaluated by Westinghouse Electric Company whose composition is identified as MPD₂-PMDA-BTDA where MPD is meta-phenylene diamine, PMDA is pyromellitic dianhydride, and BTDA is benzophenone tetracarboxylic dianhydride.

I-11 - an experimental polyimide evaluated by Westinghouse Electric Company whose composition is identified as MPD₃-PMDA₂-BTDA where MPD is meta-phenylene diamine, PMDA is pyromellitic dianhydride and BTDA is benzophenone tetracarboxylic dianhydride.

I-12 - an experimental polyimide evaluated by Westinghouse Electric Company whose composition is identified as MPD₃-PMDA-BTDA₂ where MPD is meta-phenylene diamine, PMDA is pyromellitic dianhydride, and BTDA is benzophenone tetracarboxylic dianhydride.

I-13 - an experimental polyimide evaluated by the Westinghouse Electric Company whose composition is identified as DAPE-MPD-BTDA₂ where DAPE is 4,4'-diaminophenyl ether, MPD is meta-phenylene diamine, BTDA is benzophenone tetracarboxylic dianhydride.

I-14 - an experimental polyimide evaluated by Westinghouse Electric whose composition is identified by DAPE₃-MPD-BTDA₄ where DAPE is 4,4'-diaminophenyl ether, MPD is meta-phenylene diamine, BTDA is benzophenone tetracarboxylic dianhydride.

I-15 - an experimental polyimide evaluated by Westinghouse Electric Company whose composition is identified as DAPE-MPD₃-BTDA₄ where DAPE is 4,4'-diaminophenyl ether, MPD is meta-phenylene diamine, BTDA is benzophenone tetracarboxylic dianhydride.

IP - abbreviation for isophthalic.

IPC - abbreviation for isophthaloyl chloride (an acid salt).

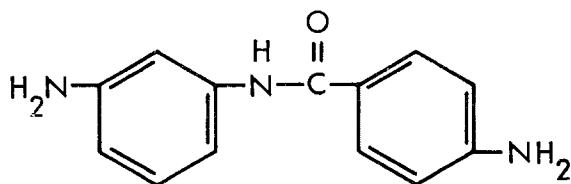
Isomid - a tradename product for a polyester-polyimide resin manufactured by the Schenectady Chemicals Incorporated, Schenectady, New York.

"Kapton" Type H - tradename for a polyimide film material marketed by the E.I. du Pont de Nemours and Company. It was formerly called H-Film.

"Kapton" HB Film - an experimental E.I. du Pont de Nemours laboratory film material containing Teflon TFE dispersed in a "Kapton" Type H-Film.

MAB - abbreviation for meta aminobenzoyl (an amino reactant material).

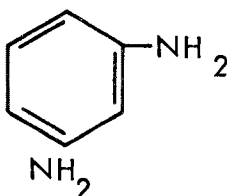
MAB-PPD - abbreviation for 3,4'-diaminobenzanilide.



MDA - abbreviation for 4,4'-methylenediamine. (An amino reactant material).

MK Coated Fabric - an experimental polyimide developed by the E.I. du Pont de Nemours Company around 1959 but never put into production. It was probably similar to "Pyre-ML."

MPD - abbreviation for meta-phenylene diamine (a diamine reactant material).



MPD₂IP - an experimental polyimide evaluated by the Westinghouse Electric Company and identified as N,N'-bis 3-aminophenyl isophthalamide.

MQ - a polyimide resin in solution form developed by the E.I. du Pont de Nemours and Company.

MRE-66 - a polyimide in solution form at 40% solids content developed by the E.I. du Pont de Nemours and Company.

NMP - abbreviation for N-methyl pyrrolidone (a solvent material).

PAA - polyamic acid

PAP - abbreviation for 2,2'-bis-(3,4-dicarboxyphenyl)propane (an anhydride reactant material).

PE - abbreviation for penta erythritol.

PEDA - abbreviation for bis(3,4-dicarboxyphenyl)ether dianhydride. (An anhydride reactant material).

PI-1100 - an E.I. du Pont de Nemours Company thermoplastic high temperature binder solution containing 18.6% solids with a viscosity of 35-55 poises at 77°F.

PI-1101 - an E.I. du Pont de Nemours Company thermosetting high temperature binder solution containing 18.3% solids with a viscosity of 10-20 poises at 77°F.

PI-1200 - an E.I. du Pont de Nemours Company thermoplastic high temperature binder solution containing 18.4% solids and a viscosity of 13-35 poises at 77°F.

PI-1501 - an E.I. du Pont de Nemours Company polyimide containing 45% solids in N-methylpyrrolidone/xylene solvent. The resin is "B-staged" by baking 15-25 minutes at 275°F before bonding.

PI-2101 - a thermoset polyimide binder solution developed by the E.I. du Pont de Nemours Company containing 18.3% solids and viscosity of 10-20 poises at 77°F. The solvent used is NMP/DMF.

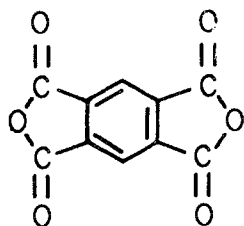
PI-2501 - an E.I. du Pont de Nemours Company "B-Staged" polyimide fiberglass prepreg with 181E Glass-All100 finish.

PI-3301 - an E.I. du Pont de Nemours Company polyimide thermosetting high temperature binder solution containing 53.0% solids and a viscosity of 60-90 poises at 77°F. The solvent system used is NMP/xylene.

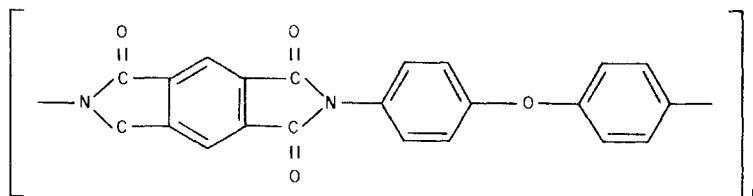
PI-3301 FGR - an experimental E.I. du Pont de Nemours Company polyimide pre-preg fiber glass roving for filament wound constructions.

PMA - abbreviation for pyromellitic acid (an anhydride acid reactant material).

PMDA - abbreviation for pyromellitic dianhydride (an anhydride reactant material).



PMI - abbreviation for poly-4,4'-oxydiphenylpyromellitimide (a polymeric polyimide resin material).



POP - abbreviation for 4,4'-diamino-diphenyl ether (a diamine reactant material).

POP-PI - abbreviation for poly[N,N'(p,p'-oxydiphenylene) pyromellitimide] (chemical name for "Kapton" Type H Film).

PP - abbreviation for benzidine (a diamine reactant material).

PPD - abbreviation for para-phenylenediamine (a diamine reactant material).

PPDA - abbreviation for 2,2'-bis(3,4-dicarboxyphenyl) propane dianhydride (an anhydride reactant material).

PPMI - abbreviation for polypyromellitimide.

PSO₂P - abbreviation for 4,4'-diamino-diphenyl sulfone (a diamine reactant material).

PSP - abbreviation for 4,4'-diamino-diphenyl sulfide (a diamine reactant material).

"Pyre-ML" - tradename of E.I. du Pont de Nemours for their polyimide binder solution resin compositions.

RC-675 - a "Pyre-ML" polyimide solution containing 16.5% solids and a viscosity of 60-80 poises at 25°C manufactured by the E.I. du Pont de Nemours Company.

RC-5019 - a "Pyre-ML" polyimide solution containing 17.0% solids and a viscosity of 6.25-10.75 poises at 25°C. It is manufactured by the E.I. du Pont de Nemours Company.

RC-5034 - a "Pyre-ML" polyimide solution containing 18.5% solids and a viscosity of 60-80 poises at 25°C; manufactured by the E.I. du Pont de Nemours Company.

RC-5060 - an E.I. du Pont de Nemours Company experimental polyimide insulating varnish containing 19% solids and a viscosity of 5-9 poises at 25°C.

RC-B-2495 - an E.I. du Pont de Nemours Company experimental polyimide insulating varnish containing 45% solids and a viscosity of 2-5 poises at 25°C.

RK-692 - an E.I. du Pont de Nemours Company insulating varnish with a solids content of 13% and a viscosity of 6-10 poises at 25°C.

RK-5034 - a "Pyre-ML" polyimide varnish identical to RC-5034 except that a flow-agent has been added to minimize "eyeholing." It is manufactured by the E.I. du Pont de Nemours Company.

Skygard 700 - a heat reactive aromatic polyimide (unformulated) system containing active amino and carboxyl groups. It has a solids content of 60-64%, viscosity of 4000-10 000 cps, specific gravity of 1.15-1.18 and a pH ranging from 4.0-4.7. It is a product of the Monsanto Chemical Company.

SP-Polymer - a moldable polyimide product developed by the E.I. du Pont de Nemours Company. It has been available as a thick opaque sheet.

Terebec (FH, FN) - tradename for a series of polyester-imide resins developed in Germany by the Dr. Beck and Company.

TMA - abbreviation for trimellitic anhydride and used in the formulation of polyamide-imide resins (e.g. AI-220 and Amoco AI Polymer Type 10)

TPC - abbreviation for terephthaloyl chloride (an acid salt).

Tufflite - tradename designation of the Tensolite Insulated Wire Company for their "Kapton" Type HF polyimide film insulated wire construction.

"Vespel" - tradename designation for molded parts made from polyimide SP-1 polymers by the E.I. du Pont de Nemours Company.

W-91173 - an unformulated polyimide resin of the E.I. du Pont de Nemours Company.

II. PROPERTIES

INTRODUCTION

The polyimides (including the polyamide-imides and polyester-imides)' as a class of plastics possess a unique combination of properties hitherto unavailable among the polymeric materials.

From the review of the property data compiled in this section, it is apparent that the bulk of the data is on "Kapton" Type H-Film polyimide material. Comparatively little data are available on the polyamide-imide and polyester-imide resins. A considerable amount of thermal and elevated temperature mechanical property data has been generated on the polyimides in general as a result of its potential high temperature capabilities.

A few general property characteristics for some polyimide forms are stated here and the detailed data given below. Thermal stability combined with retention of physical properties at high temperatures appears to be the most outstanding properties common to all forms of the polyimide materials. Without question, the practical continuous service temperature range of the materials is roughly from 300° to about 700°F. The thermal stability behaviour is demonstrated by the TGA (Thermal gravimetric analysis) and DTA (Differential thermal analysis) weight loss of which the literature was prolific as well as of other thermal properties and thermal aging effects on various mechanical and electrical properties.

"Kapton" Type H-Film material is an infusible extremely tough, polymeric film, similar to Mylar. It exhibits an extremely high degree of thermal stability and also maintains flexibility at cryogenic temperatures. A significant advantage of "Kapton" Type H-Film over currently available materials is the retention of properties over a wide range of temperature. Perhaps the most outstanding property of "Kapton" Type H-Film is its stability at very high temperatures. It has no melting point, is nonflammable, and only chars when exposed to direct flame. "Kapton" Type H-Film is comparable to commercially available Mylar film mechanically and electrically, but exhibits far superior thermal and dimensional stabilities and resistance to organic solvents and chemicals. Abrasion resistance and flame resistance are also outstanding. The remaining properties and applications of "Kapton" Type H-Film are discussed and treated in the individual property sections and in the applications section.

"Pyre-ML" insulating varnish has the same thermal stability, electrical properties, radiation resistance and chemical resistance associated with "Pyre-ML" wire enamel since the film-forming solids are the same.

"Pyre-ML" wire enamel has an excellent balance of thermal, chemical, electrical and mechanical properties and is widely used in high temperature wire applications (see Applications Section IV).

Mechanical and electrical properties of Polymer SP - the moldable form of polyimide - under severe environments surpass those of most other rigid plastics, both thermosetting and thermoplastic. Polymer SP does not melt and cannot be fabricated by conventional injection molding or extrusion methods. When exposed to direct flame, the polymer gradually chars, rather than melts or burns. It is a strong, rigid material which has excellent resistance to heat, abrasion, and nuclear radiation. It has very good electrical properties and exhibits low outgassing at high vacuum.

"Vespel" polyimide parts are stiff, hard and are designed for high temperatures - 500°F and up - depending upon the environment and service life required. They have outstanding mechanical and electrical properties including: excellent high temperature stability, low creep, high wear resistance, good low temperature properties (at -423°F), low coefficient of friction, good fatigue strength, high bearing PV limit, high dielectric strength, highly resistant to ionizing radiation, and good thin-wall stiffness.

The Sterling amide-imide varnishes (AI-220 and Alvar-220) possess excellent electrical, chemical and mechanical properties, as well as retention of high bond strength and low weight loss at elevated temperatures.

Amoco AI Polymer, also a poly(amide-imide) offers the thermal stability, very high thermal plastic flow temperatures, high dielectric strength, good chemical resistance, and low loss in weight and electrical properties that are obtained with polyimides, polybenzimidazoles, and other recently developed aromatic polymers.

The newly introduced polyester-polyimide "Isomid" by the Schenectady Chemicals Company has been investigated only superficially and apparently there is insufficient information at this time to provide a comparison of properties with polyimide and polyamide-imide enamels. Schenectady says particular attention should be directed to the heat shock, cut-through, and thermal flexibility property characteristics which are superior to the well-known polyester Isonel 200/17 enamels.

II. PROPERTIES

A. CHEMICAL

For electrical insulation to be generally useful, it must perform its functions under a wide variety of atmospheric conditions and in environments containing solvents, oils, and other chemicals.

H-Film

Tatum and co-workers at the DuPont Company found H-Film to be insoluble in all common solvents with no change in physical properties after six months immersion in hexane, benzene, dimethyl formamide, toluene, chloroform, methanol, chlorobenzene and acetone at room temperature and in hydrocarbon and chlorinated biphenyl transformer oils at 150°C. The hydrolytic stability of the film is very good with 75% of the elongation and 90% of the impact value being retained after 16 days in boiling water. In alkali and concentrated acid, however, H-Film is subject to degradation. After exposure to air containing 2% ozone for 3700 hours, H-Film still retains 50% of its elongation. Exposure to sunlight in Florida resulted in embrittlement of H-Film in approximately six months, indicating a limit to its usefulness in applications requiring constant exposure to sunlight. The DuPont Company has published the following table of chemical properties for its H-Film polyimide:

PROPERTY	TYPICAL VALUES			TEST CONDITION	TEST METHOD
	% Tensile Retained	% Elongation Retained	% Modulus Retained		
CHEMICAL PROPERTIES					
CHEMICAL RESISTANCE TO:				Days Immersed at Room Temperature	
Benzene	100	82	100	365	
Toluene	94	66	97	365	
Methanol	100	73	140	365	
Acetone	67	62	160	365	
10% Sodium Hydroxide		Degrades		5	
Glacial Acetic Acid	85	62	102	36 days at 110°C	
p-Cresol	100	77	102	22 days at 200°C	
"Arochlor"®	100	53	142	365 days at 200°C	
Transformer Oil	100	100	100	180 days at 150°C	
Water pH = 1	65	30	100	14 days at 100°C	
pH = 4.2	65	30	100	14 days at 100°C	
pH = 7.0	65	30	100	70 days at 100°C	
pH = 8.9	65	20	100	14 days at 100°C	
pH = 10.0	60	10	100	4 days at 100°C	
FUNGUS RESISTANCE	Inert			12 months	Soil Burial
MOISTURE ABSORPTION	1.3%			50% Relative Humidity at 23.5°C	Constant Environment Room
	2.9%			Immersion for 24 hrs. at 23.5°C	ASTM D-570-59T
HYGROSCOPIC COEFFICIENT OF EXPANSION	2.2 x 10 ⁻⁵ in/in/% Relative Humidity			72°F 20% - 80% Relative Humidity	
PERMEABILITY					
Gas	cc/(100 in ²) (24 hrs.) (atm/mil)			23°C	ASTM D-1434-58
Carbon Dioxide	45				
Hydrogen	250				
Nitrogen	6				
Oxygen	25				
Helium	415				
Water Vapor	gm/(100 in ²) (24 hrs.)/mil				ASTM E-96-53T
	5.4				

[Ref. 29]

SP Polymer

Todd and Wolff (1965) state that the moisture absorption of Polymer SP parallels that of many thermosetting resins--absorption in 24 hours at room temperature (ASTM-D-570) is 0.31%. The material is insoluble in organic solvents, and resists attack from acids. However, it is attacked by strong bases, and will not resist prolonged exposure to boiling water or steam. Exposure to steam or high temperature water produces a rapid decrease in tensile and flexural strength. However, after 300 hours, the properties level off at about 45% of their original values, after which the resins still offer considerable useful properties. Water absorption of the unfilled polyimide (according to ASTM D570) parallels that of many thermosetting resins: 0.317% for 24-hour immersion at 73°F and 0.82% for 48-hour immersion at 122°F. Equilibrium water absorptions at 50% RH is 1%. The dimensional changes accompanying these water absorptions are in the range of 0.001 to 0.002 inch/inch. Prolonged boiling in water produces a maximum water absorption of 3% with accompanying changes up to 0.006 inch/inch. The polymer is also resistant to hydraulic fluid, transformer oil, benzene, chloroform and hexane. It is, moreover, not suitable for prolonged exposure to hydrazine and nitrogen tetroxide.

Campbell and Cheney (1965) claim that Polymer SP is highly resistant to attack by most organic materials, including aircraft fuels and oils, even up to 700°F. It is not resistant to strong alkalis and is affected by high-temperature water; it will lose about 40% of its tensile strength, for example, after 100 hours in boiling water.

Cheney (DuPont Co.) contributed the following unpublished data on the effect of various chemicals on the tensile strength of unfilled SP Polymer:

<u>Chemical</u>	<u>Temp.</u>	<u>Time</u>	<u>Retention of Tensile Strength</u>
15% Nitric Acid	R.T.	720 Hours	66%
70% Nitric Acid	R.T.	120 Hours	37%
38% Hydrochloric Acid	R.T.	120 Hours	71%
UDMH Vapor	R.T.	113 Hours	48%
UDMH Liquid	R.T.	78 Hours	42%
Aerozine 50 Vapor	R.T.	120 Hours	67%
5% Sodium Hydroxide	R.T.	120 Hours	56%
"Skydrol" 500A	R.T.	233 Hours	100%
	356°F	24 Hours	88%
N ₂ O ₄ Vapor	R.T.	120 Hours	60%
	R.T.	720 Hours	43%
O Dichlorobenzene + 1% Tricresyl Phosphate	342°F	720 Hours	100%
Water	212°F	400 Hours	48%
	212°F	1000 Hours	44%

[Ref. 31]

"Pyre-ML" and Binder Solutions

The DuPont Company has conducted an extensive test program on the resistance of "Pyre-ML" coated fabrics to a wide range of solvents, acids, bases, and fluorinated hydrocarbons:

<u>Solvent</u>	<u>Condition</u>	<u>Exposure Period</u>
Acetone	No Change ↓	4 Weeks
Methyl Ethyl Ketone		↓
Formaldehyde		
Methanol		
Ethanol		
Ethyl Acetate		
"Skydrol"		
Carbon Disulfide		
Carbon Tetrachloride		
Chloroform		
Perchlorethylene		
O-Dichlorobenzene		
Benzene		
Toluene		
Hexane		
Naptha		
Xylene		
Dimethyl Formamide		1 Day
Dimethyl Acetamide		↓
N-Methyl Pyrrolidone		
γ-Butyrolactone		
"Freon-22"		4 Hours

<u>Reagent</u>	<u>Condition</u>	<u>Exposure Period</u>
NaOH (5%, aq.)	Somewhat Stiffened	4 Weeks
NaOH (1%, aq.)	Slight Stiffening	↓
NH ₃ (5%, aq.)	Slight Stiffening	
H ₂ SO ₄ (Conc., 96%)	Coating Dissolved	1 Day
HNO ₃ (Conc., 70%)	Coating Embrittled, Deteriorated	↓
HCL (Conc., 37%)	Coating Became Cloudy, Softened	
H ₂ SO ₄ (5%, aq.)	Somewhat Stiffened	↓
HNO ₃ (5%, aq.)	Slight Discoloration, No Softening	
HCL (5%, aq.)	Slight Stiffening	↓
H ₂ SO ₄ (1%, aq.)	Slight Stiffening	
HNO ₃ (1%, aq.)	No Softening, Slight Discoloration	4 Weeks
HCL (1%, aq.)	No Softening, Slight Discoloration	↓

[Ref. 34]

Schweitzer (1964) reports that moisture-resistance tests show no failure for four months at 100°F and 100% RH for "Pyre-ML" Binder Resin Solutions. Baked films showed no signs of blistering, swelling, or softening after exposure for 24 hours at room temperature to such solvents as xylene, cresylic acid, carbon tetrachloride, naphtha, acetone, alcohol, ethyl acetate, hexane, transformer oil, "Freon" fluorocarbons, and askarel. However, strong acids and bases cause film deterioration. The coatings are not affected by exposure to standard synthetic greases, either for three months at 70°F or 6 hours at 545°F.

Anaconda Cable and Wire Company claims that their polyimide-amide polymer enamel "AI-220" possesses good chemical resistance properties. The film is said to have high resistance to chemicals likely to be encountered during processing and operation; and it will not soften when exposed to most varnish thinners, cleaners, and degreasers. In addition, it is claimed to have high resistance to softening from the catalyst systems of epoxy encapsulation compounds. High resistance to solvent shock is another feature cited for the "AI-220" polymer. It is reported that severe stressing followed by immersion in boiling xylol will not cause cracking of the film. Anaconda also states that the wire is highly resistant to solvent crazing when the stressed film is exposed to xylol, alcohol, naphtha, mineral spirits, and other common varnish thinners. This minimizes the possibility of the enamel crazing or cracking due to inadequate prebake. A prebake at 150°C before varnishing is recommended as good practice to remove moisture and stress relieve the film against long term effects of residual solvents. A prebake of at least 200°C is recommended when using varnishes containing dimethylacetamide, dimethylformamide, and n-methylpyrrolidone. These solvents can craze the enamel if it is not stress relieved.

Hand-in-hand with the new wire's thermal and chemical stability is its reported high order of compatibility with other insulations. Several troublesome areas of compatibility sometimes found with other high temperature wires apparently have been minimized; specifically hydrolysis, and degradation in the presence of "Freon" refrigerants and chlorine-containing insulations. Anaconda offers the following table showing the excellent chemical resistance of the "AI-220", Heavy "Isonel-200/17", and Heavy "Pyre-ML" magnet wire coatings:

Comparative Properties of No. 18 AWG Magnet Wires			
	Standard "AI-220"	Heavy "Isonel-200/17"	Heavy "Pyre-ML"
Chemical Properties			
Solvent Shock, 15% Prestretch 3× Wrap in Boiling Xylol	OK	OK	OK
Solvent Resistance, 24 hr Immersion Followed by Cheesecloth Wipe			
Toluol	OK	OK	OK
Xylol	OK	OK	OK
5% H ₂ SO ₄	OK	OK	OK
Perchloroethylene	OK	OK	OK
Ethanol	OK	OK	OK
1% KOH	OK	OK	OK
Methylene Chloride	OK	Fails	OK
Hermetic Data			
Scrape Abrasion under Liquid, R-22— Strokes to Failure	1000+	225	90
Soxhlet Extraction in Toluol-Methanol, %	0.5	1.4	0.5
Soxhlet Extraction in R-22, %	0.2	0.3	0.3
Blister Test in R-22	OK	Blisters	OK

[Ref. 8]

Cont. Comparative Properties of No. 18 AWG Magnet Wires			
	Standard "A.I.-220"	Heavy "Isonel-200 17"	Heavy "Pyre-M.L."
Chemical Properties			
Varnish Compatibility, IEEE 57 Test Methods	Excellent	Good	Excellent
Epoxy Encapsulation, Softening in Curing Systems			
Amine Curing Agents	None	Severe	None
Anhydride Curing Agents	None	None	None
Sealed Tube Tests, Moisture Added— 1 Week at 150°C	Film Darkens	Severe Degradation	Film Cracks

[Ref. 8]

The Sterling Varnish Company reports that amide-imide varnishes should be stored in stainless steel, or in steel containers with a polyethylene liner. Common steel and black iron will corrode when exposed to amide-imide varnishes.

Leppla and Carryer (1964) report that the solvent and oil resistance of polyimide-coated fabrics are excellent and essentially unaffected by fluorinated hydrocarbon refrigerants. There was no change after exposure for four weeks to acetone, methyl ethyl ketone, formaldehyde, methanol, ethanol, ethyl acetate, "Skydrol", carbon disulfide, carbon tetrachloride, chloroform, perchlorethylene, o-dichlorobenzene, benzene, toluene, hexane, naphtha, and xylene. There was no change after exposure for one day to dimethyl formamide, dimethyl acetamide, n-methyl pyrrolidone, and γ -butyrolactone. There was also no change after exposure for four hours to DuPont's "Freon-22" fluorinated hydrocarbon. The authors present the following table of chemical resistance to various concentrated and dilute acids:

Chemical Resistance		
Polyimide coated fabrics are essentially unaffected by dilute aqueous acids, although they are attacked by concentrated acids and bases.		
Reagent	Condition	Exposure Period
NaOH (5%, aq.)	Somewhat stiffened	4 weeks
NaOH (1%, aq.)	Slight stiffening	4 weeks
NH ₃ (5%, aq.)	Slight stiffening	4 weeks
H ₂ SO ₄ (Conc., 96%)	Coating dissolved	1 day
HNO ₃ (Conc., 70%)	Coating embrittled, deteriorated	1 day
HCl (Conc., 37%)	Coating became cloudy, softened	1 week
H ₂ SO ₄ (5%, aq.)	Somewhat stiffened	3 weeks
HNO ₃ (5%, aq.)	Slight discoloration—no softening	3 weeks
HCl (5%, aq.)	Slight stiffening	4 weeks
H ₂ SO ₄ (1%, aq.)	Slight stiffening	4 weeks
HNO ₃ (1%, aq.)	No softening, slight discoloration	4 weeks
HCl (1%, aq.)	No softening, slight discoloration	4 weeks

[Ref. 67]

Hydrolytic stability is good. No change was detected in the coated glass fabric after seven weeks in a sealed system at 200°C in the presence of water.

The DuPont Company describes the moisture resistance of its "Pyre-ML" varnishes as being excellent over treated and untreated steel and aluminum; no failure for four months at 100°F/100% RH. Free films have been boiled in water for a year with good retentions of properties, but good moisture resistance depends on having a proper cure. Baked films of "Pyre-ML" varnish immersed in the following reagents for 24 hours at room temperature showed no signs of blistering, swelling or softening: xylene, cresylic acid, carbon tetrachloride, naphtha, acetone, alcohol, ethyl acetate, hexane, transformer oil, pyranol. The chemical resistance is fairly good, but sulfuric and nitric acids, alkali, ammonia and hydrazine cause severe film deterioration of "Pyre-ML." Using standard (oleic acid/cottonseed oil: 1/1) synthetic grease:

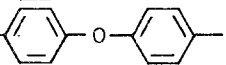
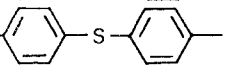
<u>Time</u>	<u>Temp.</u>	<u>Chemical Resistance</u>
6 hours	285°C (545°F)	no effect
3 months	23°C (77°F)	no effect

was noted for "Pyre-ML."

Monsanto "Skygard" 700 Laminates (1/8" thick, 181 glass-cloth with A-1100 prefinish at 250 psi):

<u>Exposure</u>	<u>Water Absorption</u>
24 hours water immersion	0.70%

Scroog and co-workers at the DuPont Company reported that the hydrolytic stability of the polypyromellitimides is, with certain exceptions, of a high order and may be considered quite unusual. The polypyromellitimides derived from bis(4-aminophenyl) ether and bis(4-aminophenyl) thioether, which already possess an outstanding level of thermal stability, retain toughness after one year and after three months respectively in boiling water. However, polyimides derived from diamines such as m-phenylenediamine and p-phenylenediamine exhibit poor hydrolytic stability, becoming embrittled after one week or less in boiling water:

HYDROLYTIC STABILITY OF POLYPYROMELLITIMIDES (R = DIAMINE COMPONENT)	
R	RETENTION OF FILM FLEXIBILITY IN BOILING H ₂ O
	1 WEEK
	1 WEEK
	1 YR
	> 3 MO

[Ref. 123]

Chemical Properties

Isomid

Solvent Resistance, R.T. for 24 hours (NEMA)

VM&P Naphtha

OK

Ethyl Alcohol

Toluol

Trichlorethylene

Butyl Acetate

Mild Alkalies (KOH 1%)

Mild Acids (H_2SO_4 5%)

Acetone Wipe, 10 minute R.T.

Alcohol/Toluol, 50/50, 10 minute boil

Extractables

Alcohol/Toluol, %

<0.25

Refrigerant, F-22

<0.25

[Ref. 134]

II. PROPERTIES

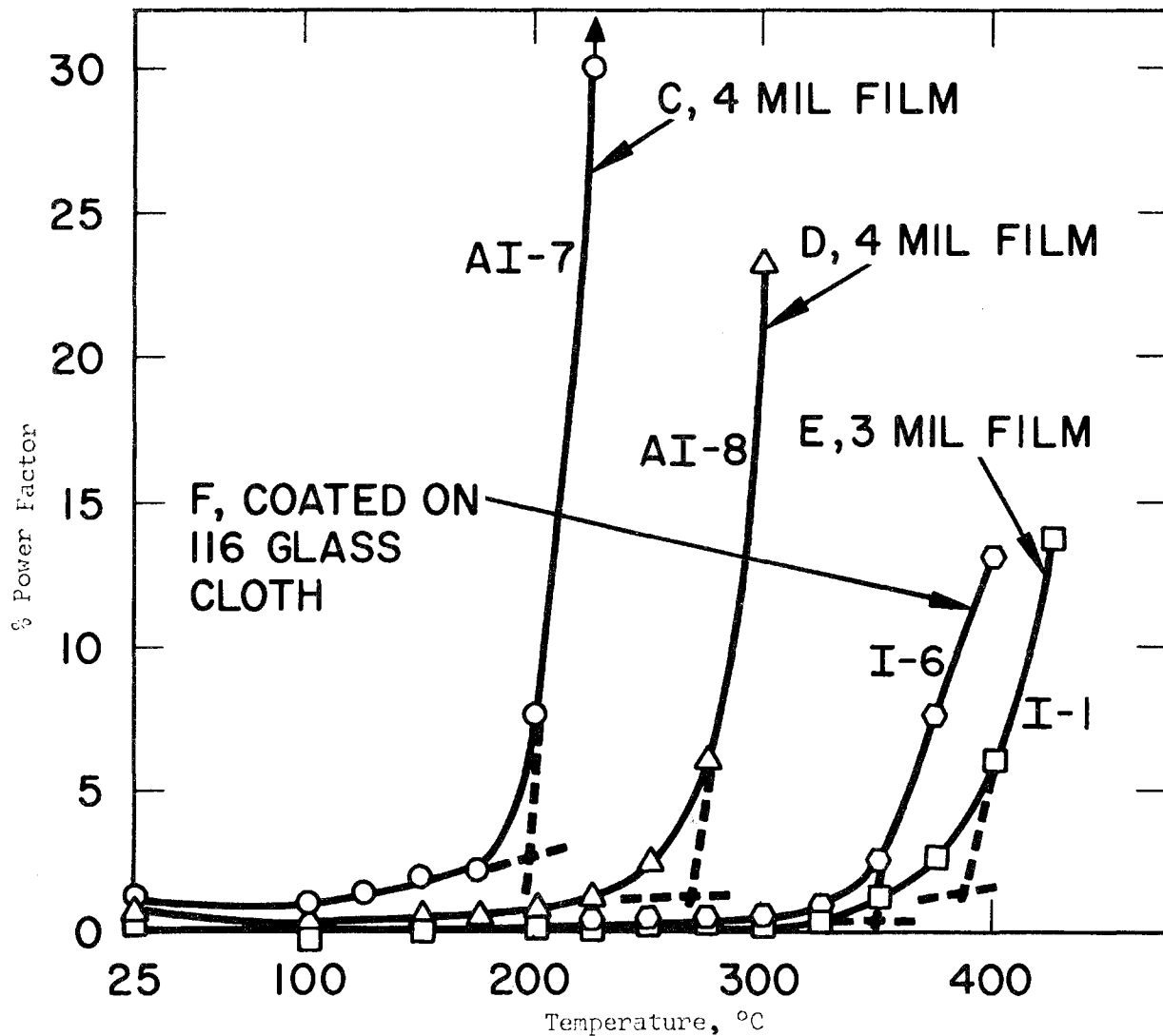
B. ELECTRICAL

When the various polyimides are compared with other plastics, they exhibit superior electrical properties, especially at high temperatures. They also represent a good combination of high dielectric strength, low dielectric constant, and low dissipation factors over wide temperature ranges. Furthermore, data indicate that their electrical properties change more slowly than the mechanical properties with thermal aging. Only in a few cases has the author included comparative properties with other plastics; but, in most instances they were omitted for space and time savings in view of the large amount of polyimide data which became available. The various polyimide forms are separated and grouped together by film; enamels, varnishes, and binder solutions; solid polymer; laminates, composites: etc., to facilitate resin form intercomparison purposes on the part of the user of this report. By such grouping practice, it is possible to visualize or determine the amount of property data available on each form, the range of temperature and frequency tested, the effects of moisture, thermal aging, thickness, etc. on each electrical property.

Long-time elevated temperature or thermal aging effects on the electrical properties were determined for most of the polyimide forms and represent a considerable and important body of information. Data on the effects of irradiation on the electrical properties are also very important and are reported separately in Section G: Irradiation Effects on Properties.

Since "Pyre-ML" and "Kapton" Type H-Film polyimide resins have such a wide potential in wire and cable applications, a large bulk of electrical property data have been generated on these materials.

Electrical property data are available on every form of polyimide: film, laminate, solid polymer, varnish and enamel coatings, filled and unfilled. Measurements of the electrical properties over a wide range of temperatures were available in the literature and from the various suppliers and are detailed below. Of special interest in the use of the change in the dissipation factor with temperature to determine T_g (glass transition temperature):



Effect of temperature on power factor ($100 \tan \delta$) for several aromatic polyimides at 1 Kc. Determination of T_g . Intersection of dotted lines approximates T_g .

[Ref. 45]

These in turn were correlated to the minimum temperature required to produce laminates by Westinghouse Electric Corporation workers.

1. Arc Resistance

"Kapton" Type H Film (ASTM D 495-61)

<u>Thickness</u>	<u>Temperature</u>	<u>RH</u> **	<u>Volts</u>	<u>Current</u>	<u>Lifetime</u>	<u>Ref.</u>
5 mil	23°C	50%	15 kv	10 m.a.	183 sec.*	31

* Failed by tracking ** Relative Humidity

SP-1 Polymer (ASTM D 495-61)

unfilled, as received

<u>Temperature</u>	<u>Lifetime</u>	<u>Ref.</u>
23°C	230 sec.*	97

* Failed by tracking

When SP-1 polymer is exposed to arcing, a carbon track is formed. The DuPont Company reports that SP-1 was also tested in a "wet tracking" test which is a new electrical measurement under consideration by ASTM. Tracking resistance is measured by the amount of power in the discharge which causes failure. For the SP-1 samples, failure occurred at the lowest discharge power which can be used in the test. This result places SP-1 in the category with insulators having low tracking resistance and indicates that applications where tracking is a problem should be avoided. They state further..."In actual field experience to date, our only failure in an electrical application has been one of tracking in a relay accuator."

2. Corona Effects

The corona resistance of the polyimide resins is superior to that of fluorocarbons and polyethylenes. For example, Todd and Wolff report, that at 1000 v/mil and room temperature, corona life for polyimide film which has electrical properties similar to polyimide parts, is in the range of 5 to 25 hours.

Freeman, et al, exposed a sample laminate of a polyamide-imide copolymer resin D to a continuous stress at a voltage of 255 volts per mil over 59 mils using 2.25-inch diameter electrodes; Continuous corona was visible. They further reported that two test points withstood over 600 hours each before failing by burn-through under the electrode.

"Kapton" Type H-Film:

<u>Temperature</u>	<u>Relative Humidity</u>	<u>Thickness</u>	Corona Threshold Voltage (ASTM D-1869-61T)	<u>Ref.</u>
25°C	50%	1 mil	465 volts	31
		2 mil	550 volts	
		3 mil	630 volts	
		5 mil	800 volts	

The General Electric Company conducted a study of the effect of corona on "H"-Film and found that pressure, temperature, and whether or not the system is dynamic, all influence the corona damage done by the available energy:

Corona Damage to "H" Film

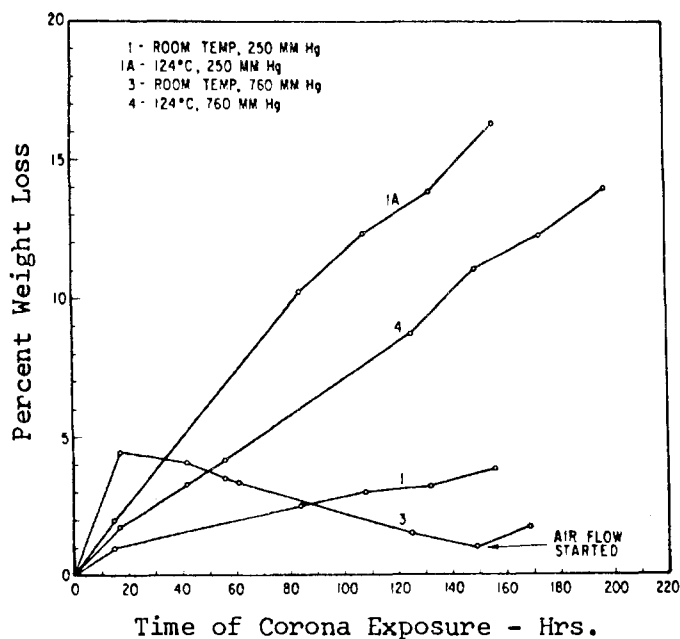
<u>Pressure (mm Hg)</u>	<u>Temperature (°C)</u>	<u>Voltage (kv rms)</u>	<u>Wt. Loss (%/hr)</u>	<u>Corona Power (watts)</u>	<u>Rate of Wt. Loss (%/w-hr x 10²)</u>
400	124	10.3	0.263	2.70	9.75
760	70	18.0	.186	4.0	4.65
760	124	10.3	.168	1.72	9.73
760	22	10.3	.00625	1.2	0.521
760	50	18.0	.0964	3.95	2.44
760	70	10.3	.0567	3.10	1.83
760	22	18.0	.0532	4.10	1.30
760	110	14.0	.183	3.75	4.88
200	124	10.3	.157	3.4	4.62
760	22	14.0	.0187	2.6	0.72
500	22	10.3	.114	--	.785
200	22	10.3	.0125	0.94	1.33

[Ref. 55]

Rate of Weight Loss/Corona Energy at 22° and 124°C. "H" Film

<u>Pressure (mm Hg)</u>	<u>Temperature (°C)</u>	<u>Voltage (kv)</u>	<u>Rate of Wt. Loss/Corona Energy (x 10²)</u>
200	124	10.3	4.62
400	124	10.3	9.73
760	124	10.3	9.75
200	22	10.3	1.33
500	22	10.3	0.785
760	22	10.3	.521

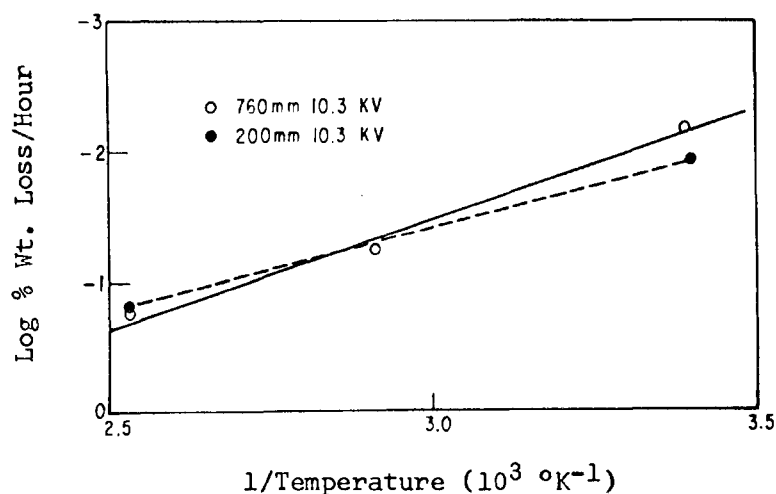
[Ref. 55]



Weight loss of "H" film
at 10.3 kv.

+ [Ref. 56]

↓



Percent weight loss of "H" film as a function of
temperature and pressure.

From the above weight loss rate measurements at various temperatures for various pressures, it will be noted that there are no significant differences between the activation energy for "H"-Film damaged by corona at the two pressures: 200 and 760 mm.

Horizontal-cut SP-1 samples from a brick were tested by the DuPont Co. for corona resistance at 23°C, 50% R. H., 0% elongation, 360 cps at 200 volts/mil. The 5/10 (fifth of ten) failure occurred at 366 hours; this is the equivalent of 2197 hours at 60 cps. The failure time for polyethylenes and "Teflon" at 60 cps is between 1200-1350 hours. Under these test conditions, therefore, SP-1 is somewhat more resistant to corona effects than "Teflon" or polyethylene.

3. Dielectric Constant

SP-1 Polymer, as-received (ASTM D-150):

<u>Temperature (°C)</u>	<u>Frequency (cps)</u>	<u>Dielectric Constant (ϵ')</u>	<u>Ref.</u>
23	10^2	3.41	31
↓	10^3	3.47	
↓	10^4	3.46	
↓	10^5	3.41	
50	10^2	3.39	
↓	10^3	3.50	
↓	10^4	3.49	
↓	10^5	3.45	
100	10^2	3.32	
105	10^3	3.38	
↓	10^4	3.36	
↓	10^5	3.33	
148	10^2	3.17	
150	10^3	3.23	
↓	10^4	3.23	
↓	10^5	3.22	
213	10^2	3.07	
205	10^3	3.12	
↓	10^4	3.10	
↓	10^5	3.05	
250	10^2	3.06	
↓	10^3	3.05	
↓	10^4	3.05	
↓	10^5	2.98	
255	10^2	3.06	
↓	10^3	3.05	
↓	10^4	3.05	
↓	10^5	3.03	
260	10^3	3.05	
↓	10^4	3.05	
↓	10^5	3.01	
264	10^2	3.13	
↓	10^3	3.05	
↓	10^4	3.04	
↓	10^5	2.99	
23	2000 mc	3.2	31
			19

Thermal aging has little effect on the dielectric constant of SP-1 Polymer. After 30 minutes aging at 400°C (752°F), the dielectric constant increases from its 3.48 at room temperature to 3.56 at a test frequency of 1 Kc.

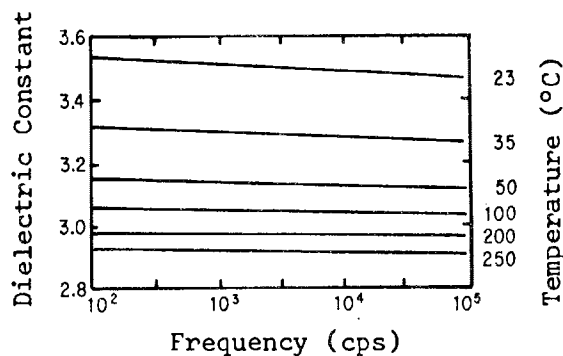
The dielectric constant increases with increasing moisture content up to a value of 4.8 after prolonged boiling in water. However, drying the boiled samples at 572°F for two hours reduces the value down to 3.4. The DuPont Company reports the following data on SP-1 polymer samples which were first dried to constant weight at 150°C and immersed in a water bath at 23°C for 48 hours. The measured water absorption was 1.4%:

Condition of Sample	Frequency (cps)	ϵ'
As received	10^3	3.38
	10^6	3.35
1.4% H ₂ O	10^3	3.73
	10^6	3.67

[Ref. 31]

"Kapton" Type "H"-Film

a) Effect of frequency on dielectric constant:



Dielectric constant of "H"-film (1 mil thick) as a function of frequency, tested at various temperatures.

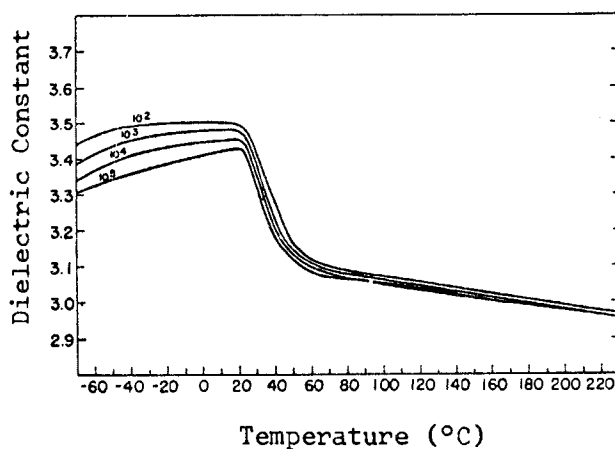
[Ref. 2, 68, 94]

"Kapton" Type "H"-Film:

Thickness	Temperature	R. H.	Freq. (cps)	Dielectric Constant
1 mil ↓	25°C ↓	50% ↓	10^2	3.52
			10^3	3.50
			10^4	3.47
			10^5	3.45
			10^6	3.43
			10^7	3.40
1 mil ↓	200°C ↓		10^2	2.99
			10^3	2.97
			10^4	2.95
			10^5	2.93

[Ref. 31]

b) Effect of temperature on dielectric constant:



Dielectric constant as a function of temperature.
Frequency 10^2 to 10^5 cps, 0.001-inch "H" film.

[Ref. 2, 58, 94]

Thickness	Cycles	Test Temperature	Original	Dielectric Constant (after aging)		
				8 weeks at 300°C	5 days at 350°C	8 hours at 400°C
1 mil	10^3 cps	23°C	3.7	3.8	3.9	4.1

[Ref. 31]

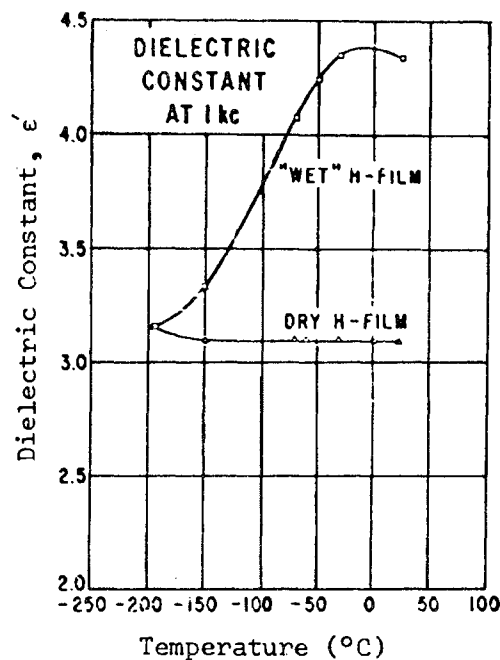
"Kapton" Type "H"-Film:

Temperature (°C)	Sample No.	Dielectric Constant*		
		0.005" H Film		
		0.1Kc	1Kc	10Kc
23	1	3.76	3.75	3.73
	2	3.74	3.73	3.71
	3	3.69	3.68	3.66
-196	1	3.32	3.31	3.30
	2	3.25	3.24	3.24
	3	3.28	3.27	3.26
-269	1	3.24	3.24	3.23
	2	3.18	3.18	3.18
	3	3.17	3.17	3.17
60	1	3.42	3.41	3.40
	2	3.42	3.41	3.42
	3	3.38	3.38	3.38
60 after 18 hours	1	3.30	3.29	3.28
	2	3.26	3.25	3.24
	3	3.22	3.21	3.20
100	1	3.24	3.23	3.22
	2	3.22	3.20	3.19
	3	3.18	3.17	3.16
150	1	3.22	3.21	3.19
	2	3.19	3.18	3.17
	3	3.15	3.14	3.13
200	1	3.18	3.18	3.17
	2	3.15	3.15	3.14
	3	3.12	3.11	3.10
200 after 17 hours	1	3.13	3.13	3.12
	2	3.11	3.10	3.09
	3	3.08	3.07	3.07
250	1	3.09	3.09	3.08
	2	3.05	3.05	3.05
	3	3.04	3.04	3.03
300	1	3.01	3.00	2.99
	2	2.97	2.97	2.96
	3	2.96	2.95	2.95
100 after above (held overnight)	1	3.09	3.08	3.08
	2	3.06	3.05	3.05
	3	3.04	3.03	3.02
25	1	3.40	3.39	3.38
	2	3.28	3.27	3.25
	3	3.14	3.13	3.12

*Approximate value uncorrected for edge effects or changes in specimen dimension with change in temperature.

[Ref. 72]

"Kapton" Type H-Film:



Dielectric constant as a function of temperature for a dry H-film, heated 2 days at 200°C and wet H-film, heated 2 days at 200°C plus 2 days in water at 80°C.

[Ref. 72]

c) Effect of film thickness on dielectric constant:

Temperature	R. H.	Frequency	Thickness	Dielectric Constant
25°C	50%	1Kc	1 mil	3.5
↓	↓	↓	2 mil	3.6
			3 mil	3.7
			5 mil	3.7

[Ref. 31]

d) Effect of moisture on dielectric constant:

Temperature	Thickness	Frequency	R. H.	Dielectric Constant
25°C	1 mil	1Kc	0%	3.0
↓	↓	↓	30%	3.3
			(1% H ₂ O) 50%	3.5
			80%	3.7
			(3% H ₂ O) 100%	3.9

[Ref. 31]

"AMANIM" Film (Westinghouse Res. Labs.):

<u>Temperature</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
25°C	60 cps	3.8
200°C	60 cps	4.3
25°C	1 Kc	3.8
200°C	1 Kc	4.5
25°C	100 Kc	4.5

[Ref. 44]

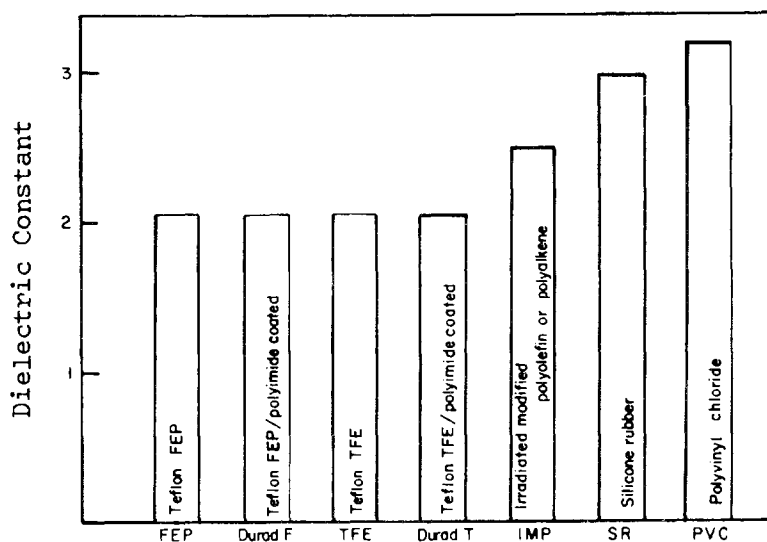
Polyimide films (Westinghouse Res. Labs.):

<u>Polymer*</u>	<u>Temperature</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
C	25°C	60 cps	4.86
		1000	4.77
D	25°C	60 cps	4.54
		1000	4.51
E	25°C	60 cps	3.21
		1000	3.21

* Polymers not identified in source report

[Ref. 51]

"DURAD" (American Super-Temperature Wires, Inc.):



[Ref. 3]

"Kapton" Type HF Film (Polyimide-Teflon FEP) Composites:

<u>Temperature</u>	<u>Frequency</u>	<u>Type</u>	<u>Dielectric Constant</u>
23°C	1Kc	*1H/1/2F	3.0
23°C	1Kc	**1/2F/1H/1/2F	2.6
23°C	1Kc	***2H/2F	2.8

Construction: * = 1-mil Kapton Type H/ 1/2-mil Teflon FEP
 ** = 1/2-mil Teflon FEP/1-mil Kapton Type H/ 1/2-mil Teflon FEP
 *** = 2-mil Kapton Type Type H/ 2-mil Teflon FEP

[Ref. 29]

Polyimide Foam (DuPont):

<u>Temperature</u>	<u>Frequency</u>	<u>Density</u>	<u>Dielectric Constant</u>
23°C	1Kc	2-30 lbs/ft ³	1.2 - 1.7

[Ref. 28]

PYRE-M.L. coated glass fabric:

<u>Temperature</u>	<u>Frequency</u>	<u>Sample Thickness</u>	<u>Dielectric Constant</u>
25°C	1Kc	4-mil	3.5

[Ref. 89]

Polyimide-coated fabrics (ASTM D-150-54T):

<u>Temperature</u>	<u>Frequency</u>	<u>Fabric Designation</u>	<u>Nominal Thickness</u>	<u>Dielectric Constant</u>
25°C	1 Kc	6500	4.0 mil	3.5
25°C	1 Kc	6507	5.5 mil	3.4
25°C	1 Kc	6507	7.0 mil	3.3
25°C	1 Kc	6508	10.5 mil	3.3
25°C	1 Kc	6509	8.75 mil	3.3

[Ref. 34, 67]

DuPont P-2101 resin-181E-A1100 glass fabric reinforced laminates:

<u>Temperature</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
77°F	9360Mc	4.15
300°F	↓	4.17
500°F		4.22
700°F		4.35

[Ref. 37]

<u>Sample Binder Solutions</u>	<u>Resin Content</u>	<u>Solvent System</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dielectric Constant</u>
"ML"	16.5% solids	NMP/DMAC	1 Kc	23°C	3.5
PI-2100	25%	DMF	↓	↓	2.0
PI-2102	25%	DMF			2.7
PI-3101	60%	NMP/Xylene			2.8
PI-1100	18.6%	DMAC			2.0
PI-1101	18.3%	DMAC	↓	↓	2.8

[Ref. 31,90]

<u>Sample Varnish (DuPont)</u>	<u>Resin Content</u>	<u>Solvent System</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dielectric Constant</u>
Polyimide RK-692	13% solids	NMP/(1)	100 cps	23°C	3.8
			1000 cps	25°C	3.5
RC-B-24951	45% solids	NMP/(2)	1000 cps	25°C	3.0
RC-5060	19% solids	NMP/(2)	1000 cps	25°C	2.8

1 aromatic hydrocarbon
2 xylene

[Ref. 33]

<u>Sample</u>	<u>Film</u>	<u>Thickness</u>	<u>Freq.</u>	<u>Dielectric Constant (ASTM D-150)</u>
DuPont "Pyre-ML"	enamel	10 mil	60 cps-1 Mc	3.8
TFE/"Pyre-ML"	composite	↓	↓	2.05
FEP/"Pyre-ML"	composite	↓	↓	2.1

[Ref. 66]

<u>Sample</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dielectric Constant</u>
Standard AI-220 (#18 AWG)	1 Kc	25°C	3.32
Magnet Wire Coating	↓	100°C	3.54
		200°C	3.20

[Ref. 8]

Heavy "Pyre-ML" (#18 AWG)		25°C	3.40
Magnet Wire Coating	↓	100°C	3.28
		200°C	2.72

[Ref. 5]

<u>Sample Binder Solutions</u>	<u>Solvent System</u>	<u>Temperature</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
ML	16.5% solids, NMP/DMAC	23°C	1 Kc	3.5
PI-2100	25% solids, DMF	23°C	1 Kc	2.0
PI-2101	25% solids, DMF	23°C	1 Kc	2.7
PI-3101	60% solids, NMP/Xylene	23°C	1 Kc	2.8
PI-1100	18.6% solids, DMAC	23°C	1 Kc	2.0
PI-1101	18.3% solids, DMAC	23°C	1 Kc	2.8

[Ref. 31, 90]

<u>Sample Varnish (DuPont)</u>	<u>Solvent System</u>	<u>Temperature</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
RK-692 polyimide	13% solids, NMP/aroma- tic hydrocarbon	23°C	100 cps	3.8
		25°C	1000 cps	3.5
RC-B-24951	45% solids, NMP/Xylene	25°C	1000 cps	3.0
RC-5060	19% solids, NMP/Xylene	25°C	1000 cps	2.8

[Ref. 33]

<u>Sample</u>	<u>Film</u>	<u>Thickness</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
DuPont "Pyre-ML"	enamel	10 mils	60 cps - 1 Mc	3.8
TFE/Pyre-ML	composite	10 mils	60 cps - 1 Mc	2.05
FEP/Pyre-ML	composite	10 mils	60 cps - 1 Mc	2.1

[Ref. 66]

<u>Sample</u>	<u>Temperature</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
Standard AI-220 (#18 AWG) Magnet Wire Coating	25°C	1 Kc	3.32
	100	↓	3.54
	200	↓	3.20
[Ref. 8]			
Heavy PYRE-M.L. (#18 AWG) Magnet Wire Coating	25°C	1 Kc	3.40
	100	↓	3.28
	200	↓	2.72

[Ref. 5]

Polyimide-glass fabric laminates: (Brunswick Corp. Defense Products Div.)

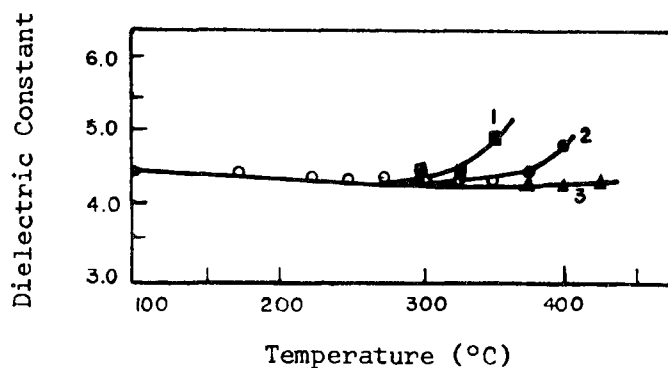
<u>Temperature</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
77°F	9360 Mc	4.20
350	↓	4.26
700		4.35

[Ref. 17]

DuPont PI-3301 Binder solution-filament wound panels:

<u>Aging Conditions</u>	<u>Temperature</u>	<u>Frequency</u>	<u>Dielectric Constant</u>
None	77°F	9375 Mc	3.94
	250°F	↓	3.98
	550°F		4.02
	700°F		4.06
	77°F		3.90
16 hours at 700°F			4.01
32 hours at 700°F			3.93

[Ref. 30]



Dielectric constant as a function of temperature for aromatic polyimide I-6 (4,4'-diaminophenyl sulfide and pyromellitic anhydride) (7 mils total) on type 116 glass cloth (4 mils).

- 1) 60 cps
- 2) 1 Kc
- 3) 100 Kc

[Ref. 45]

Polymer I-6 Glass Laminates:

Sample No.	Freq. cps	Temperature		Dielectric Constant	Comment
		°C	°F		
1	10 ¹⁰	25	77	4.15	as received
2				4.11	
2				4.14	
3				4.07	
3				4.08	
1		25	77	5.31	immersed 24 hr in water
2				4.60	
3				4.44	
1		25	77	4.02	after immersion: dried 24 hr at 115°C plus one hr at 300°C
2				4.04	
3				4.04	
3				4.11	
1		150	302	4.16	sample heated
3				4.05	
1		265	530	4.13	
2				4.15	
3				4.14	
1		315	600	4.17	
2				4.01	
3				4.20	

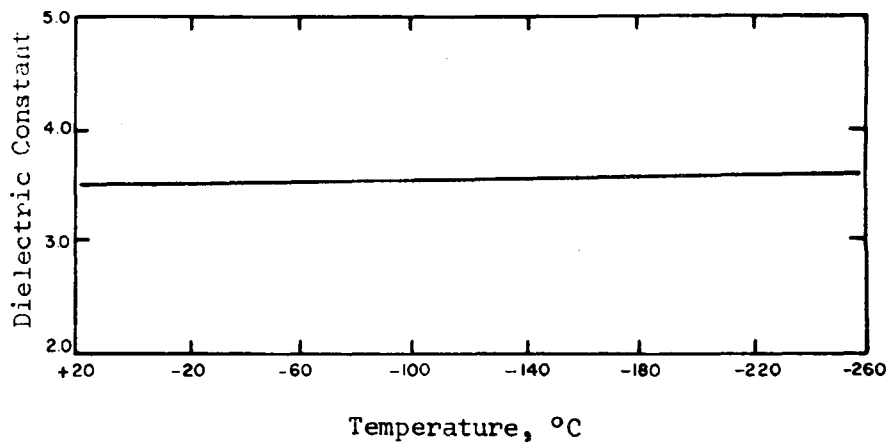
Dielectric constant was found to be independent of temperature up to 600°F.

[Ref. 45]

Dielectric Constant - 1 Kc

<u>Temperature</u>	<u>Polyester Isonel 200</u>	<u>AI-10 Polyamide- Imide</u>	<u>DuPont Polyimide</u>
25°C	3.34	3.32	3.40
100°C	3.46	3.54	3.28
150°C	3.31	2.41	2.53
200°C	4.08	3.20	2.72

[Ref. 104]



Variation of a dielectric constant with temperature for SP at 1 Kc.

[Ref. 103]

Isomid (Polyester-imide):

<u>Frequency</u>	<u>Temperature</u>	<u>Dielectric Constant</u>
1 Kc	25°C	3.44

[Ref. 134]

Polyvinyl Formal/urethane: polyimide Magnet Wire Composite Coating

<u>Thickness</u>	<u>Frequency</u>	<u>Temperature</u>	<u>Relative Dielectric Constant K'</u>
3.0 mils (build)	60 cps ↓	25°C	3.66
		50	3.45
		75	3.38
		100	3.37
		125	3.43
		150	4.12
		175	4.88
		200	6.14

Polyimide Homogeneous Magnet Wire Coating

<u>Thickness</u>	<u>Frequency</u>	<u>Temperature</u>	<u>Relative Dielectric Constant K'</u>
2.9 mils (build)	60 cps ↓	25°C	3.55
		50	3.45
		75	3.40
		100	3.40
		125	3.41
		150	3.42
		175	3.42
		200	3.44

Polyvinyl Formal/phenolic: polyimide Magnet Wire Composite Coating

<u>Thickness</u>	<u>Frequency</u>	<u>Temperature</u>	<u>Relative Dielectric Constant K'</u>
3.0 mils (build)	60 cps ↓	25°C	3.47
		50	3.33
		75	3.25
		100	3.25
		125	3.71
		150	4.31
		175	4.51
		200	5.20

[Ref. 43]

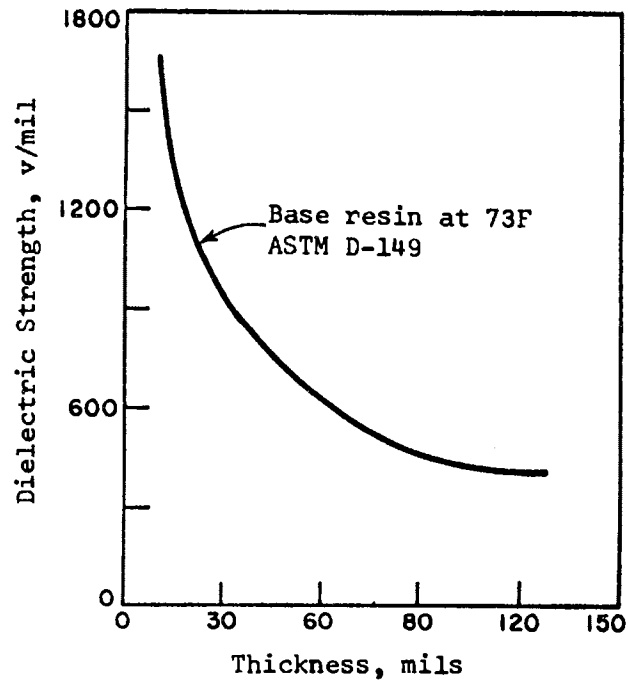
The effects of irradiation on the dielectric constant of polyimide plastics is detailed in a later section under IRRADIATION EFFECTS ON PROPERTIES.

B. Electrical

4. Dielectric Strength

<u>Type</u>	<u>Thickness</u>	<u>Voltage</u>	<u>Ref.</u>
SP-Polymer		570 volts/mil	19
SP-I Polymer (ASTM D-149)	1/8"	400 volts/mil	98

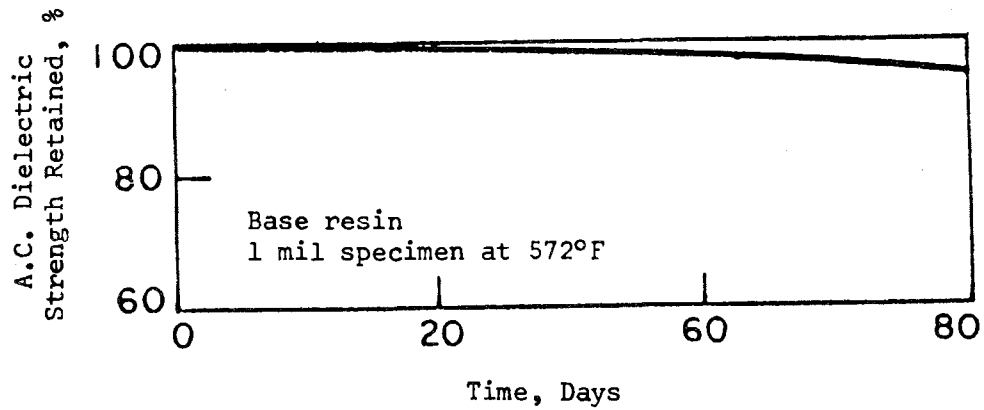
The short-time dielectric strength of the SP Polymer is high and varies with thickness as follows:



Short-time dielectric strength (at room temperature) of polyimides is high and varies with thickness.

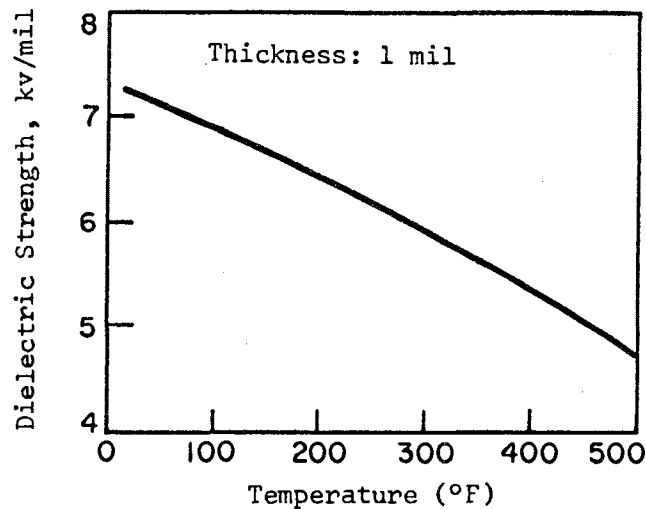
[Ref. 98]

Dielectric strength of SP-Polymer is relatively unaffected by thermal aging; there is only about a 10% loss after 120 days at 572°F:



[Ref. 98]

Todd and Wolff report the following graph showing the effect of temperature on the dielectric strength of SP-Polymer:



Dielectric strength of polyimides decreases steadily with increase in temperature but is still quite high at temperatures as high as 500°F.

[Ref. 98]

<u>Sample Binder Solution</u>	<u>Solids Content and Solvent System</u>	<u>Temperature</u>	<u>Dielectric Strength</u>
ML (DuPont RC-675)	16.5% solids, NMP/DMAC	23°C	4500-5000 volts/mil
PI-2100	25% solids, DMF	23°C	4000 volts/mil
PI-2101	25% solids, DMF	23°C	3300 volts/mil
PI-3101	60% solids, NMP/Xylene	23°C	4500 volts/mil
PI-1100	18.6% solids, DMAC	23°C	4000 volts/mil
PI-1101	18.3% solids, DMAC	23°C	3500 volts/mil

[Ref. 90,30]

<u>Sample</u>	<u>Aging Conditions</u>	<u>Temp.</u>	<u>Dielectric Strength</u>
AI-220 Magnet Wire (Standard Build, 18 gage Cu Wire)	16 hours at 100% RH	23°C Dry	4500 volts/mil
		23°C Wet	4000 volts/mil
		220°C	4100 volts/mil
		225°C	3000 volts/mil

[Ref. 4,5,8]

It is reported for the AI-220 polyimide that direct immersion in 70°C water for three months still showed adequate insulation resistance.

<u>Sample</u>	<u>Condition</u>	<u>Dielectric Strength</u>
AI-220 Sterling Varnish (unmodified polyamide-imide)	dry	4700 volts/mil
	wet	4300 volts/mil
AIvar-220 Sterling Varnish (modified polyamide-imide)	dry	4400 volts/mil
	wet	4000 volts/mil

Twisted pairs made with No. 18 AI-220 wire, given two coats of AI varnishes, aged at 250°C., after 40 days showed very little drop in electric breakdown strength.

	<u>AI-220</u>	<u>AIvar 220</u>
Initial Electric Strength	8,000 volts	9,000 volts
After 40 days at 250°C	7,100 volts	7,800 volts

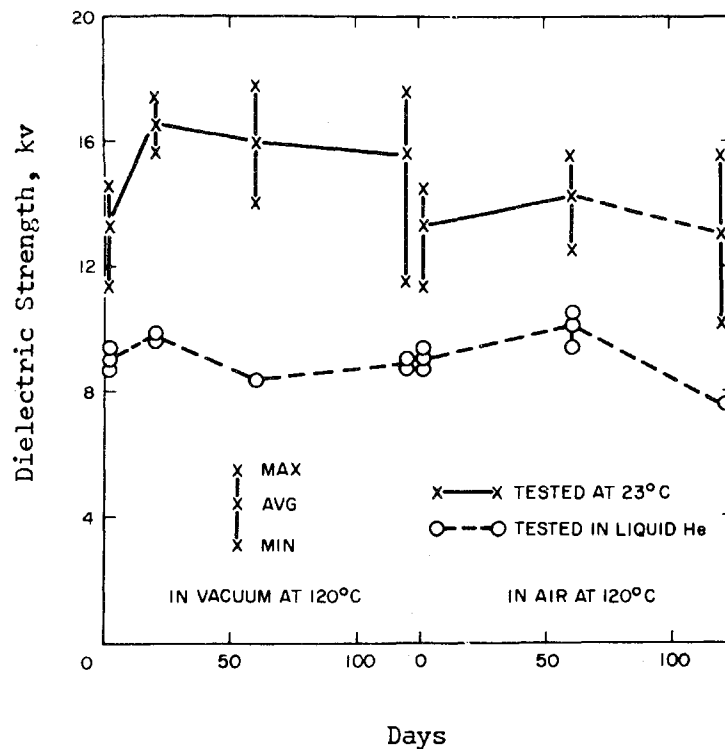
[Ref. 92]

<u>Sample</u>	<u>Thickness</u>	<u>Dielectric Strength</u> (ASTM D-149)
Pyre-M.L.	10 mil	3000 volts/mil

[Ref. 66]

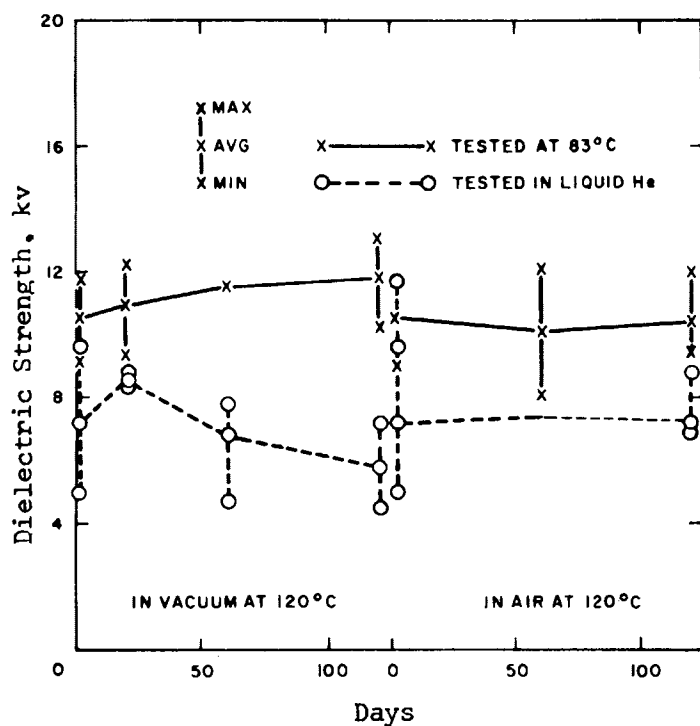
<u>Sample</u>	<u>Temp.</u>	<u>Aging Condition</u>	<u>Dielectric Strength</u>
Pyre-ML Magnet Wire (18 gage Cu wire)	23°C, Dry	16 hours at 100% RH	4000 volts/mil
	23°C, Wet		3000 volts/mil
	200°C		2500 volts/mil
	225°C		2500 volts/mil

[Ref. 8]



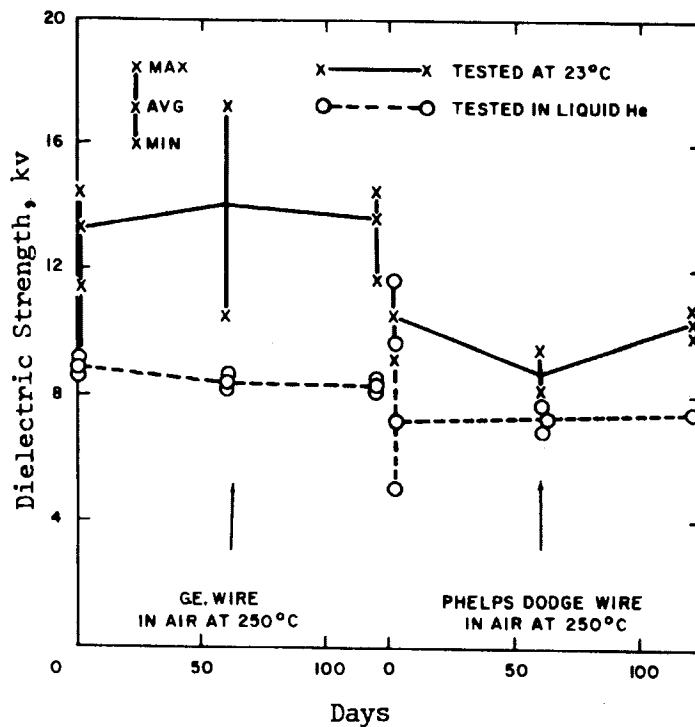
Voltage breakdown as a function of thermal aging at 120°C heavy ML (G.E.).

[Ref. 72]



Voltage breakdown as a function of thermal aging at 120°C heavy ML (Phelps Dodge).

Voltage breakdown as a function of thermal aging at 250°C heavy ML.



[Ref. 72]

Mathes, at the General Electric Company, has evaluated a variety of round wire insulations (comprised of polyimide solutions and enamels) for cryogenic applications. The following table lists the various polyimide insulations tested, their code designations (used in the next series of graphs and tables), and their manufacture source:

Description of Round Wire Insulation

(All wire is single conductor .0253 nominal copper)

<u>Designation</u>	<u>Description</u>	<u>Manufacturer</u>	<u>Nominal Insulation Wall Thickness</u>
HML(GE)	Heavy aromatic polyimide enamel (DuPont Pyre-ML)	General Electric Schenectady, N.Y.	0.0014"
HML(FW)	Heavy aromatic polyimide enamel	General Electric Fort Wayne	0.0010"
HML(PD)	Heavy aromatic polyimide enamel	Phelps Dodge Fort Wayne	0.00105"
Asbestos (ML)	Polyimide solution (ML) coated, felted asbestos	General Electric Lowell, Mass.	0.00635"
HML Asbestos	Aluminum Phosphate impregnated, felted asbestos over HML enamel (see above)	General Electric Lowell, Mass.	0.0052"
Glass/ML	Polyimide solution (DuPont ML) coated double glass fiber serving	General Electric Schenectady, N.Y.	0.0132"

[Ref. 72]

Breakdown Voltage of Film Coated Wire (Kilovolts)
As a Function of
Ambient Test Condition

Wire	Avg. Wall Thick.-In.	-26°C				-253C Liq. H ₂	-196C		-60C	23C		120C	250C
		Liq. H _e	Vac.				Liq. N ₂			Air	Vac.*	Air	V
HML(GE)	.0010	5.8	5.3	5.5	5.2	5.7	5.7	5.6		6.1	5.6		
		6.3	5.5	5.7	5.3	6.7	6.1	6.2		6.2	5.8		
		<u>6.3</u>	<u>5.9</u>	<u>6.1</u>	<u>5.7</u>	<u>6.9</u>	<u>6.7</u>	<u>6.9</u>		<u>6.7</u>	<u>5.9</u>		
		Avg. 6.1	5.6	5.8	5.4	6.4	6.2	6.2		6.3	5.8		
HML(GE)	.0014	7.2	7.9	7.4	7.4	8.6	7.4	8.8	8.3	10.9	9.8	11.8	7.5
		7.7	8.0	7.5	7.7	9.0	8.5	9.2	9.2	11.5	10.3	13.8	7.7
		<u>8.6</u>	<u>8.1</u>	<u>7.5</u>	<u>8.4</u>	<u>9.3</u>	<u>8.7</u>	<u>9.2</u>	<u>9.2</u>	<u>13.0</u>	<u>10.7</u>	<u>14.6</u>	<u>8.2</u>
		7.8	8.0	7.5	7.8	9.0	8.2	9.1	8.9	11.8	10.3	13.3	7.8
HML(PD)	.00105	5.0							5.8	10.7	5.3	5.3	5.9
		7.0							6.1	11.7	6.0	5.4	5.9
		<u>9.6</u>							<u>8.3</u>		<u>6.5</u>	<u>7.0</u>	<u>6.3</u>
		Avg. 7.2							7.1	11.2	5.9	5.9	6.0
Triple ML(PD)	.0016	10.8							10.0	11.2	7.4	10.7	7.5
		11.2							11.5	14.6	7.5	11.5	7.7
		<u>11.5</u>								<u>15.1</u>	<u>8.1</u>	<u>13.8</u>	<u>8.4</u>
		Avg. 11.2							10.8	13.6	7.7	12.1	7.9
HML Asbestos	.0069	5.8	5.4			7.1	7.6			4.3			
		6.0	5.6			7.7	7.7			4.8			
		<u>6.1</u>	<u>5.7</u>			<u>7.8</u>	<u>8.3</u>			<u>5.9</u>			
		Avg. 6.0	5.6			7.3	7.9			5.0			

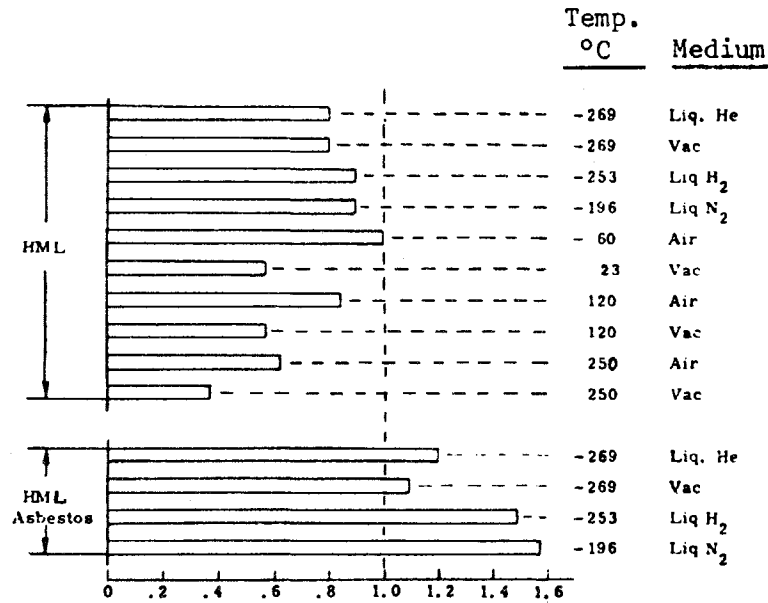
* A vacuum of about 10⁻⁵ Torr was obtained

Breakdown Voltage of Extruded and Fibrous Coated Wire (Kilovolts)
As a Function of
Ambient Test Condition

Wire	Avg. Wall Thick.-In.	-269C		-253C Liq. H ₂	-196C Liq. N ₂	-60C Air	23C		120C	250C
		Liq. H _e	Vac.				Air	Vac.*		
Asbestos ML Coated	.0052	2.8	2.4			1.5	1.2	3.3	0.9	3.8
		3.2	2.5			1.5	1.4	3.8	1.1	3.9
		3.4	2.6			2.5	1.4	4.0	1.1	4.2
		Avg. <u>3.1</u>	<u>2.5</u>			<u>1.8</u>	<u>1.3</u>	<u>3.7</u>	<u>1.0</u>	<u>4.0</u>
Glass Fiber ML Coated		1.3	7.4				2.0			
		1.3	8.1		11.2		2.0			
		1.4	8.4		11.3		2.3			
		Avg. <u>1.3</u>	<u>8.0</u>		<u>11.3</u>		<u>2.1</u>			

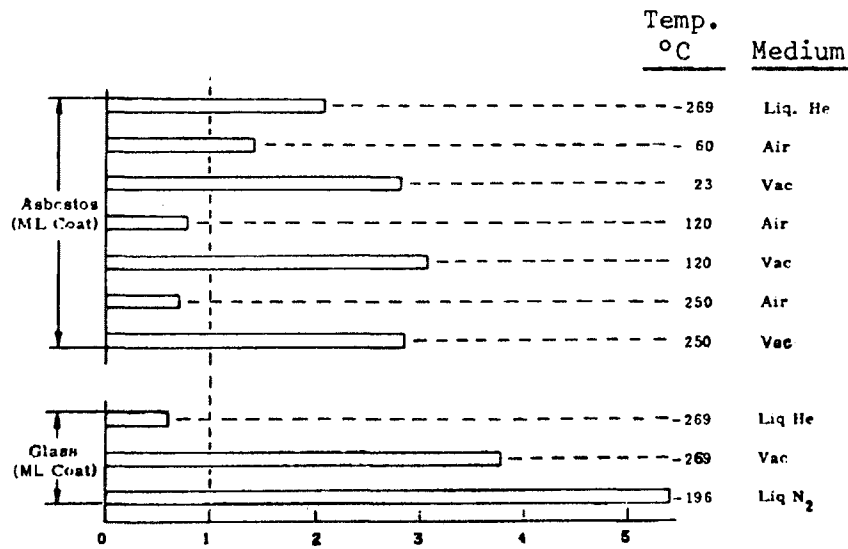
* Maximum test voltage was limited to 20.6 KV

[Ref. 72]



Ratio to Value at 23°C in Air, 50% RH

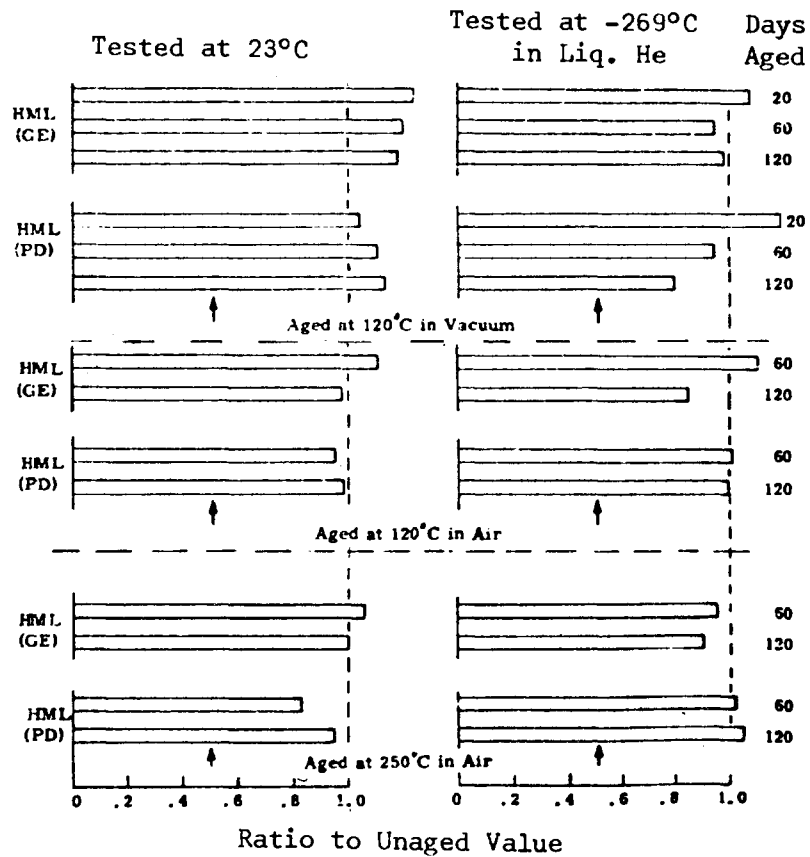
Comparison of breakdown voltage under different test ambients for film coated wires.



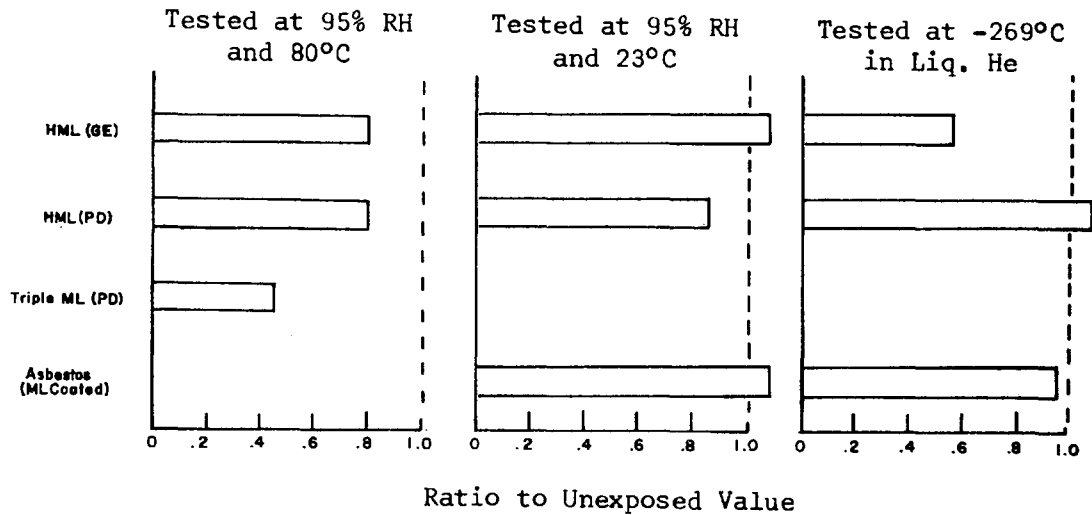
Ratio to Value at 23°C in Air, 50% RH

Comparison of breakdown voltage under different test ambients for fibrous coated wires.

[Ref. 72]



Comparison of breakdown voltage after thermal aging for film coated wires.



Comparison of breakdown voltage after exposure for 15 days at 95% RH and 80°C.

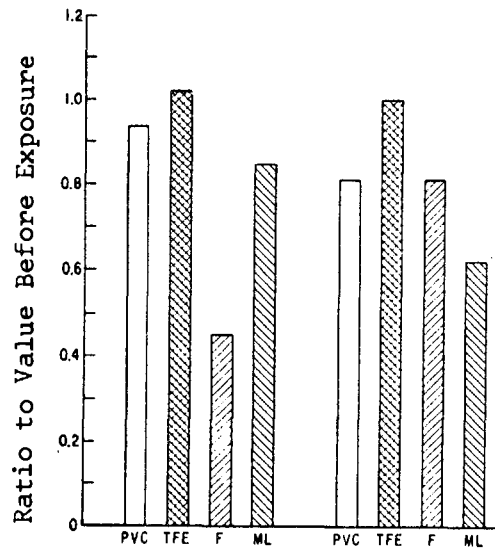
[Ref. 72]

Summary of Thermal and Humidity Aging Effects on the Voltage Breakdown (KV)
Of Insulated Wires at Room and Cryogenic Temperatures

Insulated Wire (Temperature of Test) ➡		Original 25C 4K	Thermal Aging												Moisture Aging for 15 Days (95% Relative Humidity at 80 C)			
			Vacuum at 120 C						Air at 120 C				Air at 250 C				Tested At: 25C 4K 95% R. H. 80 C	
			20 Days 25C 4K	60 Days 25C 4K	120 Days 25C 4K	20 Days 25C 4K	60 Days 25C 4K	120 Days 25C 4K	20 Days 25C 4K	60 Days 25C 4K	120 Days 25C 4K	20 Days 25C 4K	60 Days 25C 4K	120 Days 25C 4K				
Heavy ML (G. E.) (0.0014" Wall)	Avg.	13.3 9.0	16.6 9.8	15.9 8.4	15.6 8.9	14.2 10.1	13.1 7.6	14.1 8.5	13.6 8.3	14.3 7.3	10.8							
	Min.	11.4 8.7	15.6 9.7	14.0 8.4	11.6 8.8	12.5 9.4	10.2 7.6	10.4 8.3	11.6 8.1	14.0 5.0	9.5							
	Max.	14.6 9.4	17.4 9.8	17.8	17.6 9.0	15.6 10.6	15.6	17.2 3.6	14.6 8.6	14.6 9.5	11.6							
Heavy ML (Phelps-Dodge) (0.0011" Wall)	Avg.	10.5 7.2	10.9 8.7	11.5 6.8	11.9 5.8	10.1 7.4	10.4 7.2	8.7 7.4	10.0 7.6	9.0 8.0	8.5							
	Min.	9.1 5.0	9.3 8.3	11.3 4.7	10.2 4.4	8.0 7.3	9.5 6.8	8.1 6.8	9.7 7.6	7.4 8.0	7.8							
	Max.	11.7 9.6	12.2 9.0	11.6 7.8	13.2 7.2	12.1 7.5	12.0 8.8	9.3 7.8	10.3 7.7	10.2	9.1							
Asbestos (ML Coated) (0.0064" Wall)	Avg.	1.3 2.5	1.1 2.4	1.2 2.4	1.1 2.6	1.1 2.3	1.1 2.4	1.2 2.4	1.1 2.4	1.4 2.4	0.5							
	Min.	1.2 2.4	1.0 2.3	1.1 2.1	1.1 2.5	1.0 2.2	1.0 2.3	1.1 2.3	1.0 2.3	1.4 2.3	0.5							
	Max.	1.4 2.6	1.2 2.4	1.3 2.5	2.6	1.2 2.6	1.1 2.6	1.3 2.6	1.2 2.4	2.4								
Triple ML (Phelps-Dodge) (0.0016" Wall)	Avg.	13.6 11.2									6.1							
	Min.	11.2 10.8									5.4							
	Max.	15.1 11.5									7.1							
Test Conditions: Average of 3 Breakdowns (20 KV/Minute Rise, 60 Cycles) of Twisted Pairs of Wire in Room Temperature Air or Under Liquid Helium at 4K.																		

Test Conditions: Average of 3 Breakdowns (20 KV/Minute Rise, 60 Cycles) of
Twisted Pairs of Wire in Room Temperature Air or Under
Liquid Helium at 4K.

[Ref. 72]

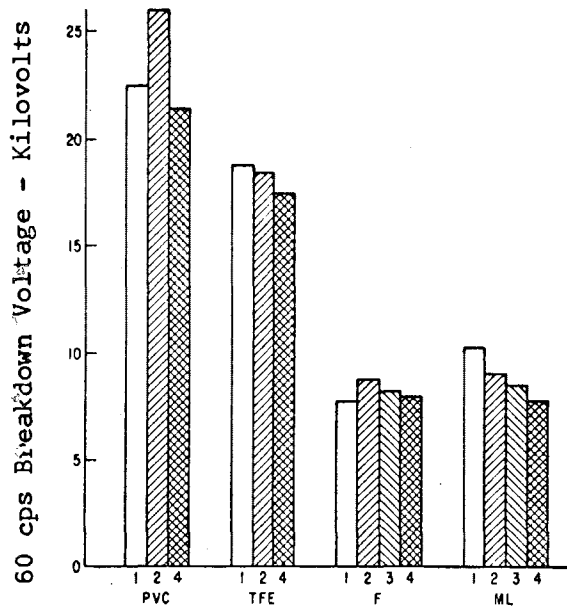


Tested at 80°C- 95% RH Tested in Liquid He at -269°C

Effect of exposure for 15 days at 80°C - 95% RH
on voltage breakdown.
(NEMA twisted pair, wire samples)

PVC - Polyvinyl chloride F - Polyvinyl formal
TFE - Polytetrafluoro-ethylene ML - Aromatic Polyimide

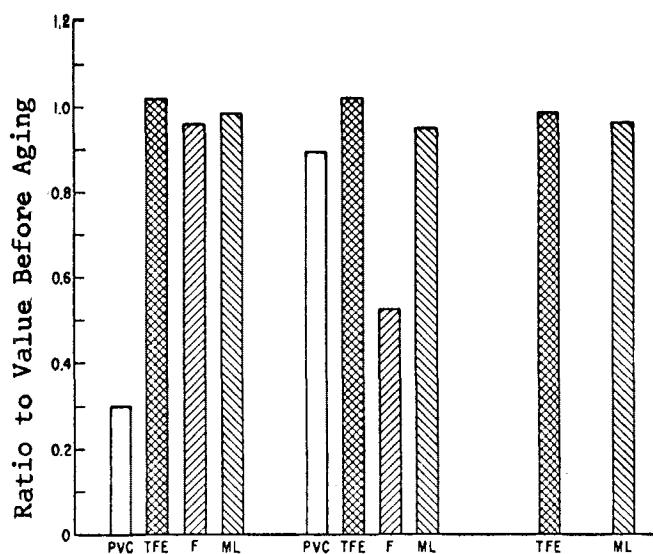
[Ref. 70]



Breakdown voltage at room and cryogenic temperatures.

- 1 - Tested in air at 23°C
- 2 - Tested in liquid nitrogen at -196°C
- 3 - Tested in liquid hydrogen at -253°C
- 4 - Tested in liquid helium at -269°C

PVC - Polyvinyl chloride, .007" wall
 TFE - Polytetrafluoroethylene, .014" wall
 ML - Aromatic polyimide, .0014" wall
 F - Polyvinyl formal, .0014" wall



Effect of thermal aging on voltage breakdown at -269°C in liquid He. (NEMA twisted pair, wire samples)

PVC - Polyvinyl chloride
 TFE - Polytetrafluoroethylene
 ML - Aromatic polyimide
 F - Polyvinyl formal

Aged in Vacuum 120 Days at 120°C Aged in Air 120 Days at 120°C Aged in Air 120 Days at 250°C

[Ref. 70]

Polyimide Varnish	Temp.	Solids Content (%)	Viscosity	Electric Strength volts/mil
DuPont RK-692	25°C Dry	13	6-10 poises	4000
	25°C Wet			1500
DuPont RC-B-24951	23°C	45	2-5 poises	4500
DuPont RC-5060	23°C	19	5-9 poises	4500

[Ref. 33]

Polyimide-coated fabrics:

<u>Property</u>	<u>Fabric Designation</u>				
	<u>6500</u>	<u>6507</u>	<u>6507</u>	<u>6508</u>	<u>6509</u>
Nominal thickness, mils	4.0	5.5	7.7	10.5	8.75
Electric Strength, vpm, short time, initial, ASTM D-295 55T, 2" electrodes	500	700	900	600	700
Heat Aged, 9 weeks at 300°C, ASTM D-902, mandrel test, 1/4" electrodes	375	-	450	-	-
Electric Strength at 250°C, ASTM D-295 55T, 2" electrodes	450	400	550	600	-

[Ref. 34, 67]

The room temperature electric strengths of polyamide-polimide coated glass cloth [4 mil, (No. 116) built up to 6 mils total thickness by resin and thoroughly cured], after aging in air at 300°C:

<u>Condition</u>	<u>Electric Strength volts/mil</u>	<u>Percent of Original</u>
As received	1150	100
After 5 days	1220	106
After eighteen days	1260	110
After twenty-seven days	1116	97
After forty-two days	945	82

1/4" electrodes: voltage 0.5 kv rise per second until breakdown.

[Ref. 45]

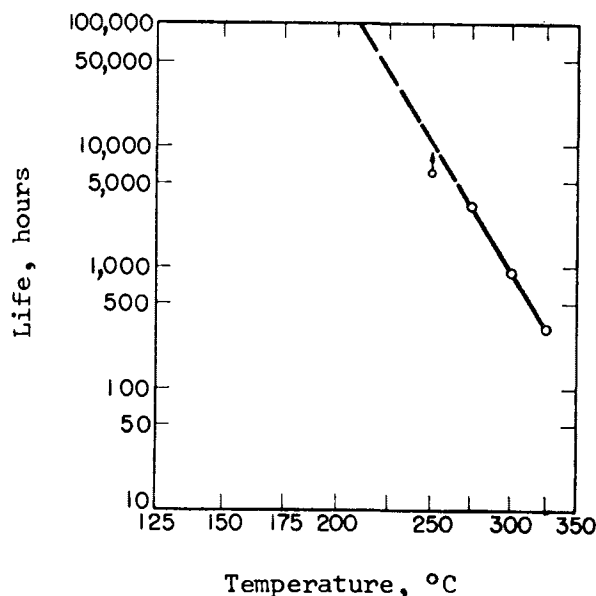
Freeman, et al, report dielectric strength data on polyamide-imide D co-polymer thin wall tubing, after various aging periods at different temperatures:

Electric Strength (volts/mil) of Thin
Wall Tubes (0.032 inch wall thickness) After Aging

<u>Weeks</u>	<u>Aging Temperature, °C</u>			
	<u>250</u>	<u>275</u>	<u>300</u>	<u>325</u>
0	912	910	910	830
1	-	-	-	657
2	-	-	-	145
3	-	-	833	-
4	-	-	960	-
6	-	920	120	-
12	-	740	-	-
17	-	640	-	-
19	1040	-	-	-
20	-	330	-	-
62	533	-	-	-

[Ref. 45]

These same authors present additional life data on this copolymer in graphical form as follows:



Extrapolated life as a function of
T for tube of polyamide-imide
D on 116 glass cloth
(retention of 250 v/mil).

[Ref. 49]

The Monsanto Co. revealed that translucent, four-ply glass-cloth laminates made from polyimide Sygard 700 have been produced which have dielectric strengths higher than 300 volts/mil. High dielectric strengths are maintained after successive aging for 3 hours in boiling water, 100 hours at 575°F and 3 additional hours in boiling water.

Mathes and McKannon have tested the short-time electric breakdown of impregnated polyimide-glass fiber magnet wire insulation (twisted wire pair, 0.0013-inch wall thickness samples) at cryogenic temperatures as follows:

<u>Temperature (°K)</u>	<u>Dielectric Strength</u>
296	10.3 KV average
77 (liquid nitrogen)	9.1
4 (liquid helium)	7.8
4 (vacuum)	7.5

[Ref. 71]

Frost and Bower of Westinghouse Research Labs. tested polyimide laminates made with 181 glass cloth (1/16" thick) and found the dielectric strengths above 900 volt/mil and maintained values above 480 volts/mil after two months at 300°C.

[Ref. 51]

PYRE-ML coated glass fabric:

Temperature

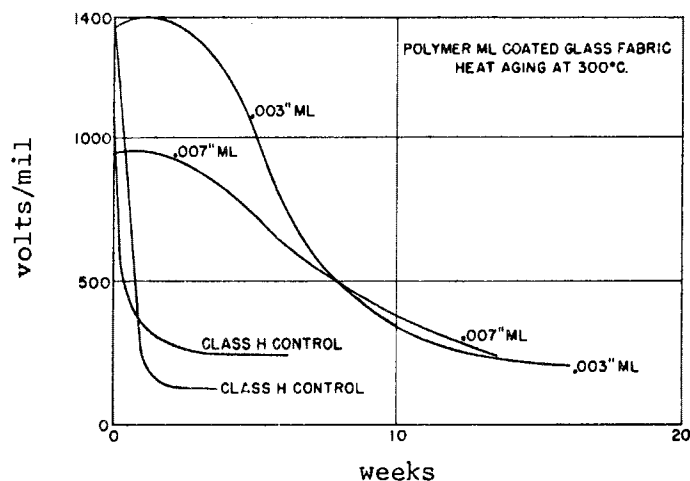
23°C

Dielectric Strength

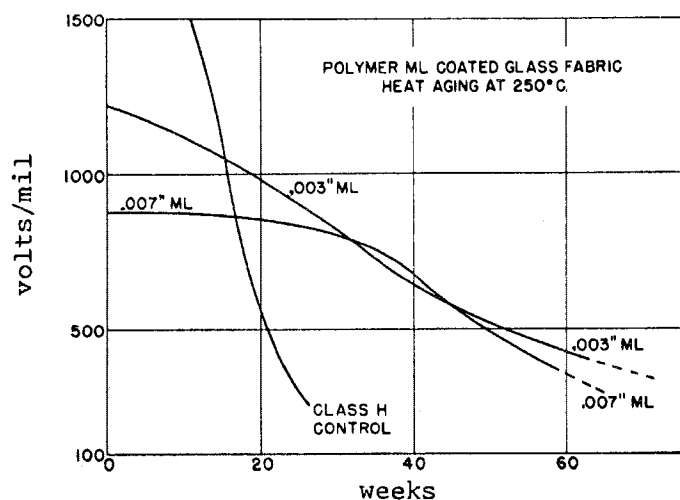
1000 volts/mil

[Ref. 89]

Schweitzer et al reported the following thermal aging data on PYRE-ML coated glass fabric:



[Ref. 67, 89]



[Ref. 31]

<u>Insulation</u>	<u>Temperature</u>	<u>Thickness</u>	<u>Dielectric Strength (ASTM D 149)</u>
TFE-Pyre-ML	23°C	10 mils	1000-2000 volts/mil
FEP-Pyre-ML	23°C	10 mils	1000-2000 volts/mil

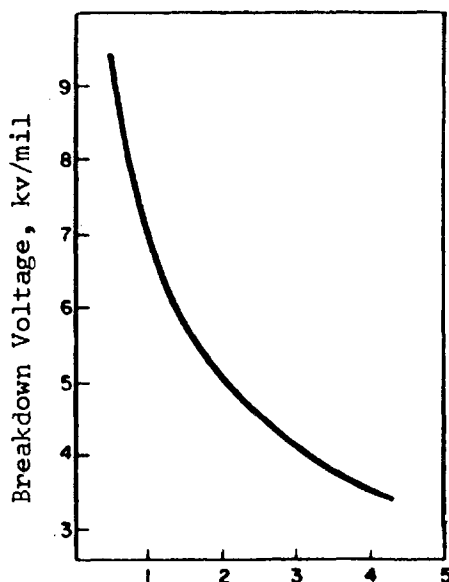
[Ref. 66]

DuPont "Kapton" Type H Film

Effect of film thickness on dielectric strength:

<u>Test Conditions</u>	<u>Film Thickness</u>	<u>AC Dielectric Strength (ASTM D149-61)</u>
1/4" Electrodes, 25°C, 50% RH, 60 cps	1 mil	6,600 volts/mil
	2 mil	5,100 volts/mil
	3 mil	4,400 volts/mil
	5 mil	3,600 volts/mil

[Ref. 31]



1/4-inch electrodes, 500 volts per sec., rate of voltage rise, 23°C, 60% RH, H-film.

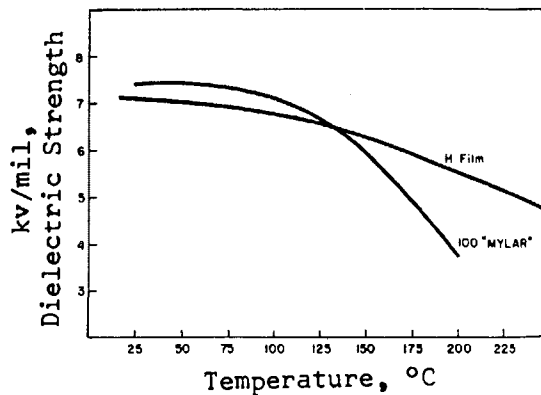
a.c. dielectric strength as a function of film thickness.

[Ref. 2]

Effect of temperature on dielectric strength:

<u>Test Conditions</u>	<u>Temperature</u>	<u>AC Dielectric Strength (ASTM D149-61)</u>
1 mil, 60 cps, 1/4" Electrodes	-195°C	10,800 volts/mil
	25°C	7,000 volts/mil
	200°C	5,600 volts/mil
	300°C	2,500 volts/mil
5 mil, 60 cps, 1/4" Electrodes	25°C	3,600 volts/mil
	200°C	3,000 volts/mil
	300°C	1,200 volts/mil

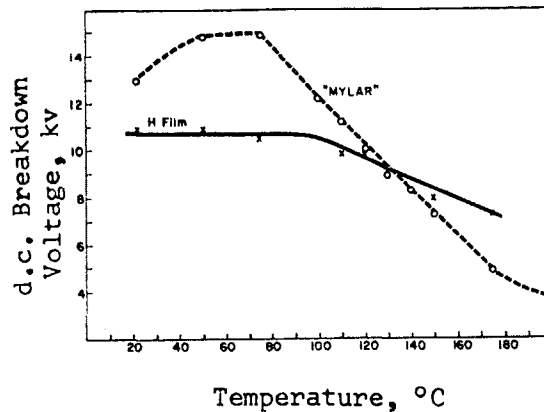
[Ref. 31]



a.c. dielectric strength as a function of temperature.

0.001-inch H-film; 1/4-inch electrodes; 500 volts per sec. rate of voltage rise.

[Ref. 2]

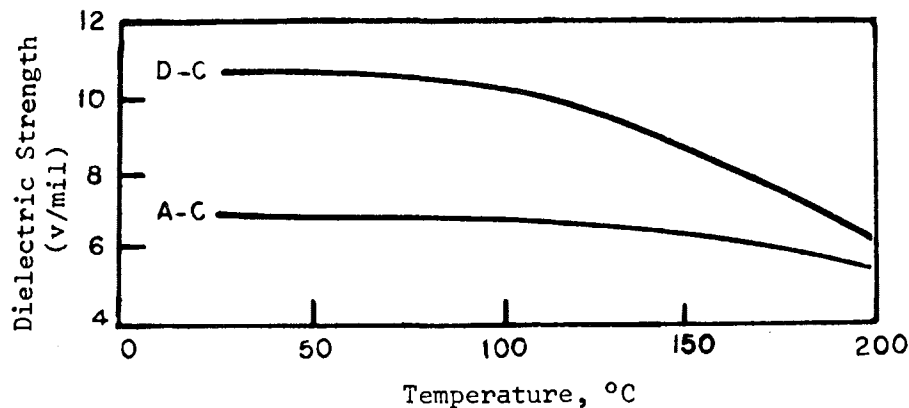


d.c. breakdown as a function of temperature.

1/4-inch electrodes, 500 volts per sec., rate of voltage rise; 0.0012-inch H-film; 100 Mylar C polyester films

[Ref. 2]

Mallouk has combined the d.c. and a.c. electric breakdown strengths of H-film into one graph for easy comparison purposes:

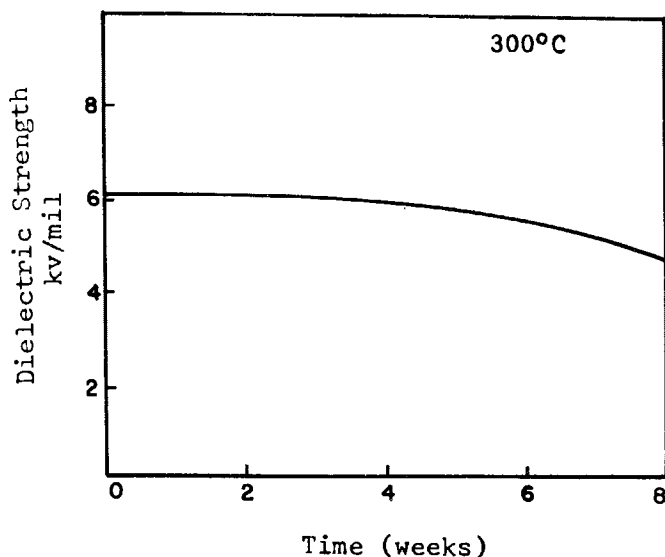


Dielectric strength of H-film as a function of temperature. Test were made on a 1-mil film using 1/4-inch diameter electrodes. Test method: ASTM D149.

and quotes that the dielectric strength of H-film containing 3% moisture is 6000 volts/mil.

[Ref. 68, 94]

Tatum and co-workers present data showing the effect of thermal aging at 300°C up to 8 weeks on 1-mil H-film:



[Ref. 94]

<u>Thickness</u>	<u>Aging Time</u>	<u>Aging Temp.</u>	<u>Test Temp.</u>	<u>Dielectric Strength</u>
1 mil	unaged	23°C	23°C	6000 volts/mil
1 mil	8 weeks	300°C	23°C	5200 volts/mil
1 mil	5 days	350°C	23°C	5100 volts/mil
1 mil	8 hours	400°C	23°C	5300 volts/mil

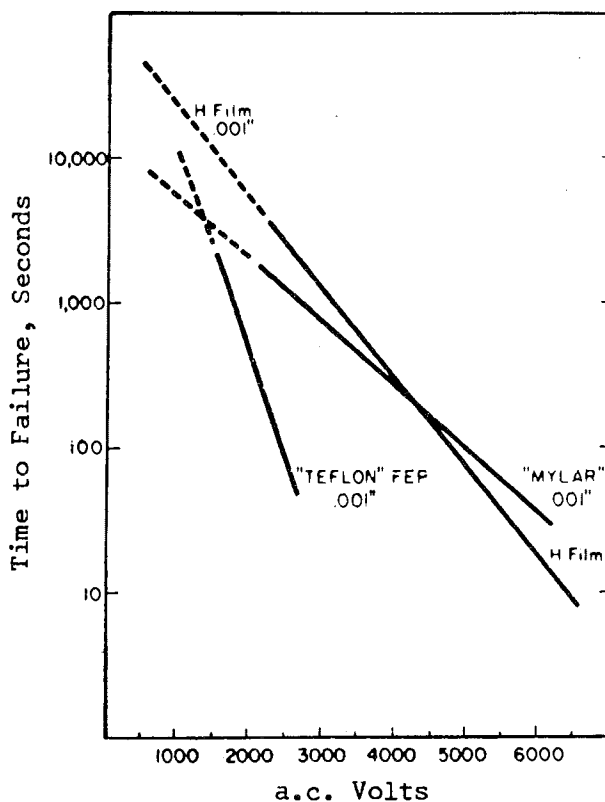
[Ref. 31]

Effect of humidity on dielectric strength:

<u>Test Conditions</u>	<u>Water Absorption</u>	<u>% Relative Humidity</u>	<u>a.c. Dielectric Strength</u>
1 mil, 25°C, 1/4" Electrodes		0	7,800 volts/mil
		30	7,300 volts/mil
		50	7,000 volts/mil
	1%	80	6,500 volts/mil
		100	6,200 volts/mil
5 mil, 25°C, 1/4" Electrodes		0	4,200 volts/mil
		30	3,850 volts/mil
		50	3,600 volts/mil
	1%	80	3,250 volts/mil
		100	3,000 volts/mil

[Ref. 31]

Amborski made an extensive comparison of DuPont H-film with other plastic film materials and reported his dielectric life results in the following graph:



Dielectric life as a function of a.c. voltage.
(1/4-inch electrodes, 23°C, 60% RH)

[Ref. 2]

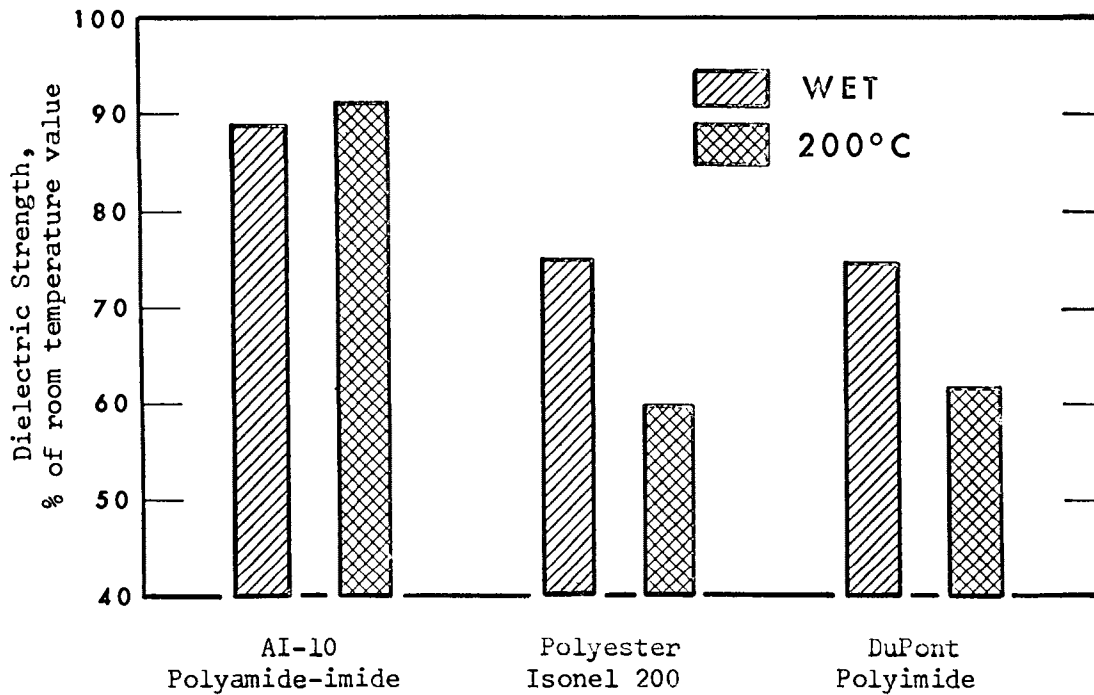
<u>Test Conditions</u>	<u>a.c. Volts</u>	<u>Time to Failure (seconds)</u>
ASTM D-2275-64T 1 mil, 25°C, 1/4" Electrode, 60% RH	6,500	9
	6,000	18
	5,500	33
	5,000	72
	4,500	144
	4,000	265
	3,000	1,258
	2,500	2,991
	1,000	~30,000

[Ref. 31]

Westinghouse Electric "Amanim" Polyamide-imide Film

<u>Time</u>	<u>% Relative Humidity</u>	<u>Frequency</u>	<u>Temp.</u>	<u>Dielectric Strength</u>
90 Hours	90	60 cps	35°C	4200 volts/mil
90 Hours	90	60 cps	200°C	3100 volts/mil

[Ref. 44]



Dielectric strength after exposure to moisture or 200°C.

[Ref. 104]

<u>Polyimide Polymer Film*</u>	<u>Temp.</u>	<u>Dielectric Strength, kv (rms)/mil</u>
C	25°C	2.4
	250°C	2.45
	300°C	2.4
D	25°C	2.5
	250°C	2.5
	300°C	2.4
F	25°C	2.4
	250°C	2.3
	300°C	1.8

*Film thickness 1.5 - 3.5 mils. Tested in air. Rate of rise 2 kv/sec.
Frequency 60 cps.

[Ref. 51]

B. Electrical

5. Dissipation Factor

<u>Specimen</u>	<u>Temp.</u>	<u>Freq.</u>	<u>Dissipation Factor</u> <u>(ASTM D-150)</u>
SP-Polymer	77°F	1 Mc	0.002
	(73-437°F)	1 Mc	0.003
	77°F	2000 Mc	0.0088
			[Ref. 19]
SP-I Polymer	77°F	1 Mc	0.003
			[Ref. 98]

Thermal aging has relatively little effect on the dissipation factor of SP-Polymer. After 30 minutes at 752°F, dissipation factor decreases from 0.0034 to 0.0012 at 1 Kc.

<u>Specimen</u>	<u>Temp.</u>	<u>Freq. (cps)</u>	<u>Dissipation Factor</u> <u>(ASTM D-150)</u>
SP-I Polymer	23°C ↓	10 ²	0.0015
		10 ³	0.0017
		10 ⁴	0.0035
		10 ⁵	0.0052
	50°C ↓	10 ²	0.0012
		10 ³	0.0014
		10 ⁴	0.0020
		10 ⁵	0.0052
	100°C 150°C ↓	10 ²	0.0011
		10 ³	0.0013
		10 ⁴	0.0013
		10 ⁵	0.0015
	148°C 150°C ↓	10 ²	0.0010
		10 ³	0.0011
		10 ⁴	0.0011
		10 ⁵	0.0015
	213°C 205°C ↓	10 ²	0.0012
		10 ³	0.0010
		10 ⁴	0.0010
		10 ⁵	0.0010
	250°C ↓	10 ²	0.0197
		10 ³	0.0089
		10 ⁴	0.0016
		10 ⁵	0.0010
	255°C ↓	10 ²	0.0370
		10 ³	0.0046
		10 ⁴	0.0012
		10 ⁵	0.0013

[Ref. 31]

Table Cont.

<u>Specimen</u>	<u>Temp.</u>	<u>Freq. (cps)</u>	<u>Dissipation Factor (ASTM D-150)</u>
SP-I Polymer	260°C ↓	10 ³	0.0173
		10 ⁴	0.0024
		10 ⁵	0.0011
	264°C ↓	10 ²	0.0815
		10 ³	0.0103
		10 ⁴	0.0017
		10 ⁵	0.0010

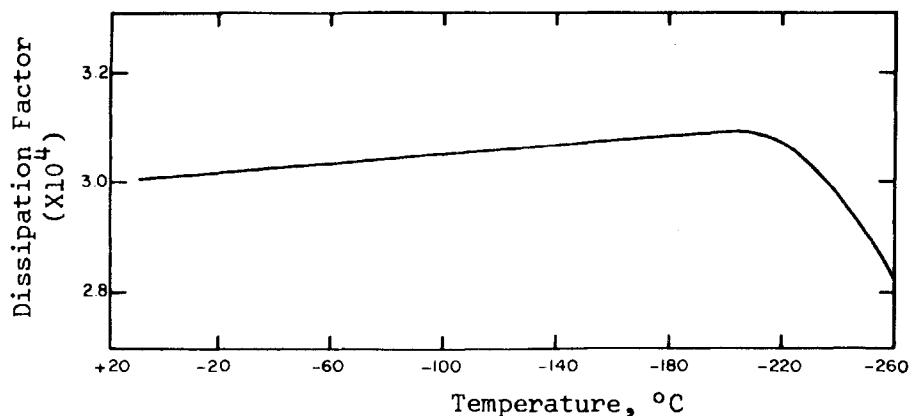
[Ref. 31]

The dissipation factor of SP-Polymer increases (up to a value of 0.04) with increasing moisture content after prolonged boiling. However, drying the boiled samples at 572°F for 2 hours reduces the value down to 0.0008.

In another set of experiments, the DuPont Company dried SP-I molded samples to constant weight at 150°C and immersed them in a water bath at 23°C for 48 hours. The water absorption was found to be 1.4%. The following tabulation shows the effect of this water content on the dissipation factor at two frequencies:

<u>Condition of Sample</u>	<u>Freq.</u>	<u>Dissipation Factor</u>
As Received	10 ³	0.0015
	10 ⁶	0.0069
1.4% H ₂ O	10 ³	0.0017
	10 ⁶	0.0068

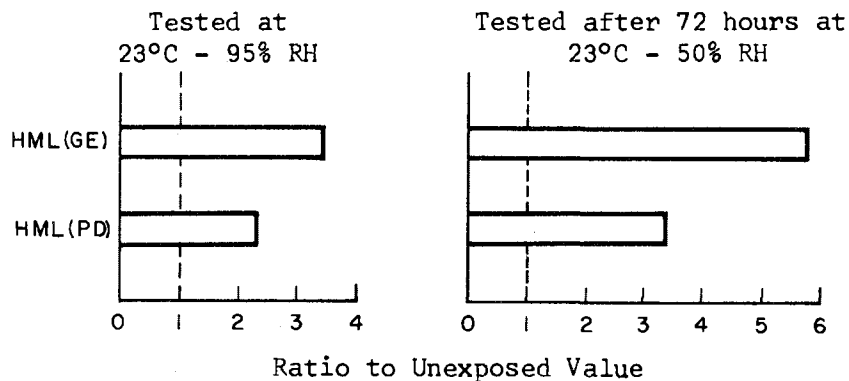
[Ref. 31]



Variation of dissipation factor with temperature for SP at 1 Kc.

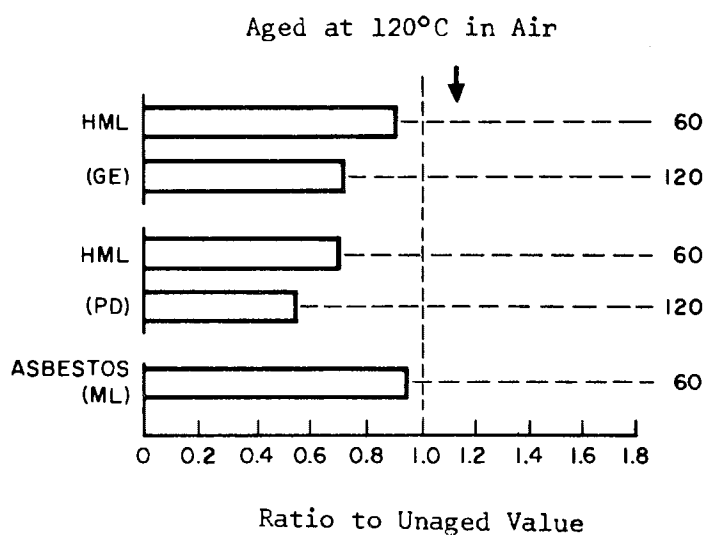
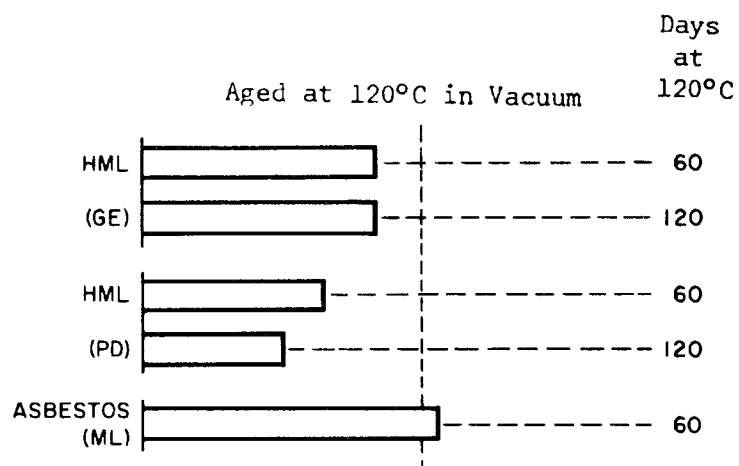
[Ref. 103]

<u>Specimen</u>	<u>Freq. & Temp.</u>	<u>Solid Content</u>	<u>Solvent System</u>	<u>Dissipation Factor</u>
Binder Solution ML (DuPont RC-675)	1 Kc, 23°C ↓	16.5%	NMP/DMAC	0.002
Binder Solution PI-2100		25%	DMF	0.001
Binder Solution PI-3101		60%	NMP/Xylene	0.001
Binder Solution PI-1100		18.6%	DMAC	0.002
Binder Solution PI-1101		18.3%	DMAC	0.001
				[Ref. 90]
DuPont RK-692 Polyimide Varnish	100 cps, 23°C			0.003
				[Ref. 33]
AI-220 (No. 18 AWG) Magnet Wire Coating	1 Kc, 25°C ↓	100°C		0.0075
		200°C		0.0117
				0.0024
Heavy "Pyre-ML" (No. AWG) Magnet Wire Coating	↓	25°C		0.0033
		100°C		0.0067
		200°C		0.017
				[Ref. 5,8]
Shawinigan RS-5303 and RS-5305 Polyimide Solutions	60 cps, 25°C ↓	50°C		0.0084
		75°C		0.0088
		100°C		0.0105
		125°C		0.0105
		150°C		0.0134
		175°C		0.0135
		200°C		0.0150
				0.0173
				[Ref. 112]



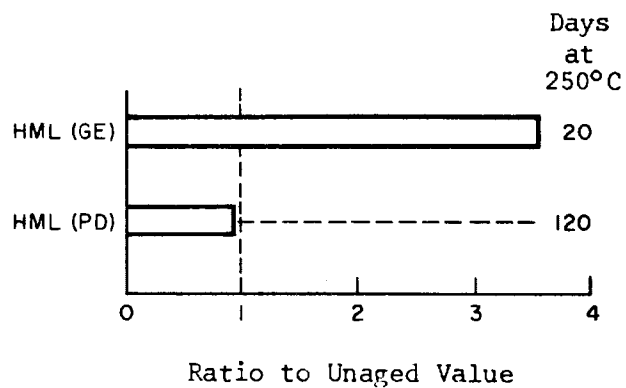
Comparison of dissipation factors at 1 Kc after exposure for 15 days at 80°C and 95% RH.

[Ref. 72]



Comparison of dissipation factors at 1 Kc and 23°C - 50% RH
after thermal aging at 120°C.

[Ref. 72]



Comparison of dissipation factors at 1 Kc and 23°C - 50% RH
after thermal aging at 250°C.

[Ref. 72]

Effect of Thermal Aging on Dissipation Factor at 1 KC

Wire	At 23 C	At 23 C	At 4.2 K	At 23 C	At 77°K	At 4.2 K	At 23 C	At 4.2 K	At 23 C	At 77°K	At 4.2 K	At 23 C	At 4.2 K	At 23 C	At 77°K	At 4.2 K
	Before Aging	After 60 d 120 C/Air	After 60 d 120 C/Air	After 120 d 120 C/Air	After 120 d 120 C/Air	After 120 d 120 C/Air	After 60 d 120 C/Vac	After 60 d 120 C/Vac	After 120 d 120 C/Vac	After 120 d 120 C/Vac	After 120 d 120 C/Vac	After 60 d 250 C/Air	After 60 d 250 C/Air	After 120 d 250 C/Air	After 120 d 250 C/Air	After 120 d 250 C/Air
HML (G.E.)	.00097	.00087	.00000													
	.00096	.00089	.00000													
	.00116			.00097	.00080	.00000										
	.00147			.00089	.00080	.00000										
	.00088						.00076	.00000								
	.00115						.00097	.00000								
	.00121								.0011	.00070	.0003					
	.00114								.00093	.00076	.0006					
	.00092											.00165	.00000			
	.00083											.00117	.00000			
	.00076													.0029	.0004	.00000
	.0011													.0036	.0005	.00005
HML (PD)	.0013	.00096	.00000													
	.0016	.0011	.00000													
	.0014			.00066	.00070	.00000										
	.0012			.00070	.00068	.00000										
	.0021						.00132	.00000								
	.0018						.0012	.00000								
	.0019								.00063	.00059	.00000					
	.0023								.00115	.00074	.00004					
	.0015													.0021	.0012	.00000
	.0018													.0144	.0011	.00000
ML Coated Felted Asbestos	0.26	0.243	.00000													
	0.22	0.211	.00000													
	0.26						0.275	.00000								
	0.20						0.215	.00000								

Dissipation Factor Before and After Moisture Exposure

Specimen	Sample No.	Test Condition			
		1	2	3	4
HML/GE	1	0.00164	0.0054	0.0001	0.0098
	2	0.00159	0.0059	0.0008	0.0095
HML/PD	1	0.00120	0.0030	0.0003	0.0057
	2	0.00114	0.0024	0.0002	0.0019
	3	0.00117	0.0029	0.0006	0.0054

Condition 1 As received - 23°C and 50% RH.

Condition 2 After exposure at 80°C-95% for 15 days, specimen removed from chamber and placed immediately in chamber at 23°C, 95% RH and measured after 36 hours at 23°C-95% RH.

Condition 3 Removed from Condition 2 above and plunged directly into liquid nitrogen before measurement.

Condition 4 Removed from Condition 3 above and kept at 23°C, 50% RH for 72 hours before measurement.

[Ref. 72]

Specimen	Freq.	Temp.	Dissipation Factor
Polyimide Homogeneous Magnet Wire Coating (build 2.9 mils)	60 cps	25°C	0.00803
		50°C	0.00607
		75°C	0.00624
		100°C	0.00675
		125°C	0.0066
		150°C	0.0082
		175°C	0.0116
		200°C	0.044
Polyvinyl Formal/Phenolic: Polyimide*Magnet Wire Coating (build 3.0 mils)	60 cps	25°C	0.00832
		50°C	0.00581
		75°C	0.00462
		100°C	0.00501
		125°C	0.0110
		150°C	0.0303
		175°C	0.116
		200°C	0.279
Polyvinyl Formal/Urethane: Polyimide*Magnet Wire Coating (build 3.0 mils)	60 cps	25°C	0.0121
		50°C	0.00913
		75°C	0.00692
		100°C	0.00611
		125°C	0.00940
		150°C	0.0480
		175°C	0.138
		200°C	0.260

*Note: Principal components of enamels are separated by virgule (/).
Multi-layer coatings are indicated by top layer:bottom layer.

[Ref. 43]

<u>Specimen</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
Polyimide-Glass Fiber Magnet Wire Insulation (twisted wire pair, 0.0013-inch wall thickness sample)	1 Kc ↓	269°K 77°K 4°K	0.0016 0.0072 0.00012 [Ref. 71]
Brunswick Corporation, Polyimide Glass Fabric Laminates	9360 Mc ↓	77°F 350°F 700°F	0.015 0.010 0.009 [Ref. 17]
DuPont P-2101 Polyimide Resin-181E-All100 Glass Fabric Reinforced Laminates	↓	77°F 300°F 500°F 700°F	0.0065 0.0068 0.0070 0.0090 [Ref. 37]
DuPont "Pyre-ML" Coated Glass Fabrics (4-mil specimen)	100 cps 1 Kc 10 Kc 100 Kc	25°C ↓	0.0149 0.0062 0.0042 0.0055 [Ref. 89]

Polyimide Coated Fabrics:

<u>Fabric Designation</u>	<u>Nominal Thickness</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
6500	4.0 mil ↓	100 cps 1 Kc 10 Kc 100Kc	23°C ↓	0.0049 0.0062 0.0042 0.0055
6507	5.5	100 cps		0.0024
6507	7.0 ↓	100 cps 1 Kc 10 Kc 100 Kc		0.0018 0.0016 0.0026 0.0044
6508	10.5 ↓	100 cps 1 Kc 10 Kc 100 Kc		0.0014 0.0013 0.0021 0.0040
6509	8.75	1 Kc	↓	>0.0050 [Ref. 67]

PI-3301 Polyimide-Filament Wound Panels (20 end-S-994, Hts):

<u>Aging Conditions</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
None	9375 Mc	77°F	0.0143
↓	↓	250°F	0.0165
		550°F	0.0187
		700°F	0.0194
16 hours at 700°F		77°F	0.0122
32 hours at 700°F		77°F	0.0197
		77°F	0.0197

[Ref. 26]

Electrical Properties of Glass Laminates at 10^{10} cps (Polymer I-6)

<u>Condition of Sample</u>	<u>Sample No.</u>	<u>Temp.</u>		<u>Dissipation Factor Tan δ</u>	
		<u>°C</u>	<u>°F</u>		
As received	1	25	77	0.0083	
	2	↓	↓	0.0077	
	2	↓	↓	0.0078	
	3	↓	↓	0.0072	
	3	↓	↓	0.0063	
Immersed 24 hours in water	1			0.101	
	2			0.037	
	3			0.026	
After immersion: dried 24 hours at 115 °C plus one hour at 300°C	1			0.0071	
	2			0.0062	
	3			0.0064	
	3	↓	↓	0.0067	
Sample heated	1	150	302	0.0045	
	3	↓	↓	0.0044	
	1	265	530	0.0044	
	2	↓	↓	0.0045	
	3	↓	↓	0.0068	
	1	315	600	0.0057	
	2	↓	↓	0.0075	
	3	↓	↓	0.0051	

Dissipation factor was found to be independent of temperature up to 600°F.

[Ref. 45]

<u>Specimen</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Insulation Thickness</u>	<u>Dissipation Factor (ASTM D-150)</u>
TFE - "Pyre-ML"	60 cps- 1 Mc	23°C	10 mil	0.0002
FEP - "Pyre-ML"	60 cps- 1 Mc	23°C	10 mil	0.0003

[Ref. 66]

Aromatic Imide Laminates;

Polymer	<u>EI-2</u>	<u>EI-2</u>	<u>I-8</u>
Laminate No.	019 (TC107)	006 (TC 107)	020 (TC 110)
Thickness	1.665	1.73	1.64
Press Temp. (°F)	662	428	662
Pressure (psi)	2,000	1,000	2,000
Resin Content (%)	25.9	25.9	28.6

Temperature				
°C	°F	<u>Tan δ*</u>	<u>Tan δ*</u>	<u>Tan δ*</u>
25	77	0.0089 .0098	0.0109	0.0094 .0084
150	311	.0071	.0056	.0044
200	392	.0077	.0057	.0047
250	482	.0064	.0066	.0059
300	572	.0079	.0076	.0061
350	662	.0072	.0076	.0048

After Aging 24 Hours at 600°F

25	77	.0097	.0085	
350	662	.0083	.0069	.0048

After Aging 100 Hours at 600°F

25	77	.016	.0085	.0095
350	662	.019	.0056	.0054

* Measured at 10 KMC.

DuPont "Kapton" Type HF Film (Polyimide-Teflon FEP) Composites

<u>Temp.</u>	<u>Type Designation</u>	<u>Dielectric Strength</u>
23°C	1H/ 1/2F*	4500 volts/mil
23°C	1/2F/1H 1/2F**	4500 volts/mil
23°C	2H/2F***	3000 volts/mil

Construction: * 1-mil Kapton Type H/ 1/2-mil Teflon FEP
 ** 1/2-mil Teflon FEP/1-mil Kapton Type H/ 1/2-mil Teflon FEP
 *** 2-mil Kapton Type H/2-mil Teflon FEP

[Ref. 27]

<u>Specimen</u>	<u>Temp.</u>	<u>Density</u>	<u>Dielectric Strength</u>
DuPont Polyimide Foam	23°C	2-30 lbs/ft ³	100 volts/mil

[Ref. 28]

<u>Isomid</u>	<u>Dielectric Strength, Dry, vpm</u>
At R.T.	4400
Wet, 24 hours in H ₂ O	2700
Unvarnished	<u>Dielectric Strength/Thermal Stability MIL-W-583C, Type K, vpm</u>
Before heat aging	4400
After 168 hours at 250°C	4200
Varnished [Isonel 31, Meets MIL-24092(SHIPS)]	
Before heat aging	4870
After 168 hours at 250°C (800 vpm minimum)	3670

[Ref. 134]

The effect of irradiation on the dielectric strength of polyimide plastics is detailed in a later section on IRRADIATION EFFECTS ON PROPERTIES.

"Kapton" Type H Film

Effect of thickness on dissipation factor:

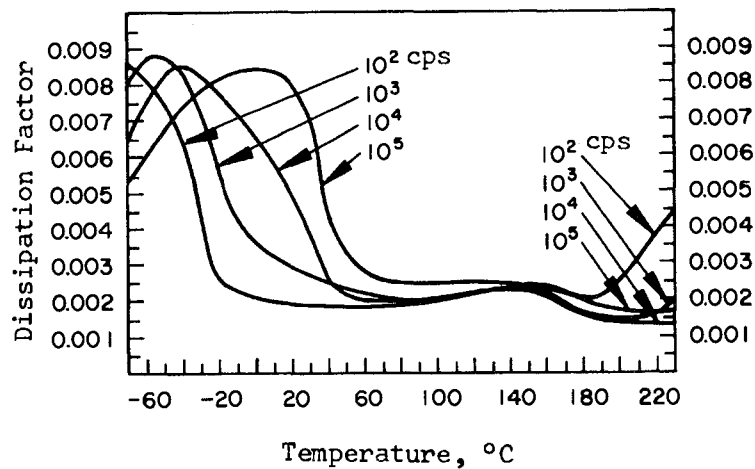
<u>Test Conditions</u>	<u>Thickness</u>	<u>Dissipation Factor</u>
10 ³ cps, 25°C, 50% RH	1 mil	0.0025
	2 mil	0.0019
	3 mil	0.0017
	5 mil	0.0017

Effect of temperature on dissipation factor:

<u>Thickness</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
1 mil ↓	10 ³ cps ↓	-50°C	0.0083
		0°C	0.0031
		25°C	0.0025
		100°C	0.0017
		200°C	0.0010
		300°C	~0.1000
	10 ⁵ cps ↓	-50°C	0.0062
		0°C	0.0081
		25°C	0.0078
		100°C	0.0028
		200°C	0.0018
		300°C	~0.0040

[Ref. 31]

Mallouk of the DuPont Company has also measured the effect of temperature on 1 mil thick H-Film:



Dissipation factor of H-Film
(1 mil thick)
as a function of temperature.

[Ref. 2]

<u>Aging Condition</u>	<u>Thickness</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
as received	1 mil	1 Kc	23°C	0.003
8 weeks at 300°C	↓	↓	↓	0.0014
5 days at 350°C				0.002
8 hours at 400°C				0.003

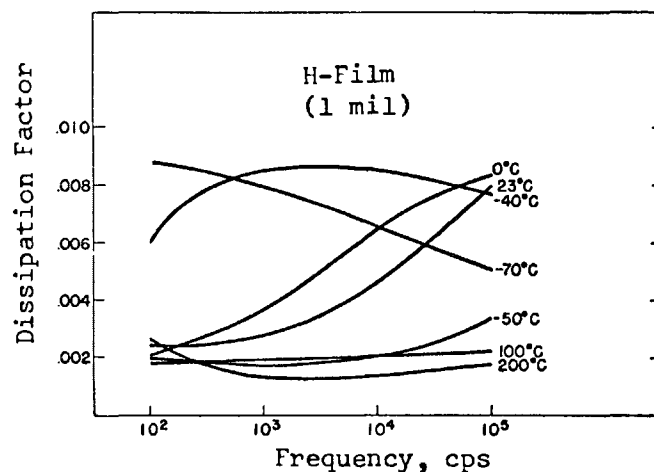
[Ref. 31]

Effect of frequency on dissipation factor:

<u>Thickness</u>	<u>RH</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
1 mil	50%	10 ² cps	25°C	0.0020
↓	↓	10 ³	↓	0.0025
		10 ⁴		0.0042
		10 ⁵		0.0078
		10 ⁶		0.0098
		10 ⁷		0.0118
		10 ²	200°C	0.0028
		10 ³	↓	0.0010
		10 ⁴		0.0014
		10 ⁵		0.0018

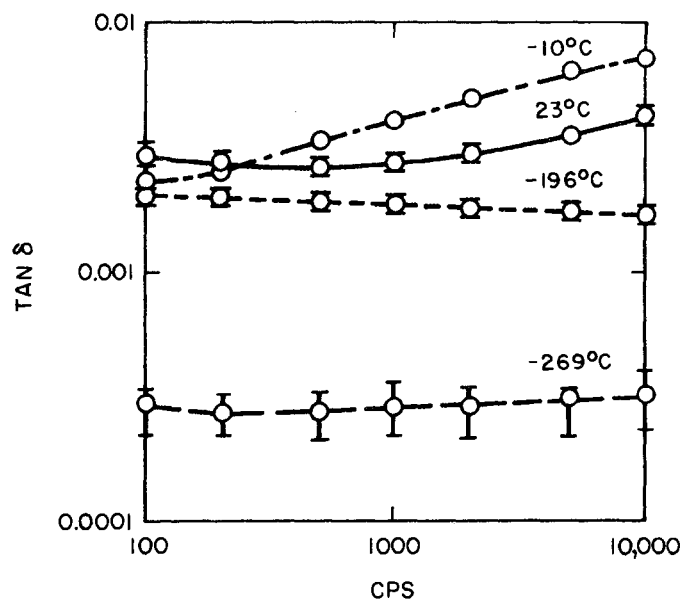
[Ref. 31]

Tatum et al have plotted similar data:



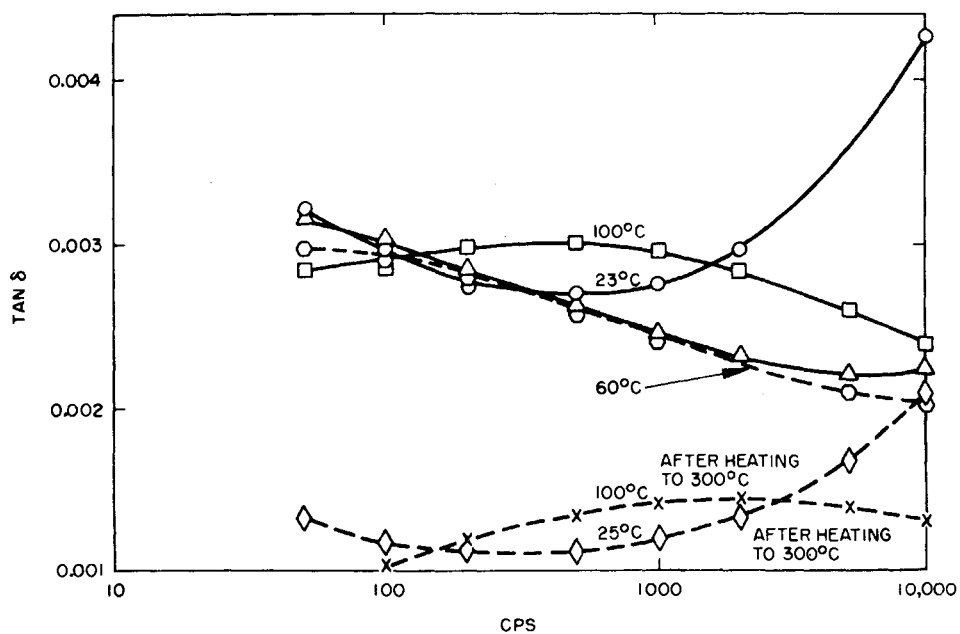
Dissipation factor as a function of frequency.

[Ref. 2, 58]



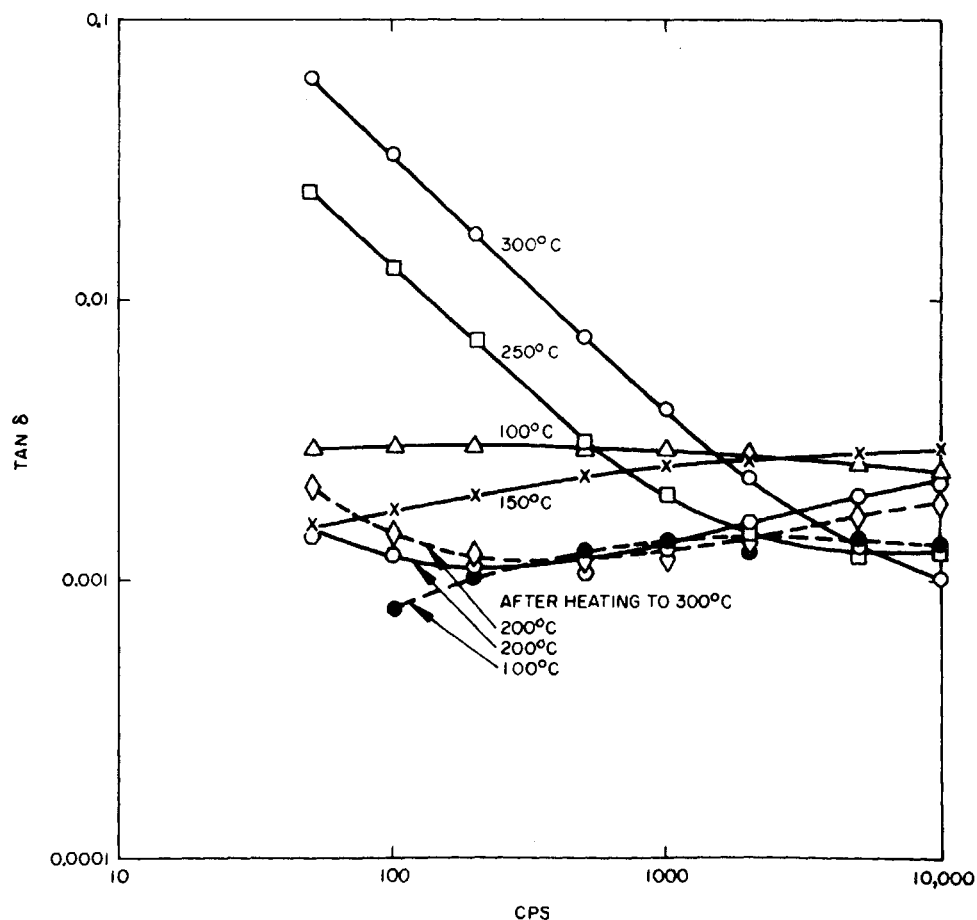
Dissipation factor ($\tan \delta$) as a function of frequency for FEP and H-Film at 23°C, -196°C and -269°C.

[Ref. 72]



Dissipation factor ($\tan \delta$) as a function of frequency for H-Film - expanded scale.
(dashed curves indicate extended heating for 18 hours)

[Ref. 72]



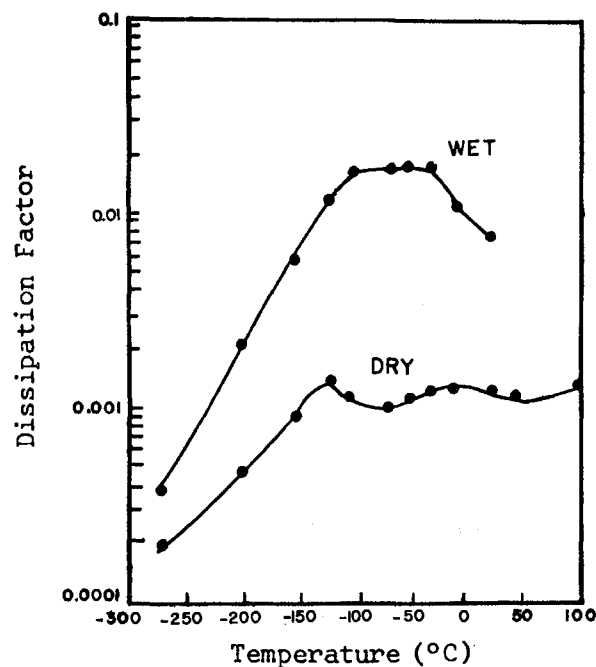
Dissipation factor ($\tan \delta$) as a function of frequency
for H-Film at elevated temperatures.
(dashed curves indicate extended heating for 17 hours)

[Ref. 72]

Effect of moisture on dissipation factor:

<u>Specimen</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
Dry H-Film	10° cps	-269°C	0.0008
	1 Kc		0.000086- 0.000101
	10 Kc		0.000141- 0.000142
Wet H-Film	10° cps		0.00022- 0.00031
	1 Kc		0.000300- 0.000317
	10 Kc		0.000357- 0.000363

[Ref. 72]



Dissipation factor at 1 Kc as a function of temperature.
 "Wet" H-Film (as above plus 2 days in water at 80°C)
 "Dry" H-Film (heated 2 days at 200°C)

[Ref. 72]

<u>Test Conditions</u>	<u>RH</u>	<u>Dissipation Factor</u>
1 mil, 25°C, 10 ³ cps	0%	0.0018
	30%	0.0021
	(1% H ₂ O) 50%	0.0025
	80%	0.0037
	(3% H ₂ O) 100%	0.0047

[Ref. 31]

<u>Specimen</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
"Amanim" Film (Westinghouse Electric)	60 cps ↓	25°C	0.006
		100°C	0.005
		200°C	0.020
	1 Kc ↓	25°C	0.007
		200°C	0.010
	100 Kc	25°C	0.016

[Ref. 44]

<u>Specimen</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
Polyimide Polymer	60 cps	25°C	0.008
Film - C	1000 cps	↓	0.013
Polyimide Polymer	60 cps		0.005
Film - D	1000 cps		0.009
Polyimide Polymer	60 cps		0.0005
Film - E	1000 cps	↓	0.0008

[Ref. 51]

DuPont "Kapton type HF (Polyimide-Teflon FEP) Composites:

<u>Type Designation</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
1H/ 1/2F *	1 Kc	23°C	0.0035
1/2F/1H/ 1/2F **	↓	↓	0.001
2H/2F ***	↓	↓	0.005

Construction: * 1-mil Kapton Type H/ 1/2-mil Teflon FEP
 ** 1/2-mil Teflon FEP/ 1-mil Kapton Type H/ 1/2-mil Teflon FEP
 *** 2-mil Kapton Type H/ 2-mil Teflon FEP

[Ref. 27]

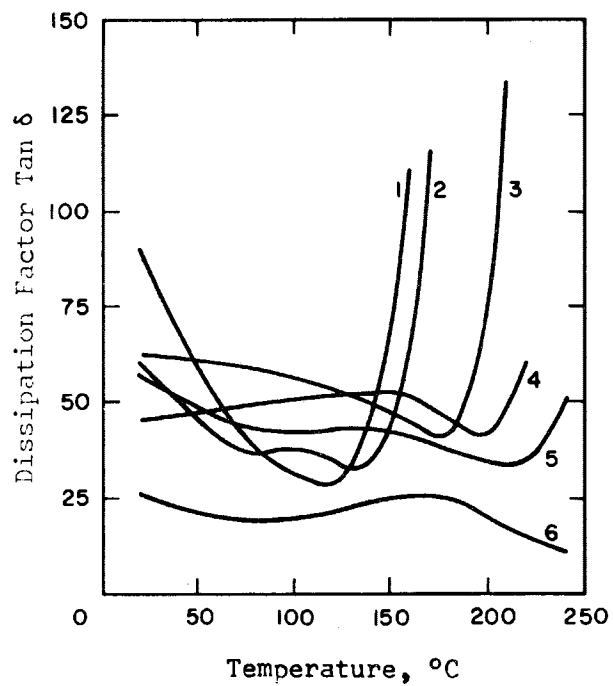
<u>Specimen</u>	<u>Density</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Dissipation Factor</u>
DuPont Polyimide Foam	2-30 Lbs/ft ³	1 Kc	23°C	0.0008

[Ref. 28]

Isomid (Polyester-Imide):

<u>Temperature</u>	<u>Frequency</u>	<u>Dissipation Factor</u>
25°C	1 Kc	0.0064
150°C	1 Kc	0.0170

[Ref. 134]



The dissipation factor of polyester-polyimide magnet wire coatings as a function of temperature at 800 cps.

- 1 - Polyester Terebec F
- 2 - Polyester-polyimide Terebec FN
- 3 - Polyester-polyimide Terebec FH
- 4 - Polyester-polyimide Research Material 109
- 5 - Polyester-polyimide Research Material 115
- 6 - Polyimide Research Material P437

[Ref. 88]

II. PROPERTIES

B. ELECTRICAL

6. Electrical Resistivity

<u>Sample</u>	<u>Temperature</u>	<u>Volume Resistivity</u>	<u>Ref.</u>
SP-Polymer	23°C	10^{13} ohm-cm	19
SP-1 Polymer	23°C	10^{17} ohm-cm (ASTM-D257)	98

The volume resistivity of SP-Polymer decreases linearly from 10^{17} ohm-cm at room temperature to 10^{11} ohm-cm at 572°F. Moisture content appears to have little effect on the volume and surface resistivities of the SP-1 polyimides as shown in the following table:

<u>Condition of Sample</u>	ρ <u>(ohm-cm)</u>	σ <u>(ohms/square)</u>	<u>Ref.</u>
as received	5.0×10^{16}	8.0×10^{15}	31
1.4% H ₂ O	6.0×10^{16}	4.5×10^{16}	31

<u>Sample</u>	<u>Condition</u>	<u>Insulation Resistance</u>	<u>Ref.</u>
Standard AI-220 (#18 AWG) Magnet Wire (Twisted Pair)	10-min. water boil	500,000 megohms	8
Heavy PYRE-M.L.(# 18 AWG) Magnet Wire (Twisted Pair)	10-min. water boil	500,000 megohms	5

<u>Sample</u>	<u>Temperature</u>	<u>Apparent Specific Resistance (RA)</u>	<u>Ref.</u>
Polyimide Magnet Wire Coating (#18 AWG, heavy build, 2.9 mil)	25°C	1.05×10^{12}	43
	50	1.47×10^{12}	
	75	1.40×10^{12}	
	100	1.30×10^{12}	
	125	1.32×10^{12}	
	150	1.07×10^{12}	
	175	7.59×10^{11}	
	200	2.00×10^{11}	
Polyvinyl Formal/phenolic Polyimide Magnet Wire coating* (#18 AWG, 3.0 mil build)	25°C	1.04×10^{12}	43
	50	1.55×10^{12}	
	75	2.00×10^{12}	
	100	1.84×10^{12}	
	125	7.35×10^{11}	
	150	2.30×10^{11}	
	175	5.73×10^{10}	
	200	2.07×10^{10}	

*Note: Principal components of enamels are separated by virgule (/).
Multi-layer coatings are indicated by top layer: bottom layer.

<u>Sample</u>	<u>Temperature</u>	<u>Apparent Specific Resistance (R_A)</u>	<u>Ref.</u>
Polyvinyl Formal/urethane:	25°C	6.77×10^{11}	43
Polyimide Magnet Wire	50	9.51×10^{11}	
Coating* (#18 AWG, 3.0 mil build)	75	1.28×10^{12}	
	100	1.45×10^{12}	
	125	9.23×10^{11}	
	150	1.52×10^{11}	
	175	4.47×10^{10}	
	200	1.88×10^{10}	

*Note: Principal components of enamels are separated by virgule (/)
Multi-layer coatings are indicated by top layer: bottom layer.

Polyimide Magnet Wire Coating:

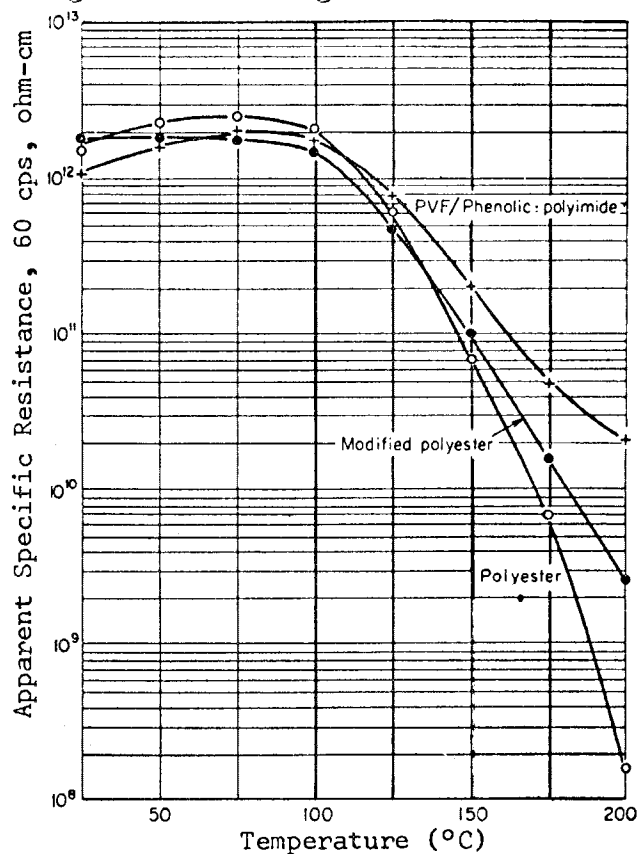
Magnet-Wire Coatings in Order of Temperatures at which Apparent Specific Resistance is 10^{10} ohm-cm.

<u>Type of Coating</u>	<u>t_r, °C</u>	<u>$K \tan \delta$ (at t_r)</u>
Polyimide	<200	(>0.2)
PVF/phenolic: polyimide	<200	(>1.5)
PVF/urethane: polyimide	<200	(>1.6)

[Ref. 43]

Fitzhugh and Taylor have compared the apparent specific resistance of Polyvinyl Formal/Phenolic:Polyimide magnet wire coatings with other wire coatings:

Apparent specific resistance of wire coatings classified at 155-180°C by AIEE procedure #57.



[Ref. 43]

<u>Sample</u>	<u>Temperature</u>	<u>Fabric Designation</u>	<u>Nominal Thickness</u>	<u>Volume Resistivity</u>	<u>Ref.</u>
Polyimide coated fabrics	25°C	6500	4.0 mils	10 ¹⁴ ohm-cm	34, 67
		6507	5.5	10 ¹⁴	
		6507	7.0	10 ¹⁴	
		6508	10.5	10 ¹⁴	
PYRE-M.L. coated glass fabrics	25°C	-	4.0 mil	10 ¹⁴ ohm-cm	89

<u>Sample</u>	<u>Condition</u>	<u>Insulation Resistance</u>	<u>Ref.</u>
DuPont RK-692 polyimide varnish	Dry	infinite	35
	Wet (after boiling 10-min. in water)	5 x 10 ⁵ megohms	

	<u>Surface Resistance</u>	<u>Surface Resistance after 15 days at 80°C-95% RH</u>		<u>Volume Resistance after 15 days at 23°C-95% RH</u>	
	<u>at 23°C-50% RH</u>	<u>at 23°C-95% RH</u>	<u>In liq. N₂</u>	<u>at 23°C-95% RH</u>	<u>In liq. N₂</u>
HML(GE)*	1.7 x 10 ¹⁶	2.2 x 10 ¹³	2.7 x 10 ¹⁶	6.5 x 10 ¹²	1.1 x 10 ¹⁴
HML(PD)**	1.2 x 10 ¹⁶	1.7 x 10 ¹³	8.3 x 10 ¹⁵	1.2 x 10 ¹²	2.1 x 10 ¹⁴

* General Electric Company

** Phelps Dodge Company

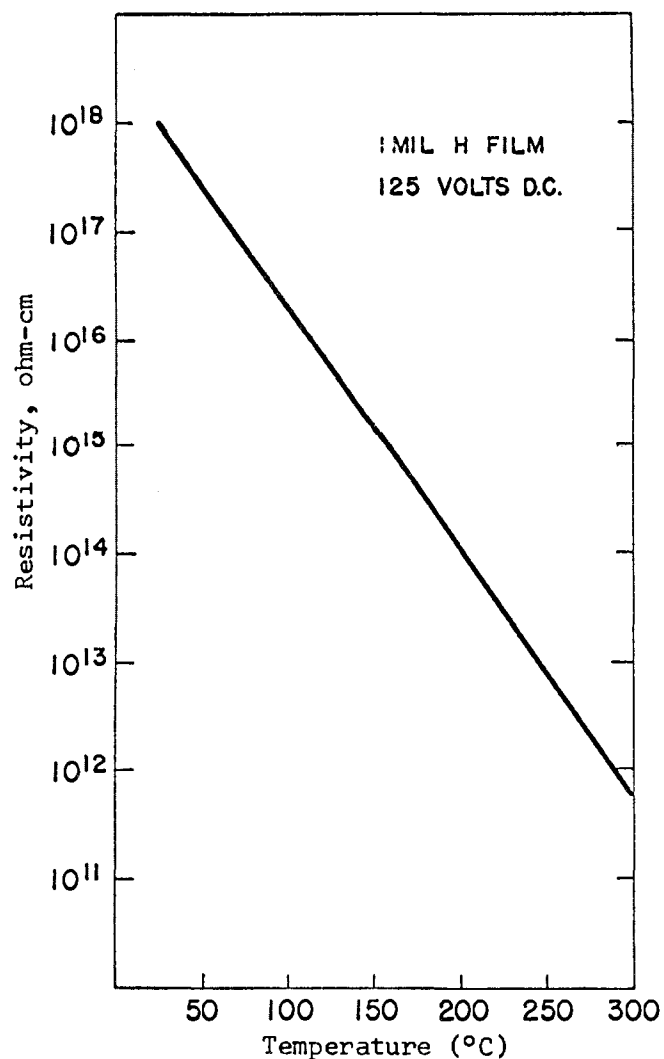
[Ref. 72]

<u>Sample</u>	<u>Thickness</u>	<u>Temperature</u>	<u>Resistivity (ASTM D-257)</u>	<u>Ref.</u>
PYRE-M.L.	10 mils wire enamel film	23°C	10 ¹⁷ ohm-cm	66
Teflon TFE/ PYRE-M.L.	10 mil	23°C	10 ¹⁸ ohm-cm	66
Teflon FEP/ PYRE-M.L.	10 mil	23°C	10 ¹⁸ ohm-cm	66

<u>Sample</u>	<u>Temperature</u>	<u>Volume Resistivity</u> <u>(ASTM-D257-61)</u>	<u>Ref.</u>
H-Film	25°C 200°C	10^{18} ohm-cm 10^{14}	29, 68
		<u>Surface Resistivity</u> <u>(ASTM-D257-61)</u>	
H-Film (50% RH)	25°C	10^{16} ohm-cm	29, 68
		(ASTM-D257-52T)	
H-Film (50% RH) (1000 volts)	23°C	10^{17} ohm-cm	2, 68

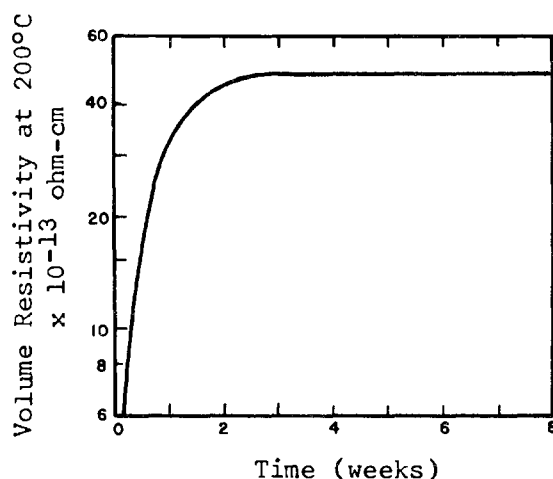
Mallouk and Tatum, et al have measured the volume resistivity of 1-mil H-film over a wide range of temperature:

Volume Resistivity as a function of temperature.



[Ref. 94,68,58]

Mallouk and Tatum, et al, also studied the effect of thermal aging at 300°C of 1-mil H-Film on the 200°C volume resistivity:



Effect of thermal aging at 300°C of 1-mil H-Film on volume resistivity. Resistivity was measured at 200°C.

[Ref. 94, 58]

DuPont "Kapton" HF-Film
(Polyimide-Teflon FEP Composites):

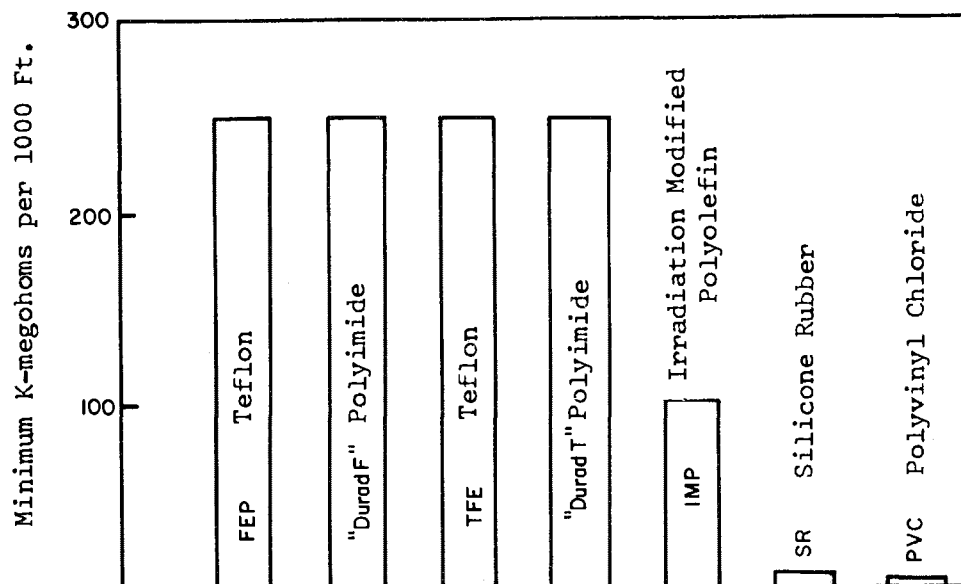
<u>Sample</u>	<u>Temperature</u>	<u>Volume Resistivity</u>	<u>Ref.</u>
1H/ 1/2F *	25°C	10 ¹⁸ ohm-cm	27
1H/ 1/2F *	200°C	10 ¹³	
1/2F/1H/ 1/2F **	25°C	10 ¹⁸ ohm-cm	
1/2F/1H/ 1/2F **	200°C	10 ¹³	
2H/2F ***	25°C	10 ¹⁸ ohm-cm	27
2H/2F ***	200°C	10 ¹³	

Construction: * 1-mil "Kapton" Type H/ 1/2-mil Teflon FEP
 ** 1/2-mil Teflon FEP/1-mil "Kapton" Type H/ 1/2-mil Teflon FEP
 *** 2-mil "Kapton" Type H/2-mil Teflon FEP

"Kapton" Type H-Film

<u>Temperature</u>	<u>Thickness</u>	<u>Volume Resistivity</u>	<u>Ref.</u>	
25°C	1 mil	1×10^{18} ohm-cm	31	
100°C	↓	1×10^{17}		
200°C		9×10^{13}		
300°C	↓	1×10^{11}		
200°C	1 mil	9×10^{13} ohm-cm		
	2 mil	4×10^{13}		
	3 mil	4×10^{13}		
	5 mil	2×10^{13}		
<u>Temperature</u>	<u>Thickness</u>	<u>Aging</u>	<u>Volume Resistivity</u>	<u>Ref.</u>
200°C	1 mil	unaged (as received)	4×10^{14} ohm-cm	31
↓	↓	8 weeks at 300°C	4×10^{14}	
		5 days at 350°C	7×10^{14}	
		8 hours at 400°C	4×10^{14}	

Insulation Resistance



Comparison of "Durad" insulations with Teflon and other common wire insulations.

[Ref. 3]

AMANIM Polyamide-imide Film (Westinghouse Electric):

<u>Temperature</u>	<u>D-C Volume Resistivity</u>
25°C	7.7×10^{16} ohm-cm
150	1.2×10^{14}
200	9.2×10^{11}

[Ref. 44]

Freeman, et al, have measured the effect of relative humidity on the surface resistivity of aromatic polyamide-imide Film D samples (all values were taken at 25°C, 9.1 volts d-c):

Effect of RH on Surface Resistivity* of Aromatic Polyamide-imide Film D

<u>Sample</u>		<u>1</u>		<u>2</u>		<u>3</u>		<u>4</u>	
<u>Conditioning</u>	<u>%RH</u>	<u>1 min.</u>	<u>2 min.</u>	<u>1 min.</u>	<u>2 min.</u>	<u>1 min.</u>	<u>2 min.</u>	<u>1 min.</u>	<u>2 min.</u>
as received	30		22.3		12.4		49.5		41.3
72 hrs. at	70-75	3.6	4.5	2.7	3.5	2.1	2.4	1.6	1.8
96 hrs. at	70-75	9.1	7.8	1.6	2.1	2.5	3.1	1.9	2.2
120 hrs. at	70-75	3.3	5.4	2.5	2.9	2.4	2.7	1.4	1.7
120 hrs. at	80-90	2.2	2.4	0.6	0.7	0.4	0.5	0.6	0.7
144 hrs. at	80-90	4.8	5.2	0.7	0.9	0.6	0.7	0.8	0.9

*Values are ohms/square $\times 10^{-13}$.

[Ref. 45]

Isomid (Polyester-Imide):

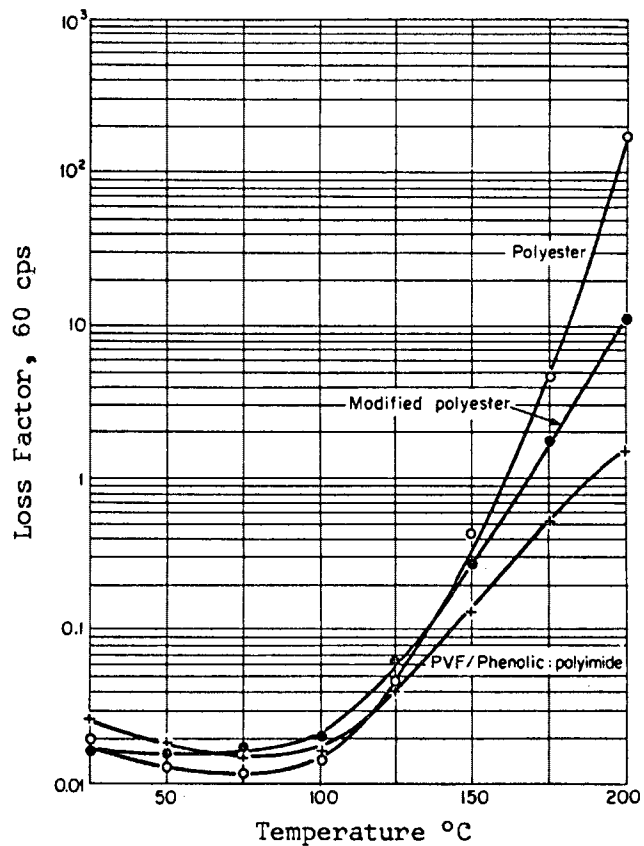
<u>Sample</u>	<u>Insulation Resistance</u> <u>(megohms)</u>
Twisted pair, 10 minute water boil	500,000

[Ref. 134]

B. Electrical

7. Loss Factor

Fitzhugh and Taylor have compared the loss factor at elevated temperature of a poly (vinyl formal)/phenolic:polyimide magnet wire coating with polyester magnet wire coatings. The coating consisted of a thin base of DuPont "Pyre-ML" jacketed by a poly (vinyl formal) phenolic coating:



[Ref. 43]

The same authors present the following loss factor data for other polyimide magnet wire coatings:

<u>Specimen</u>	<u>Freq.</u>	<u>Temp.</u>	<u>Loss Factor</u>
Polyimide Magnet Wire Coating (2.9 mils build)	60 cps	25°C	0.0285
		50°C	0.0210
		75°C	0.0212
		100°C	0.0229
		125°C	0.0227
		150°C	0.0282
		175°C	0.0392
		200°C	0.15
Polyvinyl Formal/Phenolic: Polyimide*Magnet Wire Coating (3.0 mils build)		25°C	0.0289
		50°C	0.0193
		75°C	0.0150
		100°C	0.0163
		125°C	0.0408
		150°C	0.130
		175°C	0.523
		200°C	1.45
Polyvinyl Formal/Urethane: Polyimide*Magnet Wire Coating (3.0 mils build)		25°C	0.0442
		50°C	0.0315
		75°C	0.0234
		100°C	0.0206
		125°C	0.0322
		150°C	0.198
		175°C	0.670
		200°C	1.60

*Note: Principal components of enamels are separated by virgule (/). Multi-layer coatings are indicated by top layer:bottom layer.

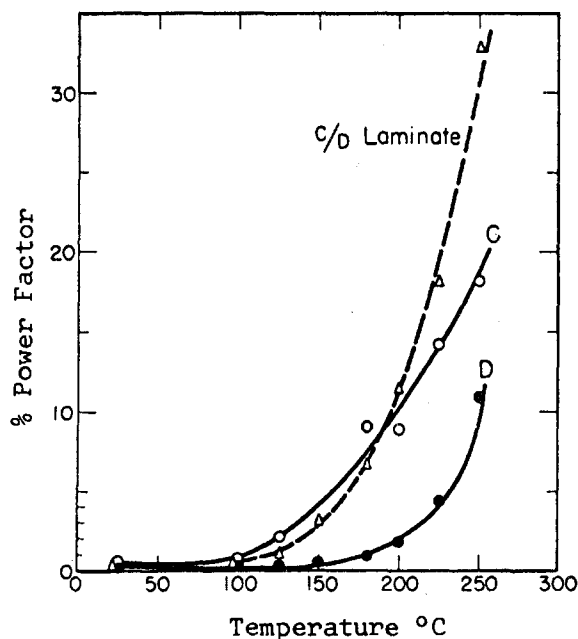
[Ref. 43]

B. Electrical

8. Power Factor

<u>Specimen</u>	<u>Fabric Designation</u>	<u>Nominal Thickness</u>	<u>Freq. & Temp.</u>	<u>Power Factor (ASTM D150-54T)</u>
Polyimide Coated Fabrics	6500	4 mil	1 Kc, 25°C ↓	0.6%
	6507	5.5 mil		0.35%
	6507	7.0 mil		0.16%
	6508	10.5 mil		0.13%
	6509	8.75 mil		0.5%
[Ref. 34,67]				
"Pyre ML" Coated Glass Fabrics		4 mil	1 Kc, 25°C	0.6%
[Ref. 89]				

Freeman et al have measured the 60-cycle power factor as a function of temperature for aromatic amide-imides on 116 glass cloth. Curves C and D represent two amide-imide copolymers differing in the proportion of imide to amide groups and also in the distribution of these linkages within the polymer chains:

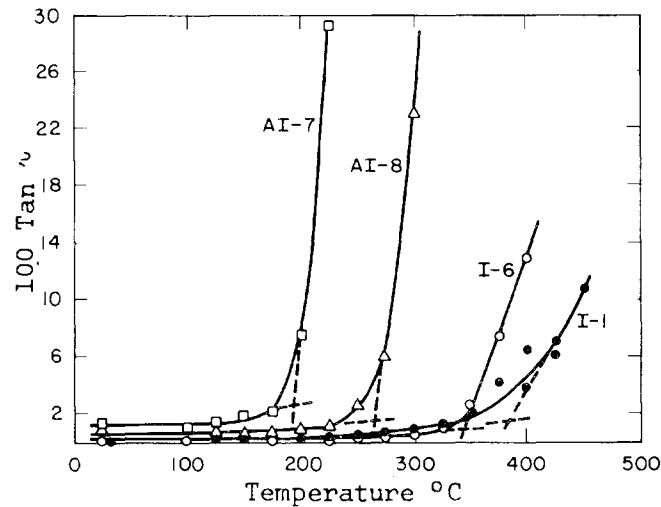


60 cycle power factor as a function of temperature. Aromatic amide-imides on 116 glass cloth.

C = 3/1 = ratio of amide to imide
D = 1/2 = ratio of amide to imide

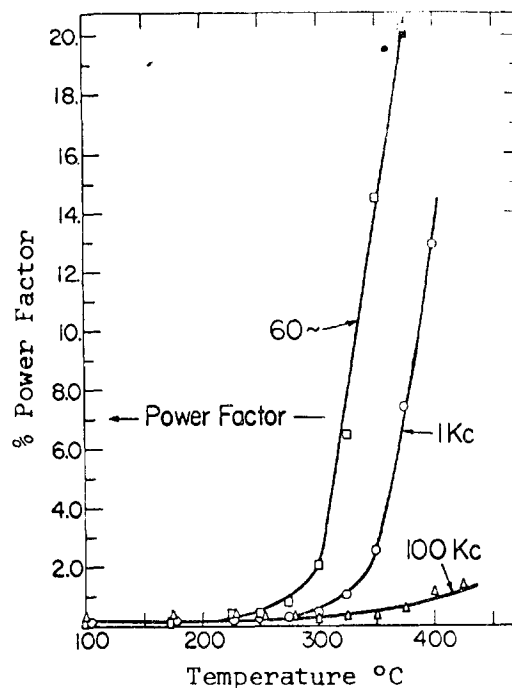
[Ref. 49]

Ruffing and Traynor, Freeman et al, have measured the effect of temperature on the power factor ($100 \tan \delta$) for several aromatic polyimides at 1 Kc. The glass transition temperature (T_g) was determined by the extrapolation of the dotted lines on the graph:



- I-1 = 4,4' - diaminophenyl ether and pyromellitic anhydride
- I-6 = 4,4' - diaminophenyl sulfide and pyromellitic anhydride
- AI-8 = 3,4' - diaminogenzanilide and pyromellitic anhydride
- AI-7 = m-phenylene diamine-isophthalic-terephthalic-pyromellitic dianhydride reaction mixture

[Ref. 51]



Electrical Properties as a function of temperature.
Aromatic polyimide I-6 (4, 4' - diaminophenyl sulfide and pyromellitic anhydride, 7 mils total).

[Ref. 46]

II. PROPERTIES

C. MECHANICAL

A considerable amount of mechanical property data have been reported in the literature and in vendor catalog sheets and practically every parameter is represented. Major attention has been directed to the properties at elevated temperature and the effects of long time thermal aging on the mechanical properties.

Retention of good properties of the polyimides at low temperatures has been demonstrated in tests in which magnet wires, coated with various types of insulation, are cooled to -450°F and bent over a mandrel. The polyimides withstood forward and reverse bends on a 1/8-inch radius; polyesters, TFE resins, and asbestos failed at 1/2-inch radius; and PVC failed at bends of 1 3/4-inch radius. The modulus and tensile strength decrease almost linearly as the temperature is raised, while the elongation goes through a maximum value at 300°C (572°F).

Like the film, the binder resins, have excellent mechanical properties throughout a wide range of temperature (liquid helium to as high as 1100°F). It ranks among the toughest polymeric films, having high tensile and impact strength, and high resistance to tear initiation. In general, its room temperature properties are comparable to those of Mylar.

The DuPont Company reports that orientation of "Kapton" Type H-Film has a moderate effect on its properties. Increases in shrinkage, crystallinity, modulus, tensile strength and decrease in elongation are encountered although the effect is not as dramatic as encountered in the orientation of polypropylene or polyethylene terephthalate (Mylar).

The effects of irradiation on the mechanical properties are treated separately in section G below.

II. PROPERTIES

C. MECHANICAL

1. Tensile Properties

a. Tensile Strength

"Kapton" Type H-Film

<u>Temp.</u>	<u>Humidity</u>	<u>Elongation Rate</u>	<u>Thickness</u>	<u>Tensile Strength (ASTM D-882)</u>
25°C	50% RH	100%/minute	1 mil	25,000 psi
↓	100% RH	↓	1 mil	24,000 psi
	50% RH		2 mil	25,000 psi
	↓		3 mil	↓
			5 mil	
25°C	50% RH	100%/minute	1 mil	25,000 psi
↓	↓	1000%/minute	↓	24,000 psi
		10000%/minute		21,000 psi
		100000%/minute		18,000 psi
		500000%/minute		16,000 psi
-195°C	--	100%/minute	1 mil	35,000 psi
-20°C	--	↓	↓	32,000 psi
25°C	--			25,000 psi
100°C	--			22,000 psi
200°C	--			17,000 psi
300°C	--			12,000 psi
450°C	--	↓	↓	5,000 psi

[Ref. 31]

<u>Aging Temp.</u>	<u>Aging Time</u>	<u>Elongation Rate</u>	<u>Thickness</u>	<u>Tensile Strength (at 25°C)</u>
250°C in air	control	100%/minute	1 mil	25,000 psi
	1 month			24,000 psi
	3 months			22,000 psi
	6 months			20,000 psi
300°C in air	control			25,000 psi
	1 week			24,000 psi
	3 weeks			21,000 psi
	6 weeks			18,000 psi
300°C in helium	control			25,000 psi
	1 week			24,000 psi
	3 weeks			22,000 psi
	6 weeks			19,000 psi
400°C in air	control			25,000 psi
	1 hour			22,000 psi
	2 hours			20,000 psi
	4 hours			18,000 psi
	6 hours			16,000 psi
	12 hours			10,000 psi
100°C in water	control			25,000 psi
	1 week			20,000 psi
	2 weeks			19,000 psi
	7 weeks			18,000 psi
	14 weeks			18,000 psi

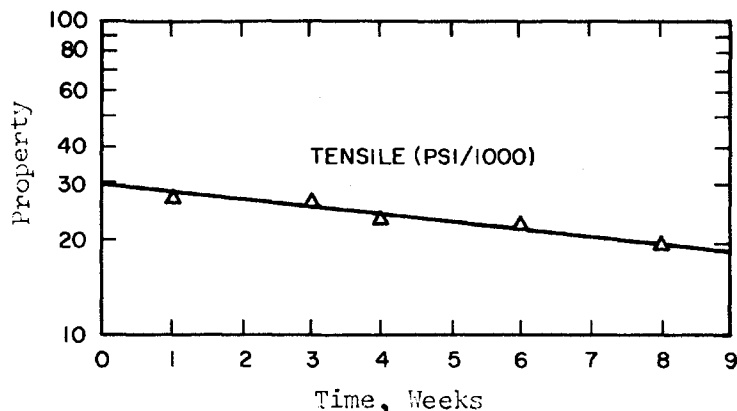
[Ref. 31]

"Amanim" Film (Westinghouse Electric)

<u>Temperature, °C</u>	<u>Direction</u>	<u>Tensile Strength</u>
25°C	Transverse	13,300 psi
25°C	Machine	18,000 psi
200°C	Machine	12,100 psi

[Ref. 44]

Heacock and Berr of the DuPont Company report the following thermal aging at 300°C data on "Kapton" Type H-Film:

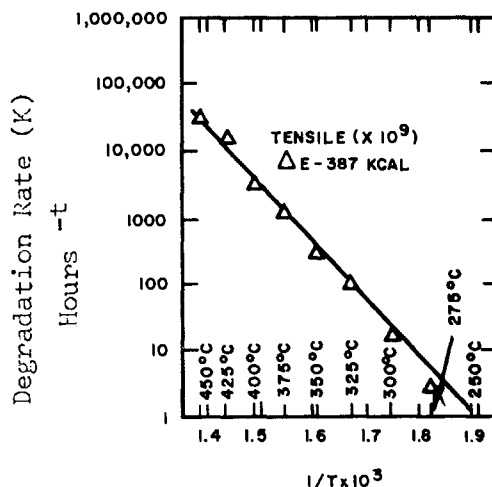


Effect of aging in air at 300°C on the physical properties of H-Film. Linear relationships are apparent for each property.

[Ref. 59]

From these above data and other temperature levels, an Arrhenius plot was constructed based on the equation: $\log P = -kt + \log P_0$

Arrhenius plot of physical property deterioration.



[Ref. 59]

where P is the elongation property at time t hours and P_0 is the initial value of the property, and K is the rate constant in reciprocal hours. From these data the authors obtained for the elongation parameter the following activation energies, initial property constant, and rate constants at 400°C:

	In Air	In Helium
Activation energy (ΔE)	38.7 ± 1.3	55.5 ± 1.3 Kcal
Initial property (P_0)	23,000	23,000 psi
Rate constant (K_{400})	0.040	0.0019 /1 hour

[Ref. 59]

"Kapton" Type HF Composites:

<u>Construction</u>	<u>Temperature</u>	<u>Tensile Strength</u>
1-mil "Kapton" Type H/ 1/2-mil Teflon FEP (1H/ 1/2F)	25°C 200°C	16,000 psi 10,000 psi
1/2-mil Teflon FEP/ 1-mil "Kapton" H/ 1/2-mil Teflon FEP (1/2F/ 1H/ 1/2F)	25°C 200°C	15,000 psi 9,000 psi
1-mil "Kapton" Type H/1-mil Teflon FEP (1H/ 1F)	25°C 200°C	15,000 psi 9,000 psi
1-mil Teflon FEP/ 1-mil "Kapton" H/ 1-mil Teflon FEP (1F/ 1H/ 1F)	25°C	10,000 psi
2-mil "Kapton" Type H/ 2-mil Teflon FEP (2H/ 2F)	25°C 200°C	14,000 psi 7,500 psi

[Ref. 27]

SP-Polymer:

<u>Temperature</u>	<u>Tensile Strength</u>
77°F	13,000 psi
482°F	7,700 psi

[Ref. 11]

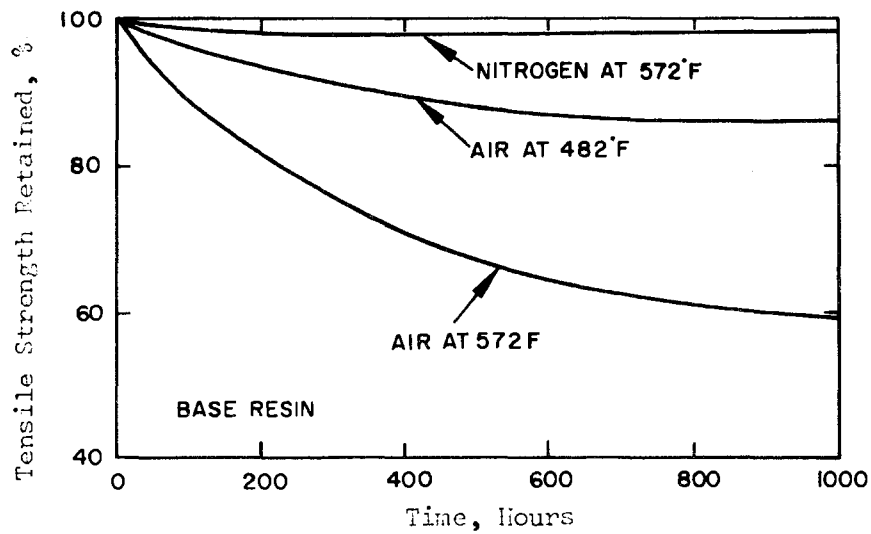
Tests have shown polyimide resins maintain 90% of tensile strength after 1000 hours of exposure to air at 570°F. For short periods of time, the material can withstand temperatures as high as 930°F.

SP-1 Polymer:

<u>Temperature</u>	<u>Tensile Strength</u> <u>(ASTM D-708)</u>
73°F	13,500 psi
302°F	9,700 psi
482°F	7,700 psi
600°F	5,000 psi
752°F	3,500 psi

[Ref. 97]

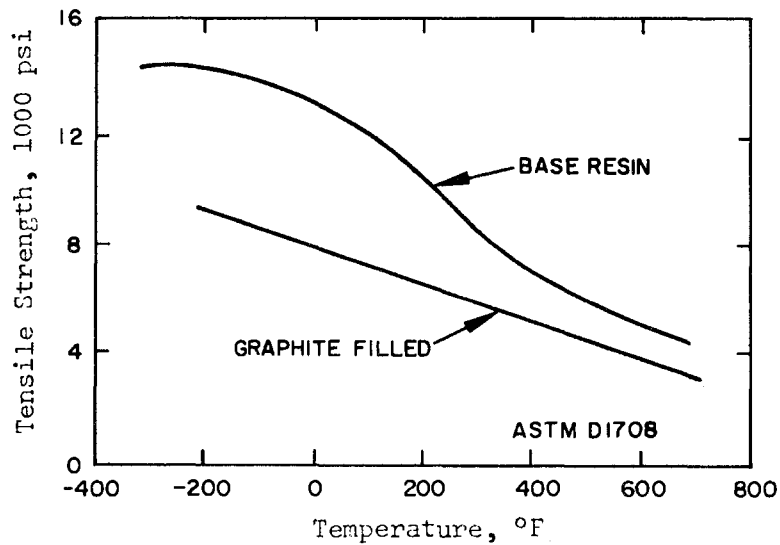
SP-Polymer:



Reduction of tensile strength in air is slight (about 15% after 1000 hour at 480°F) and insignificant in atmospheres containing no free oxygen, e.g., nitrogen.

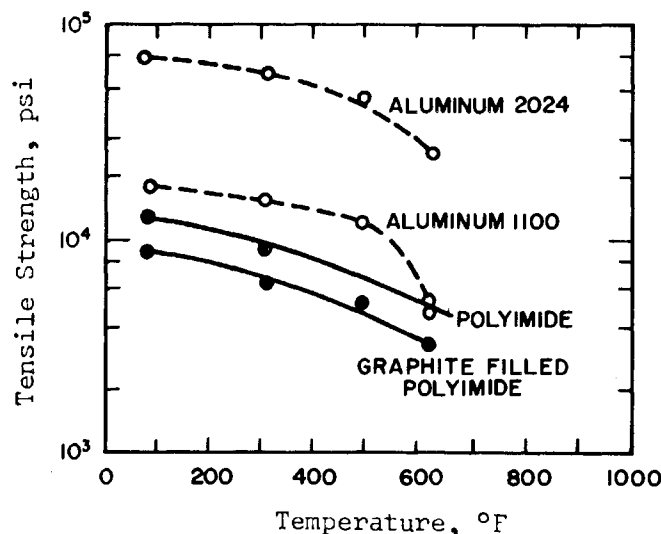
[Ref. 98]

The effect of fillers on the tensile strength of graphite-filled SP-Polymer was determined by Todd and Wolff and reported in the following graph:



[Ref. 98]

Devine and Kroll have also examined unfilled and graphite-filled polyimides with regard to the effect of temperature on the tensile properties:



Tensile Strength as a function of Temperature.
Comparison of Polyimides with Aluminum.

[Ref. 22]

"Pyre-ML" Binder Solutions:

<u>Solid Content</u>	<u>Viscosity</u>	<u>Solvent System</u>	<u>Temp.</u>	<u>Tensile Strength</u>
16.5%	70 poises	NMP/DMAC	25°C	15,000 psi
25%	35 poises	DMF	↓	13,000 psi
25%	8 poises	DMF		10,000 psi
60%	80 poises	NMP/Xylene		7,000 psi

[Ref. 90]

DuPont Insulating Varnish:

<u>Sample</u>	<u>Solid Content</u>	<u>Viscosity</u>	<u>Solvent System</u>	<u>Temp.</u>	<u>Tensile Strength</u>
RC-B-24951	45%	2-5 poises	NMP/Xylene	25°C	7,000 psi
RK-692	13%	6-10 poises	NMP/Aromatic Hydrocarbon	↓	15,000 psi
RC-5060	19%	5-9 poises	NMP/Xylene		13,000 psi

[Ref. 33]

<u>Sample</u>	<u>Thickness</u>	<u>Temp.</u>	<u>Tensile Strength (ASTM D-638)</u>
"Pyre-ML" Enamel Film	10 mil	25°C	20,000 psi
Teflon TFE/"Pyre-ML"	↓	↓	6,000 psi
Teflon FEP/"Pyre-ML"	↓	↓	3,500 psi

[Ref. 66]

DuPont Polyimide Foam:

<u>Density</u>	<u>Thickness</u>	<u>Tensile Strength</u>
2-30 lbs/ft ³	5-100 mils	40 - 400 psi

[Ref. 28]

Polyimide Laminate Evaluation ("Pyre-ML" 692)

<u>Test Temp.</u>	<u>Specimen No.</u>	<u>Width in Inches</u>	<u>Thickness in Inches</u>	<u>Area in Sq. Inch</u>	<u>Load in pounds</u>	<u>psi</u>	<u>Avg. psi</u>
RT	1	.515	.1416	.0729	1515	20,780	
RT	2	.508	.1320	.0671	1640	24,440	
RT	3	.513	.1200	.0616	1785	28,975	
							24,731
500°F	4	.517	.1416	.0732	1430	19,535	
500°F	5	.505	.1307	.0660	1315	19,925	
500°F	6	.514	.1204	.0619	1315	21,245	
							20,235
800°F	7	.519	.1385	.0719	565	7,860	
800°F	8	.504	.1300	.0655	695	10,610	
800°F	9	.509	.1266	.0644	645	10,015	
							9,495

[Ref. 23]

Aromatic Imide I-6 (4,4' - diaminophenyl sulfide and Pyromellitic anhydride) Laminates:

<u>Aging at 600°F</u>	<u>Tensile Strength (25°C)</u>	<u>Tensile Strength (600°F)</u>
As received	26,900 psi	--
0.5 hour	--	22,900 psi
24 hours	--	15,800
72 hours	--	14,900
250 hours	--	12,350
500 hours	12,900 psi	10,200
750 hours	--	10,900
1000 hours	7,450 psi	8,350

[Ref. 45,47]

All values are average of three or more samples tested in accordance with LP-406b, Amendment 1, September 25, 1952: Tensile Method 1011, Type 1. All samples 1/8 inch thick, pressed 30 minutes at 2000 psi at 365°C $\pm$ 10°C and contained 38.2% of resin I-6 on 181-A1100 glass cloth.

DuPont PI-3301 panels (20 end S-994 [HTS]):

<u>Exposure Conditions</u>	<u>Hoop Tensile Strength</u>
room temp., no age	185,000 psi
550°F, 1/2-hour age	171,700 psi

[Ref. 26]

Monsanto Skygard Laminates:

Skygard 700 Laminates (1/8 inch thick, 181 glass cloth with A-1100 pre-finish at 250 psi):

<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Ultimate Tensile Strength</u>
75°F	standard	57,000 psi
570°F	335 hours at 570°F	42,000 psi

[Ref. 73]

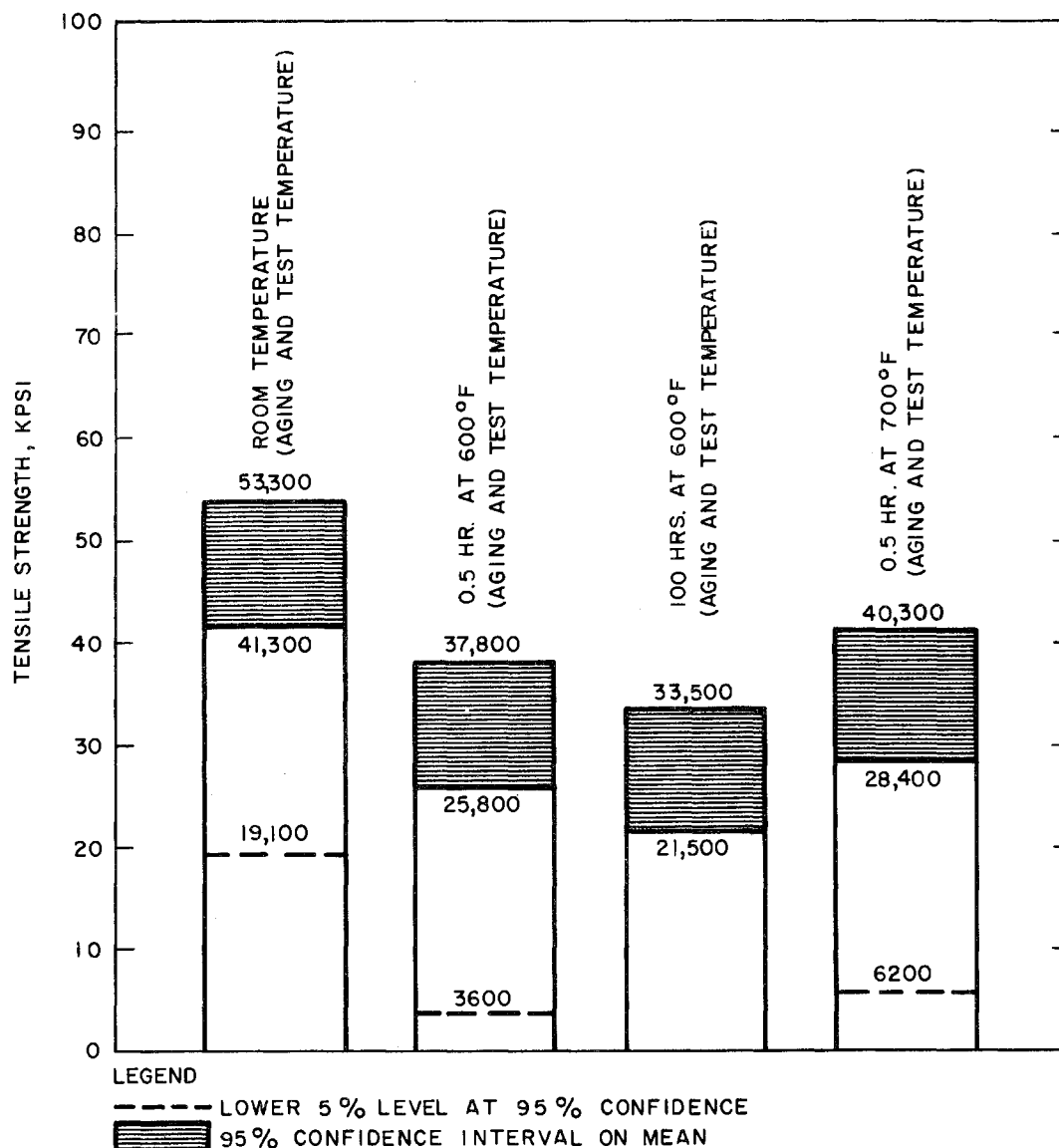
Brunswick Polyimide Glass Fabric Laminates:

Polyimide 181 A-1100 Glass Cloth Laminates:

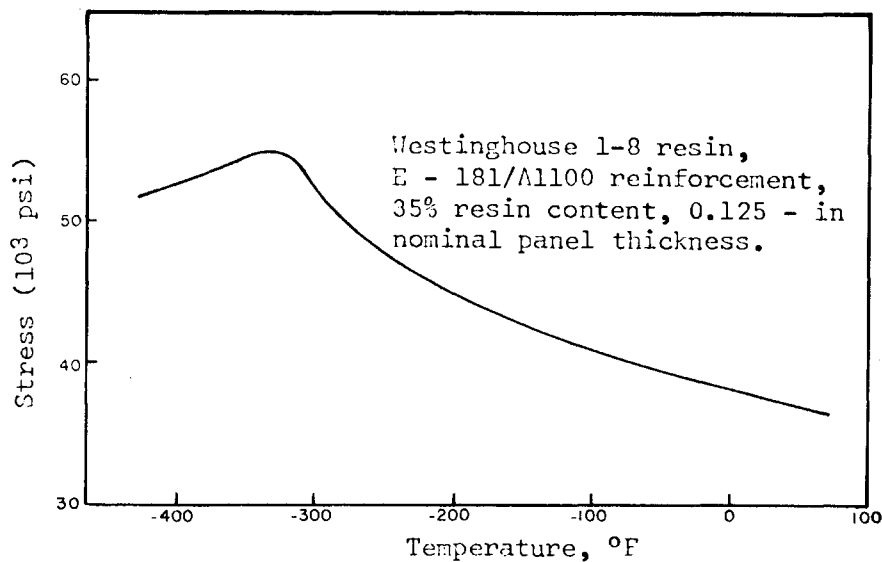
<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Tensile Strength</u>
77°F	1/2 hour soak at 77°F	39,000 psi
-65°F	1/2 hour soak at -65°F	58,000 psi
550°F	1/2 hour soak at 550°F	30,000 psi
700°C	1/2 hour soak at 700°F	30,000 psi
800°C	1/2 hour soak at 800°C	32,000 psi

[Ref. 17]

The Hughes Aircraft Company prepared several polyimide laminates using DuPont PI-2501 binder solution and 181E Fiberglass cloth. The results and statistical analysis of the tensile data are as follows:



Effect of aging temperature and time on the tensile strength of PI-2501 (DuPont) - 181E Fiberglass cloth (All00 Finish). Specimens varied from 1.84 to 2.02 in density, 3 - 23 volume % voids in resin, 18 - 32% resin content. Molding and cure conditions: 500 psi pressure, 5 minutes contact time, 4 hours at 350°F and 4 hours at 440°F; Post cure: 1 hour at 110°F and 3 minutes at 700°F.



Tensile strength of polyimide-fiberglass laminate
as a function of temperature.

[Ref. 69]

Additional tensile strength data will be found in the section on
IRRADIATION EFFECTS ON PROPERTIES.

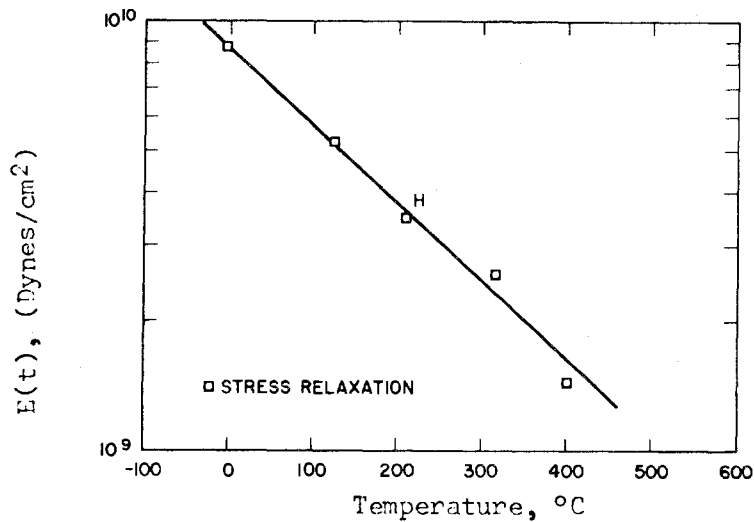
b. Tensile Modulus

"Kapton" Type H-Film:

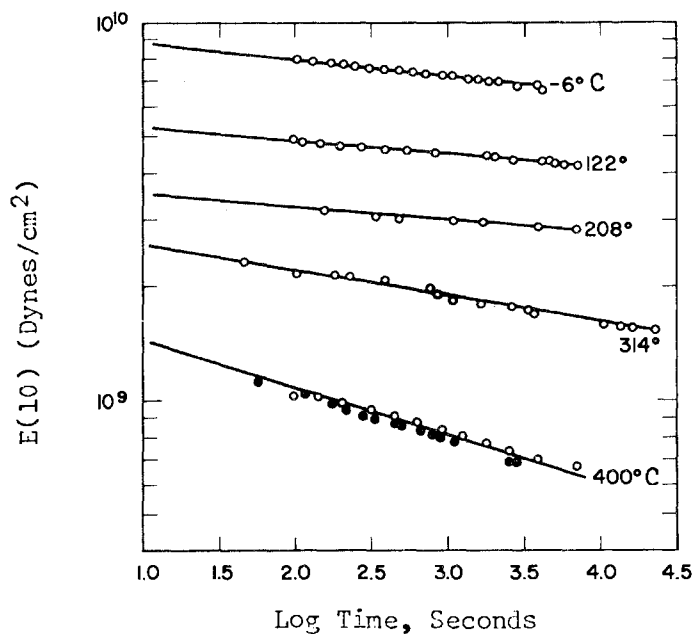
<u>Temp.</u>	<u>Humidity</u>	<u>Elongation Rate</u>	<u>Thickness</u>	<u>Tensile Modulus (ASTM D-882)</u>
25°C	50% RH	100%/minute	1 mil	430,000 psi
↓	100% RH	↓	1 mil	460,000 psi
	50% RH		2 mil	430,000 psi
			5 mil	400,000 psi
-195°C	--	100%/minute	1 mil	510,000 psi
- 20°C	--			480,000 psi
25°C	--	↓	↓	430,000 psi
100°C	--			320,000 psi
200°C	--			260,000 psi
300°C	--	↓	↓	140,000 psi
450°C	--			55,000 psi

[Ref. 31, 58, 98]

Princeton University made measurements of the tensile modulus, E , for H-Film as a function of time, at temperature ranging from -6°C to 400°C , using the stress-relaxation technique. It appears that no sharp transition region exists in this temperature region:



[Ref. 80]

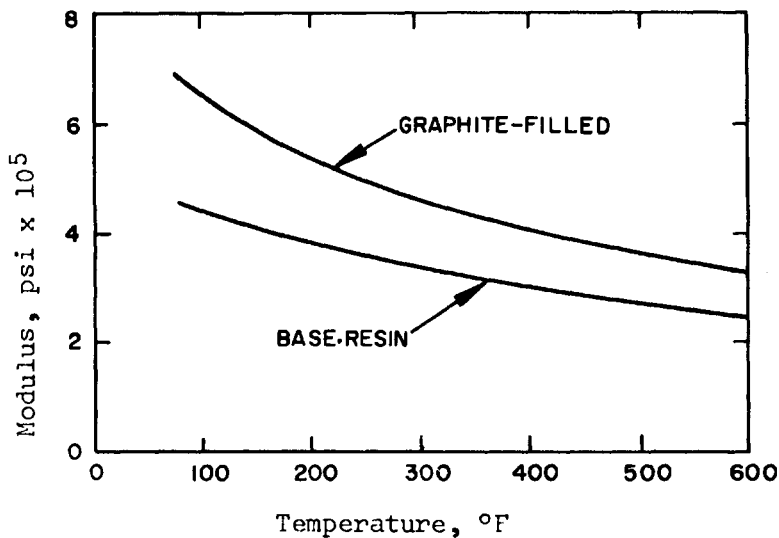


The tensile modulus $E(t)$ for H-Film as a function of time, at different temperatures.

[Ref.80]

SP-Polymer:

The effect of graphite fillers on the modulus of elasticity of SP-Polymer was measured by Todd and Wolff as follows:



Modulus of elasticity for polyimides
(at 73°F) is 450,000 psi;
graphite material has a stiffness 40% higher.

[Ref. 98]

Monsanto Skygard 700 Laminates:

Skygard 700 laminates (1/8" thick, 181 glass-cloth with A-1100 pre-finish at 250 psi):

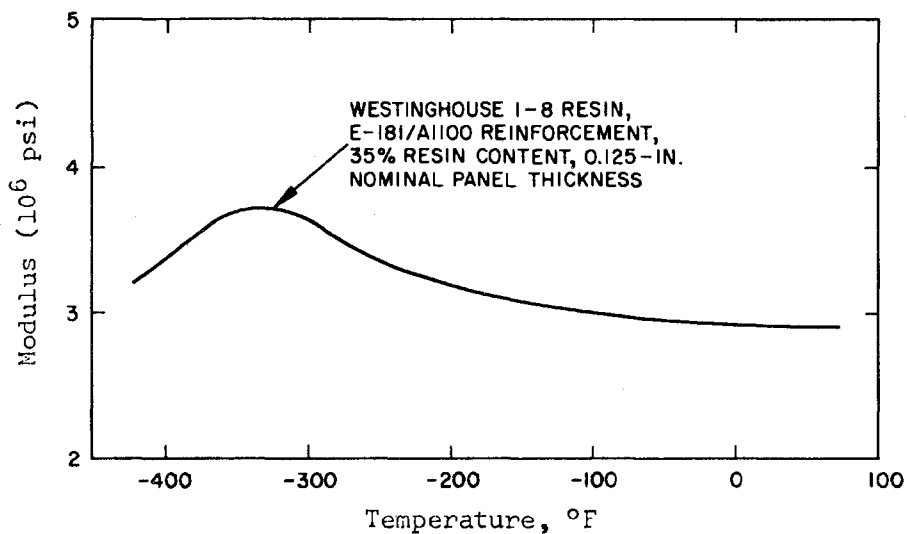
<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Modulus of Elasticity</u>
75°F	standard	3,120,000 psi
570°F	335 hours at 570°F	3,120,000 psi

[Ref. 73]

AI Polymer (Type 10) 181 glass cloth with Volan A finish, 15 ply (1/8 inch) 31% resin content, press schedule of 400°F for 25 minutes at 600 psi plus 5 minutes at 1500 psi.

<u>Temperature</u>	<u>Tensile Modulus</u>
25°C	2.76 x 10 ⁶ psi

[Ref. 4]



Modulus of elasticity of polyimide-fiberglass laminate.

[Ref. 69]

"Kapton" Type HF Composites:

<u>Construction</u>	<u>Temperature</u>	<u>Tensile Modulus</u>
1 mil "Kapton" Type H/ 1/2 mil Teflon FEP (1H/ 1/2F)	25°C	200,000 psi
	200°C	120,000 psi
1/2-mil Teflon FEP/ 1-mil "Kapton" H/ 1/2-mil Teflon FEP (1/2F/ 1H/ 1/2F)	25°C	220,000 psi
	200°C	120,000 psi
1-mil "Kapton" Type H/ 1-mil Teflon FEP (1H/ 1F)	25°C	220,000 psi
	200°C	120,000 psi
1-mil Teflon FEP/ 1-mil "Kapton" H/ 1-mil Teflon FEP (1F/ 1H/ 1F)	25°C	230,000 psi
	200°C	100,000 psi
2-mil "Kapton" Type H/ 2-mil Teflon FEP (2H/ 2F)	25°C	235,000 psi
	200°C	85,000 psi

[Ref. 27]

DuPont Polyimide Foam:

<u>Density</u>	<u>Thickness</u>	<u>Tensile Modulus</u>
2-30 lbs/ft ³	5-100 mils	1,000 - 40,000 psi

[Ref. 28]

c. Yield Point

"Kapton" Type H-Film:

<u>Temperature</u>	<u>Thickness</u>	<u>Elongation Rate</u>	<u>Yield Point Stress</u>
- 20°C	1 mil	100%/minute	16,000 psi
25°C			14,000 psi
100°C			12,000 psi
200°C			9,000 psi
300°C			5,000 psi
450°C			2,000 psi

[Ref. 31]

<u>Temperature</u>	<u>Testing Direction</u>	<u>Yield Point Stress (ASTM-D882-61T)</u>
25°C	Machine Direction	10,000 psi at 3%
200°C	Machine Direction	6,000 psi at 3%

[Ref. 27]

"Kapton" Type HF Composite:

<u>Construction</u>	<u>Temperature</u>	<u>Yield Point Stress</u>
1-mil "Kapton" Type H/ 1/2-mil Teflon FEP (1H/ 1/2F)	25°C	9,000 psi
	200°C	6,000 psi
1-mil "Kapton" Type H/ 1-mil Teflon FEP (1H/ 1F)	25°C	8,000 psi
	200°C	5,000 psi
1/2-mil Teflon FEP/ 1-mil "Kapton" H/ 1/2-mil Teflon FEP (1/2F/ 1H/ 1/2F)	25°C	8,000 psi
	200°C	5,000 psi
2-mil "Kapton" Type H/ 2-mil Teflon FEP (2H/ 2F)	25°C	14,000 psi
	200°C	4,000 psi

[Ref. 27]

d. Elongation

"Kapton" Type H-Film:

<u>Temp.</u>	<u>Thickness</u>	<u>Humidity</u>	<u>Time (in air)</u>	<u>Elongation Rate</u>	<u>Elongation (ASTM D-882)</u>
25°C	1 mil	50% RH	control	100%/minute	70%
			1 week		50%
			2 weeks		30%
			7 weeks		20%
			14 weeks		19%
			-----	1,000%/minute	70%
			-----	10,000%/minute	65%
			-----	100,000%/minute	50%
			-----	500,000%/minute	25%
	2 mil 3 mil 5 mil	100% RH	-----	100%/minute	70%
			-----		80%
			-----		85%
			-----		95%
			-----		70%
			-----		80%
			-----		85%
			-----		95%

[Ref. 31]

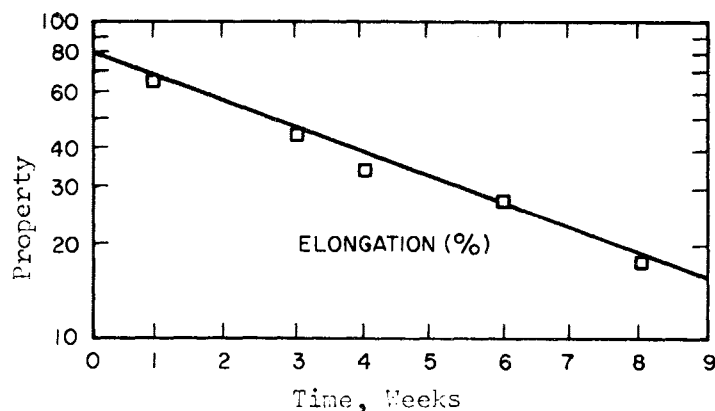
<u>Test Temp.</u>	<u>Thickness</u>	<u>Aging Temp.</u>	<u>Aging Time</u>	<u>Elongation Rate</u>	<u>Elongation (ASTM D-882)</u>	
					<u>in air</u>	<u>in helium</u>
25°C	1 mil	250°C	control	100%/minute	70%	
			1 month		61%	
			2 months		48%	
			3 months		30%	
			8 years		1%	

		300°C	control		70%	70%
			1 week		60%	60%
			3 weeks		40%	43%
			6 weeks		20%	25%

		400°C	control		70%	
			1 hour		55%	
			2 hours		40%	
			4 hours		22%	
			6 hours		14%	
			12 hours		1%	

[Ref. 31]

Heacock and Berr of the E.I. du Pont de Nemours Company report the following thermal aging at 300°C data on "Kapton" Type H-Film:

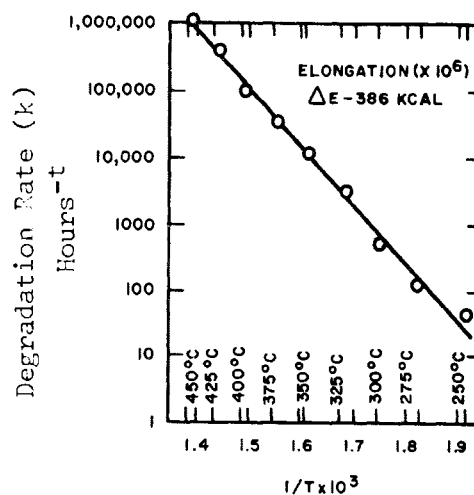


Effect of aging in air at 300°C on the physical properties of H-Film.

[Ref. 59]

From these above data and other temperature levels, an Arrhenius plot was constructed based on the equation: $\text{Log } P = -kt + \text{Log } P_0$

Arrhenius plot of physical property deterioration. A linear relationship is obtained.



[Ref. 59]

where P is the tensile property at time t hours and P_0 is the initial property value, and k is the rate constant in reciprocal hours.

From these data, the authors obtained for the tensile parameter, the following activation energies, initial property values, and rate constants at 400°C:

	In Air	In Helium
Activation Energy (ΔE)	39.6 ± 0.9	55.5 ± 1.7 Kcals
Initial Property (P_0)	70%	70%
Rate Constant (k_{400})	0.15	0.0050 /1 hour

"Kapton" Type H-Film:

<u>Temp.</u>	<u>Thickness</u>	<u>% Stretched</u>	<u>% Recovery after 1 Minute</u>	
			<u>Machine Direction</u>	<u>Transverse Direction</u>
23°C ↓	1 mil	20	67%	71%
	↓ 2 mil	33	45%	39%
		50	31%	34%
		33	39%	45%
		33	45%	51%
	5 mil			

[Ref. 31]

<u>Temp.</u>	<u>Thickness</u>	<u>Elongation Rate</u>	<u>Elongation (ASTM D-882)</u>
-195°C	1 mil ↓	100%/minute ↓	2%
- 20°C			65%
25°C			70%
100°C			87%
200°C			105%
300°C			120%
450°C			82%

[Ref. 31, 58, 94]

<u>Temp.</u>	<u>Thickness</u>	<u>Elongation Rate</u>	<u>Initial Elongation</u>	<u>Predicted time to reach 1% elongation</u>	
				<u>in air</u>	<u>in helium</u>
450°C	1 mil ↓	100%/minute ↓	70% ↓	2 hours	22 hours
425°C				5 hours	3.5 days
400°C				12 hours	2 weeks
375°C				2 days	2 months
350°C				6 days	1 year

[Ref. 31]

<u>Varnish Coating</u>	<u>Aging Temp.</u>	<u>Thickness</u>	<u>Elongation at 25°C</u>	
			<u>Original</u>	<u>After 1 Year Aging</u>
control	200°C ↓	1 mil ↓	75%	62%
DC 5651 Silicone			90%	24%
DC 997 Silicone			75%	36%
DC 1402 Mod. Silicone			14%	2%
Doryl Polyphenyl ether			35%	4%
"Pyre-ML" polyimide			45%	38%
Epon 828 epoxy			20%	pinholing in 3 months

[Ref. 31]

"Kapton" Type HF Composites:

<u>Construction</u>	<u>Temperature</u>	<u>Elongation</u>
1-mil "Kapton" Type H/ 1/2-mil Teflon FEP (1H/ 1/2F)	25°C 200°C	75% 75%
1/2-mil Teflon FEP/ 1-mil "Kapton" H/ 1/2-mil Teflon FEP (1/2F/ 1H/ 1/2F)	25°C 200°C	90% 90%
1-mil "Kapton" Type H/ 1-mil Teflon FEP (1H/ 1F)	25°C	70%
1-mil Teflon FEP/ 1-mil "Kapton" H/ 1-mil Teflon FEP (1F/ 1H/ 1F)	25°C	80%
2-mil "Kapton" Type H/2-mil Teflon FEP (2H/ 2F)	25°C 200°C	110% 110%
[Ref. 27]		

SP-1 Polymer:

<u>Temperature</u>	<u>Elongation (ASTM D-708)</u>
77°F	6 - 3%

[Ref. 19, 97]

Todd and Wolff (Ref. 98) report that the Polymer SP base resin has an ultimate elongation of about 4 - 5% at room temperature. Elongation does not go any higher even at temperatures as high as 480°F. The graphite-filled SP-Polymer has a slightly lower ultimate elongation 3 - 4% at room temperature, and, as the temperature increases, elongation drops to 3% at 300°F and 2% at 580°F.

"Pyre-ML" Binding Solutions:

<u>Solid Content</u>	<u>Viscosity</u>	<u>Solvent System</u>	<u>Temp.</u>	<u>Elongation</u>
16.5%	70 poises	NMP/DMAC	25°C	20%
25%	35 poises	DMF	↓	11%
25%	8 poises	DMF		7%
60%	80 poises	NMP/Xylene		60%

[Ref. 90]

<u>Sample</u>	<u>Thickness</u>	<u>Temp.</u>	<u>Elongation (ASTM D-708)</u>
"Pyre-ML" Enamel Film	10 mil	23°C	100%
Teflon TFE/"Pyre-ML"	↓	↓	200%
Teflon FEP/"Pyre-ML"			150%
[Ref. 66]			

DuPont Insulating Varnish:

<u>Sample</u>	<u>Solid Content</u>	<u>Viscosity</u>	<u>Solvent System</u>	<u>Temp.</u>	<u>Elongation</u>
RC-24951	45%	2-5 poises	NMP/Xylene	25°C	3%
RK-692	13%	6-10 poises	NMP/Aromatic Hydrocarbon	↓	10 - 25%
RC-5060	19%	5-9 poises	NMP/Xylene	↓	10%
[Ref. 33]					

Monsanto Skygard 700 Laminates:

Skygard 700 Laminates (1/8" thick, 181 glass-cloth with A-1100 prefinish at 250 psi:

<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Elongation</u>
77°F	standard	1.90%
75°F	335 hours aging at 570°F	1.40%
[Ref. 73]		

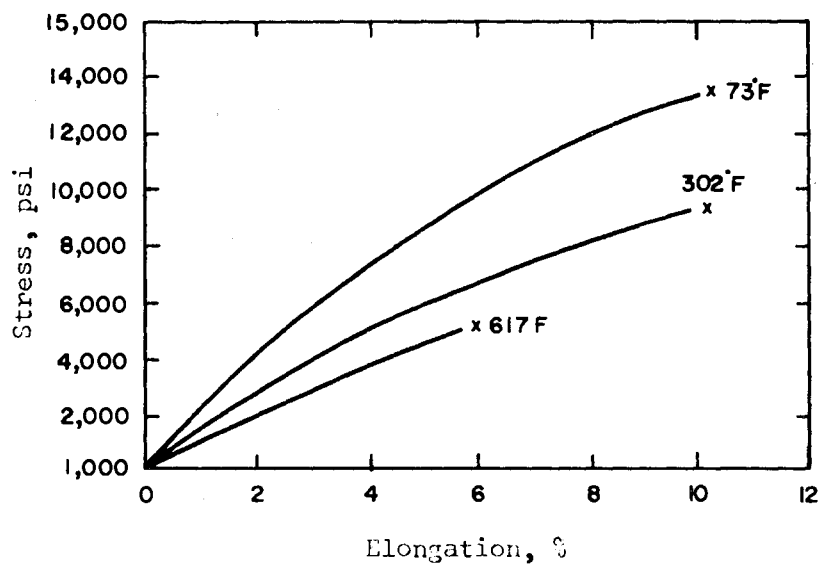
DuPont Polyimide Foam:

<u>Density</u>	<u>Thickness</u>	<u>Elongation</u>
2-30 lbs/ft ³	5-100 mils	2 - 10%
[Ref. 28]		

Additional elongation data will be found in the section on IRRADIATION EFFECTS ON PROPERTIES.

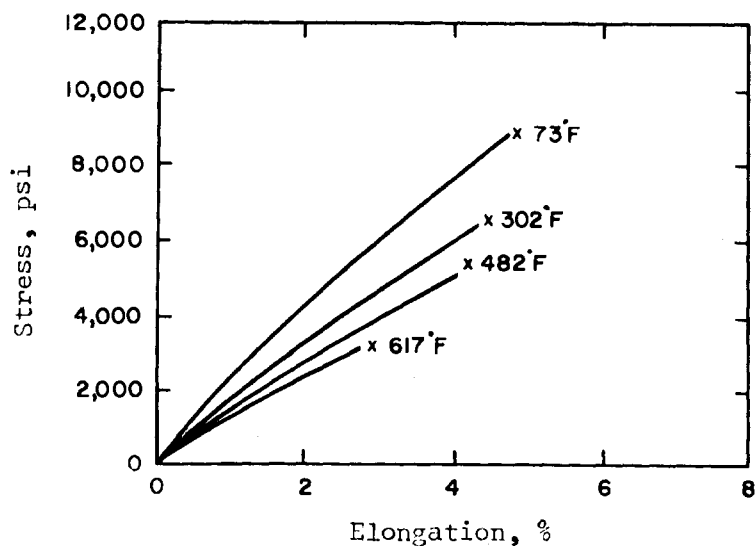
e. Stress-Strain Diagram

Devine and Kroll (Ref. 22) have examined the stress-strain behavior of un-filled polyimides over a wide range of temperatures:



Polyimide stress-strain curves based on original cross-section using a strain rate of 0.2 in/min.

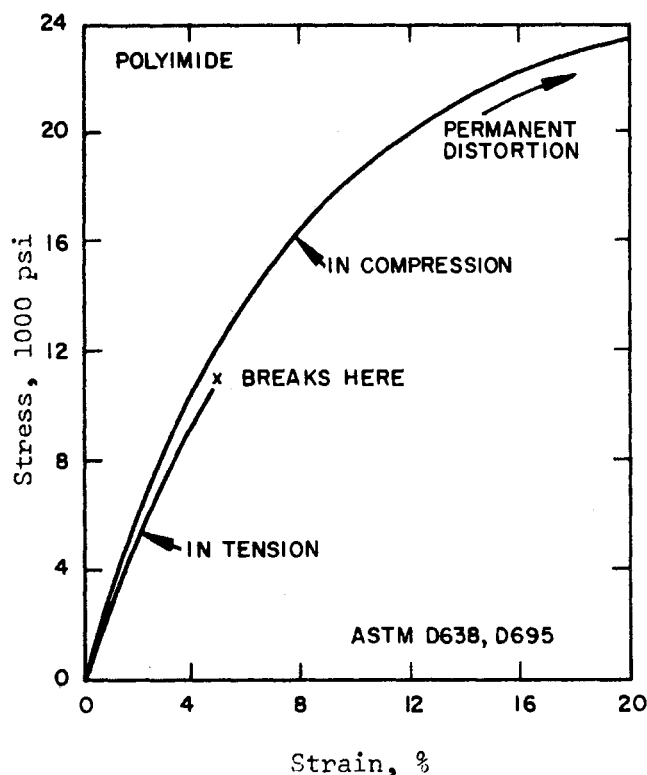
[Ref. 22]



Polyimide (graphite-filled) stress-strain curves based on original cross-section using a strain rate of 0.2 in/min.

[Ref. 22]

SP-Polymer:

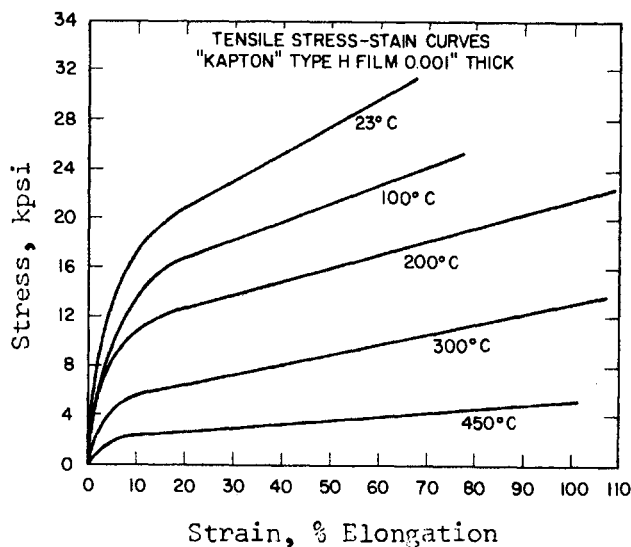


In tension, ultimate elongation of polyimides (at 73°F) is about 4 - 5%. In compression, the material is permanently distorted but does not break until at least 20% strain is reached.

[Ref. 98]

Up to 1% strain, the stress-strain behavior of polyimide SP Type can be described as Hookean, that is, stress and strain follow a nearly linear relationship. However, Hooke's Law does not apply above a 1% strain.

The DuPont Company has measured the stress-strain characteristics of their "Kapton" Type H-Film from 23°C - 450°C as shown in the following graph:



[Ref. 31]

2. Compressive Properties

a. Compressive Strength

SP-Polymer:

<u>Temperature</u>	<u>Compressive Strength</u>
77°F	24,000 psi

[Ref. 11]

SP-1 Polymer:

<u>Temperature</u>	<u>Compressive Strength (ASTM D-695)</u>
73°F	24,400 psi

[Ref. 97]

Under sufficiently high compressive stresses, Todd and Wolff (Ref. 98) report polyimides are permanently distorted, but it does not break until at least 20% strain is reached.

DuPont PI-3301 Filament Wound Panels (16.2% resin content, sp. gr. = 1.92, 20 end S-994 (HTS), bidirectionally (90°) wound according to Brunswick's BTP-600b):

<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Compressive Strength</u>
77°F		50,100 psi
250°F	1/2 hour	29,900 psi
550°F	1/2 hour	22,800 psi
77°F	1/2 hour at 700°F	52,500 psi
77°F	16 hours at 700°F	23,500 psi

[Ref. 26]

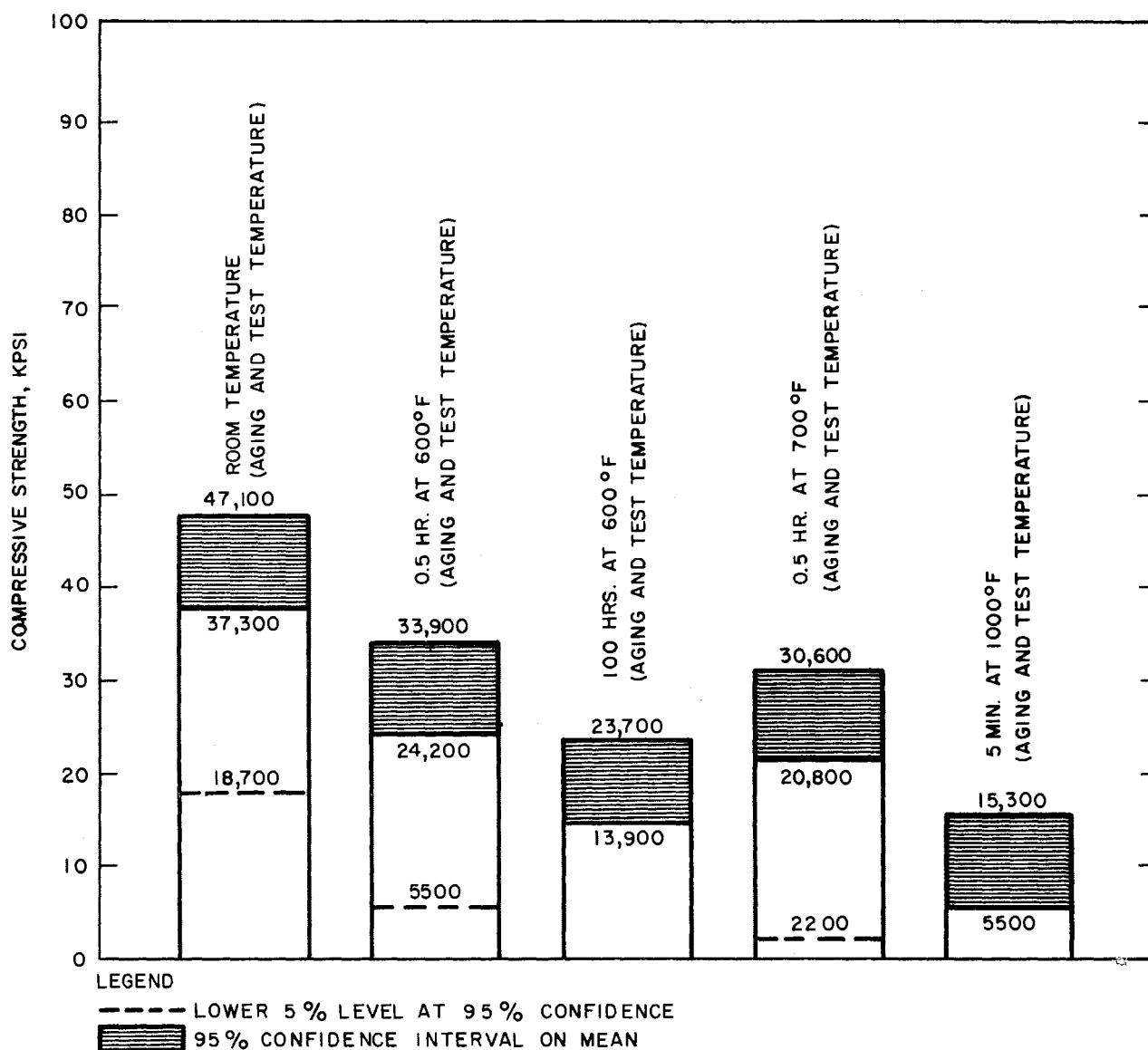
Brunswick polyimide glass fabric laminates:

Polyimide 181 A-1100 glass cloth laminates

<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Compressive Strength</u>
77°F	1/2 hour soak at 77°F	38,000 psi
-65°F	1/2 hour soak at -65°F	45,000 psi
550°F	1/2 hour soak at 550°F	23,000 psi
700°F	1/2 hour soak at 700°F	17,000 psi
800°F	1/2 hour soak at 800°F	16,000 psi

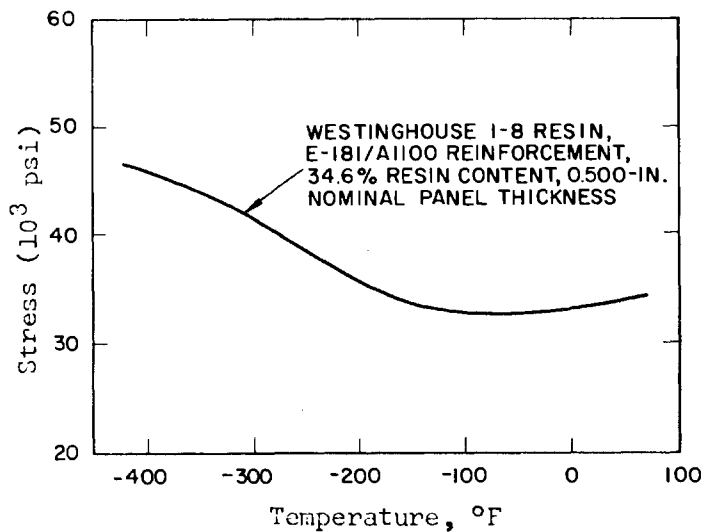
[Ref. 17]

The Hughes Aircraft Company prepared a number of laminates with DuPont PI-2501 binder solution. The compressive strength data were analyzed statistically and plotted as follows:



Effect of aging temperature and time on the edgewise compressive strength of PI-2501 (DuPont) - 181E Fiberglass cloth (All100 Finish). Specimens varied from 1.84 to 2.02 in density, 3-23 volume % voids in resin, 18-32% resin content. Molding and cure conditions: 500 psi pressure, 5 minutes contact time, 4 hours at 350°F and 4 hours at 440°F; post cure: 1 hour at 110°F and 3 minutes at 700°F.

The Martin Company tested a Westinghouse Electric Corporation I-8 Polyimide Resin reinforced with fiberglass at cryogenic temperatures:



Compressive strength of polyimide-fiberglass laminate.

[Ref. 69]

Freeman et al tested an Aromatic Imide I-6 Polyimide laminate for compressive strength at room temperature and 600°F after having been aged in air at 600°F for various time periods:

<u>Aging Time</u>	<u>Aging Temperature</u>	<u>Compressive Strength (room temperature)</u>	<u>Compressive Strength (600°F)</u>
As received	0	8,300 psi	-----
0.5 hour	600°F ↓	-----	5,900 psi
24 hours		-----	5,150 psi
72 hours		-----	6,650 psi
250 hours		-----	5,750 psi
500 hours		6,900 psi	6,250 psi
750 hours		-----	7,120 psi
1000 hours		6,350 psi	5,050 psi

[Ref. 45, 47]

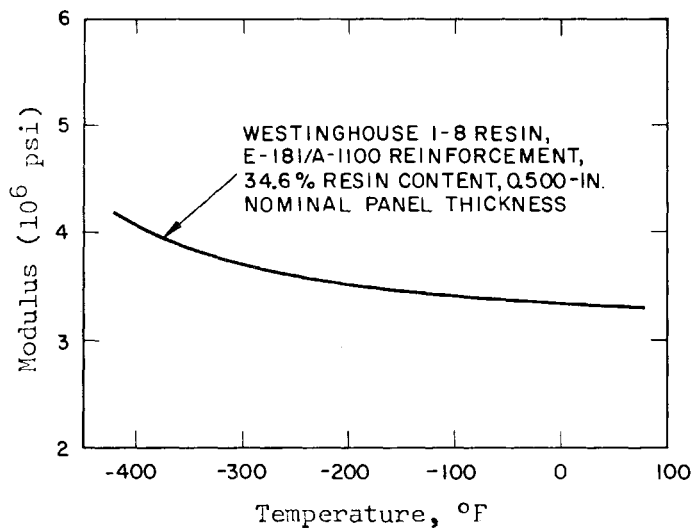
DuPont Polyimide Foam:

<u>Density</u>	<u>Thickness</u>	<u>Compressive Strength</u>
2-30 lbs/ft ³	5-100 mils	5 - 60 psi

[Ref. 28]

b. Compressive Modulus

The Martin Company tested a Westinghouse Electric Corporation I-8 Polyimide fiberglass laminate at cryogenic temperatures and found the compressive modulus to vary as follows:



Compressive modulus of polyimide-fiberglass laminate.

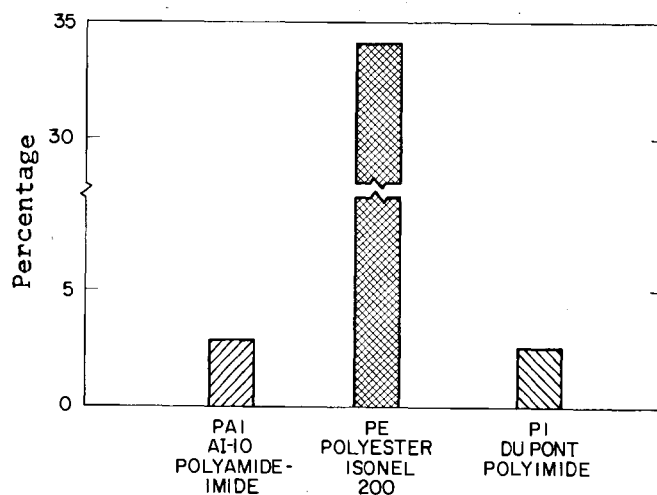
[Ref. 69]

DuPont Polyimide Foam:

Density	Thickness	Compressive Modulus
2-30 lbs/ft ³	5-100 mils	50 - 1,500 psi*

* compression at yield modulus

[Ref. 28]



Film compression at 250°C
(5 Kg load)

[Ref. 104]

3. Shear Properties

a. Shear Strength

SP-1 Polymer:

<u>Temperature</u>	<u>Shear Strength (ASTM D-732)</u>	
73°F	11,900 psi	[Ref. 97]

Polyimide Binder Solutions:

<u>Sample</u>	<u>Temperature</u>	<u>Aging Condition</u>	<u>Shear Strength</u>
Unformulated polyimide adhesive* (straight polyimide)	77°F	---	2,500 psi
	550°F	1 hour	1,800 psi
	550°F	100 hours	2,200 psi
	550°F	200 hours	1,800 psi
	700°F	1 hour	1,400 psi
	700°F	10 hours	1,300 psi
	700°F	24 hours	1,300 psi
	700°F	60 hours	0 psi
Formulated polyimide adhesive* (contains an antioxi- dant & various fillers)	77°F	---	2,200 psi
	550°F	1 hour	1,400 psi
	550°F	100 hours	1,900 psi
	550°F	200 hours	1,400 psi
	700°F	1 hour	600 psi
	700°F	10 hours	1,100 psi
	700°F	24 hours	1,200 psi
	700°F	60 hours	1,100 psi

* Substrate tested was 17-7PH stainless steel, phosphate etched, bonds were formed at 575°F under 200 psi pressure for 2 hours.

[Ref. 90]

The DuPont Company reports Polyimide Binder Solution PI-1101 data values similar to Schweitzer's and supplemented them with longer aging times and other media:

<u>Test Temperature</u>	<u>Aging Conditions</u>	<u>Tensile Shear Strength (psi)</u>	
		<u>PI-1101** Unformulated</u>	<u>PI-1101** Formulated</u>
77°F	unaged	2500	2800
550°F	1 hour at 550°F	1800	1600
	100 hours at 550°F	1800	1900
	200 hours at 550°F	1900	1400
	500 hours at 550°F	1600	1300

[Ref.30]

Table Continued

Tensile Shear Strength (psi)

<u>Test Temperature</u>	<u>Aging Conditions</u>	<u>PI-1101** Unformulated</u>	<u>PI-1101** Formulated</u>
550°F	1000 hours at 550°F	1400	1400
700°F	1 hour at 700°F	1400	1300
↓	10 hours at 700°F	1300	1100
	24 hours at 700°F	1300	1200
	60 hours at 700°F	0	1100
77°F	30 days in 77°F water	2200	2100
↓	30 days in 110°F at 100% RH	2300	1900
	30 days in 95°F, 5% salt spray	2200	2000
	7 days in JP-4 jet fuel	2300	2300
	7 days in hydraulic oil	2800	3600

*** Substrate: 17-7 PH S.S. Heat hardened to condition TH-1050 and phosphate etched. Bond Forming Conditions: 2 hours x 575°F x 200 psi.

[Ref. 30]

DuPont PI-3301 [16% resin content, sp. grav. = 1.8, 20 end S-994 (HTS)]:

<u>Temperature</u>	<u>Aging Conditions</u>	<u>Test Temperature</u>	<u>Horizontal Shear Strength</u>
R.T.		R.T.	4,600 psi
250°F	1/2 hour	↓	3,700 psi
550°F	1/2 hour		1,900 psi
700°F	1/6 hour		980 psi
550°F	1/2 hour	550°F	1,990 psi
↓	50 hours	↓	3,390 psi
	100 hours		2,310 psi
	200 hours		2,030 psi
	500 hours		1,910 psi
R.T.		700°F	3,090 psi
700°F	10 minutes	↓	980 psi
700°F	16 hours		920 psi

[Ref. 26]

DuPont PI-3301 Filament Wound Panels [resin content 16.2%, sp. grav. 1.92, 20 end S-994 (HTS), 90° bidirectionally wound]:

<u>Test Temperature</u>	<u>Exposure</u>	<u>Shear Strength</u>
77°F		4,020 psi
250°F	1/2 hour	3,370 psi
250°F	1/2 hour	1,790 psi
77°F	1/2 hour at 700°F	2,800 psi
77°F	16 hours at 700°F	2,385 psi

[Ref. 31]

Ruffing and Traynor bonded stainless steel specimens with I-8 polyimide and tested the lap-joints after various periods of aging at elevated temperature:

<u>Specimen</u>	<u>Test Temp.</u>	<u>Aging Conditions</u>	<u>Specimen No.</u>	<u>Tensile Shear Strength (ASTM D-1002)</u>
1/2" overlap		none	1	2,700 psi
	23°C		2	2,500 psi
			3	2,750 psi
			avg	2,680 psi
	260°C	192 hours at 260°C	1	800 psi
			2	900 psi
			3	1,250 psi
			4	1,100 psi
			avg	1,010 psi
1" overlap	135°C	1 hour at 315°C	1	908 psi
			2	1,375 psi
			avg	1,140 psi

[Ref. 84]

The Narmco Research and Development Division of Whittaker Corporation evaluated an experimental polyimide adhesive bonding 17-7PH stainless steel coupons and reported the following shear strength data:

Adhesive

Resin: Poly (m-phenylene-3,3' 4,4'-benzophenonetetracarboxylimide)
 Filler: 100 phr MD 105 aluminum powder plus AsAsS₄ (4/1 ratio)
 Carrier: 112 heat-cleaned E-glass
 Cure: 30 minutes at 500°F, 1 hour at 600°F under 500 psi
 Postcure: 1 hour at 750°F in N₂ as indicated

Substrate

Metal: PH 17-7 stainless steel
 Surface Preparation: Conventional phosphate etch

[Ref. 76]

Test Temperature	Aging Conditions	Avg. Tensile Shear Strength (psi)	
		No Postcure	Postcure
R.T.		1835	1805
600°F	10 minutes at 600°F	718	1492
800°F	10 minutes at 800°F	628	569
1000°F	10 minutes at 1000°F	490	528

[Ref. 76]

The Bloomingdale Department of American Cyanamid Company has developed a high temperature polyimide experimental adhesive, FXM-34-B-25 (carrier: 112 glass cloth with Volan "A" finish, 0.13 - 0.14 lbs/sq. ft. film weight, 10 - 12% volatiles after 60 minutes at 500°F), and give the following typical tensile shear values for 1/2" overlap specimens:

<u>Substrate</u>	<u>Cleaning Process</u>	<u>R.T. Shear</u>	<u>500°F Shear</u>
Type 301-1/2 hard stainless	prebond 700	3,300 psi	1,450 psi
Titanium (6Al-4V) alloy	phosphate-fluoride etch	3,100 psi	1,550 psi

Douglas Aircraft Company evaluated a DuPont polyimide solution W-97136 as an adhesive and reported the following shear strength data:

<u>Cure Cure Temp.</u>	<u>psi at R.T.</u>	<u>Avg. psi at R.T.</u>	<u>psi at 250°F</u>	<u>Avg. psi at 250°F</u>	<u>psi at 500°F</u>	<u>Avg. psi at 500°F</u>
350°F	1,470 1,040 1,700 1,320	1,382				
400°F	2,340 2,140 2,550 2,710	2,435				
450°F	2,970 2,480 2,930 2,560	2,735				
500°F			2,160 2,110 1,650 1,750	1,800		
500°F					1,380 1,560 1,160 1,940	1,510

Predry temperature - -175°F for 2 hours on all specimens

Pressure during cure - 200 psi for all specimens

Time at cure temperature - one hour for all specimens

Soak time at temperature - 10 minutes

[Ref. 23]

The Materials Technology Laboratory of the Hughes Aircraft Company (Culver City) also evaluated this same experimental DuPont and the preceding Bloomingdale polyimide adhesive tape along with other polyimide adhesives on stainless steel substrates. Various formulations with appropriate antioxidants, inert high temperature fillers, surfactants and other ingredients were explored to optimize the adhesives' high temperature resistance:

<u>Adhesive System</u>	<u>Test Temperature</u>	<u>Aging Temperature</u>	<u>Aging Time & Medium</u>	<u>Lap Shear Strength (per MIL-A-5090E)</u>
DuPont W-97136 (1)	77°F	77°F	none in air	1000 lbs
	600°F	↓	↓	400 lbs
DuPont W-97136 + 325 mesh alumina	77°F			1575 lbs
	600°F			650 lbs
Monsanto Skygard 700 (2)	77°F			650 lbs
	600°F			150 lbs
Monsanto Skygard 700 + 325 mesh alumina	77°F			1100 lbs
	600°F			150 lbs
Bloomingtondale FXM-34B-18 (3)	77°F	77°F		2500 lbs
	600°F	77°F		1150 lbs
	77°F	600°F	200 hours in air	260 lbs
	77°F	600°F	400 hours in air	0
	77°F	MIL-E-5272 cycles	30 days humidity	1315 lbs
	600°F	MIL-E-5272 cycles	30 days humidity	970 lbs
Bloomingtondale FXM-34B-25 (4)	77°F	77°F	none in air	2550 lbs
	600°F	77°F	none in air	1070 lbs
	77°F	600°F	200 hours in air	1260 lbs
	77°F	600°F	400 hours in air	660 lbs
	77°F	MIL-E-5272 cycles	30 days humidity	480 lbs
	600°F	MIL-E-5272 cycles	30 days humidity	490 lbs

(1) Cure conditions: pre-cured 2 hours at 220°F; final cure 700° ± 10°F for 3 minutes, 200 psi.

(2) Cure conditions: pre-cured 1.5 hours at 250°F; final cure 600°F for 30 minutes, 200 psi plus 3 hours at 450°F and 250 psi.

(3) Cure conditions: cold press to 500°F (in 1 hour) plus 500°F for 2 hours, 50 psi.

(4) Cure conditions: 500°F for 3 hours, 50 psi.

4. Flexural Properties

a. Flexural Strength

SP-Polymer:

<u>Temperature</u>	<u>Flexural Strength</u>
77°F	14,700 psi
509°F	8,000 psi

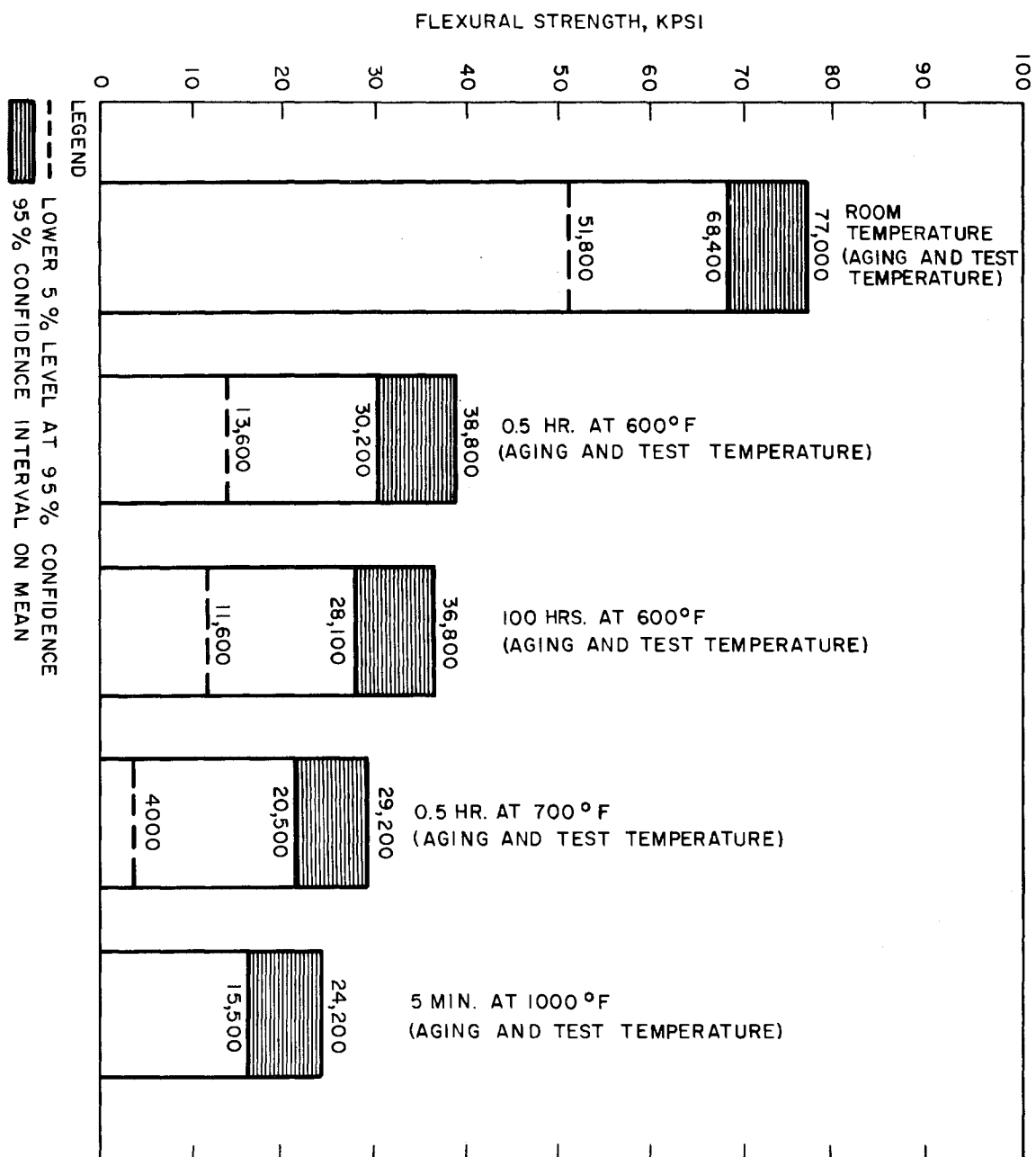
[Ref. 11]

Polyimide Resin C:

<u>Temperature</u>	<u>Aging Conditions</u>	<u>Flexural Strength</u>
77°F	---	42,000 psi
250°F	---	40,000 psi
550°F	1 hour	35,000 psi
	200 hours	33,000 psi
	600 hours	28,000 psi
	1000 hours	24,000 psi

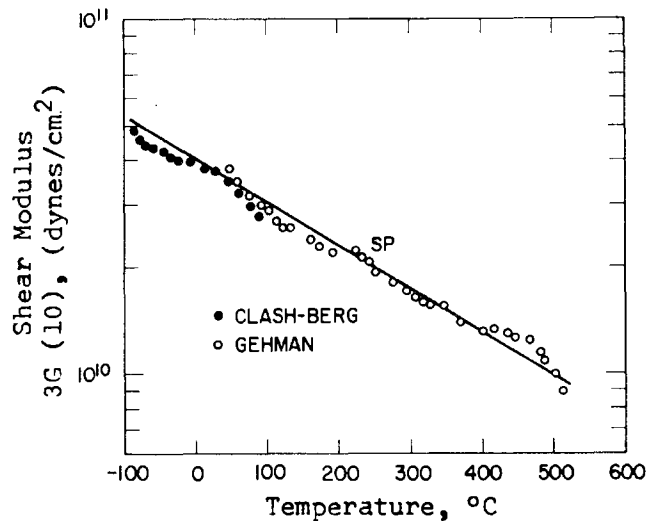
[Ref. 90]

The Hughes Aircraft Company prepared a number of polyimide laminates with DuPont PI-2501 binder solution. The laminates were prepared with 181E Fiberglass cloth with the AII00 finish. The molding and cure conditions were varied slightly to obtain specimens with optimum density and minimum voids. The resulting specimens varied in density from 1.84 to 2.02, 18 to 32% resin content. 3 to 23 volume percent voids. Molding and cure conditions were as follows: 500 psi pressure with a 5-minute contact time, 4 hours at 350°F and 4 hours at 440°F. Post cure was 1 hour at 110°F and 3 minutes at 700°F. The aging conditions on the finished laminates varied from 0.5 hour at 600°F to 5 minutes at 1000°F. The resulting flexural strength data were analyzed statistically and graphically presented in the following bar graph:



b. Shear Modulus

Princeton University made measurements on the ten-second modulus ($3G$) of SP polyimide polymer over the temperature range of -95°C up to 510°C . It was concluded that viscoelastic measurements show that the elastic modulus of the polyimide studied changes but little over a very wide temperature range, and that no sharp molecular transition takes place within this region:

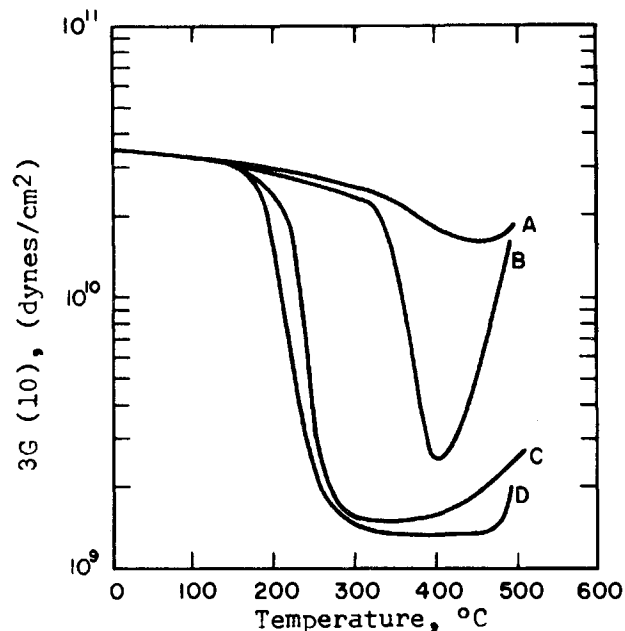


[Ref. 80]

Cooper and co-workers at the Frick Chemical Laboratories, Princeton University determined subsequently the shear modulus (or $3G(10)$ which is directly comparable to the tensile modulus $E(10)$ via the relationship: $E = 2(\nu + 1)G$, where ν = Poisson's ratio) versus temperature of four different polyimide films:

- A = DuPont H-Film
- B = Westinghouse AI-8
- C = Westinghouse I-6
- D = Westinghouse I-7

[Ref. 114]



The data were obtained on modified Gehman torsional instrument.

POLYIMIDE LAMINATE DATA

Resin	Aging Conditions	Flexural Strength, psi x 10 ⁻³				
		PI-2101	P-2101	PI-2201	PI-2501	
Glass Fabric		181-E-A-1100	181-E-112	181-E-A-1100	181-E-112	181-E-A-1100
Test Temp.	Aging Conditions					
77°F		43.5	33.8	50.0	33.1	57.1
	2 hour water boil	42.1	15.0	46.5	14.9	--
500°F	1/2 hour at 550°F	33.0	28.0	37.9	29.2	22.7
	100 hours	31.5	25.6	23.2	24.4	30.1
	200	27.9	24.5	19.1	24.5	27.5
	500	21.5	27.0	15.3	26.4	29.9
	1000	16.6	22.2	10.4	20.5	24.2
	1500	12.2	--	--	--	24.2
	2000	13.8	--	--	--	22.0
	3000	4.6	--	--	--	22.0
600°F	1/2 hour at 600°F	26.0	--	24.3	21.2	10.9
	100 hours	28.7	--	--	--	18.5
	200	23.7	--	--	--	26.6
	500	13.7	--	--	--	22.5
	1000	2.9	--	--	--	19.1
700°F	1/2 hour at 700°F	15.6	15.4	27.5	21.1	12.4
	16 hours	18.6	--	--	--	21.7
	34	13.0	--	--	--	24.1
	64	5.0	--	--	--	19.0
800°F	1/2 hour at 800°F	15.6	--	25.7	20.0	--
900°F	1 hour	--	--	--	--	11.9

[Ref. 30]

The DuPont Company has evaluated a number of their binder solutions reinforced with different fiberglass compositions under a wide range of aging temperatures and times:

DuPont PI-3301 Filament Wound Panels* (bidirectionally 90°)

<u>Temperature</u>	<u>Flexural Strength</u>
77°F	90,500 psi
250°F	75,600 psi
550°F	33,100 psi
700°F	31,400 psi

* resin content 17.5%, sp. gr. = 1.84, 20 end S-994 (HTS)

[Ref. 26]

Polyimide laminate evaluation ("Pyre-IL" 692):

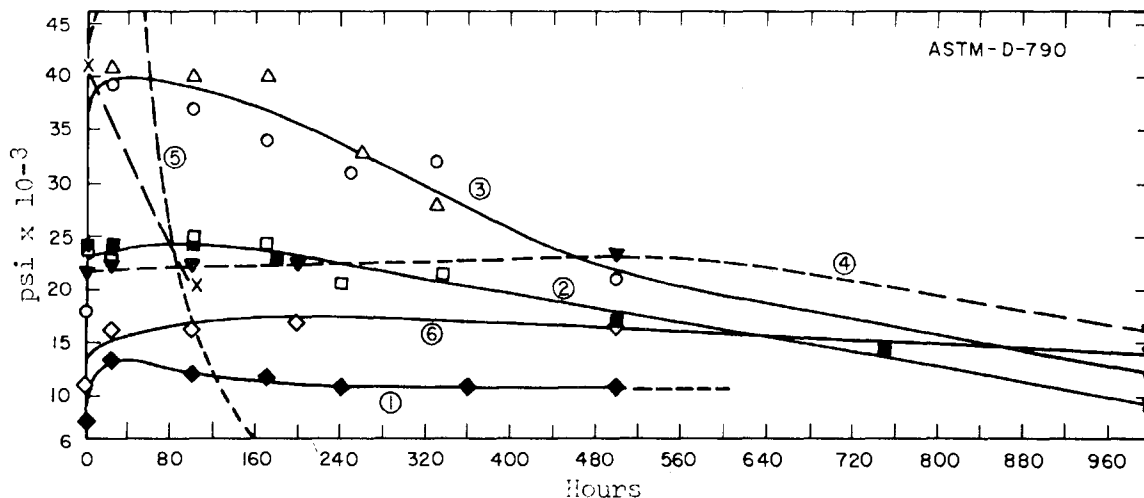
<u>Test Temp.</u>	<u>Specimen No.</u>	<u>Width in Inches</u>	<u>Thickness in Inches</u>	<u>Load in Pounds</u>	<u>Flexural Strength* psi Avg. psi</u>
R.T.	1	.998	.0904	76	27,975
	2	.937	.1079	98	26,970
	3	.937	.1106	101	26,580
					27,175
500°F	4	.982	.0876	52	20,715
	5	.936	.1085	64	17,455
	6	.936	.1102	66	17,520
					18,563
800°F	7	.965	.0894	39	15,215
	8	.935	.1072	52	14,445
	9	.934	.1078	50	12,930
					14,916

* All 9-ply laminates

[Ref. 23]

Polyamide-imide laminates:

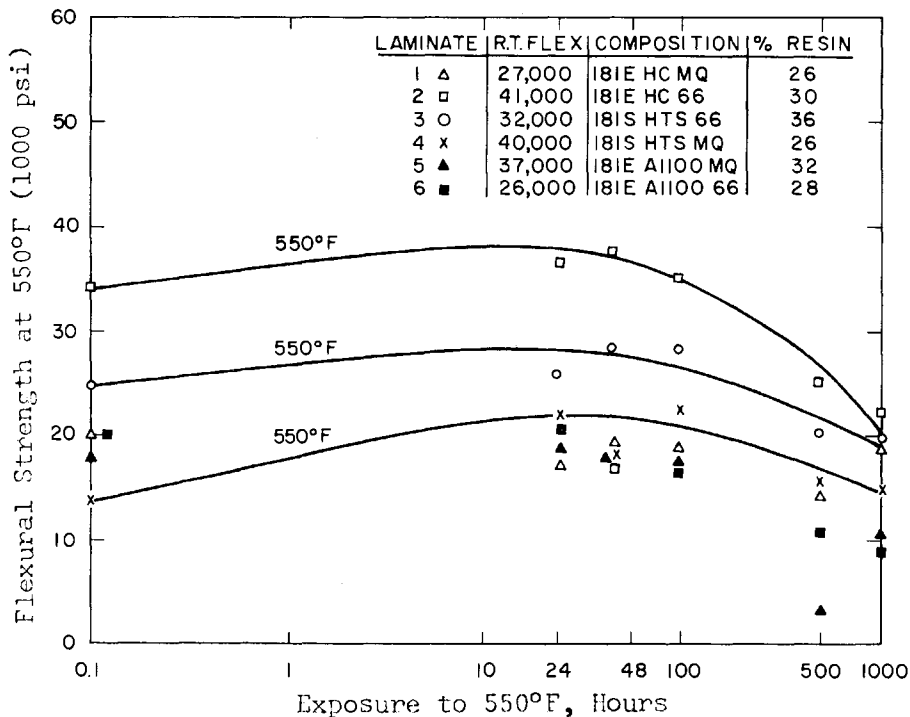
Freeman et al made a study of aromatic polyamide-imide laminates aged and tested at 300°C in air for flexural strength. Curve 3 represented the best values achieved on small pilot plant samples using a Resin C. Curve 2 represented early developed materials using small scale production samples. Curve 4 was made with a Resin D. Resin C and D differ in the proportion of imide to amide groups and also in the distribution of these linkages. The flexural strength of as-received laminates at room temperature was approximately 45,000 - 50,000 psi:



Flexural strength of 181 glass cloth laminates aged and tested at 300°C in air. Curves: 1,6 Silicones; 2,3,4 Aromatic Amide-Imides; 5 Phenolic Mil-R9299 for 260°C.

[Ref. 45]

The Air Force Materials Laboratory has conducted 550°F aging studies on DuPont MQ and MRE-66 polyimide resins reinforced with 181-E glass cloth (with different finishings) and found the flexural strength at 550°F to vary as follows:



14-ply laminates prepared from DuPont's MQ & MRE 66 polyimide resins 1 hour at 700°F at 200 psi.

Notes: HC=heat cleaned HTS=sizing at bushing

[Ref. 1]

Monsanto Skygard 700 Laminates:

Skygard 700 laminates (1/8" thick, 181 glass-cloth with A-1100 prefinish at 250 psi):

<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Flexure, Flatwise</u>
75°F	standard	75,000 - 85,000 psi
700°F	1/2 hour at 700°F	45,000 - 60,000 psi
700°F	100 hours at 700°F	20,000 - 35,000 psi

[Ref. 73]

Brunswick Polyimide Glass Fabric Laminates:

Polyimide 181 A-1100 glass cloth laminates:

<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Flexural Strength</u>
77°F	1/2 hour soak at 77°F	52,500 psi
-65°F	1/2 hour soak at -65°F	64,000 psi
550°F	1/2 hour soak at 550°F	35,300 psi
700°F	1/2 hour soak at 700°F	30,000 psi
800°F	1/2 hour soak at 800°F	22,000 psi
550°F	24 hour soak at 550°F	38,000 psi*
550°F	48 hour soak at 550°F	38,800 psi*
550°F	100 hour soak at 550°F	35,200 psi*
550°F	200 hour soak at 550°F	25,300 psi

* heat cleaned fabric

[Ref. 17]

Harmco Polyimide Glass Cloth Laminates:

Laminate Fabrication: Resin--Poly [p,p'-oxybis (phenylene) pyromellitimide];

Prepreg: solution-coated, using pyridine, dried under 20-mm Hg for 2 hours at 50°C;

Carrier: 181 heat-cleaned E-glass;

Cure Cycle: 2 hours at 700°F under 1000 psi, cooled under pressure;

Laminate Dimensions: 2 inches x 2 inches x 2 plies;

<u>Test Temperature</u>	<u>Postcure</u>	<u>Flexural Strength</u>
77°F	none	22,220 psi (avg. of 2 spec)
77°F	1 hour at 400°C (high vacuum)	21,110 psi (avg. of 2 spec)

[Ref. 75]

Narmco has had a developmental processing study project on various polyimide laminates under way for several years and report the following flexural strength data:

Laminate Fabrication

Resin:	Poly(m-phenylene-3,3',4,4' - benzophenonetetracarboxylimide)	Same
Prepreg:	Solution coated via brush using pyridine; dried 24 hours @ R.T. 12 hours @ 85°C under vacuum	Same; dried 24 hours @ R.T., 4.5 hours @ R.T. under vacuum, 2 hours @ 50°C under vacuum
Carrier:	1581 HTS-994	Same
Cure Cycle:	1 hour @ 600°F under 300 psi	Kiss cycle @ 6-second intervals for 30 seconds @ 550° - 600°F for 1 hour under 500 psi
Postcure:	None	None
Laminate Dimensions:	5 inches x 8 inches x 13 plies	5 inches x 8 inches x 14 plies
Avg Laminate Thickness:	0.140 inches	0.130 inches

<u>Test Condition</u>	<u>Avg Flexural Strength</u>	<u>Avg Flexural Strength</u>
R.T.	27,300	17,700
R.T. after 2 hour water boil	22,400	9,300
600°F after 10 min. @ 600°F	18,100	10,700
800°F after 10 min. @ 800°F	14,800	16,900

[Ref. 76]

Laminate Fabrication:

Resin: Poly(m-phenylene-3,3',4,4'-benzophenonetetracarboxylimide)

Prepreg: Solution-coated, 3 dip coats and 1 brush coat; each coat air dried 30 min. @ 176°F, 45 min. @ 437°F, and 15 min. @ 600°F

Carrier: 1581 HTS (994)

Cure Cycle: 0.5 hours @ 716°F under 500 psi, then cooled under pressure

Postcure: R.T. specimens, none; aged specimens, R.T. to 600°F @ 10°F/hour

Laminate Dimensions: 4 inches x 5 inches x 14 plies

Laminate No*	Thickness, inches	Resin Content, %	Void Content, %	Flexural Strength (psi) & Modulus (psi x 10 ⁶)			
				R.T.		After 247 hours @ 600°F	
A	0.144	32.2	12.5	57,200	3.44	22,000	2.50
	0.144			56,500	3.47	22,400	2.47
	0.145			56,700	3.34	22,100	2.34
				Avg 56,800	3.42	Avg 22,200	2.44
B	0.164	43.2	10.5	60,900	3.00	22,800	2.22
	0.165			58,100	3.12	24,300	2.26
	0.165			61,600	3.17	22,700	1.97
				Avg 60,200	3.10	Avg 23,300	2.15
C	0.134	32.0	13.5	57,600	3.66	22,300	2.44
	0.133			61,400	3.54	22,700	2.56
	0.132			62,200	3.55	21,500	2.51
				Avg 60,400	3.58	Avg 22,200	2.51
D	0.163	41.4	12.5	56,900	3.01	25,600	2.39
	0.161			57,600	3.17	24,600	2.18
	0.161			53,500	3.13	22,800	2.05
				Avg 56,000	3.10	Avg 24,300	2.21

*Laminates A and B were fabricated from prepreg solution-coated with anhydride-terminated polyamic acid in DMAC and in pyridine, respectively. Laminates C and D were fabricated from prepreg solution-coated with amine-terminated polyamic acid in DMAC and in pyridine, respectively.

[Ref. 102]

Laminate Fabrication

Resin: Poly(m-phenylene-3,3',4,4'-benzophenonetetracarboxylimide)

Prepreg: Solution coated via brush using pyridine; dried 24 hours @ R.T., 4.5 hours @ R.T. under vacuum, 2 hours @ 50°C under vacuum

Carrier: 1581 HTS (994)

Cure Cycle: Kiss cycle 5 seconds @ 550°F - 600°F for 0.5 hours under 500 psi

Postcure: 1 hour @ 750°F in N₂ as indicated

Laminate Dimensions: 2 inches x 2 inches x 2 plies

Avg Laminate thickness: 0.014 inches

<u>Test Conditions</u>	<u>Flexural Strength, psi</u>	
	<u>No Postcure</u>	<u>Postcure</u>
R.T.	90,300	80,000
600°F after 10 min. @ 600°F	-	50,600
800°F after 10 min. @ 800°F	-	40,300
1000°F after 10 min. @ 1000°F	-	28,200
600°F after 24 hours @ 600°F	53,200	27,150
700°F after 50 hours @ 700°F	18,600	-

[Ref. 76]

Freeman and co-workers at Westinghouse Electric Corporation have investigated the flexural strength of various other polyamide-imide laminates after prolonged periods of aging at elevated temperatures as well as the effect of varying the laminating conditions:

Flexural Strength of Aromatic Imide Laminates on 181-A1100 Glass Cloth

Resin	I-6	I-6	EI-2
Bond Coat	AI-8	-	None
Press Temp., °F	590	690	430
Resin Content, %	33.8	38.2	22
Aging and Test Temp., °C	315	315	300
Aging and Test Temp., °F	600	600	572
Laminating Pressure, psi	2000	2000	1000
<u>Aging Time (hours)</u>		<u>Flexural Strength, psi</u>	
	<u>R.T.</u>	<u>R.T.</u> <u>600°F</u>	<u>R.T.</u> <u>500°F</u>
Initial R.T.	20900	23500 -	39100 26000
0.5	11900	- 15400	16000 -
24	13800	- 15700	16450 -
72	15200	- 14750	- -
100	-	- -	18200 -
250	20100	- 15150	25600 -
500	18550	19000 13350	22670 -
750	16800	15300 12900	25850 -
1000	14050	12450 11200	24530 -

Average of three samples 1/8 inch thick tested according to LP 406 b,
Amend. 1, Method 1031.1

[Ref. 45, 47]

EFFECT OF LAMINATING PRESSURE ON FLEXURAL
STRENGTH OF AROMATIC IMIDE I-6 LAMINATES

<u>Pressure psi</u>	<u>Press Time & Temperature</u>	<u>Sample Thickness (inch)</u>	<u>Flexural Strength, psi</u>	
2000	30 min. 680°F	0.126 avg	23,500 avg	Sound boards
1000	30 min. 680°F	0.129 0.131 0.133 Avg 0.131	22,000 21,000 20,600 21,200	Slight edge delamination on cutting.
500	30 min. 680°F	0.147 0.147 0.149 Avg 0.148	14,400 13,400 12,200 13,330	Edge delamination on cutting. Delamination on break.
500	10 min. 734°F	0.142 0.140 0.140 Avg 0.141	20,500 20,500 21,000 20,700	Partial delamination on break.
1000	10 min. 734°F	0.128 0.130 0.129 Avg 0.129	24,100 23,400 24,000 23,830	Edges furred slightly on cutting. No delamination.
1000	10 min. 734°F	0.131 0.130 0.131	21,200 16,350* 15,700*	Aged 24 hours at 600°F. No delamination.

All samples contained 12 plies of 181-A1100 glass cloth impregnated with 38.2% by weight Resin I-6.

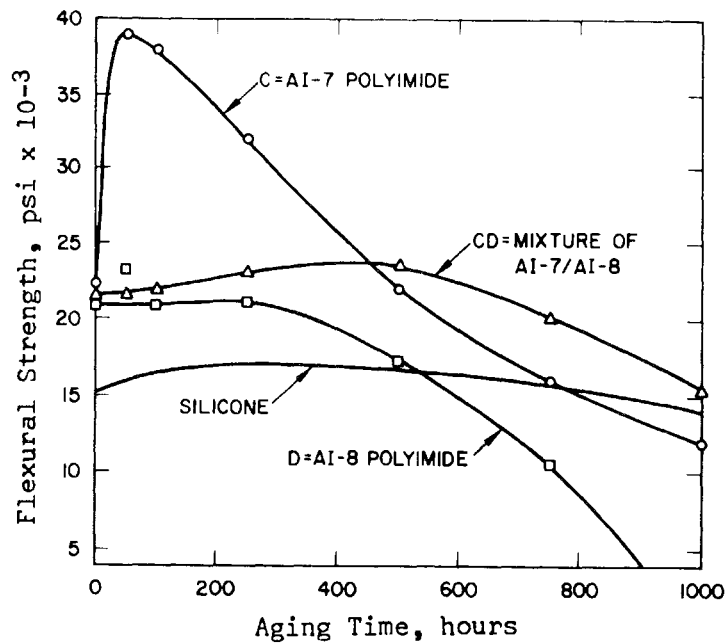
*Test conducted at 600°F.

[Ref. 47]

Twelve-ply glass cloth laminates produced with Shawinigan RS-5303 and 181-A-1100 glass cloth have shown the following flexural strength values:

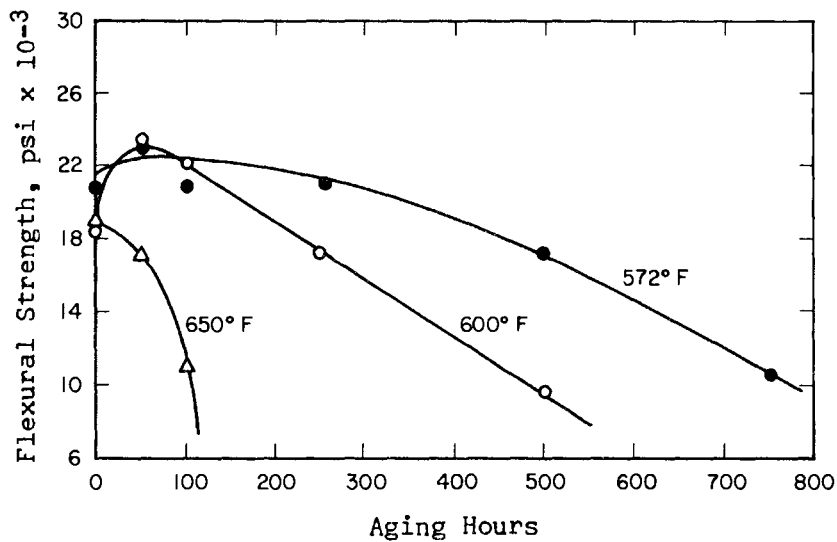
<u>Test Conditions</u>	<u>Flexural Strength</u>
At room temperature	75,000 to 85,000 psi
After 1/2 hour @ 700°F measured @ 700°F	45,000 to 60,000 psi
After 100 hours @ 700°F measured @ 700°F	20,000 to 35,000 psi

[Ref. 112]



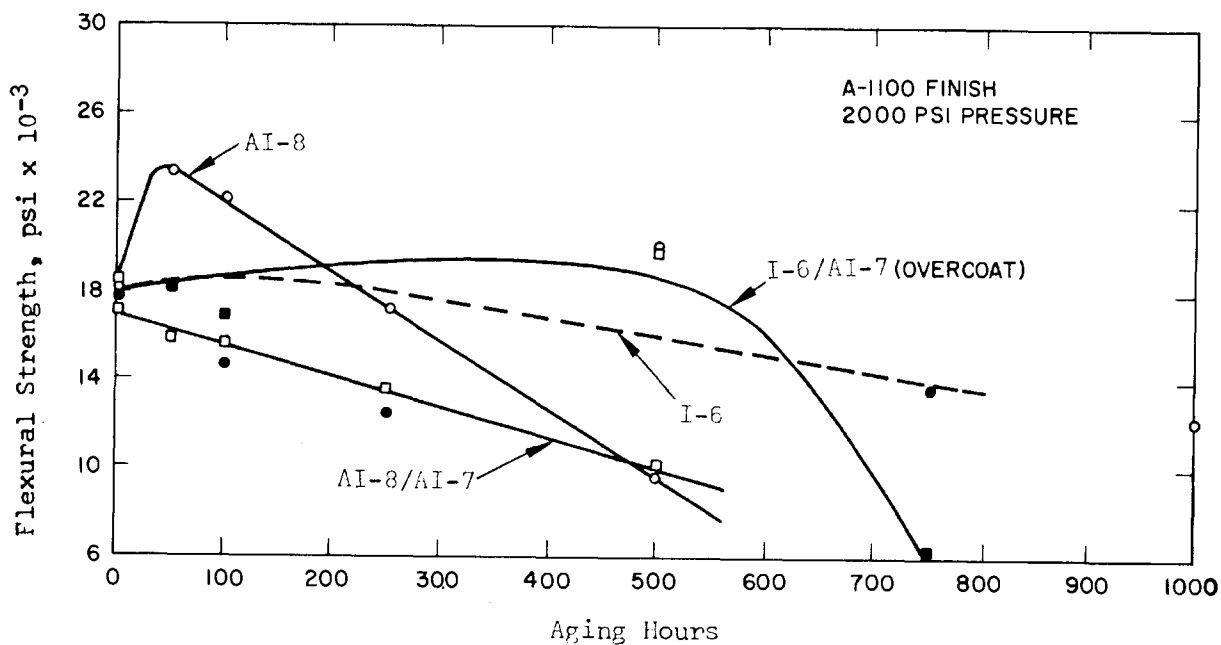
Flexural strength of 1/8 inch polyamide-imide laminates.
181 glass cloth A-1100 finish 37% resin aged and tested at 300°C

[Ref. 51]



Flexural strength of (3,4'-diaminobenzanilide +
pyromellitic anhydride) laminates aged and tested at temperature

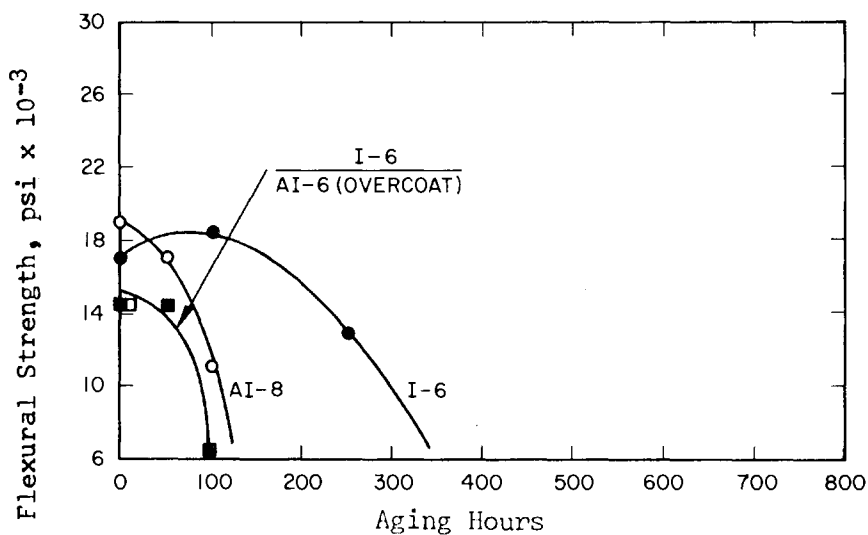
[Ref. 45]



Flexural strength of 181 glass laminates (1/8 inch thick)
aged in air and tested at 600°F (ASTM D-790).

I-6 = 4,4' - diaminophenyl sulfide and pyromellitic anhydride
AI-8 = 3,4' - diaminobenzanilide + pyromellitic anhydride

[Ref. 45]



Flexural strength of 181 glass laminates aged and tested at 650°F

I-6 = 4,4' - diaminophenyl sulfide and pyromellitic anhydride
AI-8 = 3,4' - diaminobenzanilide + pyromellitic anhydride

[Ref. 46]

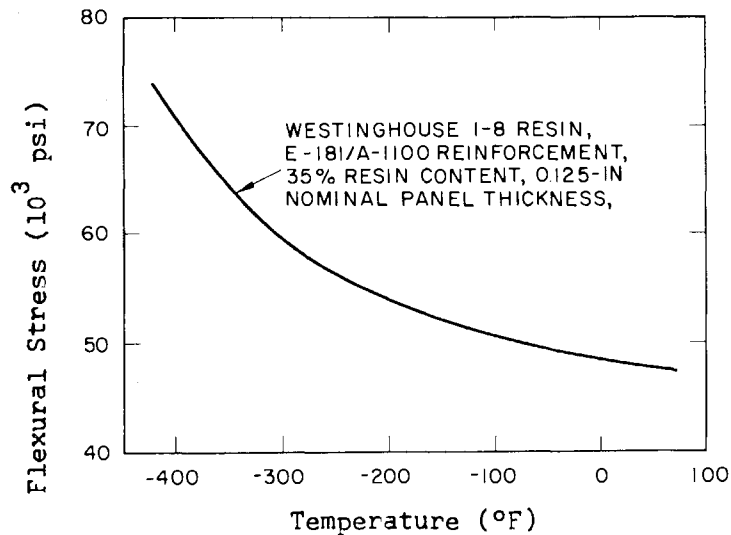
Effect of Glass Finish and Resin AI-8 & AI-7 Content
on Flexural Strength of Aromatic Polyamide-Imide Laminates

Glass Finish	A-1100(6)	172(8)	HOL-24(7)	112(9)	A-1100(6)	A-1100(6)
Resin Content (%) ⁽⁴⁾	26.4	28.0	28.0	27.0	37.5	36.3*
Resin	AI-8	AI-8	AI-8	AI-8	AI-8	AI-7
Aging Time (hours) ⁽⁵⁾	Flexural Strength					
Initial (R.T.)	23,400	10,400	26,500	13,700	30,900	45,000
0.5	16,600	10,500	19,800	12,200	20,800	22,000
50	17,300	12,200	18,900	17,300	23,100	39,000
100	19,300	12,500	20,000	22,900	20,900	38,000
250	18,200	13,600	17,500	17,400	21,100	32,000
500	14,100	10,300	14,700	12,900	17,200	22,000
750	4,900	5,780	11,700	1,900	10,500	16,000
1000	No Bond	No Strength	No Bond	No Test	No Strength	

- Note: (1) All laminates 1/8" thick on 181 glass cloth filler.
 (2) Flexural strength in psi; average of 3 samples/ASTM D-790 (LP-406b).
 (3) Postcured from 260° to 572°F at 10°F/hour rise after removal from press.
 (4) Resin AI-8, pressed 1 hour at 626°F at 2000 psi (3,4'-diaminobenzanilide and pyromellitic dianhydride).
 (5) Aged and tested at 572°F.
 * Resin AI-7, pressed at 400°F.
 (6) γ -aminopropyl end groups.
 (7) Phenolic end groups
 (8) Vinyl groups.
 (9) Heat-cleaned finish.

[Ref. 45]

The Martin Company tested the Westinghouse Electric polyimide I-8 at cryogenic temperatures and found the flexural strength to vary as follows:



[Ref. 69]

AI polymer (type 10) polyamide-imide 181 glass cloth with Volan A finish, 15 ply (1/8 inch), 31% resin content, press schedule of 400°F for 25 minutes at 600 psi plus 5 minutes at 1500 psi:

<u>Temperature</u>	<u>Flexural Strength</u>
25°C	54,320 psi

[Ref. 4]

b. Flexural Modulus

SP-Polymer:

<u>Temperature</u>	<u>Flexural Modulus</u>
77°F	450,000 psi
707	100,000

[Ref. 11]

SP-1 Polymer:

<u>Temperature</u>	<u>Flexural Modulus (ASTM D790)</u>
-310°F	515,000 psi
73	450,000
392	225,000
482	210,000
752	90,000

[Ref. 97]

DuPont PI-3301 filament wound panel*(90° bidirectionally wound):

<u>Temperature</u>	<u>Flexural Modulus</u>
77°F	5.3 x 10 ⁶ psi

*17.5% resin content, sp. grav = 1.84, 20 end S-994 (HTS)

[Ref. 26]

Brunswick Polyimide Glass Fabric Laminates:

Polyimide 181 A-1100 Glass Cloth Laminates:

<u>Test Temperature</u>	<u>Exposure Conditions</u>	<u>Flexural Modulus</u>
77°F	1/2-hour soak at 77°F	2.69 x 10 ⁶ psi
-65	1/2-hour soak at -65°F	3.11 x 10 ⁶
550	1/2-hour soak at 550°F	2.17 x 10 ⁶
700	1/2-hour soak at 700°F	1.89 x 10 ⁶
800	1/2-hour soak at 800°F	1.95 x 10 ⁶
550	24-hour soak at 500°F	2.30 x 10 ⁶
550	48-hour soak at 550°F	2.47 x 10 ⁶
550	100-hour soak at 550°F	2.32 x 10 ⁶

[Ref. 17]

The NARMCO Research and Development Division of Whittaker Corporation has had a developmental processing study project on various polyimide laminates under way for several years and report the following flexural modulus data:

NARMCO Laminate Fabrication:

Resin: Poly[p,p'-oxybis(phenylene)pyromellitimide]

Prepreg: Solution-coated, using pyridine; dried under 20-mm Hg for 2 hr @ 50°C

Carrier: 181 heat-cleaned E-glass

Cure Cycle: 2 hr @ 700°F under 1000 psi, cooled under pressure

Laminate Dimensions: 2 inches x 2 inches x 2 plies

<u>Test Conditions</u>	<u>Postcure</u>	<u>Flexural Modulus, psi x 10⁶</u>
R.T. ↓	None	1.31
	None	1.15
		Avg 1.23
	1 hr @ 400°C, high vacuum	1.01
	1 hr @ 400°C, high vacuum	1.11
		Avg 1.06

[Ref. 75]

Fourteen-Ply Polyimide Laminate Data

Laminate Fabrication:

Resin: Poly(m-phenylene-3,3',4,4'-benzophenonetetracarboxylimide)

Prepreg: Solution-coated, 3 dip coats and 1 brush coat; each coat air dried
30 minutes @ 176°F, 45 minutes @ 437°F, and 15 minutes @ 600°F

Carrier: 1581 HTS (994)

Cure Cycle: 0.5 hours @ 716°F under 500 psi, then cooled under pressure

Postcure: R.T. specimens, none; aged specimens, R.T. to 600°F @ 10°F/hour

Laminate Dimensions: 4 inches x 5 inches x 14 plies

Laminate No.*	Thickness, inch	Resin Content, %	Void Content, %	Flexural Modulus (psi x 10 ⁶)	
				R.T.	After 247 hr @ 600°F
A	0.144	32.2	12.5	3.44	2.50
	0.144			3.47	2.47
	0.145			3.34	2.34
				Avg 3.42	Avg 2.44
B	0.164	43.2	10.5	3.00	2.22
	0.165			3.12	2.26
	0.165			3.17	1.97
				Avg 3.10	Avg 2.15
C	0.134	32.0	13.5	3.66	2.44
	0.133			3.54	2.56
	0.132			3.55	2.51
				Avg 3.58	Avg 2.51
D	0.163	41.4	12.5	3.01	2.39
	0.161			3.17	2.18
	0.161			3.13	2.05
				Avg 3.10	Avg 2.21

*Laminates A and B were fabricated from prepreg solution-coated with anhydride-terminated polyamic acid in DMAC and in pyridine, respectively.

Laminates C and D were fabricated from prepreg solution-coated with amine-terminated polyamic acid in DMAC and in pyridine, respectively.

[Ref. 102]

Narmco Laminate Fabrication

Resin:	Poly (<u>m</u> -phenylene-3,3' 4,4'-benzophenonetetracarboxylimide)	Same
Prepreg:	Solution coated via brush using pyridine; dried 24 hr @ R.T. 12 hr @ 85°C under vacuum	Same; dried 24 hr @ R.T., 4.5 hr @ R.T. under vacuum, 2 hr @ 50°C under vacuum
Carrier:	1581 HTS-994	Same
Cure		
Cycle:	1 hr @ 600°F under 300 psi	kiss cycle @ 6 sec intervals for 30 sec @ 550° - 600°F for 1 hr under 500 psi
Postcure:	None	None
Laminate Dimensions:	5 in x 8 in x 13 plies	5 in x 8 in x 14 plies
Avg. Laminate Thickness:	0.140 in	0.130 in

<u>Test Conditions</u>	<u>Avg Flexural Modulus (psi x 10⁶)</u>	<u>Avg Flexural Modulus (psi x 10⁶)</u>
R.T.	2.28	2.66
after 2 hr water boil	2.35	2.54
600°F after 10 min @ 600°F	2.33	2.35
800°F after 10 min @ 800°F	1.85	2.16

[Ref. 76]

Narmco Laminate Fabrication

Resin: Poly (m-phenylene-3,3' 4,4'-benzophenonetetracarboxylimide)

Prepreg: Solution coated via brush using pyridine: dried 24 hr @ R.T.,
4.5 hr @ R.T. under vacuum, 2 hr @ 50°C under vacuum

Carrier: 1581 HTS (994)

Cure Cycle: kiss cycle 5 sec @ 550°F - 600°F for 0.5 hr under 500 psi

Postcure: 1 hr @ 750°F in N₂ as indicated

Laminate Dimensions: 2 in x 2 in x 2 plies

Avg. Laminate Thickness: 0.014 in

Table Continued

[Ref. 76]

Table Continued

Test Conditions	Aging Conditions	Flexural Modulus x 10 ⁶ psi	
		No Postcure	Postcure
R.T.	--	2.72	2.79
600°F	10 min @ 600°F	--	2.04
800°F	10 min @ 800°F	--	2.31
1000°F	10 min @ 1000°F	--	1.96
600°F	24 hrs @ 600°F	2.55	1.93
700°F	50 hrs @ 700°F	1.63	--

[Ref. 76]

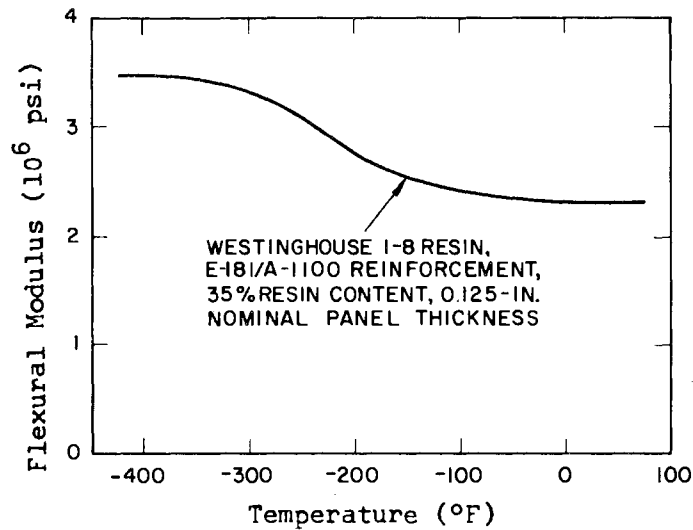
Freeman and co-workers at Westinghouse Electric Corporation have investigated various polyamide-imide (I-6) laminates after aging them for prolonged periods of time at 600°F in air and found the flexural modulus to vary as follows:

Hours at 600°F	Flexural Modulus (psi x 10 ⁶)		Test Temp.
	Sample B	Sample C	
Initial	(2.01)	(1.73)	600°F
0.5	1.09	1.34	
24	1.37	1.64	
72	1.44	1.55	
250	1.62	1.39	
500	1.67	1.40	
	(1.65)		
750	1.2		
1000	1.6		

*Values in parentheses represent tests at room temperature. All values, average of three or more samples in accordance with LP406b Amend. 1, Sept. 25, 1952: Method 1031.1 Flexural. All samples 1/8 inch thick, pressed 30 minutes at 2000 psi at 365°C ± 10° (690°F) and contained 38.2% of resin I-6 on 181-A1100 glass cloth.

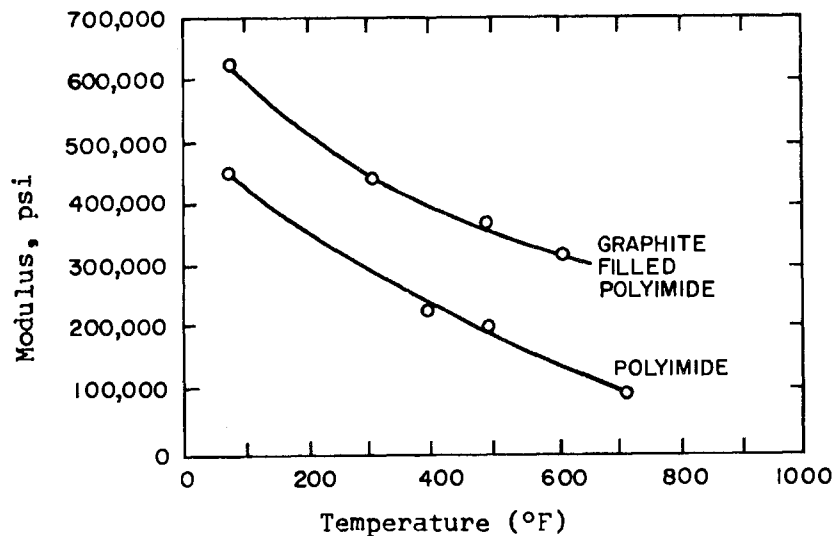
[Ref. 45]

The Martin Company tested a Westinghouse Electric Corporation polyimide (I-8) glass cloth laminate at cryogenic temperatures and found the flexural modulus to vary as shown in the following graph:



[Ref. 69]

Devine and Kroll have tested the effects of temperature on the flexural modulus of both unfilled and graphite-filled polyimides over a wide range of temperatures:



Flexural modulus as a function of temperature for polyimides

c. Flexibility

Mathes at the General Electric Company evaluated aromatic polyimide insulations (0.0014 inches thick) at cryogenic temperatures and found the flexibility to be as follows:

Mandrel flexibility of insulated .0253 inches wire before and after aging in air and vacuum (tested at 23°C in air and at -269°C in liquid helium)

Insulation		Mandrel Diameter to Cause Failure - Inches			
Aromatic polyimide	Before Aging	After Aging 120 Days at 120°C		After Aging 120 days at 250°C	
		In Air		In Vacuum	
	Tested at	Tested at		Tested at	
	23°C -269°C 23°C	23°C -269°C		23°C -269°C	
	OK-1X* 1/8	OK-1X 1/8		OK-1X 1/8	
				1/4 1/2	

*OK-1X The wire can be wrapped about its own diameter without visible damage

[Ref. 70]

Repeated Mandrel Flexibility
As a Function of Temperature
(Unaged Samples)

Wire		-269°C	-196°C	-60°C	23°C
HML(GE)	Satisfactory	OK - 1/8*	OK - 1/8	OK - 1/8	OK - IX**
HML(PD)	Satisfactory	OK - 1/8	OK - 1/8	-	OK - IX
Triple ML(PD)	Satisfactory	OK - 1/8	OK - 1/8	-	OK - IX
Asbestos	Failure	1/4	1/8	-	1/8
(ML Coat)	Cracks	1/8 - 1/4	1/8 - 1/4		1/8 - 3/4
	Satisfactory	1/2	1/2		1

* OK - 1/8 indicates no failure when reverse flexed 10 times around a 1/8 inch mandrel.

** OK - IX indicates no failure when wrapped around its own diameter.

"Failure" involves loss of adhesion with film coatings or a crack through to the copper with fibrous coatings.

GE = General Electric Company polyimide HML
PD = Phelps Dodge Company polyimide ML

[Ref. 72]

Repeated Mandrel Flexibility Test at 25°C

(Test Conditions: 10 Reverse Bands Around Mandrel Diameter Shown)

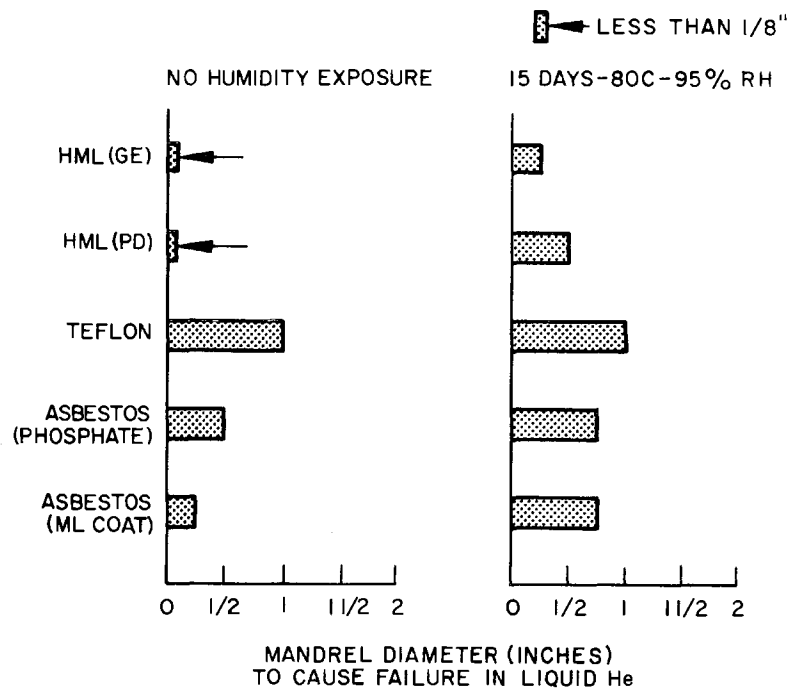
178

		Pre-Conditioning Before Test						
Insulated Wire	Overall Wire Dia. - In.	120°C/Vac		120°C/Air		250°C/Air		80°C/95% RH 15 Days
		60 Days	120 Days	60 Days	120 Days	60 Days	120 Days	
HML (G.E. Co.) (0.0014" Wall)	Failure		None		None	1/8	1/4	None
	Welts		--		--	1/2 - 3/4	1/2 - 3/4	--
	Satisfactory	OK - 1X	OK - 1X	OK - 1X	OK - 1X		1	OK - 1X
HML (Phelps-Dodge)	Failure		None		None	1/8	1/2	None
	Welts		--		--	1/2 - 3/4	3/4 - 1	--
	Satisfactory	OK - 1X	OK - 1X		OK - 1X		---	OK - 1X
Asbestos (ML over- coated) (0.0064" Wall)	Failure	Not	1/8	Not	1/8	Not	1/4	1/4
	Cracks	Tested	1/8 - 3/4	Tested	1/4 - 1/2	Tested	1/8 - 1/4	1/8 - 1/2
	Satisfactory		1		3/4		1/2	3/4

Note: OK - 1X, the wire can be wrapped on its own diameter without visible damage.

"Failure" involves loss of adhesion with film coatings or a crack through to the copper with fibrous coatings.

[Ref. 72]

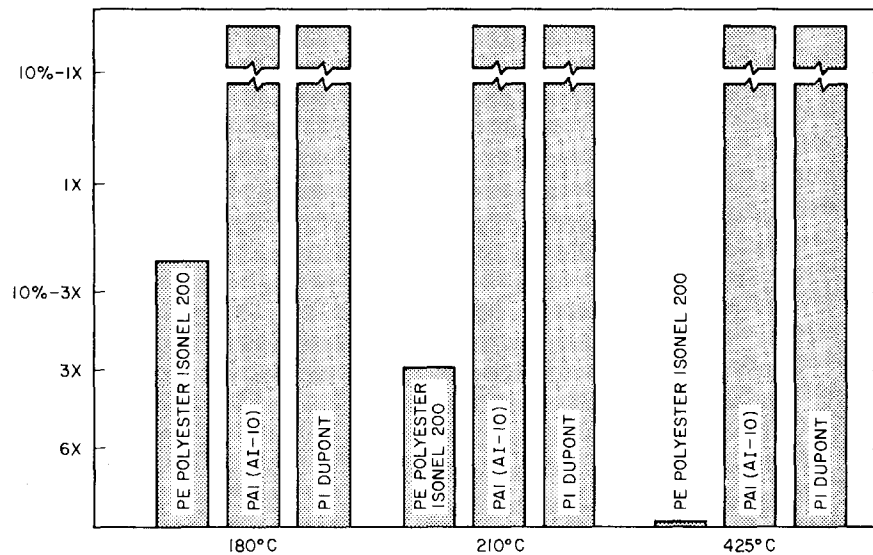


Effect of Humidity Exposure on Repeated Mandrel Flexibility at -269°C

[Ref. 72]

The Amoco Chemical Company has compared their AI-10 Polyamide-imide polymer with a DuPont Polyimide (type not identified) and a polyester (Isonal 200) with regard to its flexibility and report the following data:

Flexibility: Elongation and Wrap



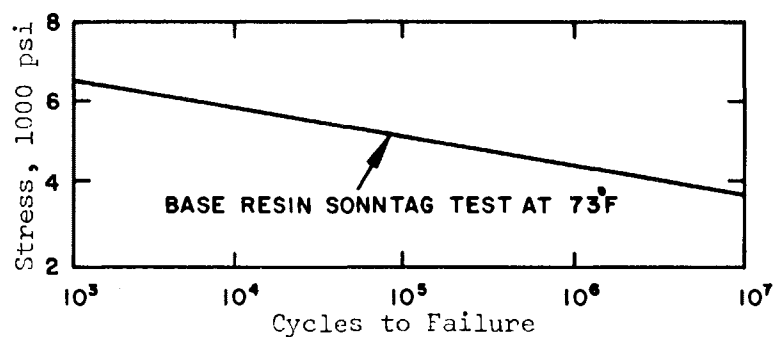
[Ref. 104]

	Polyester Isonel 200 PE	AI-10 Polyamide-imide PAI	DuPont Polyimide PI
Flexibility, 20%-1X	OK	OK	OK
Snap	OK	OK	OK
Unilateral Scrape	1400-1600	1200-1500	900-1200
Repeated	25	100+	20
Windability (field trials)	very good	excellent	good
			[Ref. 104]

5. Endurance Properties

a. Fatigue Strength

SP-Polymer:



Axial fatigue of polyimide is about 5000 psi at 10⁵ cycles.

[Ref. 98]

The above axial fatigue studies were made with Sonntag testing machine at 1800 cpm at an ambient temperature of 73°F and shows that the fatigue strength of polyimides is quite high.

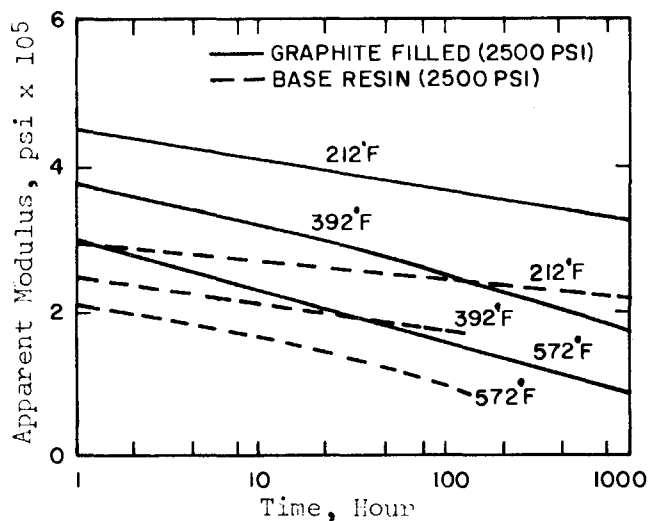
b. Folding Endurance

<u>Sample</u>	<u>Temperature</u>	<u>Method</u>	<u>Folding Endurance</u>
H-Film	25°C	ASTM-D643-43 MIT Tester, 1 Kg	10,000 cycles
Amanim (polyamide-imide)	25°C	ASTM-D643-43 MIT Tester, 1 Kg	6,000 cycles

[Ref. 98]

c. Creep

The outstanding resistance of creep displayed by SP-Polymer polyimides is shown by the apparent modulus data by Todd and Wolff:

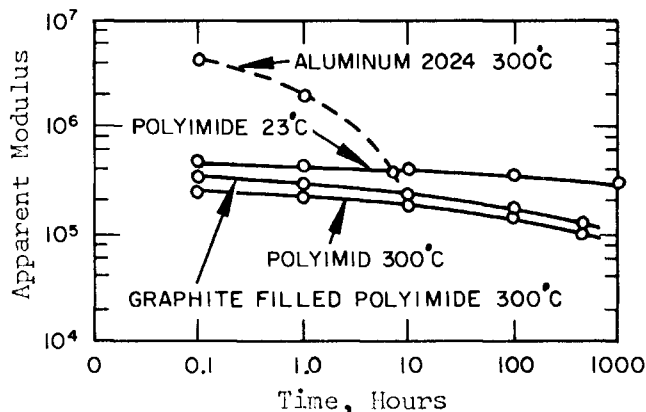


Apparent modulus data indicate the outstanding resistance to creep displayed by the polyimides.

[Ref. 98]

Apparent modulus takes into account both the initial deflection and deflection due to creep. These data, which were obtained by loading a test bar in flexure with a stress of 2500 psi, show that unfilled polyimide still has an apparent modulus of over 100,000 psi at temperatures as high as 572°F after 100 hours. The graphite-filled material has an even higher apparent modulus--close to 200,000 psi.

Devine and Kroll compared the apparent modulus in flexural creep of unfilled and graphite-filled polyimides with aluminum alloy 2024 at 300°C. The polyimides show outstanding resistance to creep:



Apparent modulus in flexural creep as a function of time for polyimides and aluminum.

<u>Sample</u>	<u>Tensile Stress</u>	<u>Time</u>	<u>Temp.</u>	<u>Change in Dimension</u>	
				<u>MD*</u>	<u>TD**</u>
1 mil "Kapton" (Type H-Film)	8,800 psi ↓	1 hour ↓	25°C	3.8%	2.8%
			100°C	5.2%	3.1%
			135°C	6.3%	4.0%
			170°C	9.0%	--
			202°C	22.0%	6.5%
	8,800 psi 11,000 psi 13,000 psi	1 hour ↓	100°C ↓	5.2%	--
				13.0%	--
				32.0%	--
	8,800 psi ↓	10 minutes ↓		4.8%	2.9%
				5.2%	3.1%
				5.8%	3.2%
				5.9%	3.3%
				6.0%	3.4%

*MD = Machine Direction

**TD = Transverse Direction

[Ref. 31]

<u>Sample</u>	<u>Compressive Stress</u> (ASTM D876-61)	<u>Temperature</u>	<u>Hour to Cut-Through</u>
1 mil "Kapton" (Type H-Film)	∞ to 12,000 psi ↓	450°C	2
		425°C	5
		400°C	16
		350°C	140
		300°C	1,300 Extrapolated
		250°C	10,000
		200°C	100,000

[Ref. 31]

6. Wear and Frictional Properties

a. Wear and Abrasion Resistance

<u>Material</u>	<u>Thickness</u>	<u>Machine</u>	<u>Conditions</u>	<u>Abrasion Resistance</u>
"Kapton" Type H	1 mil	Taber Abrader	CS-17 wheels, 500 gm load	3.5 mg/ 1,000 cps
	1 mil	G.E. Scrape Tester ↓	1 Kg, 2-side tape adhesion	10 scrapes
	1 mil		1 Kg, polyester adhesion	97 scrapes
	2 mil		1 Kg, 2-side tape adhesion	146 scrapes
	2 mil		500 gm, 2-side tape adhesion	1,000 scrapes
	5 mil		1 Kg, 2-side tape adhesion	1,000 scrapes
	5 mil		1 Kg, polyester adhesion	1,000 scrapes

[Ref. 31]

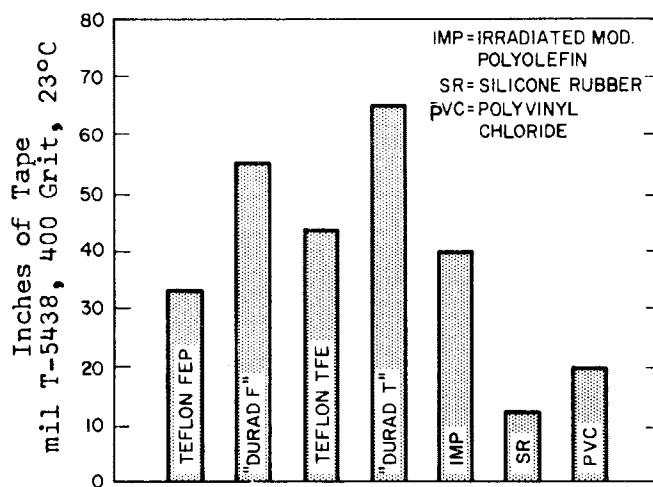
Mathes at the General Electric Company has evaluated the repeated scrape abrasion of two binder solutions:

<u>Material</u>	<u>Source</u>	<u>Temp.</u>	<u>Humidity</u>	<u>Load</u>	<u>No. of cycles to failure</u>
HML	Phelps Dodge Co.	23°C	50% RH	540 gms.	29 (avg. of 5)
HML	General Electric Co.	23°C	50% RH	540 gms.	49 (avg. of 5)

[Ref. 72]

It should be noted that the absolute abrasion resistance values are dependent upon processing method used in the insulation construction, the method of testing and variables with each test procedure (grit tapes, wall thickness, apparatus, etc.), poor repeatability from laboratory to laboratory.

The American Super-Temperature Wires, Inc. has conducted abrasion resistance tests on 10 mil wall insulation and report the following values:



[Ref. 3]

b. Coefficient of Friction

<u>Sample</u>	<u>Temperature</u>	<u>Coefficient of Kinetic Friction</u>	
H-Film: Film-to-Film (ASTM D-1894)	25°C	0.42	[Ref. 68,29]
"Pyre-ML" Films:	25°C	0.17	[Ref. 35]

Todd and Wolff studied and discussed the wear and frictional characteristics of unfilled and graphite-filled SP Polymer in nitrogen and air atmospheres and report the following data for a load condition of 25 psi:

<u>Property</u>	<u>Unfilled Molding</u>	<u>Graphite-Filled Molding</u>
Load (P), psi	25	25
Velocity (V), ft/min	1000	1000
PV	25,000	25,500
Wear Rate, in./1000 hr		
in nitrogen	0.0003 - 0.0006	0.0003 - 0.0006
in air	0.3 - 0.6	0.2 - 0.3
Coef. of Friction		
in nitrogen	0.08 - 0.10	0.05 - 0.07
in air	0.22 - 0.26	0.17 - 0.21
Hardness, Rockwell H	83 - 89	73 - 75

[Ref. 98]

"The PV limit of polyimides sliding on carbon steel without lubrication is much lighter than that of other organic materials. Since the polyimides do not melt, their PV limit is set by the PV value at which wear rates show a rapid increase. PV values as high as 100,000 psi x ft/min have been achieved, and there indications that it may be possible to operate at even higher values in gases such as nitrogen or fluorocarbon refrigerants which contain little or no free oxygen. However, in air, PV values as high as 100,000 cannot always be exploited because of rapid wear.

Under ideal conditions the wear rate of polyimides against mild steel can be as low as 0.3 micro-in/hr. These very low rates have been obtained in thrust washer tests at a PV of 25,000 where a nitrogen atmosphere surrounded the thrust washer; no lubricant was used in these tests. When run in air, wear is much more rapid and friction is increased (see above table). The wear rate in both air and nitrogen is not affected by changing the moisture content of the atmosphere. Also, the wear rate in air is not affected by the increasing the surface temperature of the base resin to 500°F.

In an air atmosphere, the graphite-filled polyimide shows lower wear and friction than the un-filled material. In nitrogen, the wear and friction behavior of the two materials, however, is nearly the same. Long-term (1,000 hr) field testing of the polyimides shows that smooth operation, free of chatter, noise and vibration, can be achieved with the graphite-filled polyimide against mild steel without lubrication and in the presence of air. Typical metals that are believed to be satisfactory with polyimides in bearing uses include: 1025 mild steel, gray cast iron, 4140 steel, 420 stainless steel, 52100 bearing steel, M-10 tool steel and tungsten carbide. Not recommended are: 316 stainless steel, 347 stainless steel, A-2 chrome steel, A-4 chrome steel, and cadmium-plated steel.

As with other materials, the dynamic coefficient of friction of polyimides does not reach a steady-state value until after several hours of operation. On start-up, friction is high but drops rapidly in the first 10 to 30 minutes and then approaches the steady-state value. This ultimate value is affected by the atmosphere and decreases with increasing load. At a PV value at 25,000 the steady-state coefficient in air is about 0.24 for un-filled polyimide and about 0.19 for the graphite-filled material. In nitrogen the steady-state coefficient of friction is 0.10 or less for both materials. At a PV value of 100,000 in air (240 psi at 417 ft/min) the coefficient drops to less than 0.05 for both materials".

[Ref. 98]

The same authors (Todd and Wolff) conducted additional experiments at higher loads (30, 60, and 240 psi) and lower velocities and obtained the following data:

Friction and Wear of SP-1 Against Carbon Steel**
(no lubrication)

<u>Material</u>	<u>Environment</u>	<u>Pressure P (psi)</u>	<u>Velocity V (fpm)</u>	<u>Coefficient of Friction*</u>	<u>Wear Rate (in./1000 hr)</u>
SP-1 Polyimide	Air	30, 60	PV=25,000 834,417	0.17	0.3
			834,417	0.10	0.0005
	Nitrogen	30, 60	PV=100,000 417		
			417	0.05	0.002
	Nitrogen	240			

* After break-in

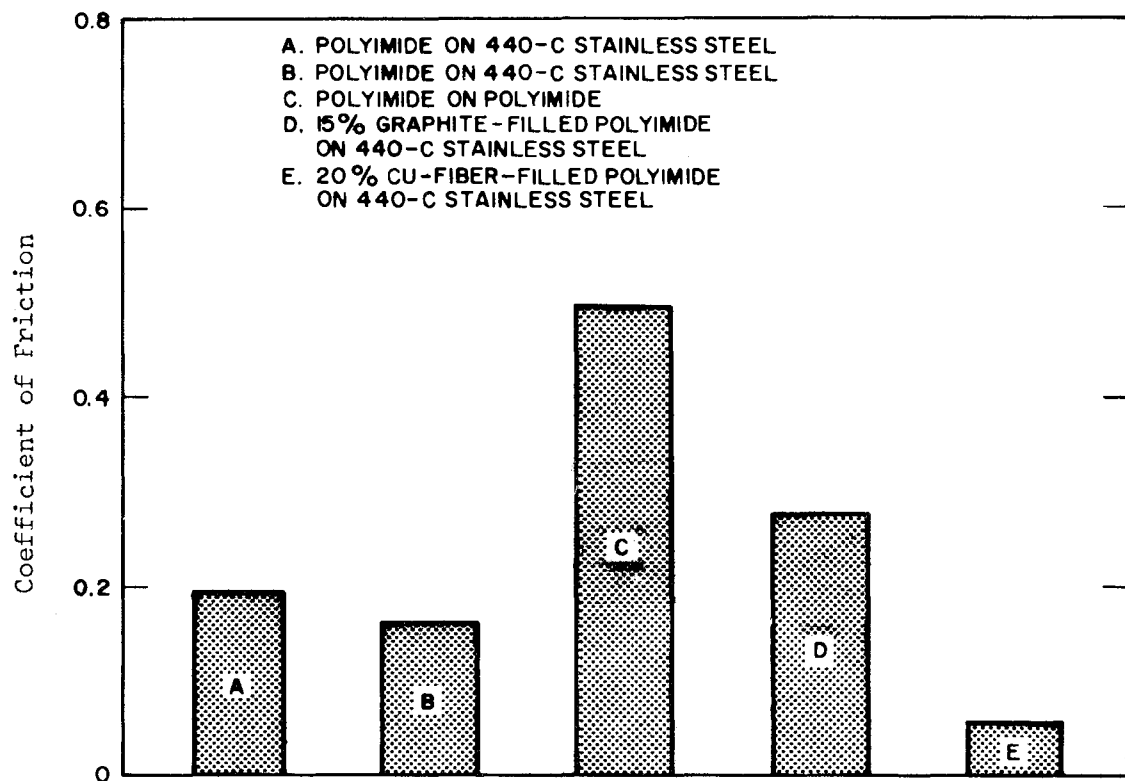
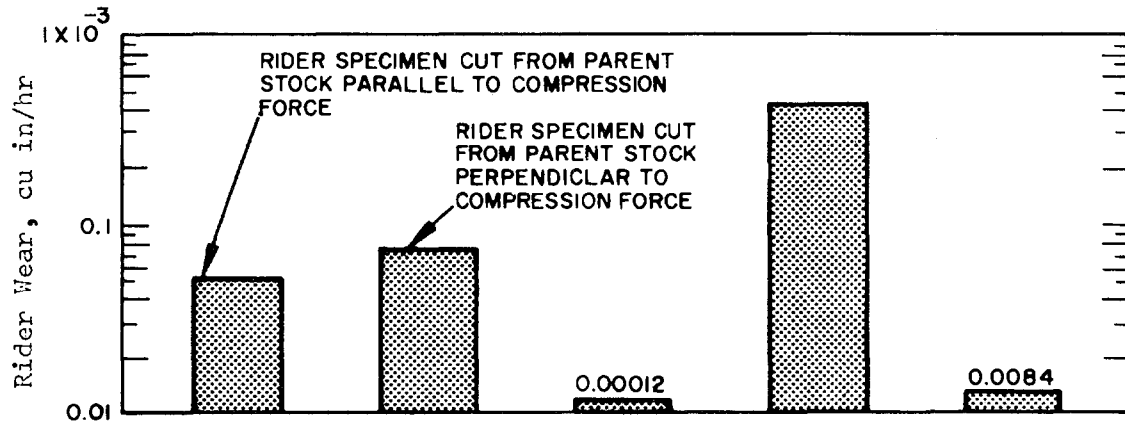
** Initial surface finish, 8 to 10 μ in. rms.

[Ref. 97]

The DuPont Company has developed (on a laboratory basis only) a "Kapton" Type HB Film which contains fluorocarbons dispersed throughout the entire thickness of a polyimide "Kapton" Type H-Film. This combination has resulted in a film with the antifriction properties of "Teflon" TFE fluorocarbon resin and the high temperature capability of "Kapton" polyimide film. The material has been evaluated by laminating it to steel and forming it into thrust and sleeve bearings. In evaluating the Type HB Film, the DuPont Company found that:

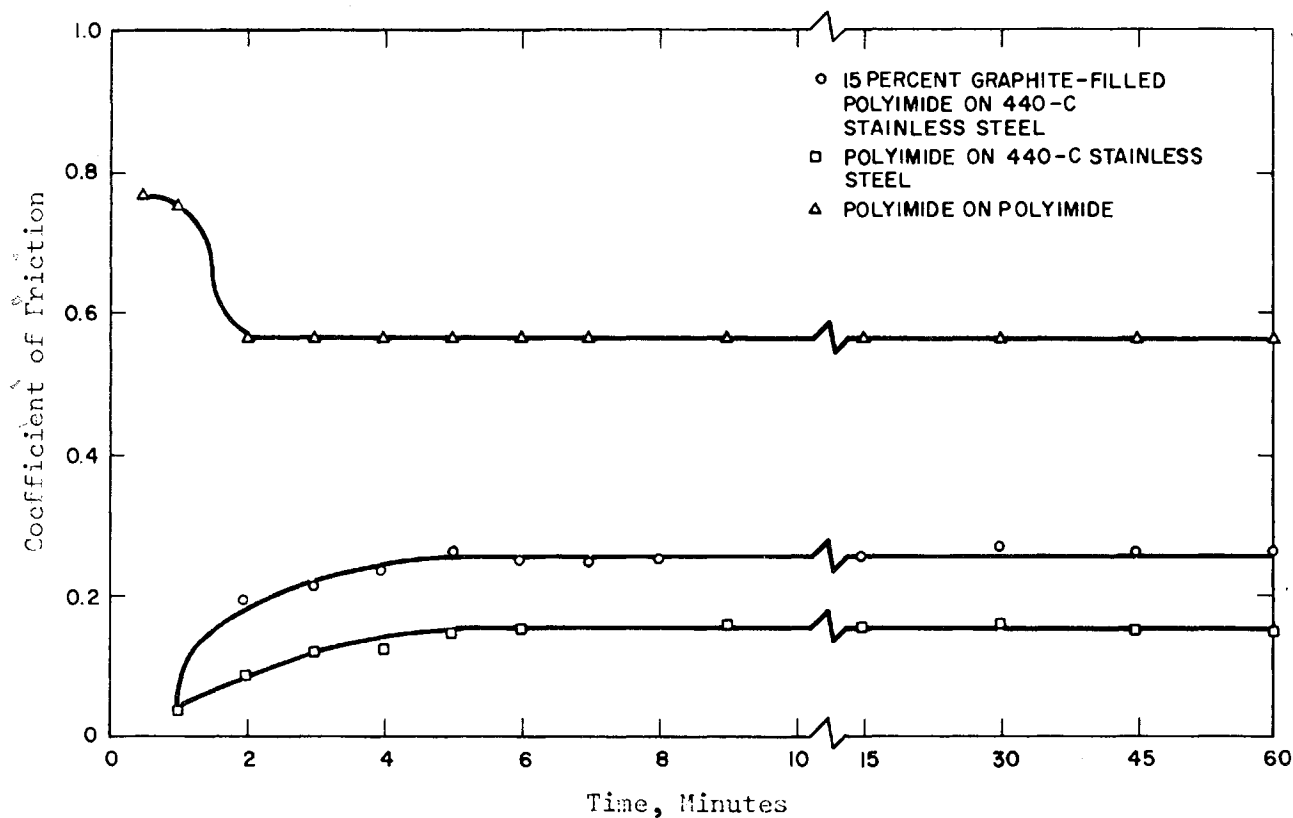
1. The dry PV limit is in excess of 50,000 (PV = product of pressure in psi times velocity in ft/min).
2. Dry wear is similar to that of other nonlubricated bearings.
3. Improved wear is obtained if the PV is greater than 10,000.
4. A PV resulting from high P and low V gives better wear than an equal PV which is the product of low P and high V.
5. Increased temperature appears to improve the wear performance.

Buckly and Johnson (Lewis Research Center, NASA) conducted friction and wear studies in vacuum (10^{-9} mm Hg) with Sp polyimide sliding on both metals and itself. Their results are presented in the following graphs:



Coefficient of friction and rider wear for various material combinations in vacuum (10^{-9} mm Hg). Sliding velocity, 390 fet/minute; load, 1,000 grams; duration of run, 1 hour; no external specimen heating.

[Ref. 18]



Coefficient of friction as a function of time for polyimide sliding on polyimide and on 440-C stainless steel in vacuum (10^{-9} mm Hg). Sliding velocity, 390 feet/minute; load, 1000 grams; ambient pressure, 10^{-9} millimeter of mercury; no external specimen heating.

[Ref. 18]

7. Miscellaneous

a. Hardness

SP-Polymer:

<u>Sample</u>	<u>Hardness</u>
Rockwell H	83-89
	[Ref. 11]
Rockwell M	112
	[Ref. 19]

<u>Sample</u>	<u>Temperature</u>	<u>Hardness</u>
"Pyre-ML" Varnish (post formed clear coating)	--	20 (Knoop) [Ref. 31]
Monsanto Skygard 700 Laminates (1/8" thick, 181 glass cloth with A-1100 prefinish at 250 psi.	73°F	70 (Barcol) [Ref. 73]

b. Peel Strength

Ruffing and Traynor at Westinghouse Electric Corporation evaluated the peel strength (using ASTM D-903-49) of a Westinghouse polyamide-imide polymer to a nickel surface at room temperature and at 300°C:

<u>Measurement No.</u>	<u>Peel Strength at 25°C lbs/in. width</u>	<u>Peel Strength at 300°C lbs/in. width</u>
1	2.6	--
2	2.8	--
3	6.5	--
4	6.2	--
5	5.6	--
6	3.8	--
7	5.9	--
avg.	4.8	
8	--	4.7
9	--	3.6
avg.		4.1

[Ref. 84]

In a similar manner, the same authors tested a DuPont "Pyre-ML" polyimide polymer adhering to a nickel metal surface for its peel strength at room temperature:

<u>Measurement No.</u>	<u>Peel Strength lbs/in. width</u>
1	1.9
2	3.2
3	4.4
4	3.6
5	2.0
6	2.0
7	4.5
8	4.0
9	3.7
10	3.6
11	1.5
avg.	3.1

[Ref. 84]

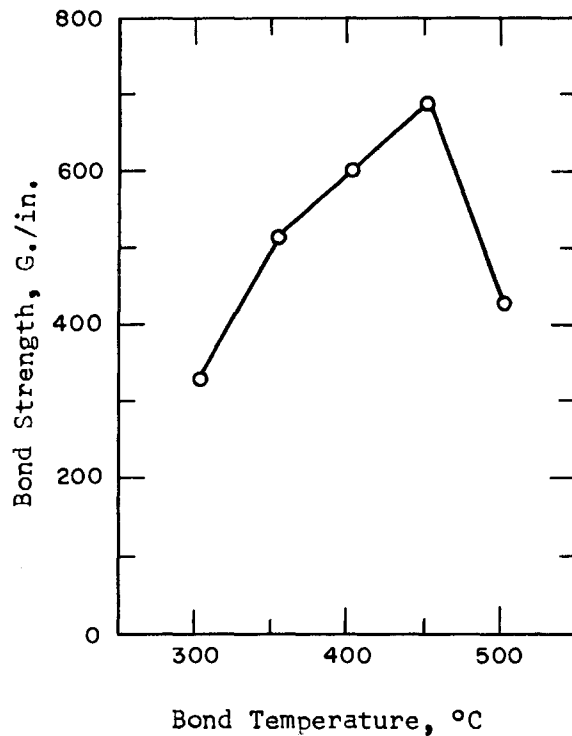
The DuPont Company, using the ASTM D-1781 climbing drum method, found the peel strength at 77°F of their PI-1101 and PI-1101 (formulated) adhesives to be as follows:

<u>Sample</u>	<u>Peel Strength</u>
PI-1101	30 in lbs/in
PI-1101 (formulated)	20 in lbs/in

[Ref. 30]

According to one author (Ref. 7) adhesive systems have been evaluated for bonding "Kapton" Type H-Film both to itself and to nonferrous metals and wood. Excellent bonds have been obtained, ranging in peel strength from 200 - 300 gm/in. width for a pressure-sensitive silicone rubber adhesive, to about 2000 gm/in width, for an epoxy. An epoxy novolac produced a bond so strong that the film itself failed in test.

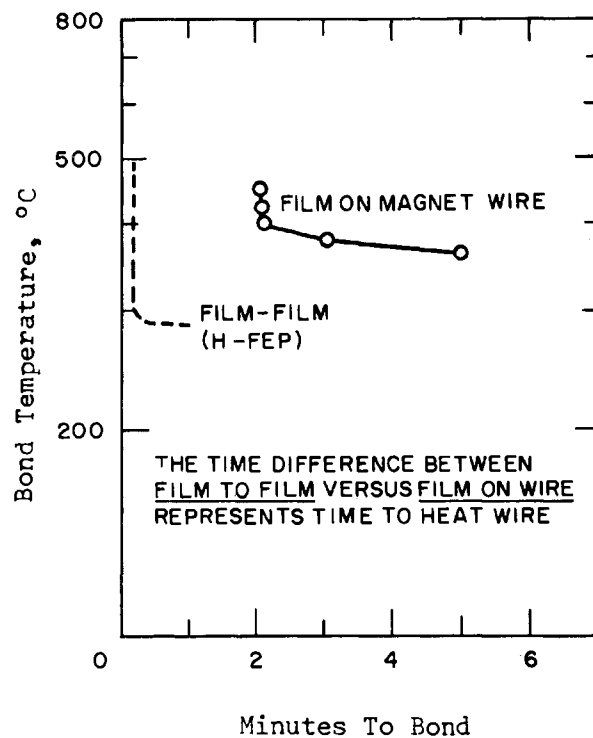
Melvin and Parish (Ref. 133) reported on the bonding characteristics of "Kapton" Type H-Film to Teflon FEP fluorocarbon and bond time relationship for "Kapton" Type HF polyimide film:



Bond strength of type H polyimide film to FEP fluorocarbon as a function of temperature; 10-second dwell at 10 psi pressure.

[Ref. 133]

Bond time-temperature relationship for type HF polyimide film. The time difference represents the time to heat the wire.



[Ref. 133]

The DuPont Company evaluated the bond strength of three polyimide insulating varnishes after various periods of elevated temperature aging:

<u>Varnish</u>	<u>Testing and Aging Conditions</u>	<u>Bond Strength</u>		
		<u>RC-B-24951</u>	<u>RK-692</u>	<u>RC-5060</u>
Solids		45% $\pm$ 0.5	13% $\pm$ 0.5	19% $\pm$ 1.0
Viscosity (poises)		2 - 5	6 - 10	5 - 9
	Initial at 25°C	35 - 70 lbs	6 - 9 lbs	40 - 70 lbs
	Initial at 180°C	20 - 30 lbs	6 - 8 lbs	20 - 30 lbs
	@ 25°C after 2 wks @300°C	20 - 40 lbs	-	25 - 45 lbs
	@180°C after 2 wks @300°C	10 - 20 lbs	-	20 - 40 lbs
	@ 25°C after 4 wks @300°C	10 - 30 lbs	3 - 5 lbs	-
	@ 25°C after 3 wks @260°C	20 - 40 lbs	5 - 7 lbs	20 - 35 lbs
	@180°C after 3 wks @260°C	10 - 20 lbs	3 - 5 lbs	-

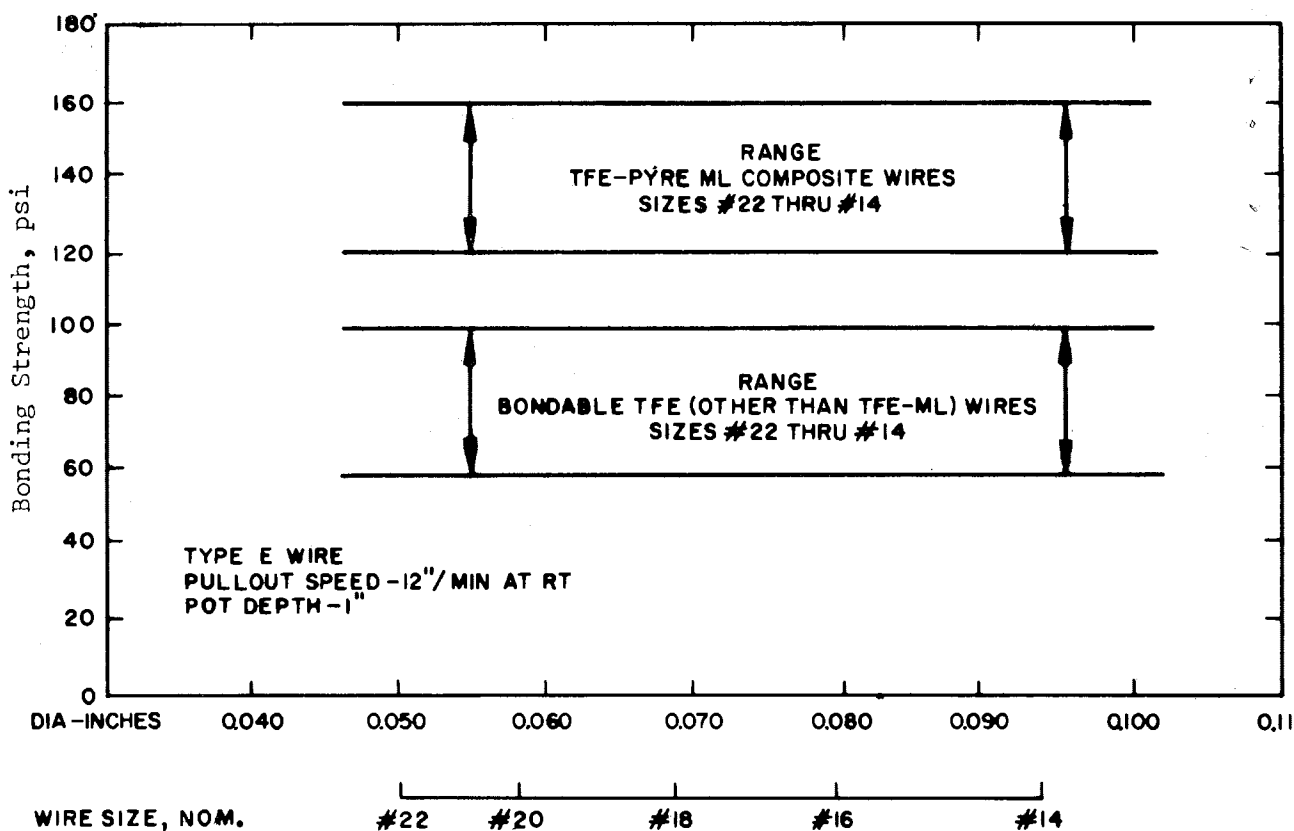
[Ref. 33]

The American Super-Temperature Wires, Inc. Company has evaluated the pull-out strengths of its "Durad" polyimides from various potting compounds:

<u>Sample</u>	<u>Bond (pull-out) Strength - psi</u>			
	<u>Hysol 4183</u>	<u>Stycast 2651</u>	<u>Pro-Seal 777</u>	<u>RTV 1018</u>
FEP	74.2	59.3	29.3	51.1
Durad-F	66.9	73.1	164.0	107.5
TFE	36.6	27.6	28.7	19.8
Durad-T	80.2	98.9	180.0	179.0

[Ref. 3]

Learn and Seegers have also determined the bonding strength of Teflon/ "Pyre-ML" wires to epoxy potting compounds and report the following data:



[Ref. 66]

The Shawinigan Resins Corporation determined the bond strength at elevated Temperatures of their polyimide resin solutions RS-5303 and RS-5305 applied over magnet wire coils coated with DuPont "Pyre-ML". The bond strength was tested using Dexter helical coil method (Insulation, Sept. 1955, page 12):

<u>Aging Time at 300°C</u>	<u>Bond Strength at 180°C</u>	<u>Bond Strength at 225°C</u>
	<u>(in pounds)</u>	
o	15	5
168 hours	35	41
336 hours	28	35
672 hours	25	26

[Ref. 112]

Amoco Chemical Company tested the bond strength of their AI-220 polyamide-imide wire coating after various hours of cure time at 200°C yielding the following results:

<u>Hours Cure at 200°C</u>	<u>Lbs. Pull at Room Temp.</u>		<u>Lbs. Pull at 200°C</u>	
	<u>AI-220</u>	<u>AIvar 220</u>	<u>AI-220</u>	<u>AIvar 220</u>
2	19.0	24.3	4.0	3.5
4	22.0	27.0	7.0	6.0
6	23.5	29.0	9.0	7.5
8	25.0	30.5	11.0	9.0
12	26.0	32.0	14.0	11.0

[Ref. 104]

In another series of tests, they aged AI-220 wire helical coils for 40 days at 250°C and determined the bond strengths at room temperature:

	<u>AI-220</u>	<u>AIvar 220</u>
Initial Bond Strength	28.0 lbs	35.0 lbs
Room Temperature Bond Strength After 40 Days at 250°C	7.0 lbs	13.5 lbs

[Ref. 104]

c. Tear Strength

"Kapton" Type H-Film:

<u>Sample</u>	<u>Temperature</u>	<u>Tear Strength</u>
Tear Strength-Propagating (Elmendorf) (ASTM D-1922-61T)	25°C	8 gm/mil
Tear Strength-Initial (Graves) (ASTM D-1004-59T)		500 gm/mil

[Ref. 29]

"Kapton" Type HF Composites:

<u>Construction</u>	<u>Tear Strength</u>
1H/ 1/2F (1-mil "Kapton" Type H/ 1/2 mil Teflon FEP)	20 gm/mil (Elmendorf) 800 gm/mil (Graves)
1/2F/ 1H/ 1/2F (1/2-mil Teflon FEP/ 1-mil "Kapton" H/ 1/2-mil Teflon FEP)	30 gm/mil (Elmendorf) 1,000 gm/mil (Graves)
1H/ 1F (1-mil "Kapton" Type H/ 1-mil Teflon FEP)	60 gm/mil (Elmendorf)
2H/ 2F (2-mil "Kapton" Type H/ 2-mil Teflon FEP)	150 gm/mil (Elmendorf) 1,150 gm/mil (Graves)

[Ref. 27]

Polyimide Coated Fabrics:

<u>Sample</u>	<u>Tear Strength (Elmendorf, W x F*)</u>
108 Fabric No. 6500 (4.0 mil thickness, total wt = 2.88 oz/sq yd, coating wt = 1.44 oz/sq yd):	100 x 80 grams [Ref. 67] 125 x 80 grams [Ref. 34]
116 Fabric No. 6507 (5.5 mil thickness, total wt = 5.5 oz/sq yd, coating wt = 2.34 oz/sq yd):	300 x 350 grams [Ref. 34,67]
116 Fabric No. 6507 (7.0 mil thickness, total wt = 7.0 oz/sq yd, coating wt = 3.84 oz/sq yd):	400 x 400 grams [Ref. 34] 400 x 450 grams [Ref. 67]
128 Fabric No. 6508 (10.5 mil thickness, total wt = 10.8 oz/sq yd, coating wt = 4.8 oz/sq yd):	1600 x 1400 grams [Ref. 34] 1900 x 1650 grams [Ref. 67]
1185 Fabric No. 6509 (8.75 mil thickness, total wt = 8.73 oz/sq yd, coating wt = 4.60 oz/sq yd):	700 x 500 grams [Ref. 34]

* W = Warp Threads

* F = Filling Threads

"Kapton" Type H-Film:

Propagating Tear Run of Elmendorf

<u>Film Thickness</u>	<u>Tear Strength</u> gm/mil	
	<u>MD*</u>	<u>TD**</u>
1 mil	7.6	8.2
2 mil	10.5	13.2
3 mil	10.3	13.7
5 mil	11.7	13.7

*MD = Machine Direction
**TD = Transverse Direction

[Ref. 31]

Cut-Through (Thermal Penetration Test)
(ASTM D-876-61)

<u>Conditions</u>	<u>Film Thickness</u>	<u>Cut-Through Temperature</u>
Temperature rise of 1/2°C/minute	1 mil	435°C
	2 mil	525°C
	3 mil	530°C
	5 mil	530°C

[Ref. 31]

<u>Film Thickness</u>	<u>Cut-Through Temperature</u>	
	<u>240°C/Minute</u>	<u>1/2°C/Minute</u>
1 mil	637°C	435°C
2 mil	711°C	525°C
5 mil	760°C	530°C

[Ref. 31]

Isomid (Polyester-imide):

Heat Shock, 1 Hour at 200°C (pre-stretch 15%) % Pass as

1x Diameter	50 - 100
2x Diameter	90 - 100
3x Diameter	100
4x Diameter	100

Cut-Through Temperature at 1000 grams, 5°C/Min Rise, °C 370 - 390

Thermal Flexibility when aged at 180°C, followed by 3x
diameter wrap, hours to failure 168 - 336

[Ref. 31]

Freeman and co-workers (Ref. 45) report the thermoplastic cut-through value for a wire enameled polyimide is in excess of 950°F. Stabler and Lewis (Ref. 91) report a cut-through temperature of 435°C for "Kapton" Type H-Film and a zero strength temperature of 815°C.

Learn and Seegers have reported typical room temperature cut-through properties of Teflon- "Pyre-ML" systems:

<u>Test Conditions</u>	<u>Cut-Through Force</u>		
	<u>FEP</u>	<u>FEP- "Pyre-ML"</u>	<u>Percentage Increase</u>
Mod. UL Test, 0.008" R. Blade (1)	0.236 lb/mil	0.326 lb/mil	38
	<u>TFE</u>	<u>TFE- "Pyre-ML"</u>	
Mod. UL Test, 0.003" R. Blade (2)	0.196 lb/mil	0.335 lb/mil	71

- (1) #22 insulated wire, Type K
(2) #32 insulated wire, Type ET

[Ref. 66]

<u>Test Conditions</u>	<u>FEP</u>	<u>FEP- "Pyre-ML"</u>
Solder iron, 1 lb load 45° tip, 800°F (1)	1 sec	over 1.67 hours*
	<u>TFE</u>	<u>TFE- "Pyre-ML"</u>
1/16" R. cutting edge incr. load at 0.75 KG/sec at 212°F	15 KG	30 KG
1/16" R. cutting edge incr. load at 0.75 KG/sec at 302°F	10 KG	20 KG

- (1) #22 insulated wire, type KT, 6 mil nom wall
* over 5 minutes required for passing

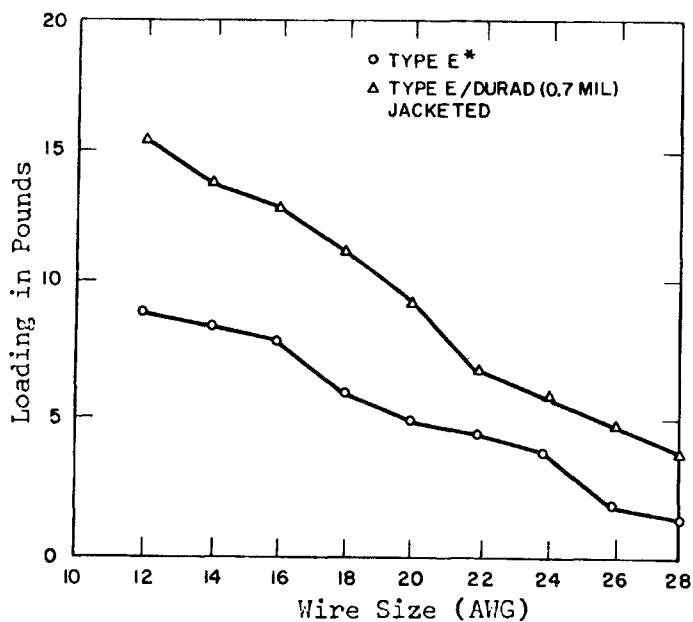
[Ref. 66]

The DuPont Company studied the effect of water (above 100°C) on the failure embrittlement of "Kapton" Type H-Film, 1 mil thick as compared to polyester, 1 mil thick film:

<u>Test Conditions</u>	<u>Material</u>	<u>Time to Failure</u>
Parr Bomb at 175°C: criterion for failure - embrittlement, as determined by the ability of the film to support vertical penetrating load against its horizontal surface.	"Kapton" H	20 - 55 hours
	Polyester	2 - 3 hours
Parr Bomb at 150°C: criterion for failure - embrittlement, as determined by a crease test.	"Kapton" H	8 days
	Polyester	5 hours

[Ref. 31]

American Super-Temperature Wires, Incorporated have investigated the maximum allowable for minimum twenty-minute penetration time of their "Durad" polyimide resin:



*Type E coating is TFE-Mil-W-16878D

[Ref. 3]

d. Bursting Strength:

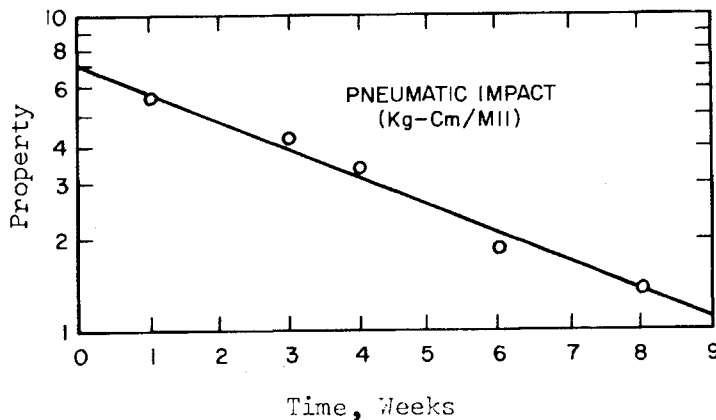
<u>Material</u>	<u>Temperature</u>	<u>Burst Strength, Mullen Test (ASTM D-774)</u>
"Kapton" Type H-Film	25°C	75 psi
		[Ref. 29,68]
"Ananim" Film (polyamide-imide)	25°C	52 psi
		[Ref. 44]

e. Impact Strength:

<u>Material</u>	<u>Temperature</u>	<u>Film Thickness</u>	<u>Impact Strength Kg-cm/mil</u>
"Kapton" Type H-Film	77°F ↓	1 mil	5.3
		2 mil	6.3
		3 mil	6.2
		5 mil	6.6

[Ref. 31]

Heacock and Berr of the E.I. du Pont de Nemours Company report the following thermal aging at 300°C data on "Kapton" Type H Film:

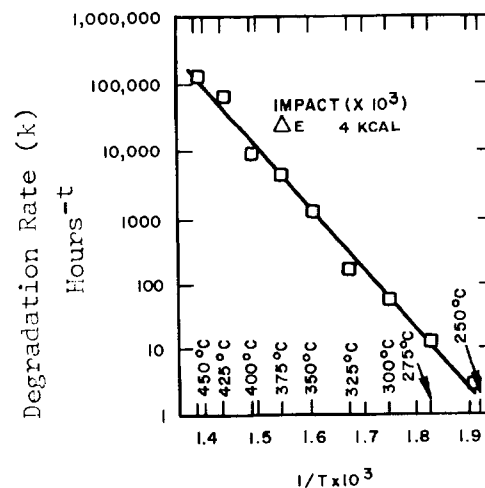


Effect of aging in air at 300°C on the physical properties of H-Film.

[Ref. 59]

From these above data and other temperature levels, an Arrhenius plot was constructed based on the equation: $\log P = -kt + \log P_0$

Arrhenius plot of physical property deterioration. A linear relationship is obtained.



[Ref. 59]

where P is the pneumatic impact (a DuPont test method which determines the energy required to rupture a film from a measure of the velocity of mechanically accelerated ball, first in free flight, and then in impeded flight after rupturing the test specimen) property at time t hours and P_0 is the initial value of the property, and k is the rate constant in reciprocal hours.

From these data, the authors obtained for the impact parameter the following activation energies, initial property values, and the rate constants at 400°C:

	In Air	I In Helium	
Activation Energy (ΔE)	41.4 $\pm$ 1.0	51.1 $\pm$ 2.7	Kcal
Initial Value (P_0)	6	6	kg-cm/mil
Rate Constant (k_{400})	0.14	0.0029	/1 hour

"Kapton" Type HF Composites:

<u>Construction</u>	<u>Temperature</u>	<u>Impact Strength</u>
1H/ 1/2F (1-mil "Kapton" Type H/ 1/2-mil Teflon FEP)	25°C	6.5 Kg-cm/mil
1/ 2F/ 1H/ 1/2F (1/2-mil Teflon FEP/ 1-mil "Kapton" H/ 1/2-mil Teflon FEP)	25°C	9 Kg-cm/mil
2H/ 2F (2-mil "Kapton" Type H/ 2-mil Teflon FEP)	25°C	16 Kg-cm/mil

[Ref. 27]

<u>Sample</u>	<u>Impact Strength</u>
SP-Polymer	0.7 - 1.1 ft-lb/in (izod) [Ref. 11]
	100 ft-lb/in ³ (tensile) [Ref. 19]

Todd and Wolff report that un-filled and graphite-filled SP-Polymer are notch sensitive. Compared to notched materials, un-notched test specimens have significantly higher impact strenghts -- 10 ft/lb/in for the un-filled material, and 1.8 ft-lb/in for the graphite-filled:

<u>Sample</u>	<u>Temperature</u>	<u>Impact Strength (izod)</u>
Un-filled SP-Polymer	77°F	0.7 ft-lb/in
	482°F	0.9 ft-lb/in
Graphite-filled SP-Polymer	77°F	0.2 ft-lb/in
	482°F	0.5 ft-lb/in

[Ref. 98]

Devine and Kroll also report impact test data on un-filled and graphite-filled polyimide plastics in the un-notched and notched conditions:

Impact Test (ASTM D-256-56)

<u>Sample</u>	Un-notched (ft/lb/in)		Notched (ft/lb/in)	
	<u>73°F</u>	<u>482°F</u>	<u>73°F</u>	<u>482°F</u>
Un-filled Polyimide	9.6	11.8	0.70	0.90
Graphite-filled Polyimide	1.8	2.8	0.23	0.47

[Ref. 22]

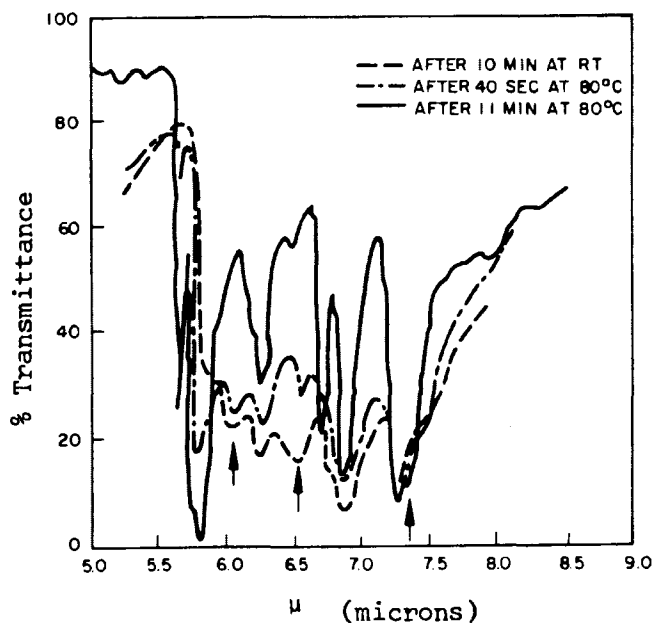
<u>Sample</u>	<u>Time, Minutes</u>	<u>Cure Temperature</u>	<u>180 in-lb Impact Resistance</u>
Amoco AI Polymer (Type 10) (on approx. 1 mil thickness on 1/8-inch steel panels)	10	250°F	Failed
	30	250°F	Passed
	30	300°F	↓
	10	350°F	
	2	600°F	

[Ref. 104]

PROPERTIES

D. OPTICAL

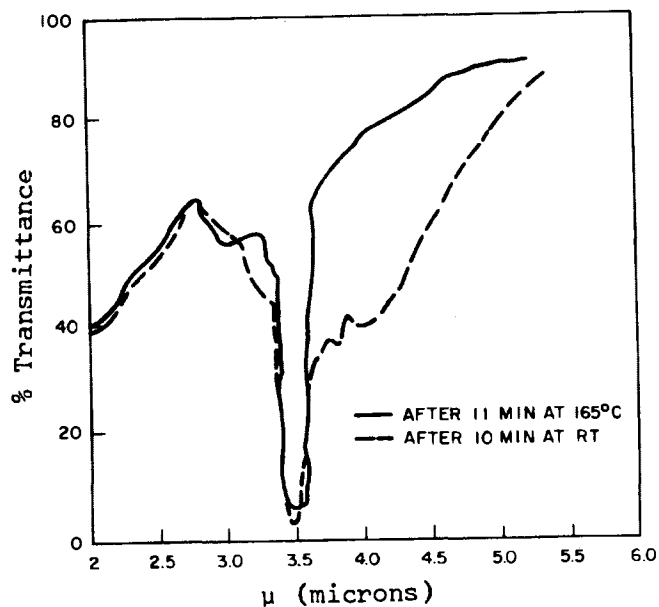
A variety of infrared analyses was conducted by Narmco Research and Development (1964) in their development of polyimides prepolymers (B-Stage) for high temperature structural adhesive purposes. The IR analyses permitted determination of N-H, imide rings, amide, carbonyl structures necessary to follow the degree and course of the polymerization reactions under various conditions:



Imidization as shown by infrared spectra. Kinetic study: condensation of Pyromellitic Dianhydride and m-phenylenediamine.

[Ref. 75-77]

Infrared spectra of reaction products indicating salt formation. Kinetic study: condensation of Pyromellitic Dianhydride and m-phenylenediamine.

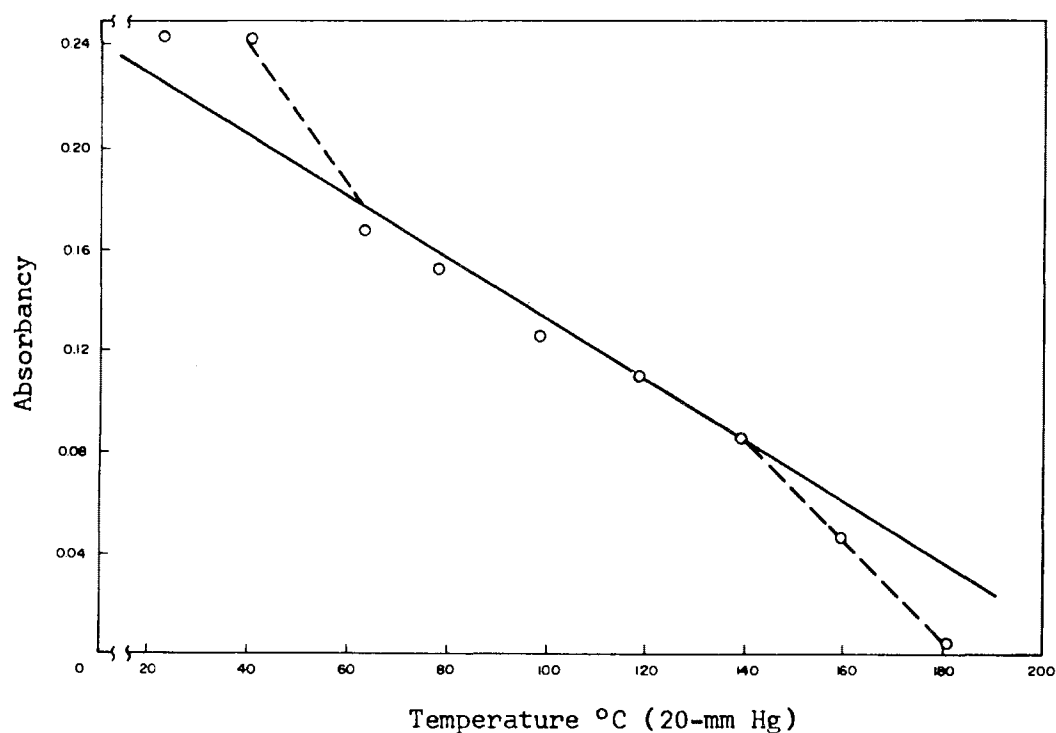


[Ref. 75-77]

Polymerization of 3,3', 4,4' - Benzophenonetetracarboxylic Dianhydride
and m - Phenylenediamine under various conditions.

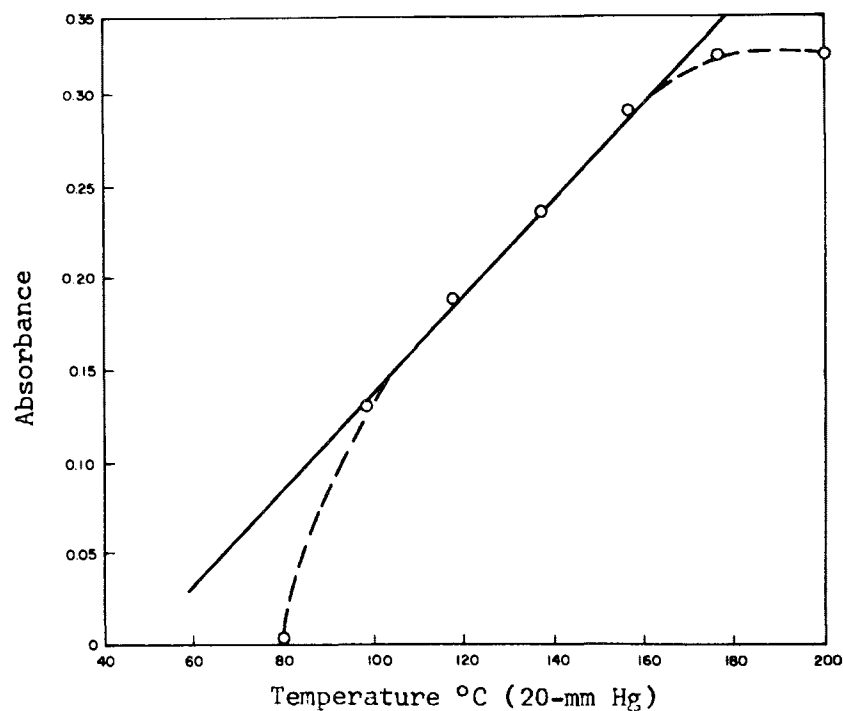
Sample No.	Reaction Conditions (Cumulative)		Infrared Absorption Bands, Relative Absorbance					Polymer Softening Temp, °C	Gel Time @ 300°C, sec	Remarks	Solubility in Pyridine @ RT
	Temp, °C	Time, min	3.05μ N-H	5.65μ (Imide Carbonyl)	5.8μ	6.55μ (Amine II Band)	7.35μ (Imide Ring)				
1	RT	5	0.244	0.000	0.174	0.284	0.125	78-101 125 140-145	-	No melt	Yes Yes Yes Yes (115°C)
	45	10	0.244	↓	↓	0.284	0.125		120	No melt	
		60	0.229			0.252	0.125		115	Semi-melt	
		120	0.229			0.232	0.143		110		
2	65	30	0.168	0.004	0.181	0.237	0.161	160 >	110		Yes
3	80	30	0.155	0.004	0.181	0.237	0.161	≈160	≈110		Yes
4	100	30	0.125	0.131	0.301	0.161	0.194	180 >	≈75	No melt	Partial
5	120	20	0.114	0.187	0.347	0.132	0.208	180 >	≈50		
6	140	20	0.086	0.237	0.456	0.102	0.347	205 >	≈50		
7	160	20	0.046	0.284	0.456	Weak	0.368	220 >	17		No
8	180	20	Weak	0.323	0.456	Weak	0.377	220-235	5		
9	200	20	Weak	0.323	--	Weak	0.377	No melt	No gel point		

[Ref. 75-77]



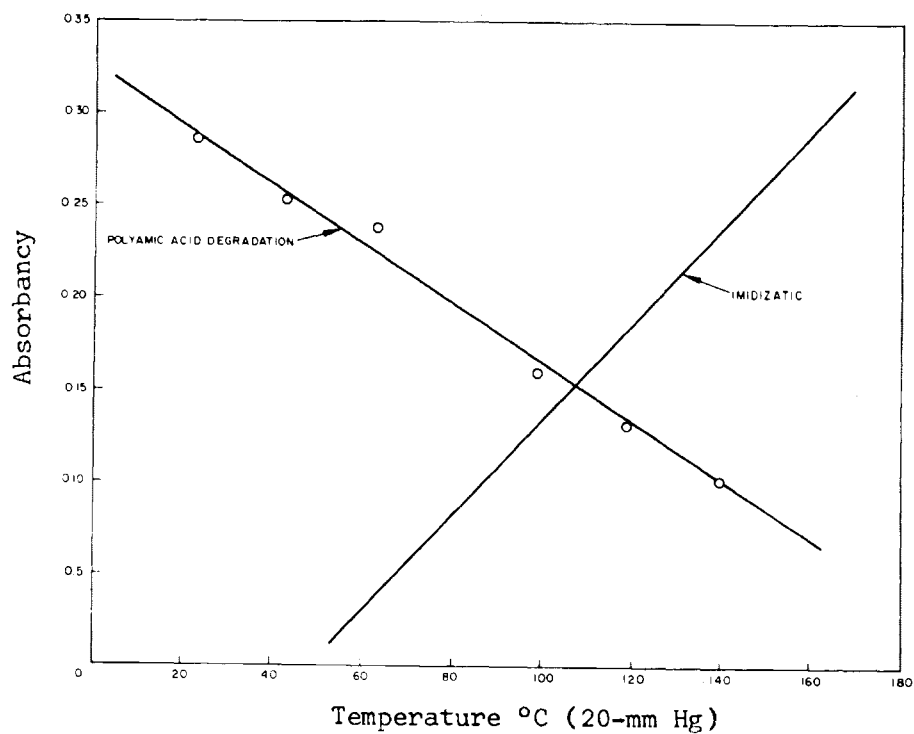
N-H (at 3.05 μ) absorbancy as a function of temperature of Poly (m-phenylene-3,3', 4,4' - benzophenonetetracarboxylimide).

[Ref. 75-77]



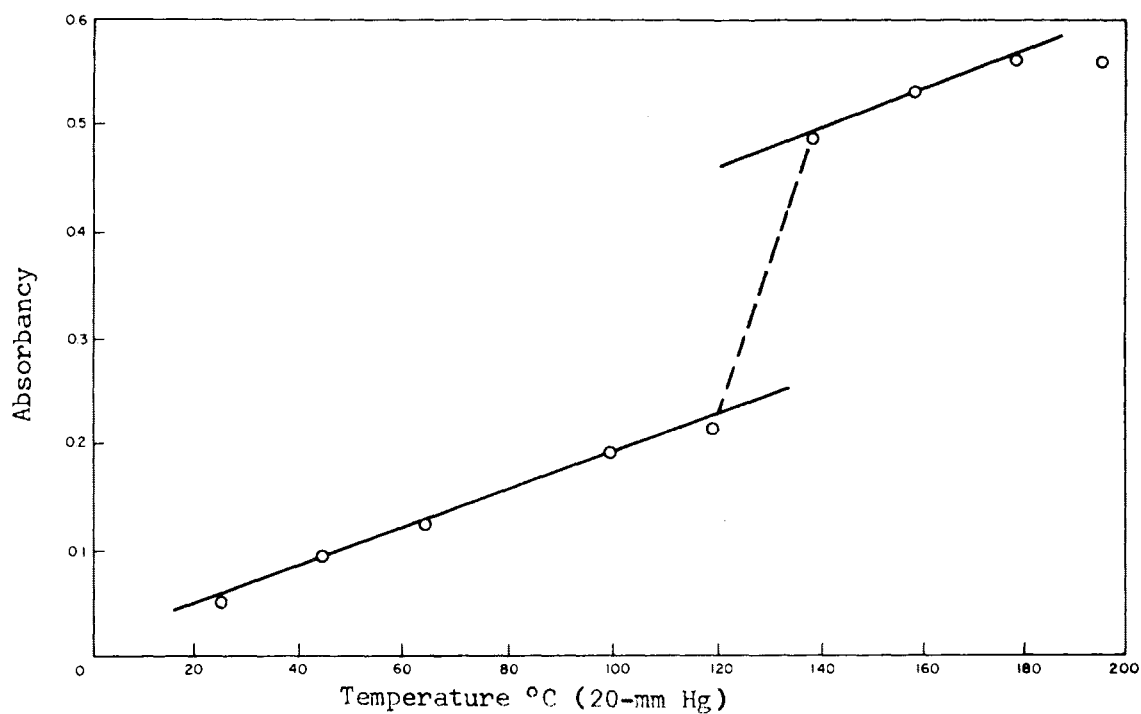
Imide carbonyl (at 5.6 μ) as a function of temperature of Poly (m-phenylene-3,3', 4,4' - benzophenonetetracarboxylimide).

[Ref. 75-77]



Amide II Band (at 6.55 μ) absorbance as a function of temperature of Poly (m-phenylene-3,3', 4,4' - benzophenonetetracarboxylimide).

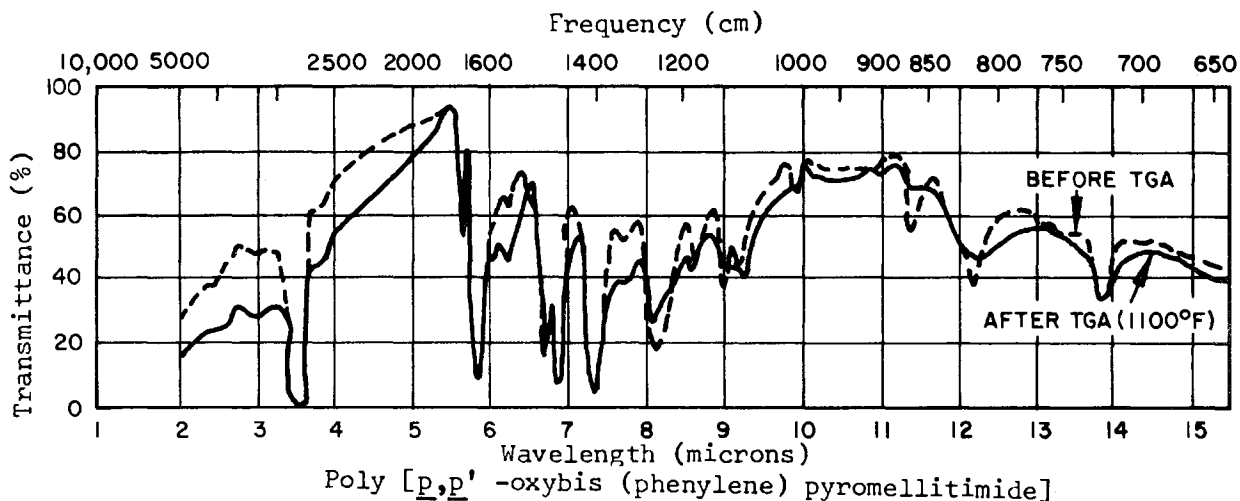
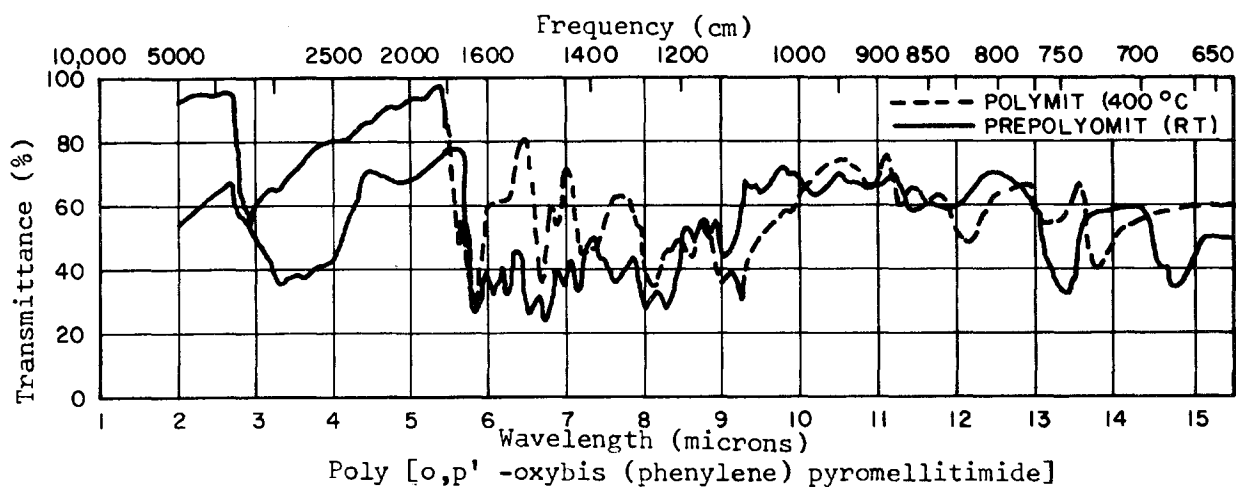
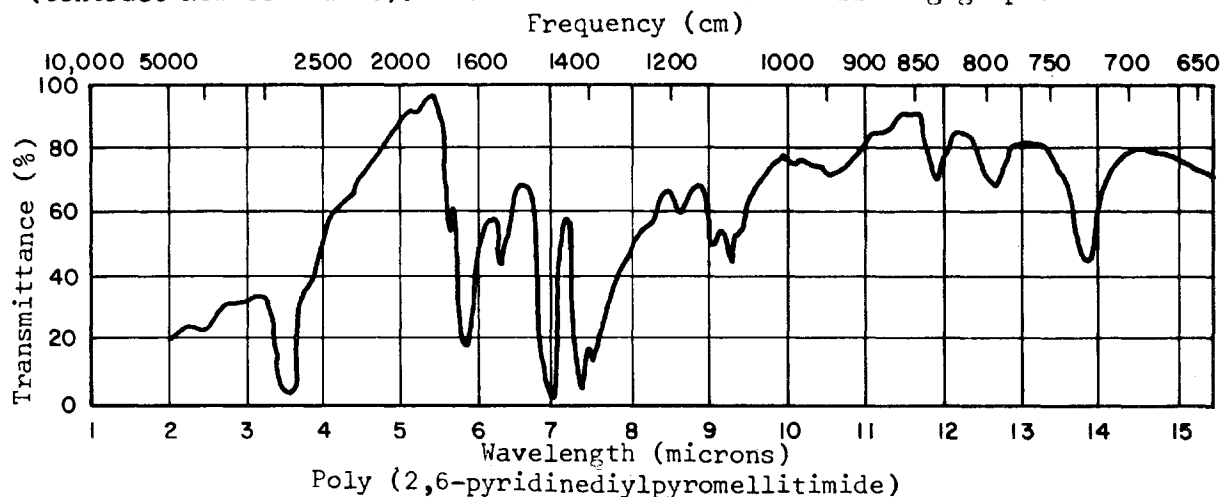
[Ref. 75-77]



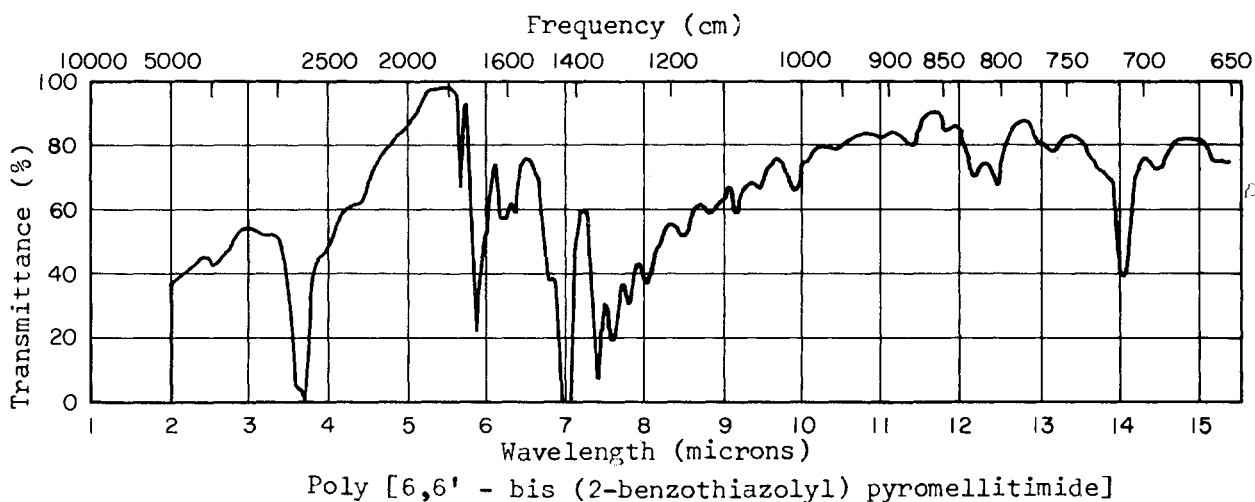
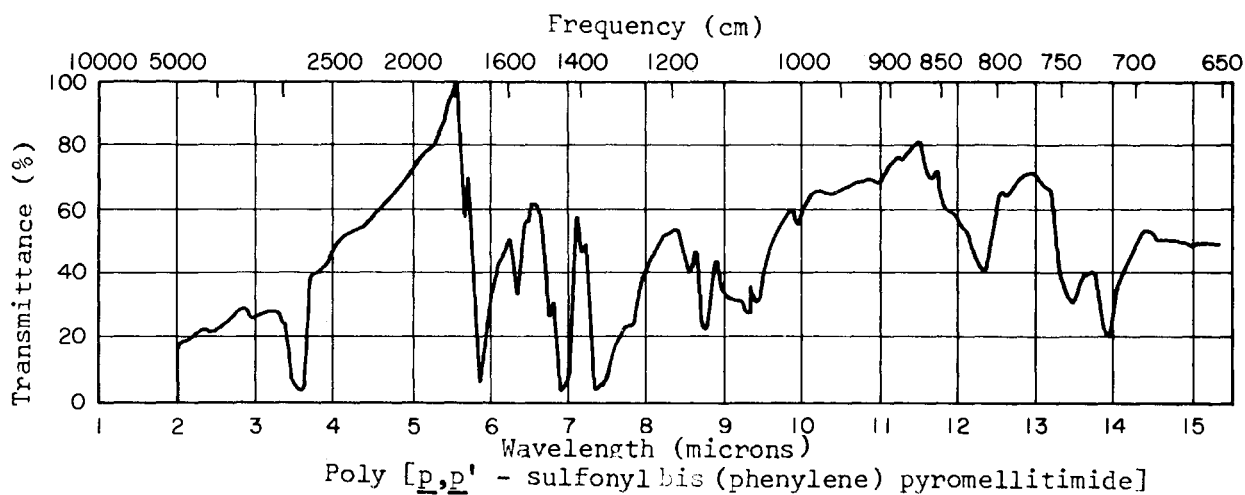
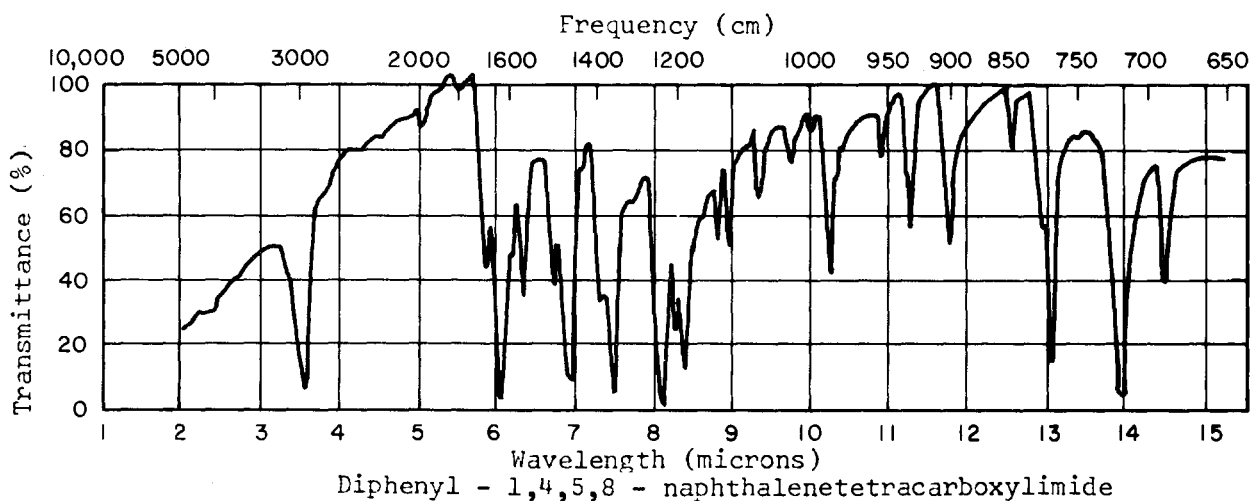
N-H (at 3.05 μ) absorbance as a function of temperature of Poly (m-phenylene-3,3', 4,4' - benzophenonetetracarboxylimide).

[Ref. 75-77]

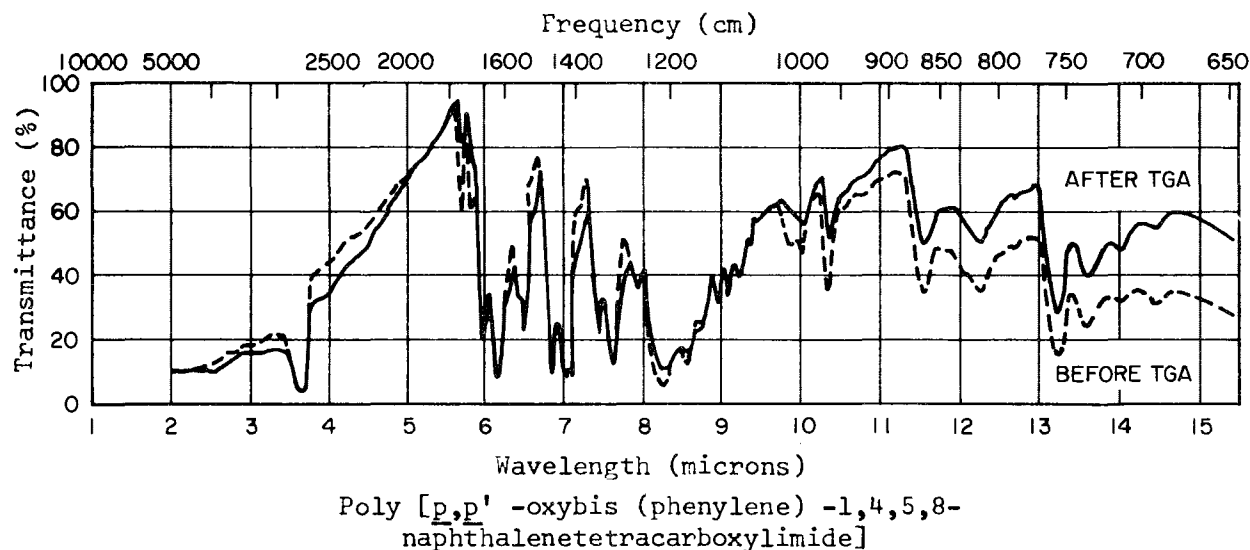
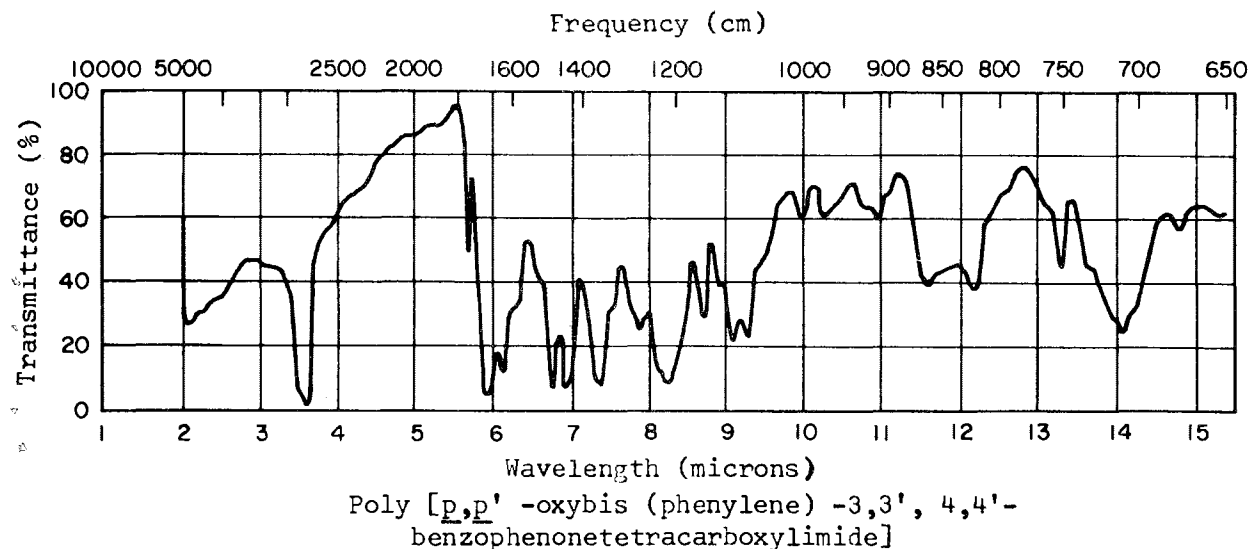
Narmco published a few infrared spectral curves of a number of reactants, model compounds, and polyimide polymers studied in their high-temperature structural adhesives contract for the Navy Department, Bureau of Weapons (Contract N0w-63-0420-C). These are shown in the following graphs:



[Ref. 75-77]



[Ref. 75-77]



[Ref. 75-77]

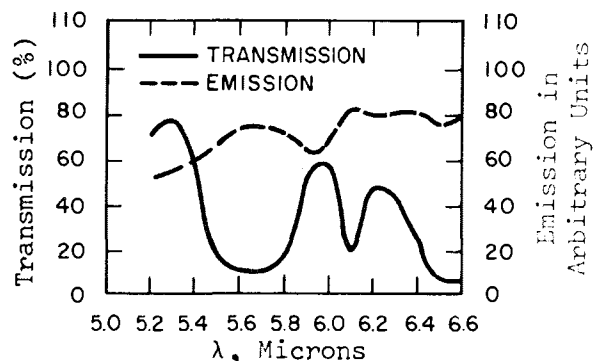
They are reported herein because of their origin and are not apt to be found in analytical chemical texts containing IR data.

Infrared absorption spectra can be used to monitor the polymerization stages in the preparation of polyamide-acid. The spectra initially show a predominating absorption band at ca. 3.1 microns due to the -NH bond. This band gradually disappears and as the reaction progresses, the polyamide absorption bands appear, a doublet at ca. 5.64 and 5.89 microns and a peak at 13.85 microns. The appearance of bands representing the -NH and -COOH groups serve to characterize the polyamide-acid formation.

Some of these polymerization reactions are discussed in detail in section I of this report.

Members of the General Electric Company Research Laboratories investigated the effects of corona on H-Film and examined the corona-damaged surface with infrared. No definite spectral differences could be noted. The sample showed a weak absorption due to amide groups, but this peak is not always apparent even in H-Film unexposed to corona.

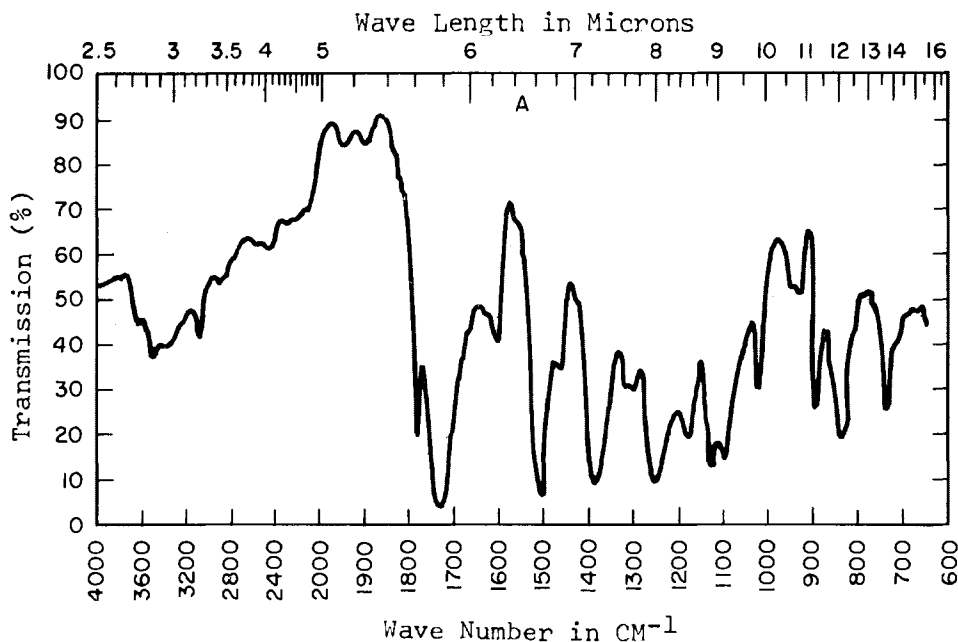
W.A. Hovis, Jr. at the Jet Propulsion Laboratory (California Institute of Technology) examined a 50-micron-thick DuPont H-Film for its infrared emission spectra in the region of 5 to 6.6 microns. The resulting emission spectrogram is shown below:



Emission and absorption of 50 μ H-Film epidermis.

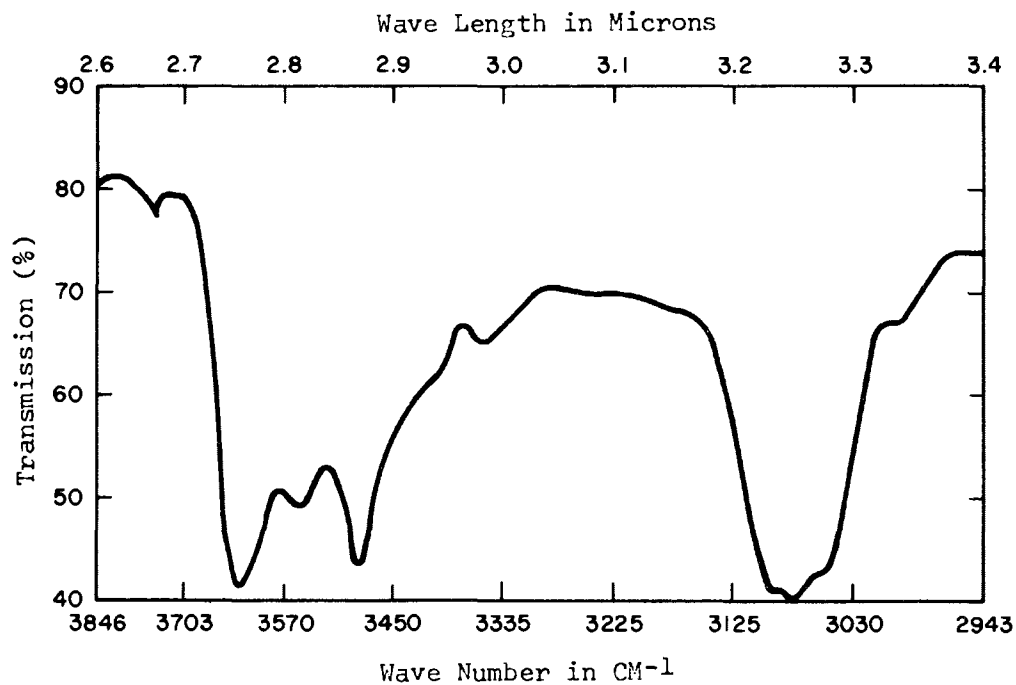
[Ref. 63]

In the course of studying the thermal degradation of commercial and purified H-Film in air and vacuum to obtain activation energy data, S.D. Bruck of the Applied Physics Laboratory, John Hopkins University, determined its infrared spectra from 2.5 to 16 microns plus a H-Film carbonized residue remaining after vacuum pyrolysis at 639°C for 20 hours (both spectra were obtained with KBr pellets). A discussion of the thermal degradation tests are discussed in the Thermal Properties section of this report:



Infrared absorption spectrum of Unpurified H-Film

[Ref. 12,13]



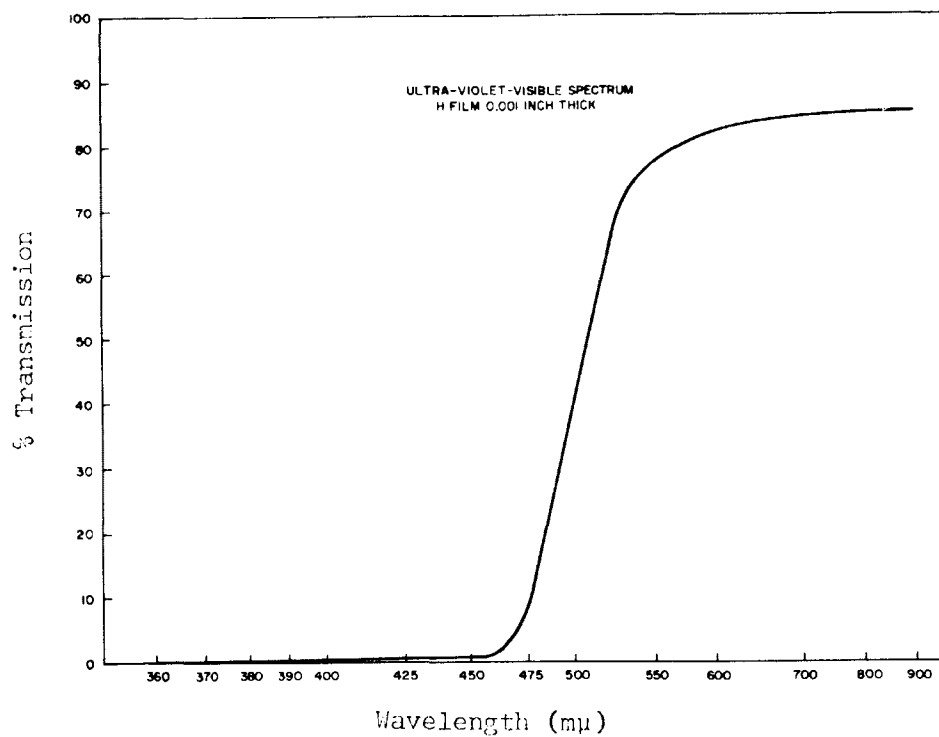
Infrared absorption spectrum of unpurified H-Film
between 2.6 and 3.4 μ (0.001 inch thick film, Beckman DK-2A)

[Ref. 13]

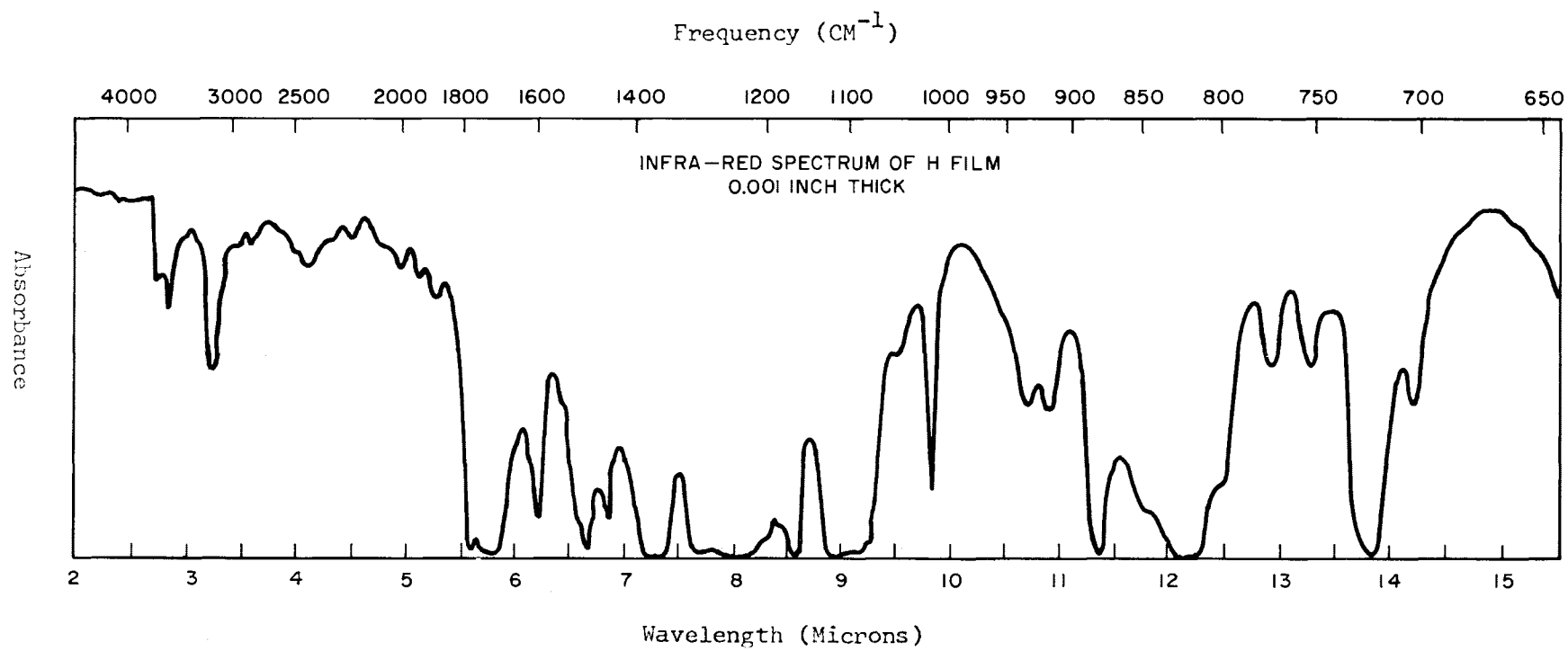
The DuPont Company report the following optical data on their H-Film:

Refractive Index (Becke Line), 25°C 1.78

[Ref. 29]

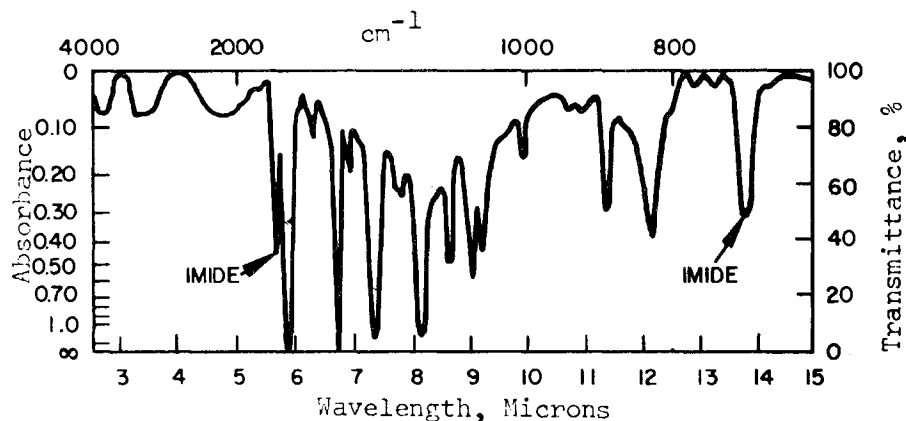


[Ref. 31]

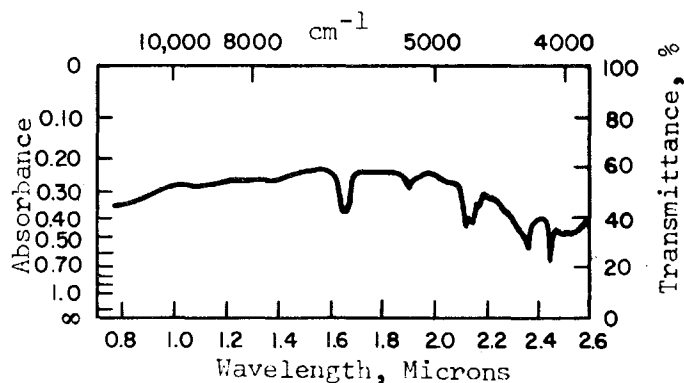


[Ref. 31]

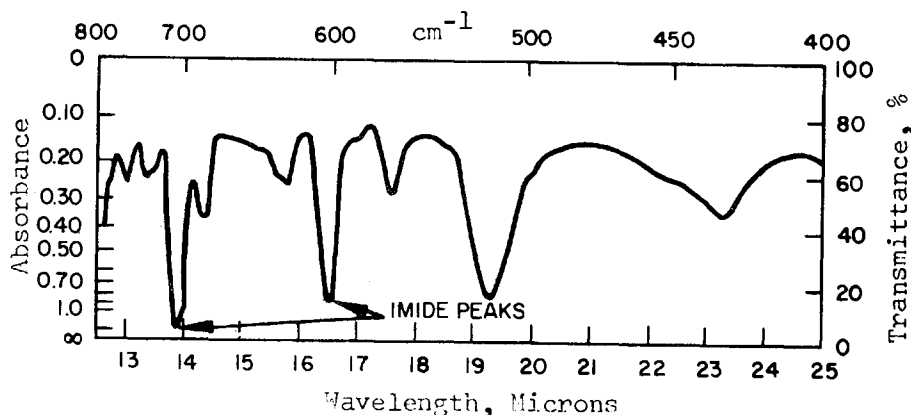
Scroog and co-workers at the DuPont Company in their comprehensive study of polyimide polymerizations made complete infrared spectra and ultraviolet spectra for poly [N,N'(p,p'-oxydiphenylene)pyromellitimide] polymers, some of which are shown in the following spectrograms:



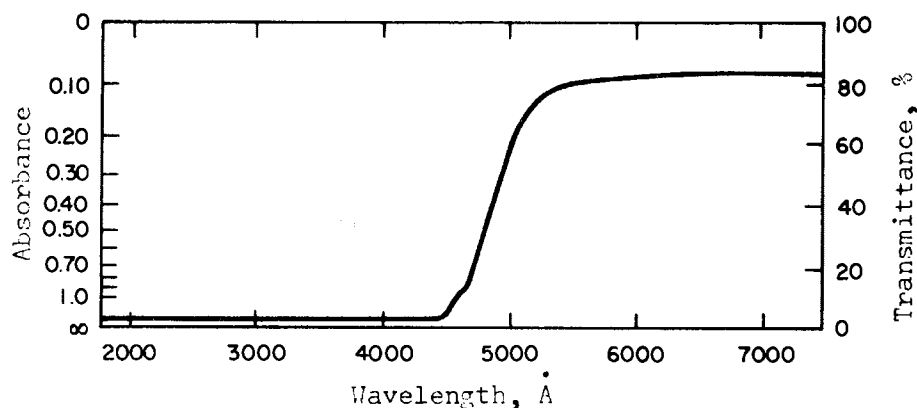
Infrared spectrum of 0.1 mil poly[N,N'(p,p'-oxydiphenylene)pyromellitimide](POP-PI) film. The film was heated to 300°C over a period of 45 minutes, then held at 300°C for 1 hour.



Near infrared spectrum of POP-PI film 14 mils thick, scan 50 Å/sec.



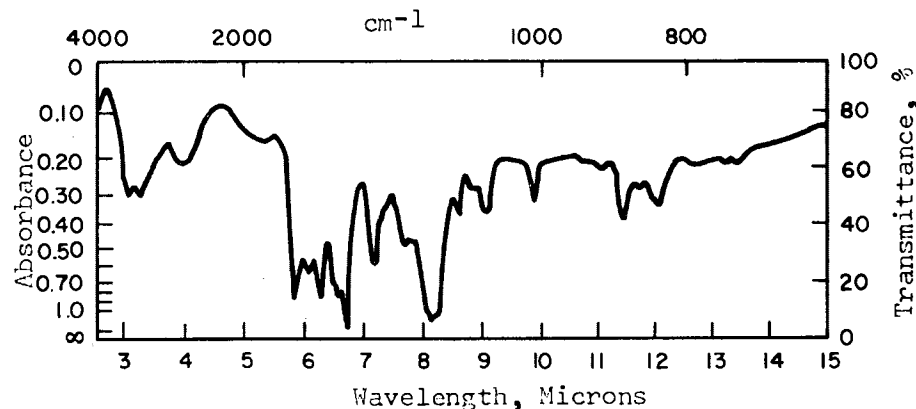
Far infrared spectrum of POP-PI film 0.5 mil thick.



Ultraviolet and visible spectrum of 0.1 mil POP-PI film.

These authors followed the polyimide cyclization spectrophotometrically by noting the disappearance of the N-H band (3.08 μ) and appearance of imide bands (5.63, 13.85 μ).

A characteristic infrared spectrum of a polyamic acid film from bis(4-aminophenyl)ether was also reported by these same authors:



Infrared spectrum of POP-PA film 0.1 mil thick,
dried 2 hours at 80°C.

From the above analytical data, it is apparent that the infrared characteristics of polyimides are now fairly well established. The N-H band shows a predominantly absorption band at 3.1 microns (3360 cm^{-1}) and a peak at 13.28 microns (720 cm^{-1}). When conversion is completed, the characteristic polyimide band predominates.

The Hughes Aircraft Company has evaluated several polyimide DuPont MQ enamels for high temperature coating applications. The following total emittance data were obtained:

<u>Sample Identification</u>	<u>Aging Condition</u>	<u>Total Emittance (ϵ 650°F)</u>
HAC #7 (MQ Resin W-87788)		0.70
HAC #7 (MQ Resin W-87788)	95 hours at 650°F*	0.65
HAC #8 (MQ Resin W-87788 + Carbon Filler)		0.84
HAC #8 (MQ Resin W-87788 + Carbon Filler)	95 hours at 650°F	0.81
HAC #8 (MQ Resin W-87788 + Carbon Filler)	190 hours at 650°F	Coating peeled off

* Coating peeled off at edges

[Ref. 105]

<u>Sample Identification</u>	<u>Aging Condition</u>	<u>a_{10}</u>	<u>ϵ_P</u>
Polyimide PI-2101 laminate	after vacuum exposure	0.75	0.87

Note: Solar absorptivity, a , is measured in an integrating sphere and represents a simulated solar distribution energy curve: a_{10} is obtained from 10 points. Emissivity, ϵ , is measured in a heated Hohlraum: the specimen is water cooled to maintain it at approximately room temperature. ϵ_P is a nine point integration of spectral values.

[Ref. 105]

Schweitzer reported the absorptivity in the visible and near infrared regions to range from 0.92 to 0.93 and the emissivity of about 0.9 from 104 to 662°F.

[Ref. 90]

The Research and Advanced Development Division of the Avco Corporation in their study of arc-heater characterization of ablative plastics reported the following total emittance data on various reinforced polyimide resins:

Specimen Code*	Emitted Radiation		Surface Brightness Temperature		True Surface Temperature		Total Emittance	
	$q_r^{(a)}$ (Btu/ft ² sec)	$q_r^{(b)}$ (Btu/ft ² -sec)	$T_B^{(c)}$ °R	$T_B^{(b)}$ °R	$T_T^{(c)}$ °R	$T_T^{(b)}$ °R	$\epsilon_T^{(c)}$	$\epsilon_T^{(b)}$
I8-35-C-1	698	655	6430	6290	6750	6950	0.71	0.589
I8-35-C-2	674	655	6430	6290	6750	6950	0.68	0.589
W-3-C-2	674	694	6450	6290	6770	6910	0.67	0.640
W-3-C-3	566	(1)	6370	(1)	6660	(1)	0.60	(1)
I8-35-R-1	154	(1)	5060	(1)	5210	(1)	0.44	(1)
I8-35-R-2	154	(1)	4840	(1)	4970	(1)	0.53	(1)
W-3-R-4	123	(1)	4920	(1)	5060	(1)	0.39	(1)
SP-1	550	549	(f)	5960	(f)	6130	(f)	0.83
SP-PT-2	266	(1)	5320	(1)	5530	(1)	0.59	(1)

*See Section I for chem. composition and molding conditions.

NOTES

- (a) Measured with an Eppley thermopile.
- (b) Based on intensities of infrared emission; brightness temperature given at 0.7 micron wavelength.
- (c) Measured with an IDL recording pyrometer (the accuracy is $\pm 50^\circ\text{F}$ at 5000°F) at 0.653 micron wavelength.
- (d) Spectrometer saturated for part of the spectrum.
- (e) Estimated - no valid data obtained; the specimens behaved in an erratic fashion.
- (f) Specimens did not ablate into the field of view of the IDL pyrometer.
- (g) The variation in emission spectra made only a 100°F difference in surface temperature so only the average value is reported.
- (h) Grey-body assumption was not justified for data of Note (b). Spectral emittance was assumed to be 0.85 to 0.95 in the 1.5 to 2.2 micron range.
- (j) Short run; insufficient time to obtain steady state data.
- (k) No values of emitted radiation or true temperature are reported because the wavelength range that was covered was small (between 1 and 2 microns)
- (l) No values because infrared emission spectra were erratic.
- (m) No brightness temperatures or emitted radiation estimated.
- (n) Malfunction of instrumentation.
- (o) Brightness temperature at 1.0 micron wavelength.

[Ref. 14]

Scroog and co-workers at DuPont Company in their extensive study of polyimide polymerization investigated the crystallinity of the resulting polymers by making Laue transmission x-ray diffraction patterns before and after various annealing heat treating conditions. Several of the polyimides were found to be crystalline, e.g., the polypyromellitimide from p-phenylenediamine. Others are more or less ordered, depending on their structure. Thus polypyromellitimides derived from m-phenylenediamine, bis(4-aminophenyl)methane, bis(4-aminophenyl)isopropylidene, bis(4-aminophenyl)sulfide, and bis(4-aminophenyl)ether normally exhibit a low degree of order as prepared. High temperature treatment will cause crystallization of film derived from m-phenylenediamine and of films based upon bis(4-aminophenyl)ether. Films, despite the slight weight loss upon heating noted previously, retain structural integrity for short exposures to 750 - 800°C in air; up to this temperature there appears, for the polymers studied, to be no evidence of crystal melting. These include the polypyromellitimide derived from bis(4-aminophenyl)ether, m-phenylenediamine, and p-phenylenediamine. No true second-order transition temperatures have been determined.

E. PHYSICAL

1. Density

<u>Specimen</u>	<u>Temp.</u>	<u>Density</u>	<u>Specific Gravity</u> <u>(ASTM D-1505-57T)</u>
H-Film	25°C	1.42 gms/cc	[Ref. 29]
Amoco AI (Type 10)			1.4 [Ref. 4]
DuPont Varnish RK-692		8.26 - 8.36 lbs/gal	[Ref. 35]
AI-220 Sterling Varnish (unmodified polyamide-imide)			1.045
Alvar-220 Sterling Varnish (modified polyamide-imide)	25°C		1.060 [Ref. 92]
"Pyre-ML" 10 mil wire enamel film	25°C		1.4
TFE Teflon/"Pyre-ML" 10 mil wire enamel film			2.1
FEP Teflon/"Pyre-ML" 10 mil wire enamel film	25°C		2.1
"Anamin" Film (Westinghouse Electric) polyamide-imide	25°C	1.40 gms/cc	[Ref. 66]

2. Viscosity

<u>Insulating Varnish</u>	<u>Solids Content</u>	<u>Viscosity</u>
DuPont RC-B-24951	45% ± 0.5	2.5 Poises
DuPont RK-692	13% ± 0.5	6-10
DuPont RC-5060	19% ± 1.0	5-9
		[Ref. 33]

<u>Insulating Varnish</u>	<u>Temp.</u>	<u>Brookfield Viscosity</u>
DuPont RK-692	25°C	625-1075 Centipoises
	↓	[Ref. 35]
Sterling AI-220		370 Centipoises
Sterling Alvar-220		490 Centipoises
		[Ref. 92]

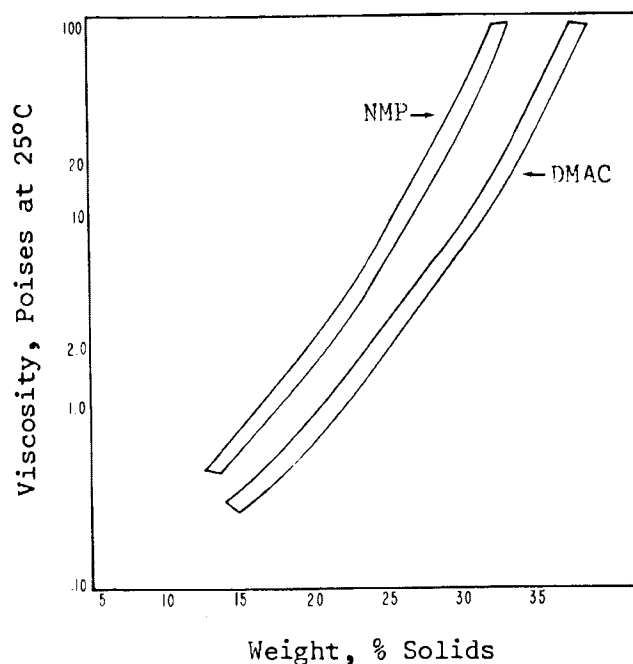
Stability of solution viscosities :

<u>Specimen</u>	<u>Hours at 100°C</u>	<u>Viscosity* (poises) at 25°C</u>	
		<u>DMAC</u>	<u>NMP</u>
Amoco AI Polymer	0	0.65	1.8
	2	0.78	2.0
	5	0.85	2.0
	20	0.65	1.5

* Brookfield

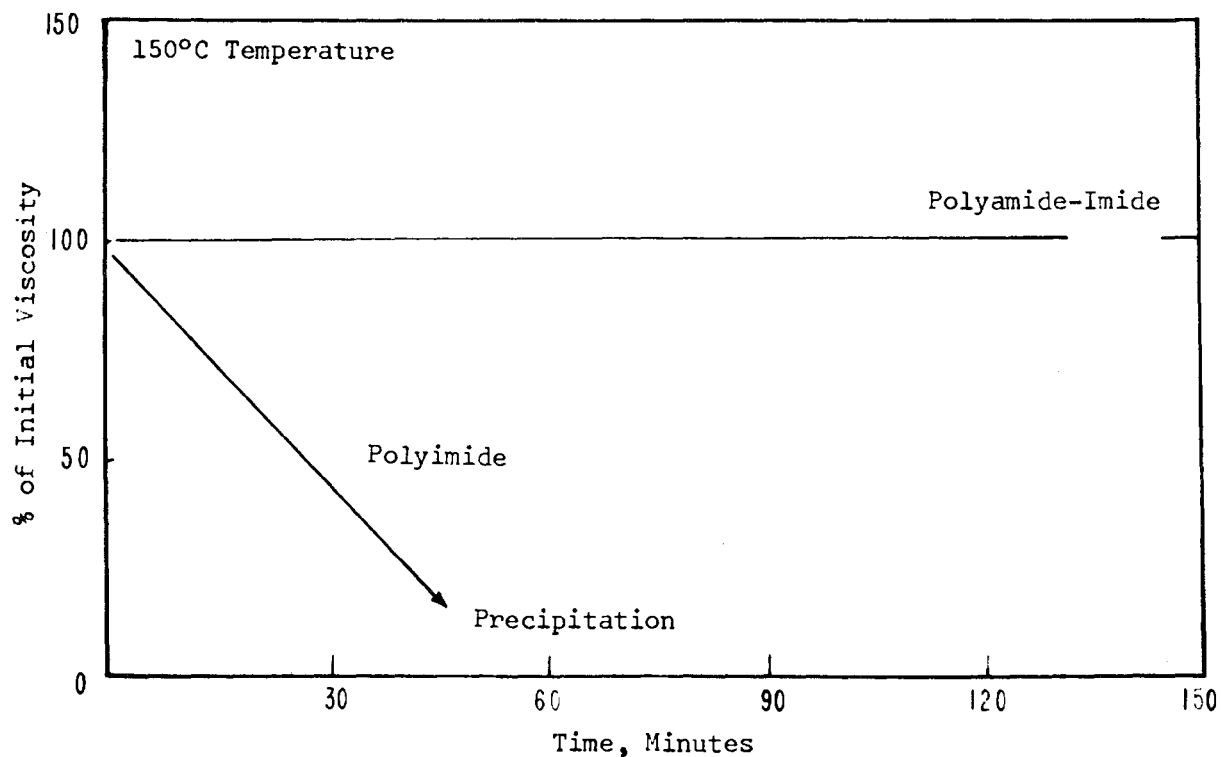
[Ref. 4]

Amoco Chemical Corporation report the following typical viscosity data for solutions of their Amoco AI Polymer (Type 10) in N-methyl pyrrolidone (NMP) and in dimethylacetamide (DMAC) solvents:



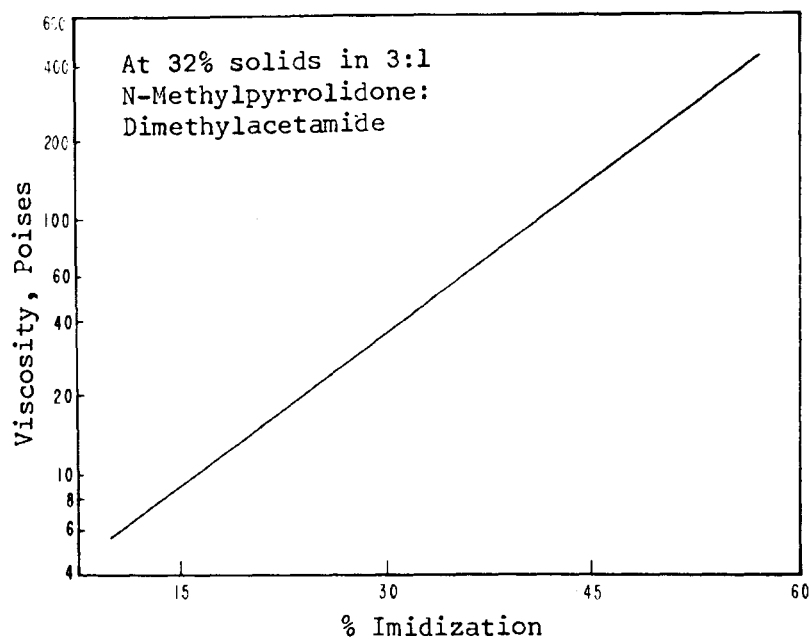
Solutions of Amoco AI Polymer (Type 10) show marked increases in viscosity with increasing resin solids.

[Ref. 4]



Solution stability in
NMP solvents of Amoco AI Polymer (Type 10)

[Ref. 104]



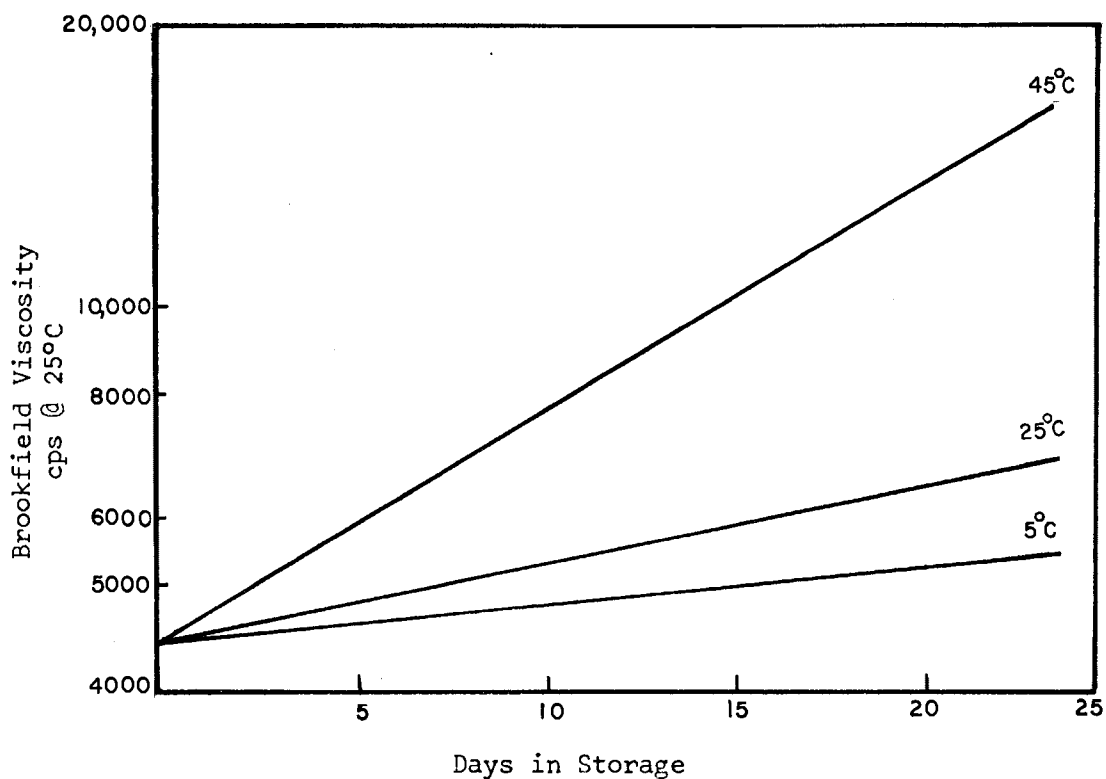
Viscosity - % imidization
relationship of Amoco AI Polymer (Type 10)

[Ref. 104]

Amoco AI Polymer (Type 10):

<u>Specimen</u>	Typical Viscosity, (at 32% solids) Poises
Dimethylformamide	4
Dimethylacetamide	14
Dimethylsulfoxide	20
N-Methylpyrrolidone	63
Dimethylacetamide/Xylene, 60/40	10
N-Methylpyrrolidone/Quinoline, 70/30	95
N-Methylpyrrolidone/Acetanilide, 70/30	280
Acetone	Insol.
Hydrocarbons	Insol.
Formamide	Insol.

[Ref. 104]



Storage characteristics of Shawinigan Polyimide
RS-5303 and RS-5305 Solutions.

[Ref. 112]

E. PHYSICAL

3. Hygroscopic Behavior

<u>Specimen</u>	<u>Thickness</u>	<u>Temp.</u>	<u>RH</u>	<u>Hygroscopic Coefficient of Expansion</u>
"Kapton" Type H-Film	1 mil	23°F	20-80%	2.2×10^{-5} in/in % RH

[Ref. 31]

<u>Specimen</u>	<u>Water Vapor Permeability</u>
"Kapton" Type H-Film	5.4 gm/ (100 square inch) (24 hours)
"Kapton" Type HF-Film	0.8 gm/ (100 square inch) (24 hours)

[Ref. 91]

4. Weathering Behavior

Todd and Wolff [Ref. 98] state that polyimides should not be used where there is a prolonged outdoor exposure. Samples exposed 6 months in Florida showed a degradation of both mechanical and electrical properties. However, the polymers do appear completely resistant to fungus attack. Polyimide parts have met the requirements of the federal specification CCC-T-191 b, Methods 5762 and 5751 for resistance to mildew and rot.

Appearance and Exterior Durability

Films of "Pyre-ML" are yellow to gold initially. This color over metals is attractive, simulating the appearance of brass.

The DuPont Company reports that resistance to discoloration (change from yellow) in the dark and in diffused sunlight has not been measured. A very slight lightening of the yellow color is observed after 1500 - 1600 hours in the weatherometer. A slight darkening of the film is observed when the film is baked at 400°C (752°F).

Further, accelerated weathering tests show that "Pyre-ML" varnish is very resistant even as a clear varnish to weathering. Its ability to absorb ultra-violet radiation also enables the varnish to protect sensitive substrates.

E. PHYSICAL

5. Gas Permeability

Bailey and co-workers at Lockheed-Georgia Company, Division of Lockheed Aircraft Corporation have measured and compared the hydrogen permeability through "Kapton" Type H-Film (0.00097 inch thick) over a range of temperatures (-140°F to 250°F) with other vapor barrier materials:

Hydrogen Permeability Values at Selected Temperatures

<u>Material</u>	Permeability, $\frac{\text{Std. cc.} - \text{cm.}}{\text{Sec.} - \text{cm.}^2 - \text{cm. Hg}} \times 10^{11}$						
	<u>200°F</u>	<u>150°F</u>	<u>100°F</u>	<u>50°F</u>	<u>0°F</u>	<u>-50°F</u>	<u>-100°F</u>
H-Film	--	58	29	14	5.4	1.7	0.17
Mylar	50	23	9.4	3.4	0.97	0.18	--
Tedlar	100	36	9.6	2.1	0.32	--	--
Polyethylene	--	--	97	25	4.7	0.75	0.05
KEL-F (KX 8105)	170	66	19	5.0	0.96	--	--
Teflon FEP	--	300	135	52	14	2.4	0.15
KEL-F (KX 8205)	172	61	19	5.2	1.1	0.13	--

[Ref. 111]

Activation Energies and Constants of Permeation

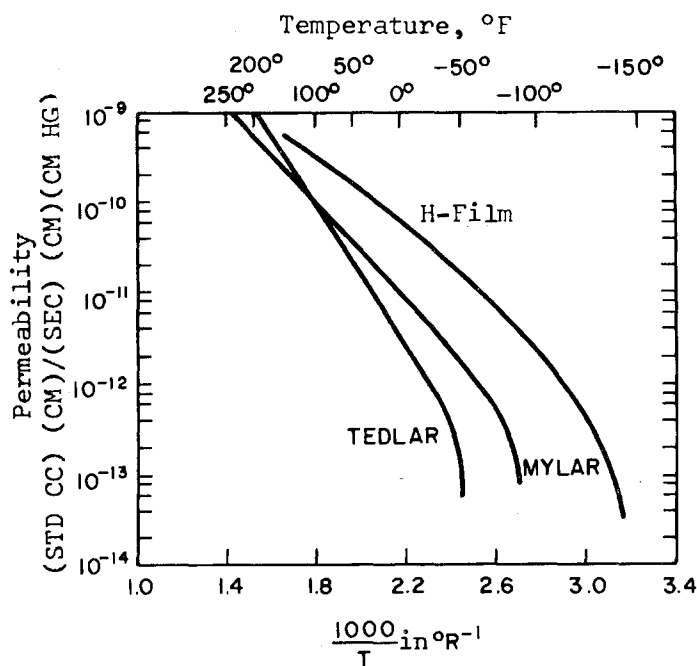
<u>Material</u>	<u>Temp.</u> <u>°F</u>	Activation Energy of Permeation, <u>Kcal/Gm. Mole</u>	Constant of Permeation, P_0 <u>std. cc. - cm.</u>
			<u>sec. - cm.² - cm. Hg</u>
H-Film	142 to -60*	4.9	0.08×10^{-5}
	-90	8.6	-
	-120	8.6	-
	-140	35.5	-
Mylar	254 to -43*	6.7	0.47×10^{-5}
	-60	10.4	-
	-80	14.3	-
	-85	28.2	-
Tedlar	200 to 6*	9.7	64.60×10^{-5}
	-37	16.8	-
Polyethylene	200 to 16*	8.5	20.82×10^{-5}
	16 to -63	7.6	15.04×10^{-5}
	-85	9.5	-
	-110	15.5	-

[Ref. 111]

Activation Energies and Constants of Permeation
(continued)

Material	Temp. °F	Activation Energy of Permeation, Kcal/gm. Mole	Constant of Permeation, P_0 Std. cc. - cm. sec. - cm. ² - cm. Hg
KEL-F (KX 8105)	230 to 23*	8.7	24.07×10^{-5}
	0	10.0	-
	-25	11.0	-
	-47	122.0	-
Teflon FEP	140	5.7	-
	80	6.3	-
	10	7.3	-
	-60	8.7	-
	-125	26.2	-
KEL-F (KX 8205)	230 to -43*	6.4	1.11×10^{-5}
	-60	15.3	-
	-70	48.0	-

* The asterisk denotes a linear section of the permeability curve. [Ref. 111]



Hydrogen permeability of H-Film, Mylar and Tedlar.

[Ref. 111]

PROPERTIES

F. THERMAL

1. Thermal Stability

A fairly extensive evaluation of the various forms of the polyimides have shown them all to possess a high thermal stability due to their aromatic cyclic or ring structure. Tests to date have included thermal aging and thermogravimetric analyses in air, inert gases and vacuum. DuPont workers in their evaluation of H-Film found that the rate of degradation of the polymer was dependent on the availability of oxygen. They found that the average lifetime at 450°C was 2 hours in air and 22 hours in helium; at 350°C it was six days and 1 year, respectively.

S.D. Bruck of the Applied Physics Laboratory at the John Hopkins University has made perhaps the most thorough study of the thermal degradation of the polyimides both in air and in vacuum. His studies included the effect of impurities and the nature of the degradation products. Purification of the polymer (H-Film) in dimethylformamide decreases the rates of degradation both in air and in vacuum, but only slightly reduces the activation energies. Infrared and mass spectrographic data suggest the participation of polypyromellitic acid (polyamide acid) impurity in the degradation process by thermal cleavage of its carboxyl and hydrolytic scission of its amide groups. He also found that as far as the vacuum pyrolysis of the main polyimide chains is concerned, the activation energy of 74 kcal/mole, bond dissociation, and elemental analysis of the residue remaining after pyrolysis suggest that the primary scission occurs at the imide bonds, most likely followed by a secondary cleavage resulting in the elimination of CO groups.

Todd and Wolff report that fabricated SP Polymer polyimide parts can be used continuously in air at temperature of 500°F. In an inert atmosphere allowable continuous exposure temperature increases to 600°F. For intermittent or short-term exposure, temperatures to 930°F are possible. The material does not melt but chars slowly at excessive temperatures according to Campbell and Cheney. These same authors say that, even after several hours at 900°F, it is still in a condition which is adequate for lightly stressed applications. Thermal degradation is even slower in the absence of oxygen, so that longer exposure times at high temperatures are possible in inert atmospheres or in vacuum.

2. Melting Point

Specimen

Temperature

"Kapton" Type H-Film

(no definite melting point)

[Ref. 31]

"Anamin" Film (Westinghouse Electric)
Polyamide-Imide

400°C (melting point)
265°C (glass transition)

[Ref. 44]

<u>Specimen</u>	<u>Temperature</u>
"Pyre-ML"	932°F (melting point)
Teflon TFE/"Pyre-ML" Composite	620°F (gel point of TFE)
Teflon FEP/"Pyre-ML" Composite	545°F - 563°F (gel point of FEP)

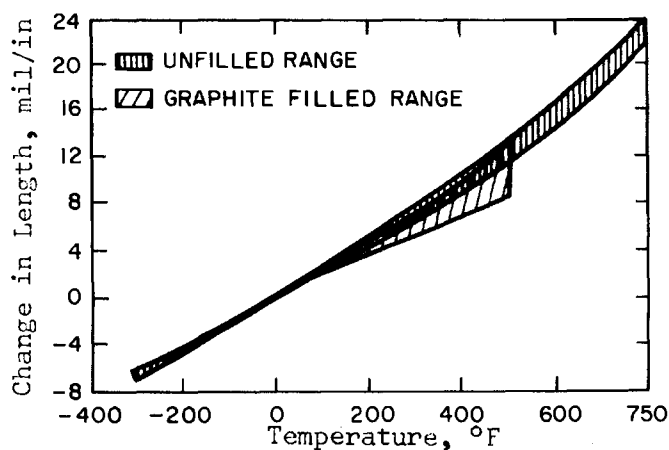
[Ref. 66]

3. Thermal Expansion

<u>Specimen</u>	<u>Temperature</u>	<u>Thermal Expansion</u>
SP-Polymer	-300° to +500°F	28.4×10^{-6} in/in/°F

[Ref. 19]

Todd and Wolff have noted that orientation during fabrication has a pronounced effect on the thermal expansion of the polyimides as reflected in the following graph:



Thermal expansion of polyimides is intermediate between that of metals and other plastics. Range shown indicates the effects of polymer orientation on expansion.

[Ref. 98]

<u>Specimen</u>	<u>Thermal Expansion</u>
"Pyre-ML" Varnish	$4.0 \times 10^{-5}/^{\circ}\text{C}$
DuPont RK-692 Varnish ("Pyre-ML")	$4.0 \times 10^{-5}/^{\circ}\text{C}$
	[Ref. 35]
"Pyre-ML" Coatings	$2 \times 10^{-5}/^{\circ}\text{C}$
	[Ref. 90]

"Kapton" Type H-Film

<u>Thickness</u>	<u>Temperature Range</u>	<u>Coefficient of Thermal Expansion</u> <u>in/in² °C x 10⁵</u>			
		<u>Annealed Film</u>		<u>Untreated Film</u>	
		<u>MD*</u>	<u>TD**</u>	<u>MD*</u>	<u>TD**</u>
(Annealed film has been exposed to 400°C for 30 minutes)					
1 mil	23-100°C	1.75	1.85	0.09	--
	100-200°C	3.32	2.90	-0.58	--
	200-300°C	4.92	4.82	--	--
	300-400°C	7.61	7.91	--	--
	23-400°C	4.52	4.52	--	--
2 mil	23-100°C	.75	1.30	.95	1.70
	100-200°C	2.37	3.31	1.19	2.82
	200-300°C	3.35	4.54	1.20	.46
	300-400°C	4.94	6.63	3.92	1.38
	23-400°C	2.99	4.13	2.14	.85
(Annealed film has been exposed to 400°C for 2 hours.)					
5 mil	23-100°C	1.00	2.32	1.36	3.03
	100-200°C	2.19	3.41	2.10	4.10
	200-300°C	3.01	4.82	1.98	4.78
	300-400°C	3.79	5.90	.25	2.93
	23-400°C	2.59	4.22	1.43	3.76

* MD=Machine Direction

** TD=Transverse Direction

[Ref. 31]

4. Thermal Conductivity

"Kapton" Type H-Film: measured on composite structure ~ 100 mils thick in order to establish equilibrium.

<u>Temperature</u>	<u>Thermal Conductivity</u>		<u>Method</u>
	<u>(cal) (cm)</u>	<u>(cm)² (sec) °C</u>	
25°C	3.72 x 10 ⁻⁴		Dynatech
63°C	3.58 x 10 ⁻⁴		Cenco Fitch
75°C	3.88 x 10 ⁻⁴		Dynatech
200°C	4.26 x 10 ⁻⁴		Dynatech
300°C	4.51 x 10 ⁻⁴		Dynatech

[Ref. 31]

<u>Specimen</u>	<u>Thermal Conductivity</u>
"Pyre-ML"	35×10^{-5} cal-cm/sec-cm ² -°C [Ref. 90]
"Pyre-ML" Varnish	75×10^{-5} cal/sec/cm ² /°C/cm [Ref. 36]
SP-Polymer (unfilled)	2.6 - 3.0 Btu/hr/ft ² /°F/in [Ref. 98]
SP-Polymer	2.20 Btu-in/ft ² -hr-°F [Ref. 98]
DuPont Polyimide Foam (Density = 2 - 30 lbs/ft ³ , 5 - 100 mils thick)	0.25 - 0.38 Btu/ft ² /in/°F/hr [Ref. 28]

5. Specific Heat

<u>Specimen</u>	<u>Specific Heat</u>
SP-Polymer (unfilled) ASTM D-648	0.27 Btu/lb-°F [Ref. 97]
SP-Polymer (122°F)	0.75 Btu/lb/°F [Ref. 98]

"Kapton" Type H-Film

<u>Temperature</u>	<u>Film Thickness</u>	<u>Specific Heat (cal/gm °C)</u>
40°C	1 mil	0.261
	5 mil	0.261

[Ref. 31]

6. Thermal Degradation and Weight Loss

<u>Insulating Varnish</u>	<u>Solids Content</u>	<u>Viscosity (poises)</u>	<u>Time (hours)</u>	<u>Temp. (°C)</u>	<u>Weight Loss (%)</u>
DuPont RC-B-24951	45%	2-5	50	300	5
			150	300	7
			3.3	400	12
			3.3	450	31
DuPont RK-692	13%	6-10	50	300	1
			150	300	1
			3.3	400	11
			3.3	450	31
DuPont RC-5060	19%	5-9	50	300	1
			3.3	400	12
			3.3	450	35

[Ref. 33]

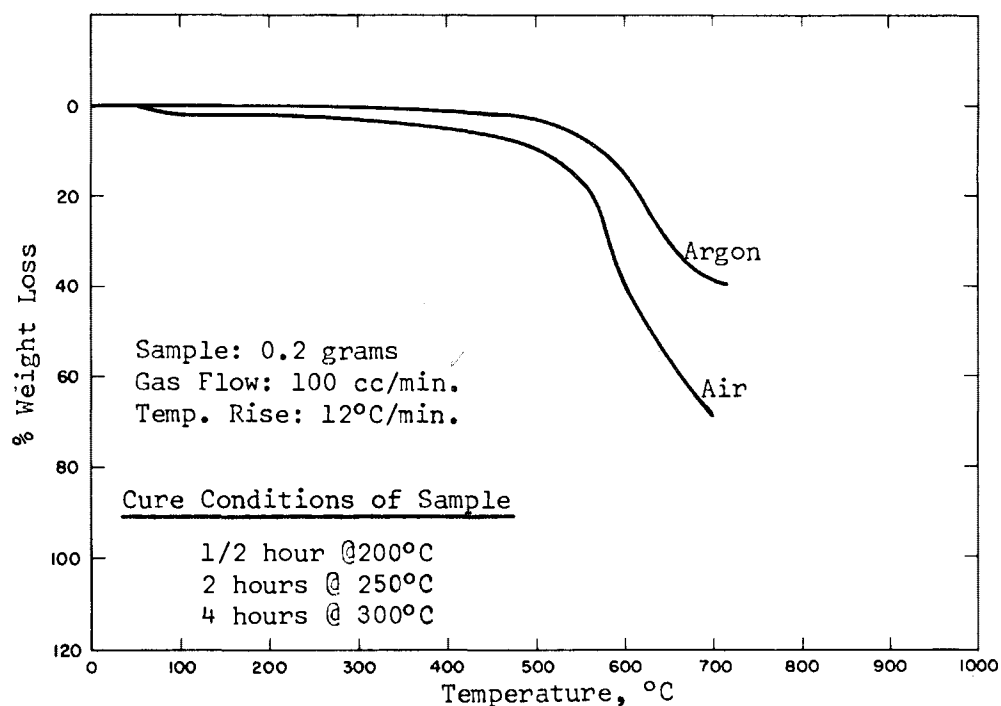
Sterling AI-220 (on glass cloth)	40 days	220	5
Alvar 220 [Sterling] (on glass cloth)	40 days	220	15

[Ref. 92]

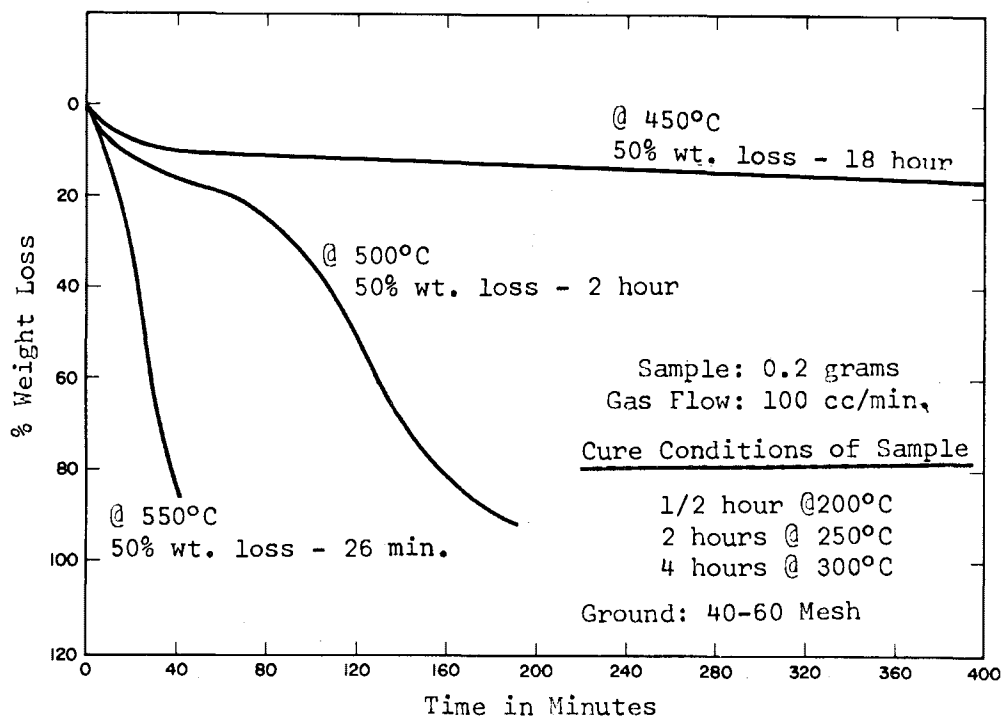
<u>Binder Solution</u>	<u>Solid Content</u>	<u>Solvent System</u>	<u>Viscosity (poises)</u>	<u>Temp. (°F)</u>	<u>Time</u>	<u>Weight Loss</u>
"Pyre-ML" (Resin)	16.5%	NMP/DMAC	70	572	50 hrs.	1%
				752	200 min.	11%
				842	200 min.	20%
"Pyre-ML" (Resin)	25%	DMF	35	572	50 hrs.	1%
				752	200 min.	12%
				842	200 min.	35%
"Pyre-ML" (Resin)	25%	DMF	8	572	50 hrs.	4%
				742	200 min.	11%
				842	200 min.	46%
"Pyre-ML" (Resin)	60%	NMP/Xylene	80	572	50 hrs	5%
				752	200 min.	12%
				842	200 min.	31%
[Ref. 90]						
"Pyre-ML" (RK-692)				572	3 wks.	1%
[Ref. 35]						
DuPont PI-1100	18.6%	DMAC	30-35	572	500 hrs.	1%
				752	200 min.	12%
				842	200 min.	35%
DuPont PI-1101	18.3%	DMAC	10-20	572	500 hrs.	4%
				752	200 min.	12%
				842	200 min.	45%
DuPont PI-2101				572	500 hrs.	4%
				752	200 min.	11%
				842	200 min.	46%
DuPont PI-3301	53%	NMP/Xylene	60-90	572	500 hrs.	5%
				752	200 min.	12%
				842	200 min.	31%
DuPont ML				572	50 hrs.	1%
				752	200 min.	11%
				842	200 min.	20%
DuPont PI-2100				572	50 hrs.	1%
				752	200 min.	12%
				842	200 min.	20%
DuPont PI-2101				572	50 hrs.	4%
				752	200 min.	11%
				842	200 min.	46%
DuPont PI-3101				572	50 hrs.	5%
				752	200 min.	12%
				842	200 min.	31%

[Ref. 31]

The Shawinigan Resins Corporation made a thermal gravimetric analysis of their polyimide RS-5303 and RS-5305 resin solutions in air and in argon and over a wide range of temperatures:



[Ref. 112]



[Ref. 112]

<u>Specimen</u>	<u>Temp. (°F)</u>	<u>Time (hour)</u>	<u>Weight Loss (%)</u>
Polyimide coating formula #13, DuPont RCW62480 polyimide + non-leafing Al pigment, P/B ratio = 200/100, DMAC solvent, on stainless steel.	800 ↓	1	-2.69
		3	0.77
		5	8.20
		10	15.0
		25	20.1
		50	6.02 (?)
		100	29.7
	1000 ↓	1	13.96
		3	15.45
		5	20.68
		10	14.48
		25	8.25
		50	9.36
		100	8.67
Polyimide coating formula #22, Poly- pyromellitimide PPMI + non-leafing Al pigment, P/B ratio = 200/100, DMAC solvent, on stainless steel.	800 ↓	1	-6.19
		3	-6.25
		5	-6.46
		10	-8.68
		25	34.7
		50	34.7
		100	50.2
	1000 ↓	1	27.4
		3	32.87
		5	39.5
		10	37.8
		25	27.9
		50	17.9
		100	25.2
Polyimide coating formula #25, DuPont RCW62480 + leafing Al pigment, P/B ratio = 200/100, DMAC solvent, on stainless steel.	800 ↓	1	9.04
		3	10.49
		5	17.11
		10	20.39
		25	28.8
		50	12.8 (?)
		100	35.2
	1000 ↓	1	11.32
		3	13.09
		5	14.63
		10	15.8
		25	8.42
		50	11.83
		100	14.48

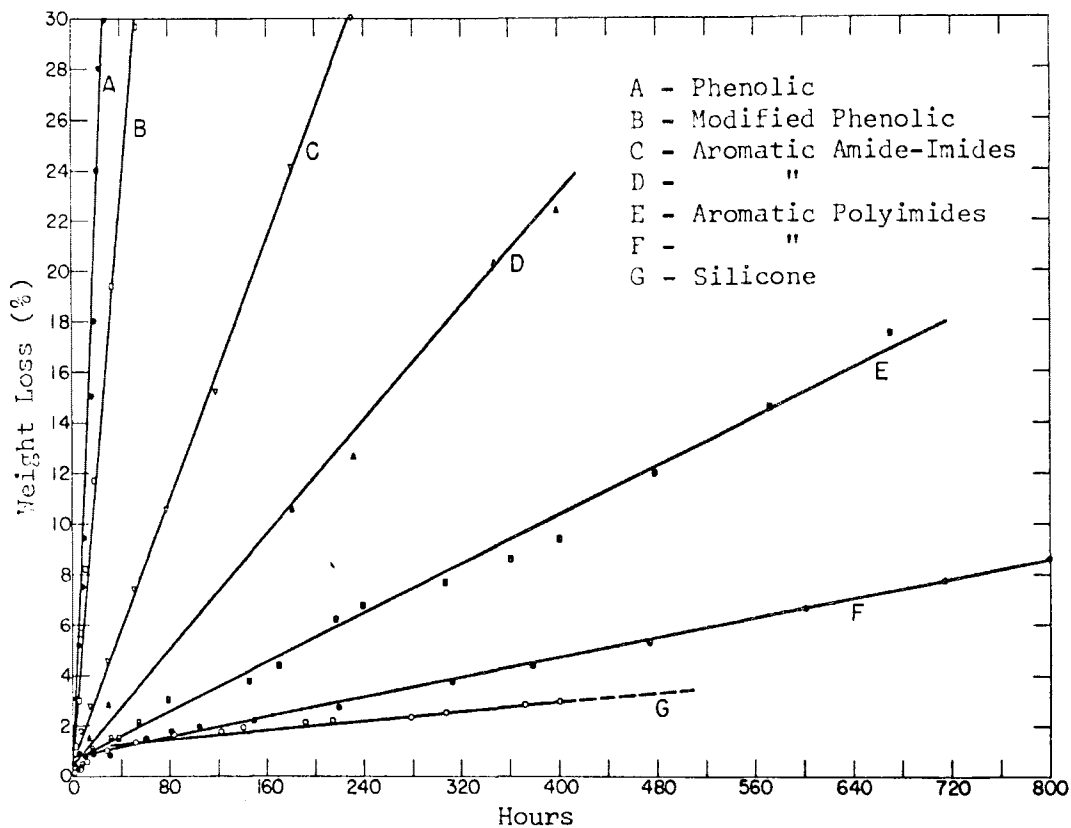
[Ref. 1]

<u>Specimen</u>	<u>Temp. (°F)</u>	<u>Time (hour)</u>	<u>Weight Loss (%)</u>
Polyimide coating formula #34, Poly- pyromellitimide PPMI + leafing AI pigment, P/B ratio = 200/100, DMAC solvent, on stainless steel.	800 ↓	1	0
		3	1.90
		5	6.72
		10	21.6
		25	37.1
		50	38.6
		100	54.2
	1000 ↓	1	27.9
		3	33.17
		5	47.1
		10	37.6
		25	28.4
		50	32.5
		100	31.3
DuPont Polyimide RCW62480 (0.5089 gram sample)	800 ↓	1	7.40
		3	16.76
		5	18.17
		10	84.0
(0.7676 gram sample)	1000 ↓	1	99.5
		3	*
Polypyromellitimide PPMI (0.3695 gram sample)	800 ↓	1	0.406
		3	1.136
		5	1.57
		10	6.03
(0.4845 gram sample)	1000 ↓	1	91.7
		3	*

* Completely gone after 3 hours.

[Ref. 1]

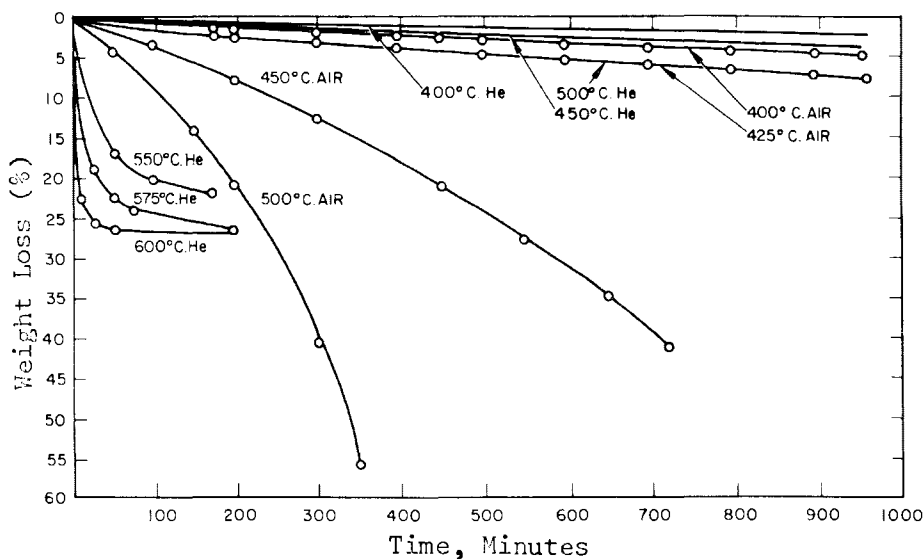
Freeman et al made weight loss measurements on various polyimide and polyamide-imides in thin film (1 - 4 mils thickness) on aluminum dishes and compared them with a number of other polymers. The weight loss measurements were made by aging samples in a circulation air oven at $320 \pm 2^\circ\text{C}$ (608°F):



Comparative percent weight loss as a function of time of thin films in air at 320°C.

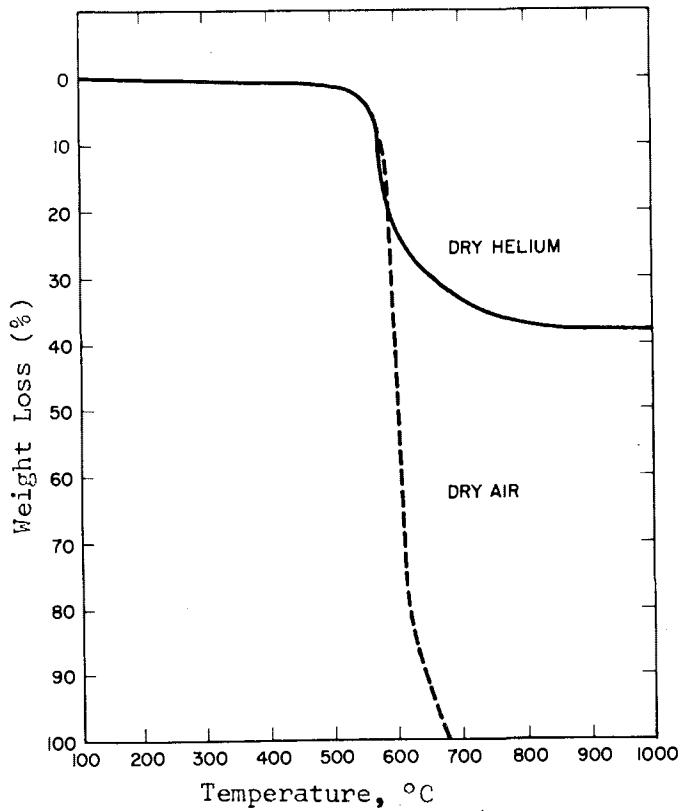
[Ref. 49]

The DuPont Company has made weight loss measurements on their "Kapton" Type H-Film over a wide range of temperatures and in various environments:



Isothermal weight loss of "Kapton" Type H-Film in air and in Helium.

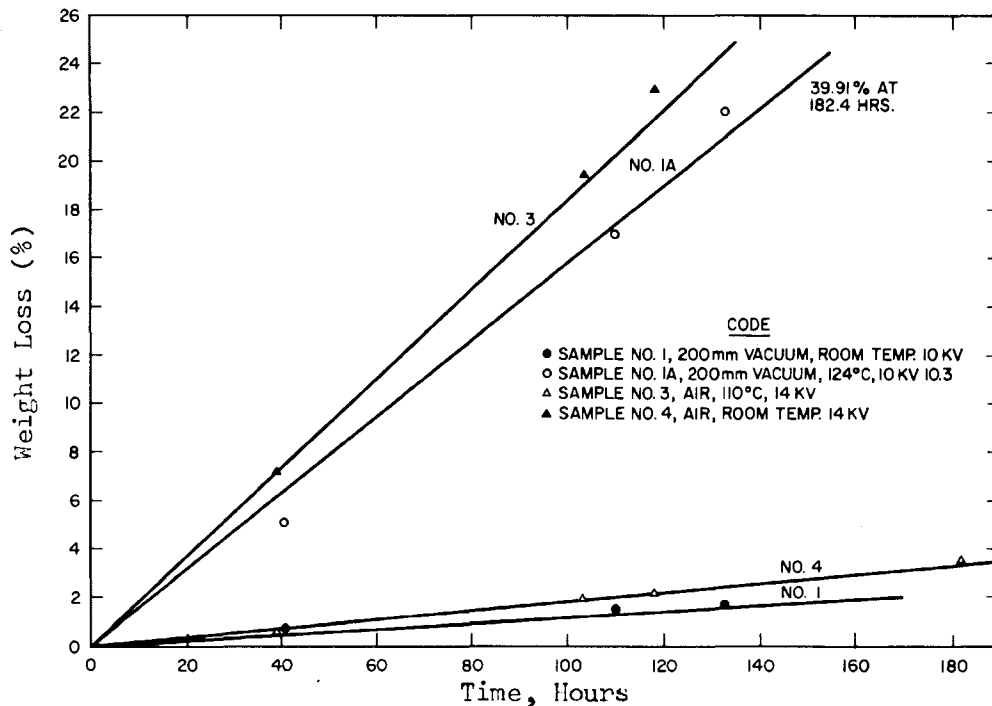
[Ref. 31]



Effect of Environment on the Weight Loss of "Kapton" Type H-Film during constant temperature rise (3°C/minute).

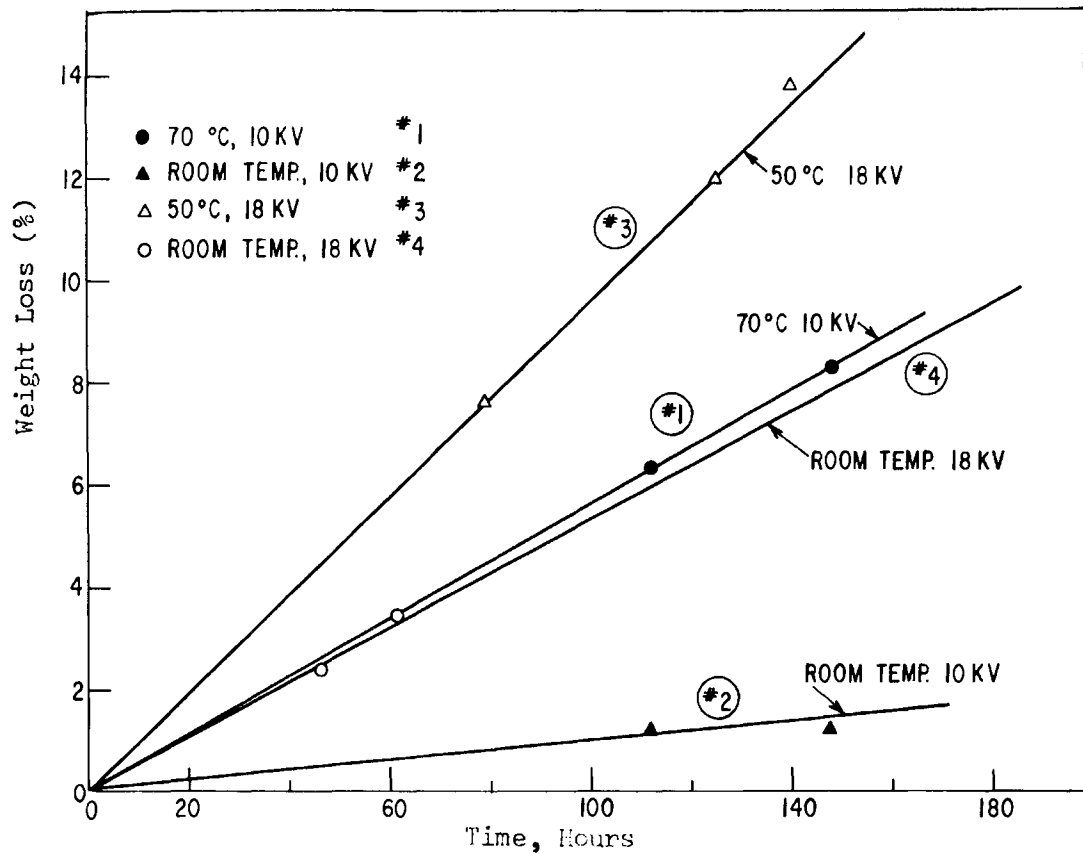
[Ref. 31]

The General Electric Company Research Laboratories conducted weight loss rate measurements on H-Film at various temperatures for various pressures. Additional weight loss (under corona conditions) data is reported in the Electrical Properties section titled "Corona Effects." Their results are shown in the following graphs:



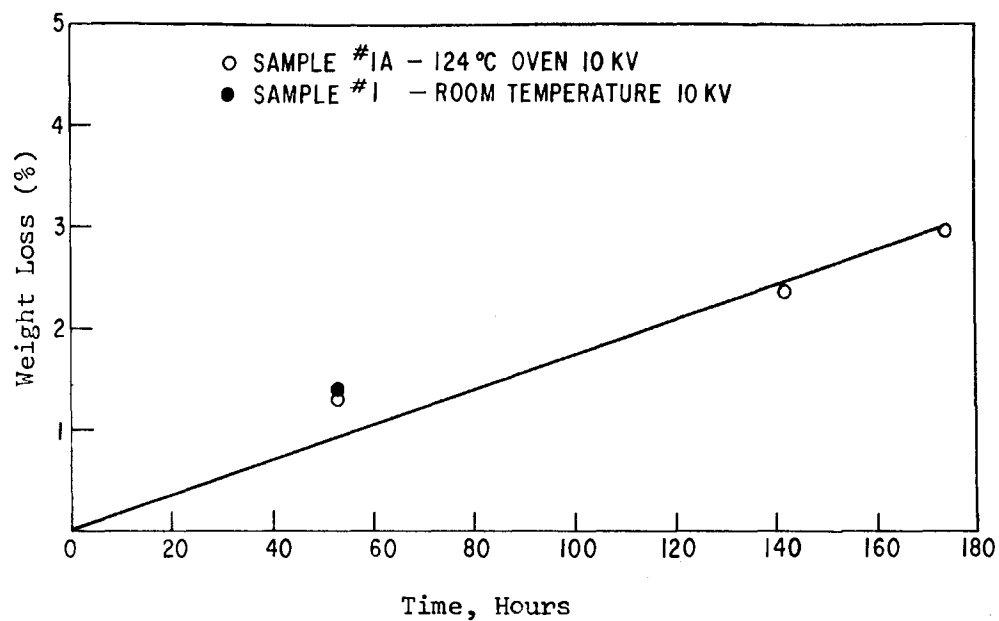
Weight Loss of "Kapton" Type H-Film.

[Ref. 56]



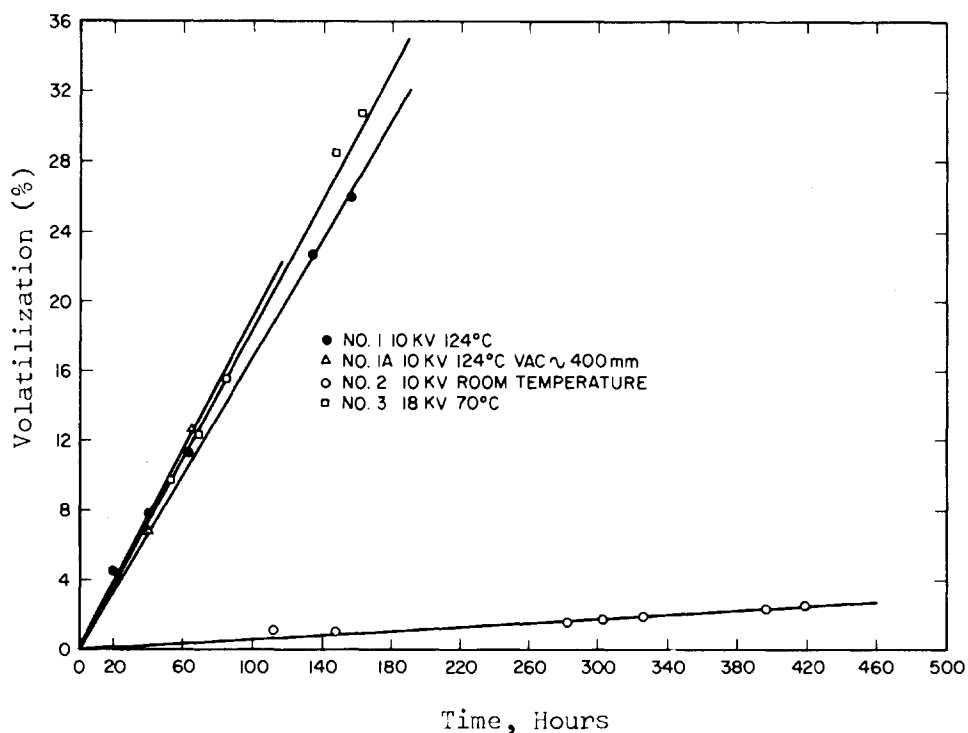
Weight Loss of H-Film.

[Ref. 56]



Weight Loss of H-Film at 10 Kv; 500-mm Vacuum.

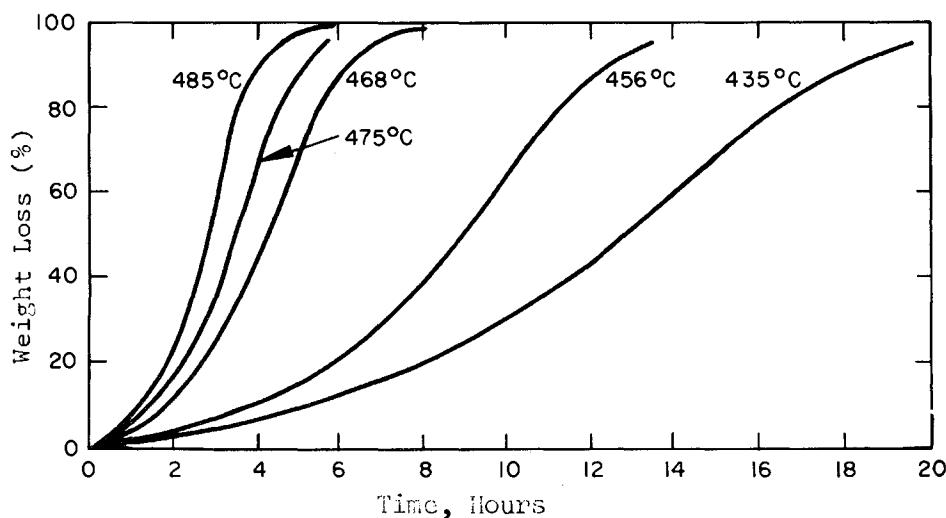
[Ref. 56]



Weight Loss of "Kapton" Type H-Film.

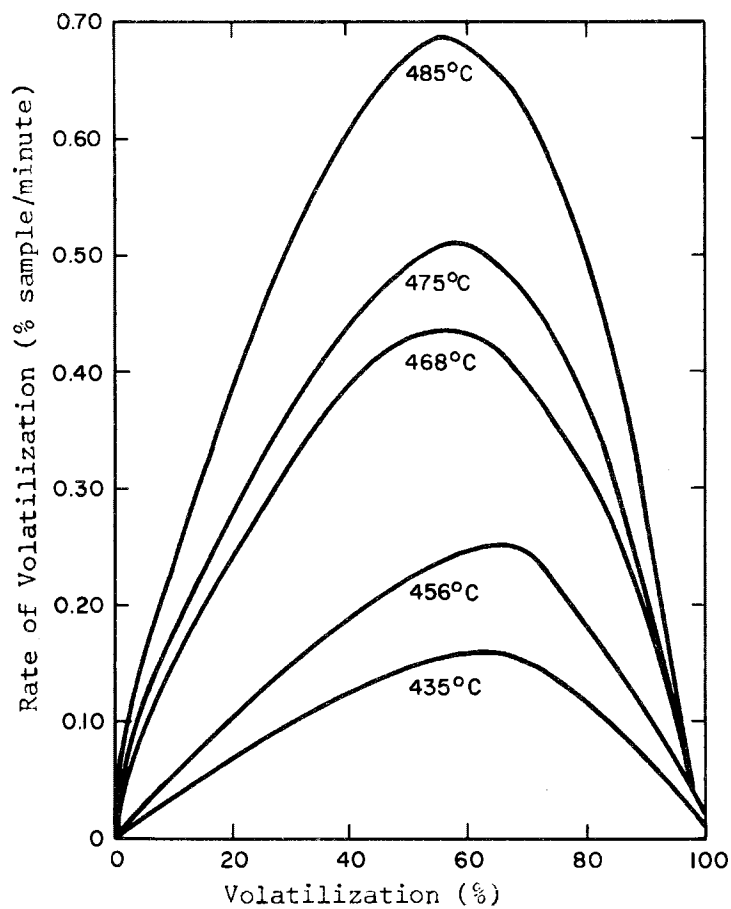
[Ref. 56]

S.D. Bruck of the Applied Physics Laboratory of John Hopkins University studied the thermal degradation of H-Film (1 mil thick) in air and vacuum and found that in (10^{-3} mm Hg) vacuum this organic polymer shows good heat stability, with no appreciable weight loss even after prolonged exposure to temperatures up to approximately 500°C. Above this temperature it begins to volatilize, leaving a brittle, carbonized residue which appears to reach a limiting weight corresponding to approximately 45% of the original sample, and showing no infrared absorption bands:



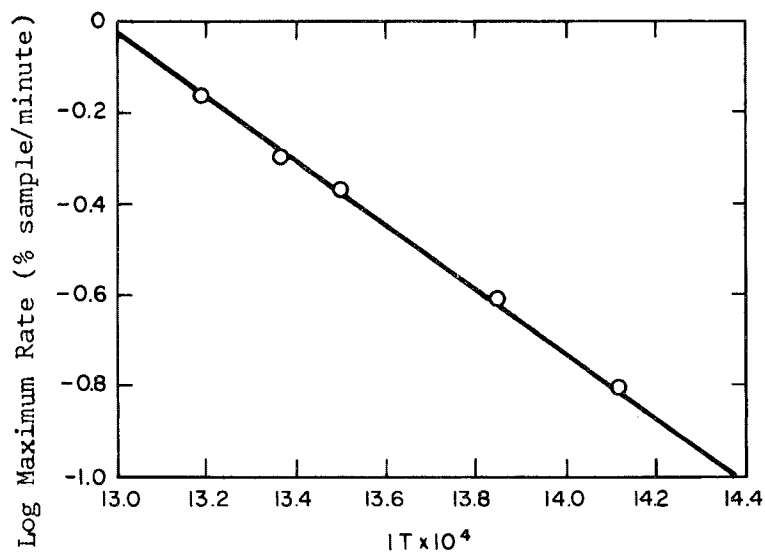
Thermal degradation of H-Film in air.

[Ref. 12]



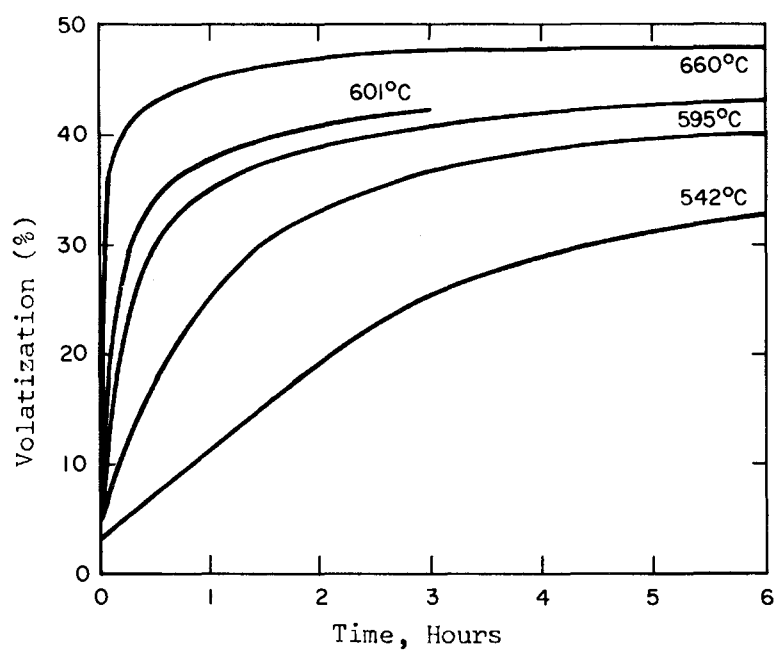
Rates of thermal degradation of H-Film
at various temperatures in air.

[Ref. 12]



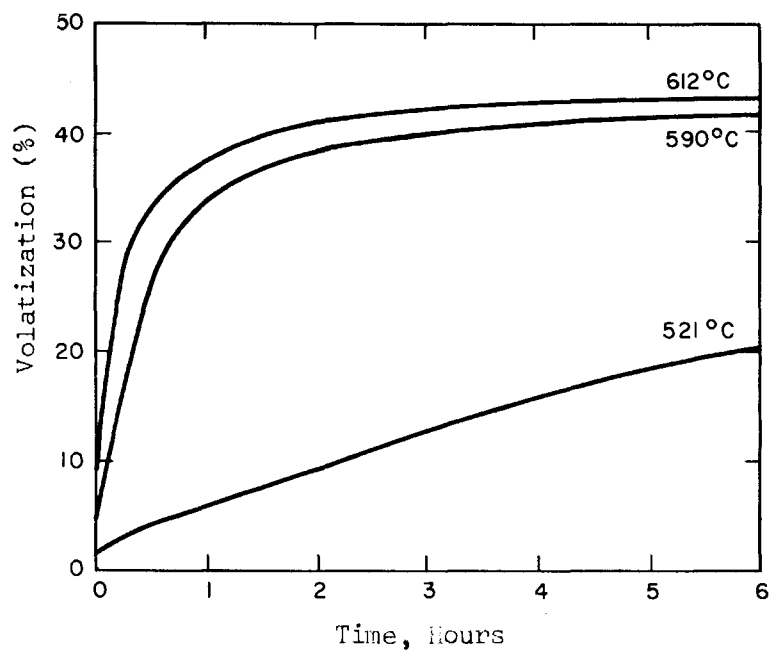
Arrhenius plot for thermal degradation
of H-Film in air.

[Ref. 12]



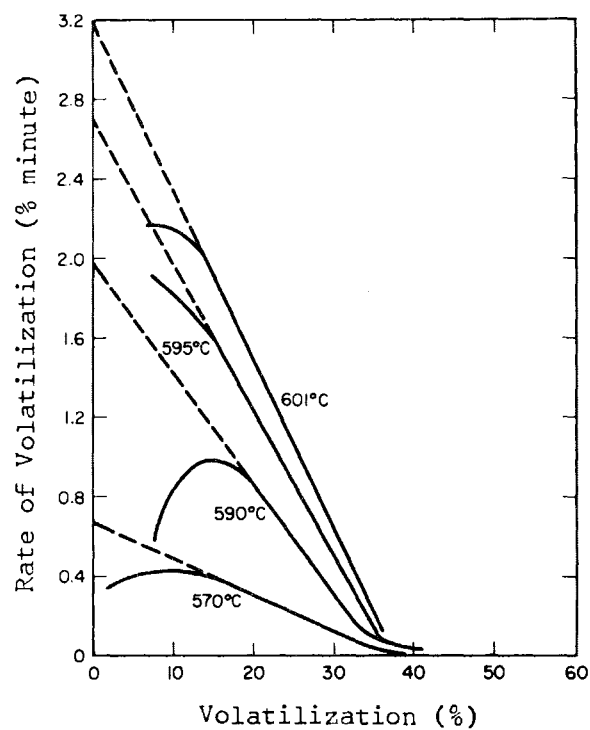
Thermal degradation of H-Film in vacuum.

[Ref. 12]



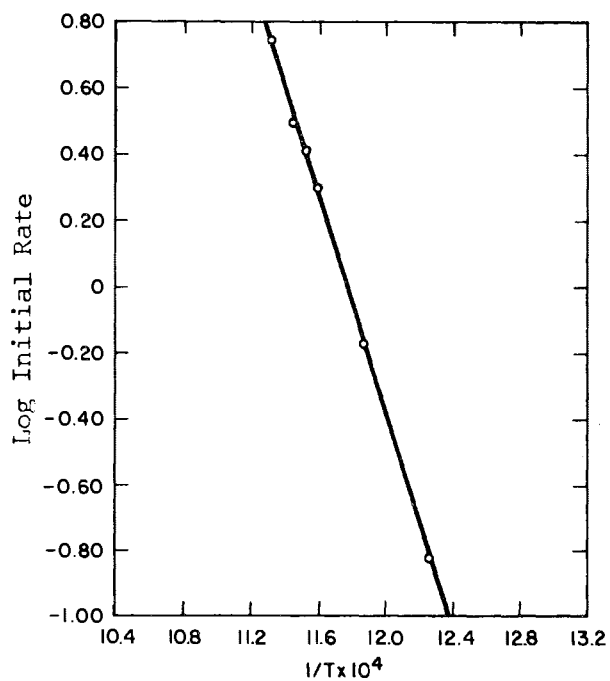
Thermal degradation of H-Film in vacuum.

[Ref. 12]



Rates of thermal degradation of H-Film in vacuum at various temperatures.

[Ref. 12]



Arrhenius plot for thermal degradation of H-Film in vacuum.

[Ref. 12]

Rates and Activation Energies for the Thermal Degradation
of H-Film in Air and Vacuum

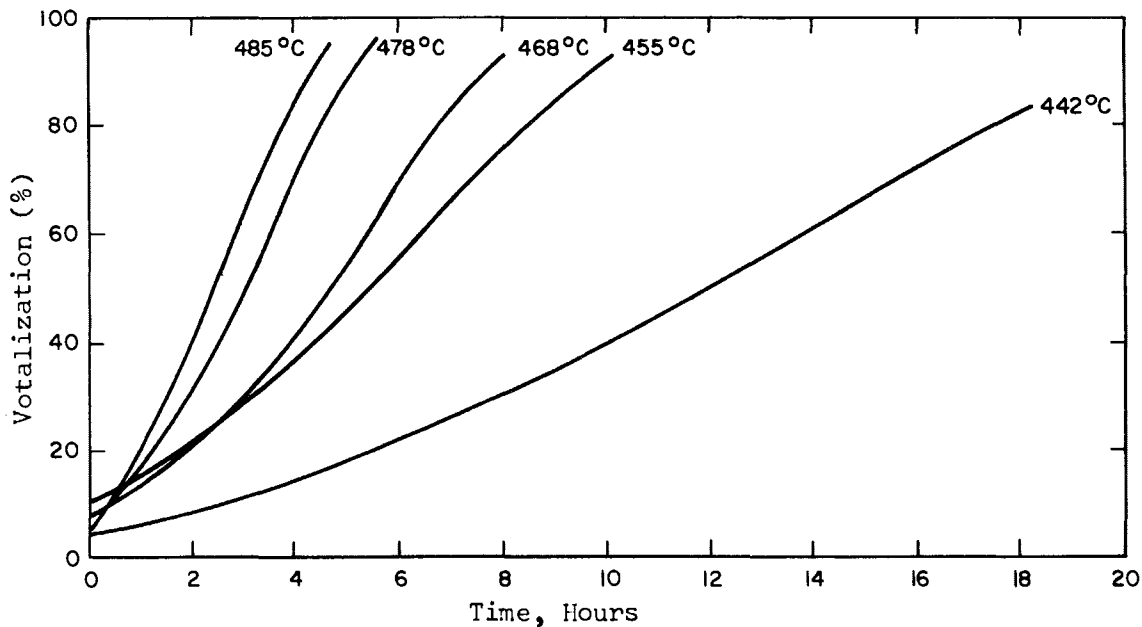
Thermal Degradation	Temperature °C	Total Time Hours	Total Volatilization %	Rates % Sample/Minute		Activation Energy kcal mole
				Extrapolated Initial	Maximum	
Air	485	5.5	98.4	---	0.685	32
Air	475	5.5	93.3	---	0.510	
Air	468	9.0	98.9	---	0.435	
Air	456	13.5	94.6	---	0.252	
Air	435	20.0	95.6	---	0.157	
Vacuum	660	20.0	49.0	*	---	74
Vacuum	612	20.0	45.2	5.620	---	
Vacuum	601	3.0	42.3	3.180	---	
Vacuum	595	10.0	43.3	2.700	---	
Vacuum	590	16.0	43.4	1.990	---	
Vacuum	570	18.0	41.9	0.680	---	
Vacuum	542	18.0	38.6	0.150	---	
Vacuum	521	18.0	32.1	**	---	

* Rates too fast for reliable measurement and extrapolation.

** Degradation complicated by side reactions at this low temperature.

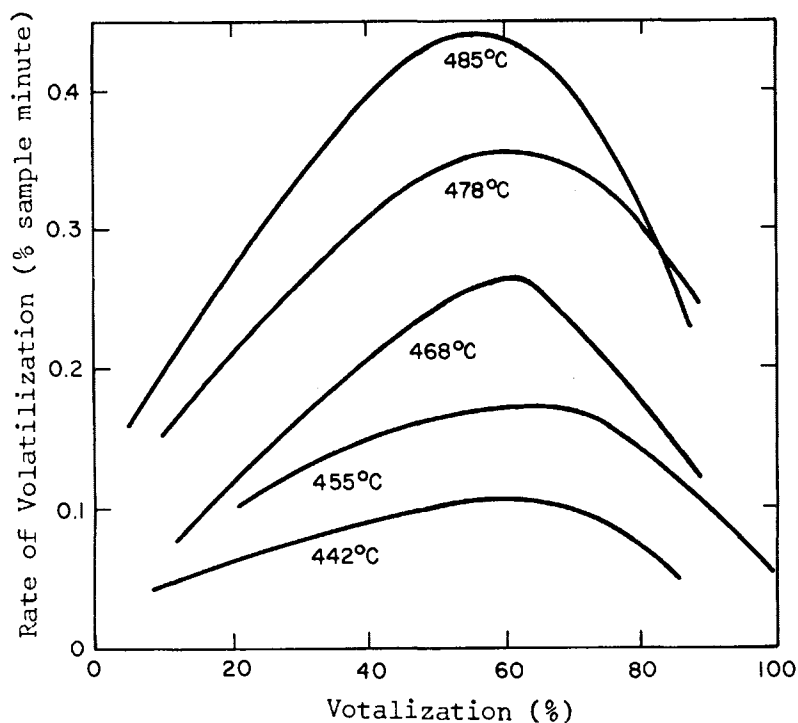
[Ref. 12]

The same author (S.D. Bruck) repeated the thermal degradation experiments with a purified H-Film material (i.e., purified at room temperature as follows: Approximately 150 mg of H-Film soaked in 50 ml of dimethylformamide for 72 hours, followed by soaking in 50 ml of distilled water for 24 hours, and finally soaking in 50 ml of ethyl alcohol for 24 hours. The polymer was dried at 120°C for 12 hours in a vacuum oven, and stored in a desiccator). The following graph shows the percent volatilization of the purified H-Film in air as a function of time between 442° and 485°C:



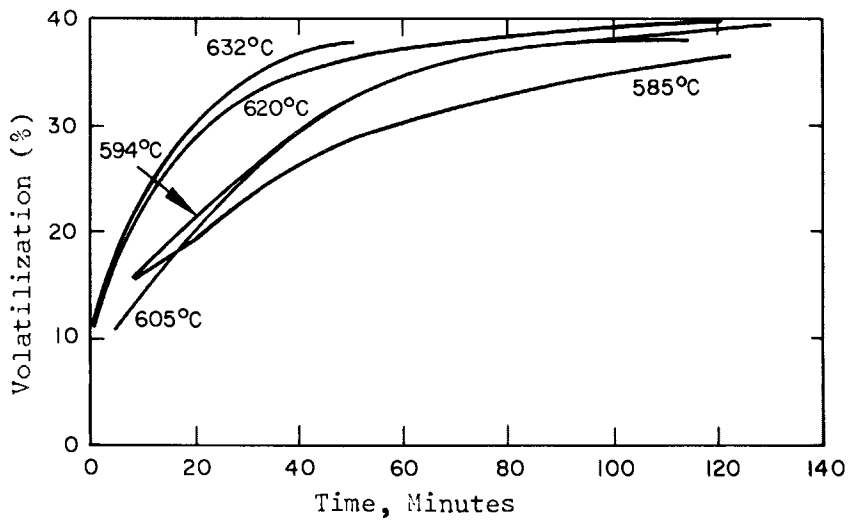
Thermal degradation of purified H-Film in air.

[Ref. 13]



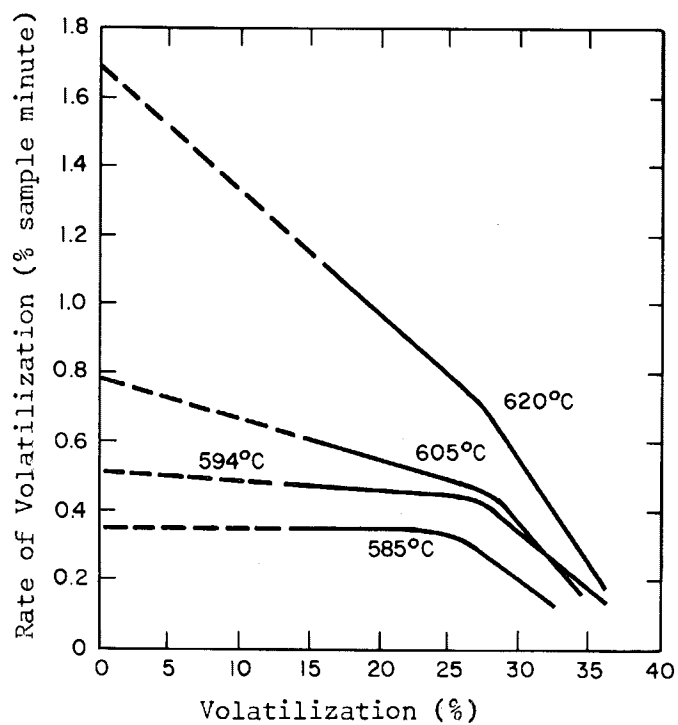
Rates of thermal degradation of purified H-Film in air.

[Ref. 13]



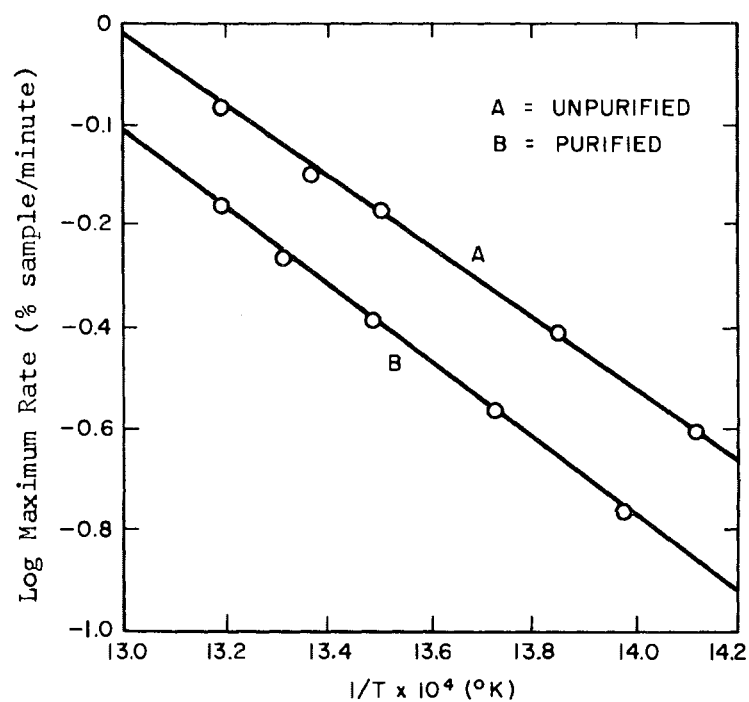
Thermal degradation of purified H-Film in vacuum.

[Ref. 13]



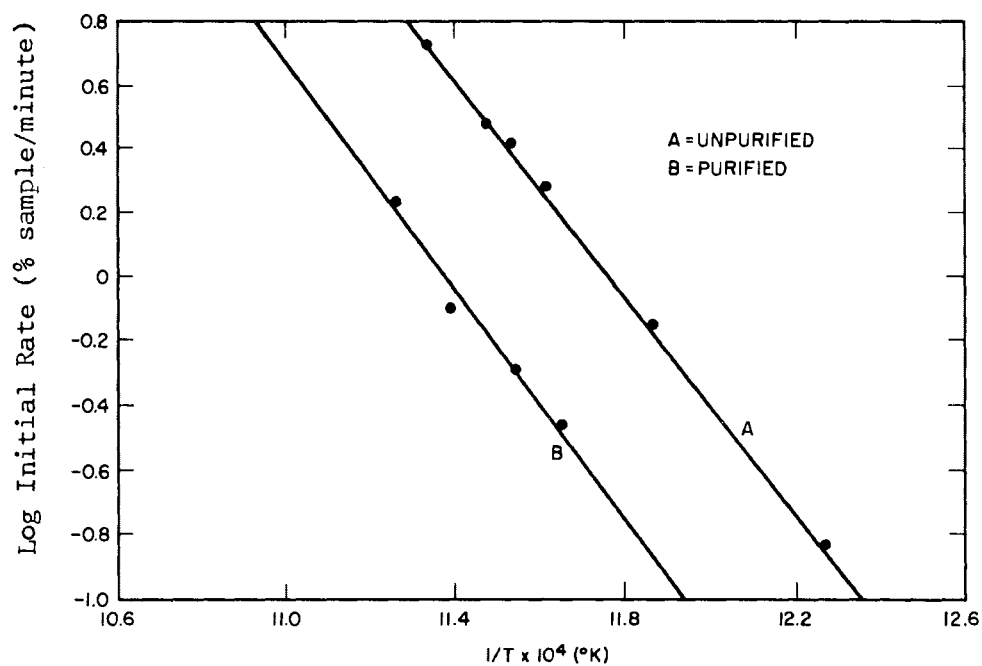
Rates of thermal degradation of purified H-Film in vacuum.

[Ref. 13]



Arrhenius plots for the thermal degradation of unpurified and purified H-Film in air.

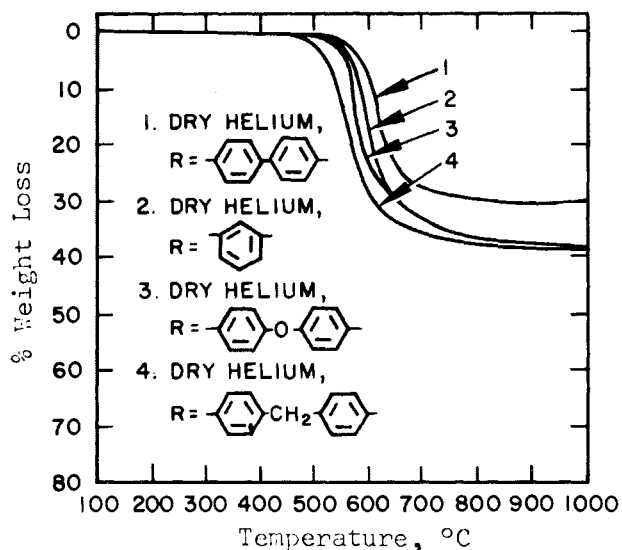
[Ref. 13]



Arrhenius plots for the thermal degradation of unpurified and purified H-Film in vacuum.

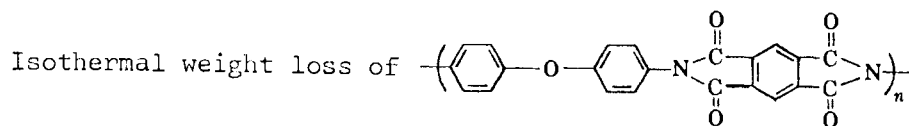
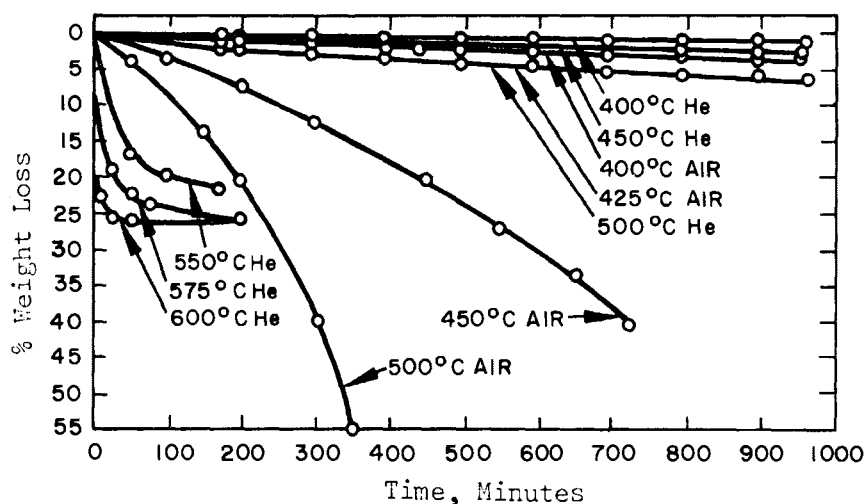
[Ref. 13]

Scroog and co-workers reported that the polypyromellitimide derived from bis(4-aminophenyl) ether showed isothermal weight loss in helium atmosphere of only 1.5% after 15 hours at 400°C., 3.0% after 15 hours at 450°C., and 7% after 15 hours at 500°C. In other weight loss measurements during constant rate of temperature increase (3°C/min) the polyimides derived from m-phenylenediamine, benzidine, and bis(4-aminophenyl) ether exhibited a 1-1.5% weight loss in the heating period to 500°C. The thermogravimetric analyses are lent additional significance by the retention of film toughness after thermal aging summarized below. Films derived from wholly aromatic polypyromellitimides retain toughness for months at elevated temperatures in air.



Effect of diamine structure on weight loss of polypyromellitimides during constant rise in temperature (3°C/min).

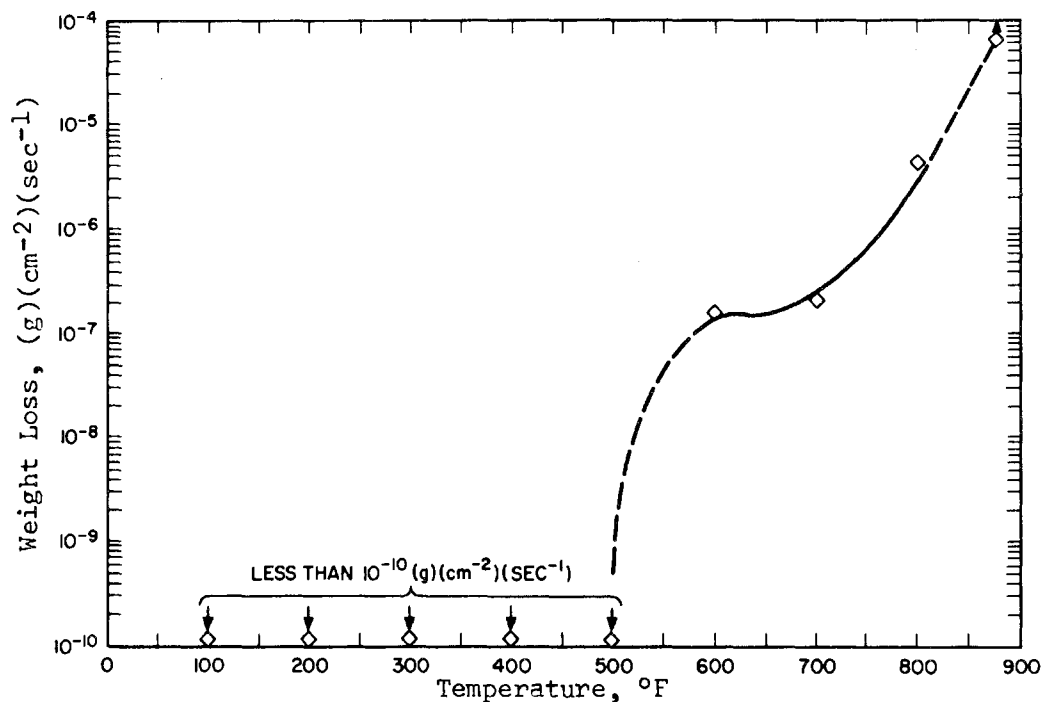
[Ref. 123]



[Ref. 123]

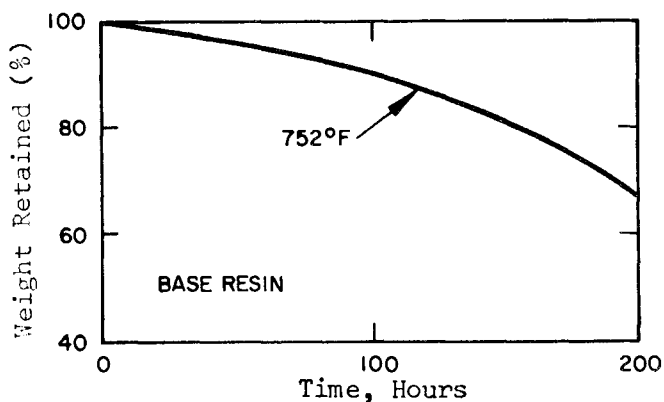
SP-Polymer:

Buckley and Johnson at NASA have studied the decomposition of polyimide plastic SP-1 at elevated temperatures in high vacuums and measured the evaporation rate:



Weight loss of SP-1 Polyimide in vacuum as a function of specimen temperature. Ambient pressure, 10⁻⁷ to 10⁻⁸ millimeter of mercury.

[Ref. 18]



Weight loss of polyimides in air at 750°F can be as little as 30% after 200 hours.

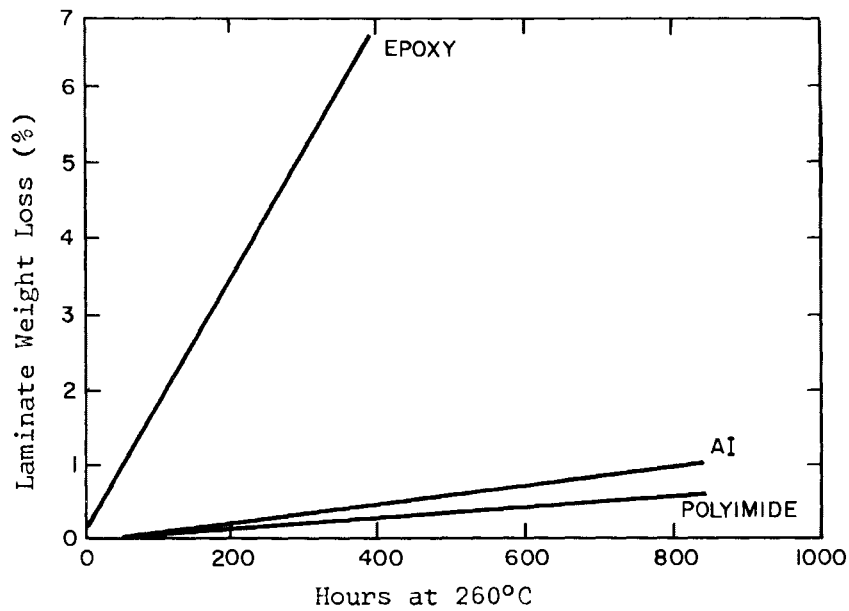
[Ref. 98]

AI-220 Magnet Wire:

1 month, 210°C

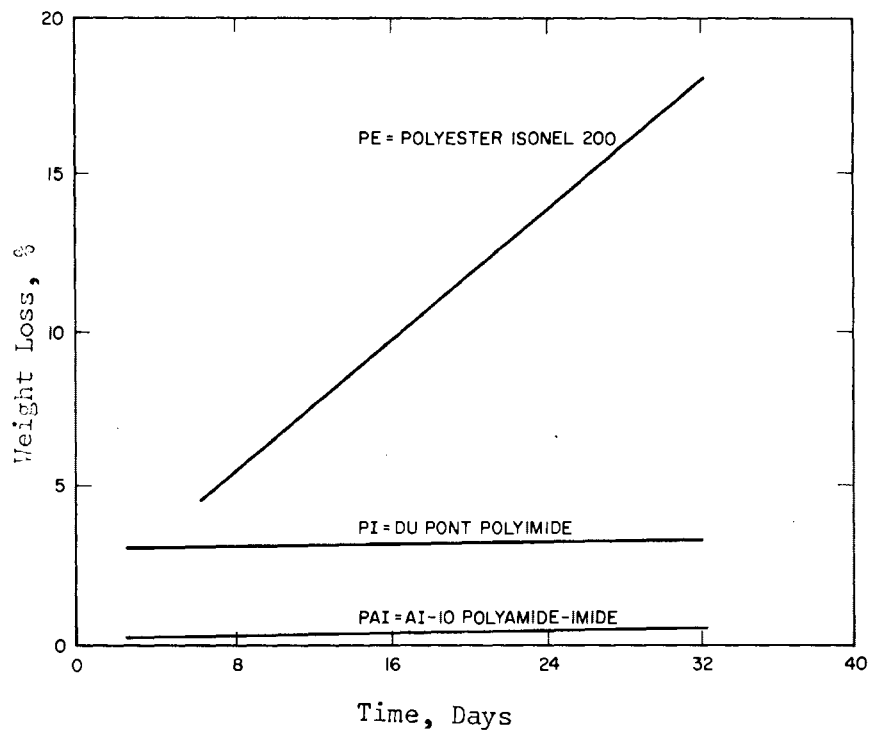
0.2 - 0.5% (weight loss)

[Ref. 5,8]



Weight loss of laminates aged at 260°C.

[Ref. 4]

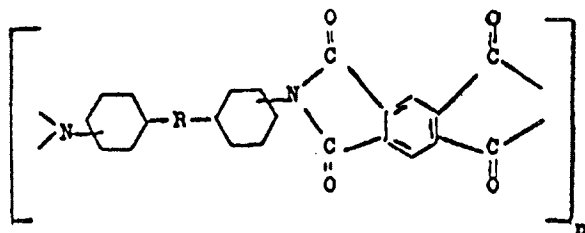


Weight loss - 1 month 210°C.

[Ref. 104]

On the basis of the following (series of tables and graphs) extensive polyamide-imide weight loss data; Freeman and co-workers at the Westinghouse Electric Corporation give the relative order of stability of polymer linkages as follows: phenyl rings; imide > phenyl-phenyl bond > ether; sulfide > amide > methylene > isopropylidene:

Relative Thermal Stability of Linkage R in Aromatic Polyimides **



Diamine	R Equals	Inherent Viscosity	Film* Property	% Weight Loss in Air at 325°C			
				100 hr	200 hr	300 hr	400 hr
m-phenylene diamine I-1	None	1.19	B	3.3	4.3	5.0	5.6
benzidine	>-<	3.22	F	2.2	3.6	5.1	6.5
4,4'-diaminophenyl ether	-O-	2.45	F	3.3	4.0	5.2	6.6
3,4'-diaminophenyl ether	-O-	1.31	F	3.4	3.8	5.1	7.2
4,4'-diaminophenyl sulfide I-6	-S-	1.83	F	4.8	5.8	6.8	7.9
3,4'-diaminobenzanilide AI-8		2.19	F	2.0	4.2	6.9	9.8
3,3'-diaminobenzanilide		1.18	F	3.2	6.5	9.8	11.2
4,3'-diaminobenzanilide		1.55	F	4.3	7.8	10.8	11.9
4,4'-diaminobenzanilide		2.20	B	5.7	8.4	11.9	12.1
isophthal 3-aminoanilide		1.48	F	4.7	6.8	8.9	10.7
isophthal 4-aminoanilide		1.46	F	6.9	9.4	14.4	20.4
4,4'-methylene dianiline	-CH ₂ -	1.86	B	9.4	12.9	14.7	16.8
N,N'-m-phenylene bis 4-aminobenzanilide		1.48	F	6.0	9.2	12.5	15.6
N,N'-m-phenylene bis 3-aminobenzanilide		1.07	F	6.2	8.3	14.0	20.3
4,4'-isopropylidene dianiline		1.48	F	16.1	26.2	31.0	36.0

I-8 m-phenylene diamine reacted with benzophenone tetracarboxylic dianhydride
 AI-7 m-phenylene diamine - isophthalic - Terephthalic - pyromellitic dianhydride reaction mixture.

* B - Brittle, F-- Flexible on creasing

** Note: 1 mil films on aluminum

[Ref. 45]

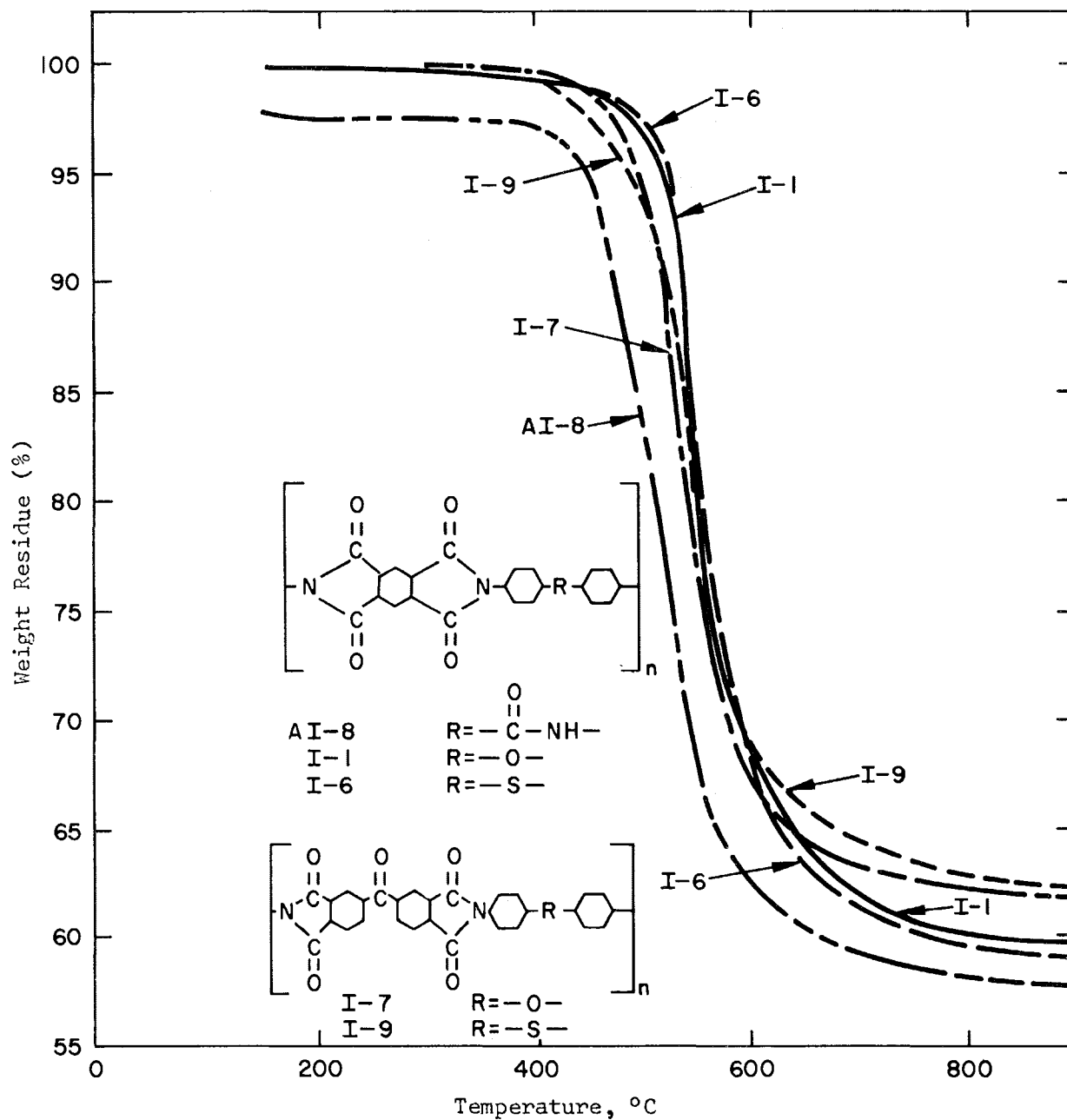
Weight Loss (%) of Aromatic Polymer Films in Air at 325°C (617°F)

<u>Resin</u>	<u>Composition*</u>	Weight Loss at		
		<u>200 Hours</u>	<u>400 Hours</u>	<u>800 Hours</u>
I-3	MPD-PMDA	---	5.6	---
I-1	DAPE-PMDA	---	6.6	---
I-6	DAPS-PMDA	---	7.9	---
AI-8	(MAB-PPD)-PMDA	---	9.8	---
AI-2	(MPD ₂ -IP)-PMDA	---	10.7	---
AI-6	(MAB ₂ -MPD)-PMDA	---	20.3	---
AI-95	(MAB-PPD) _{3.4} -IP _{2.4} -PMDA	38.0	---	---
AI-7	MPD ₁₆ -IP ₉ -TP ₃ -PMDA ₄	30.0	---	---
I-8	MPD-BTDA	2.7	3.2	3.7
I-7	DAPE-BTDA	6.3	11.2	---
I-9	DAPS-BTDA	2.0	3.3	---
AI-83	(MAB-PPD)-BTDA	2.2	4.4	8.8
AI-94	(MPD ₂ IP)-BTDA	10.5	19.5	---
AI-96	DAPE-(MAB-PPD)BTDA ₂	3.9	6.3	15.0
AI-93	MPD-(MAB-PPD) ₂ BTDA ₃	3.9	5.0	---
I-13	DAPE-MPD-BTDA ₂	4.9	7.1	---
I-14	DAPE ₃ -MPD-BTDA ₄	3.7	6.3	14.5
I-15	DAPE-MPD ₃ -BTDA ₄	2.8	3.7	5.1
I-11	MPD ₃ -PMDA ₂ -BTDA	1.8	2.7	4.8
I-10	MPD ₂ -PMDA-BTDA	1.4	2.3	4.3
I-12	MPD ₃ -PMDA-BTDA ₂	1.6	2.3	3.9

Note: Weight loss of 1 mil film determined from plots of average loss versus aging time.

* See Section 1 of this report for chemical identification if these polymers.

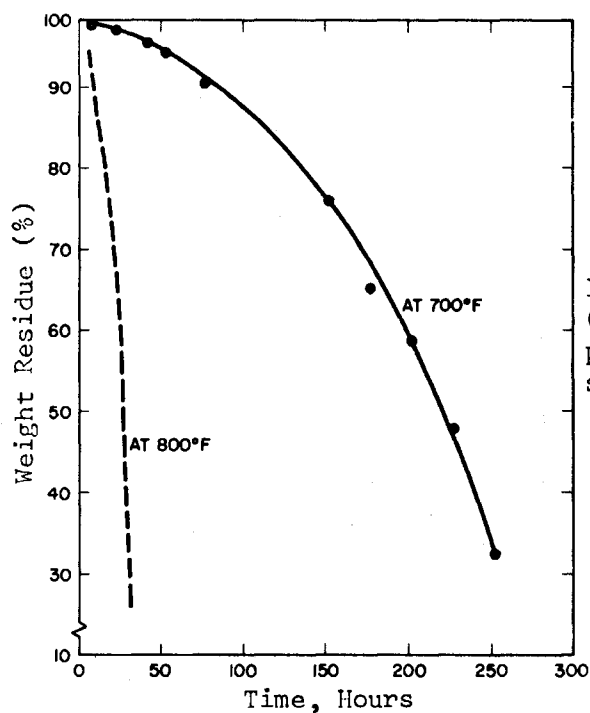
[Ref. 47]



TGA for aromatic polyimides in nitrogen T = 150°C/hour.

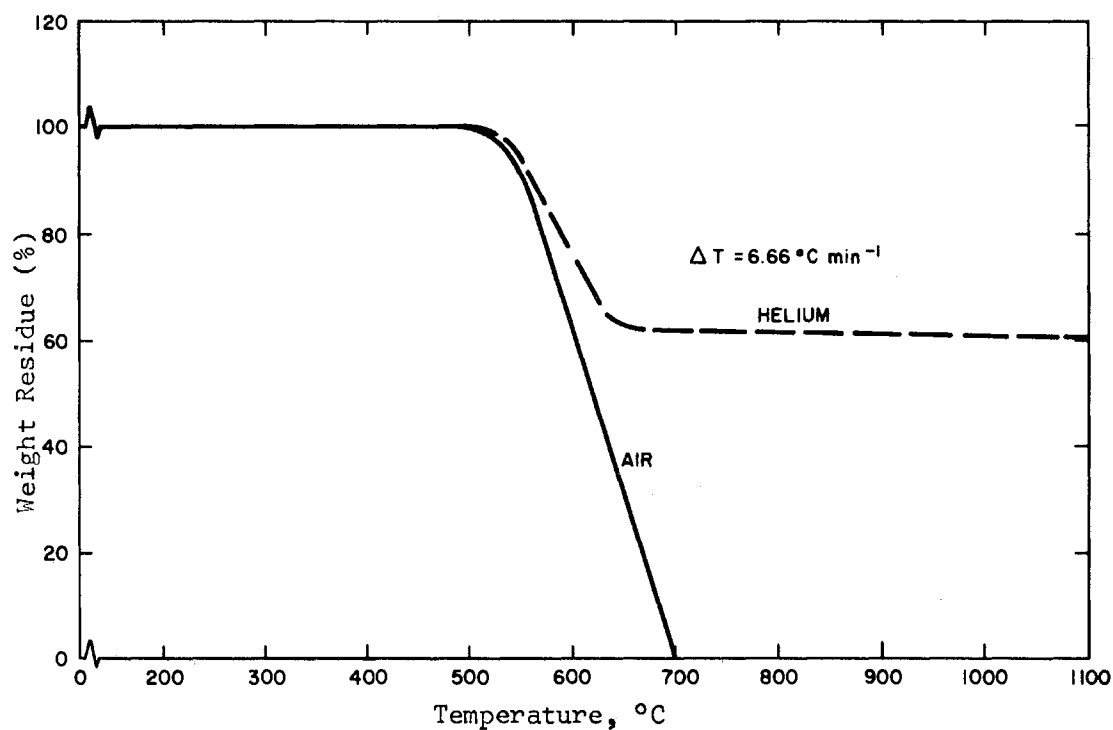
[Ref. 47]

Narmco Research and Development Division of the Whittaker Corporation evaluated the thermal degradation of an experimental polyimide resin over a wide range of temperatures and different environments:



Isothermal weight loss of Poly (m-phenylene - 3,3', 4,4', -benzophenonetetracarboxylimide) in static air.

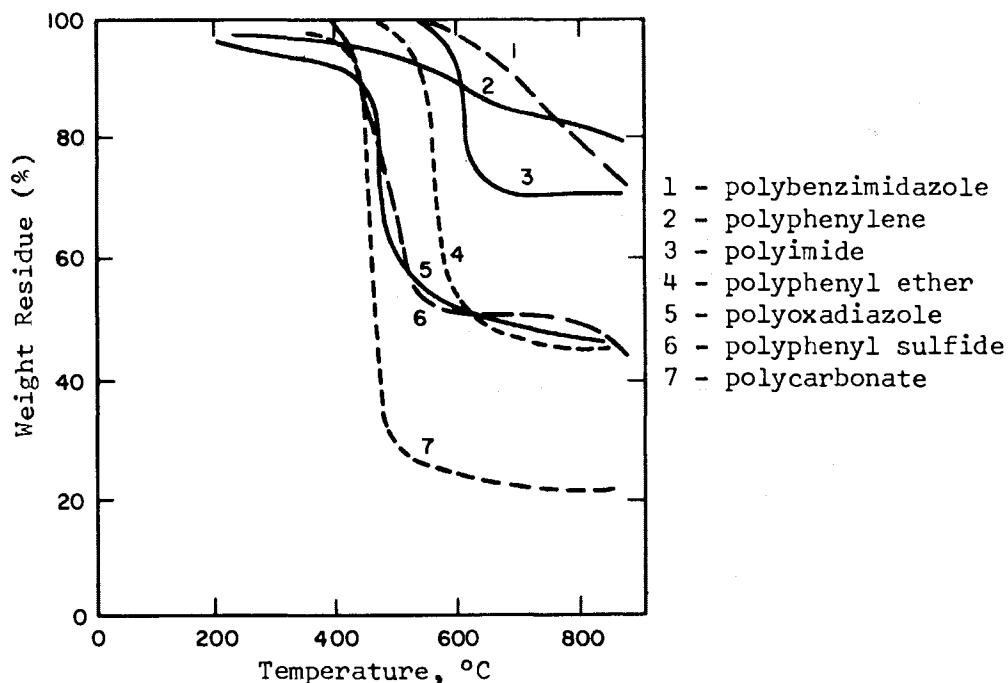
[Ref. 76]



Thermogram of Poly (m-phenylene-3,3', 4,4', - benzophenonetetracarboxylimide).

[Ref. 76]

Dunnavent and Palinchak (Battelle Memorial Institute) in a developmental study of thermally stable polymers compared the TGA curve of a polyimide with those of other polyaromatic polymers:

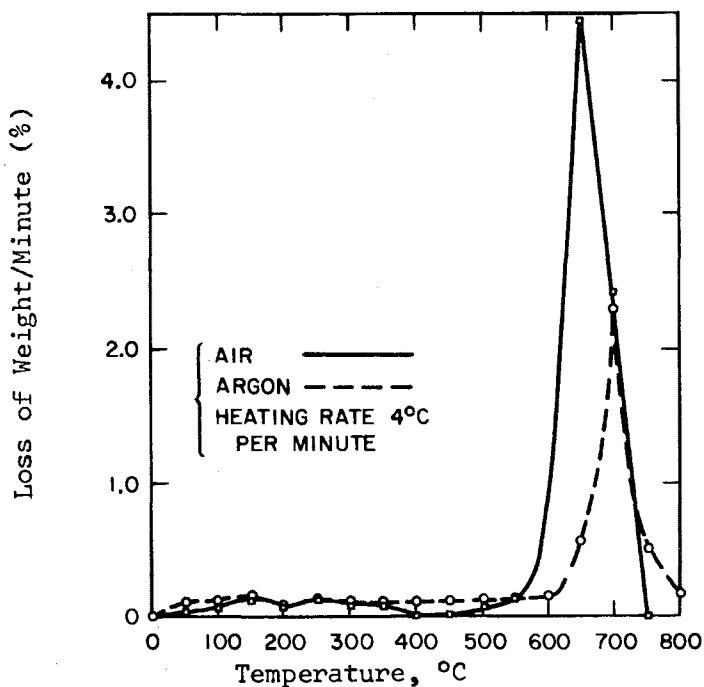


TGA curves of polyaromatic polymers.

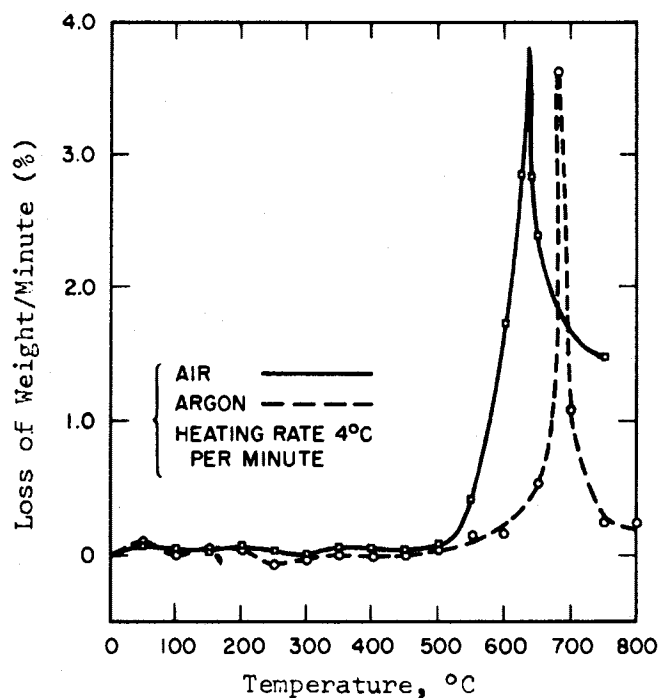
[Ref. 25]

In similar fashion, Jones and co-workers studied the thermal stability of polyimides derived from pyromellitic anhydride with three different diamines:

Differential thermogravimetric curves for polymer derived from pyromellitic anhydride and *m*-phenylene diamine.

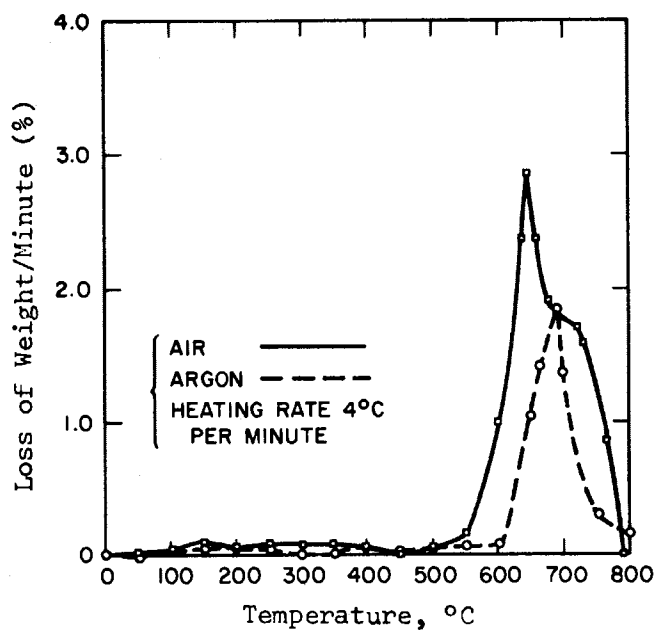


[Ref. 64]



Differential thermogravimetric curves for polymer derived from pyromellitic anhydride and benzidine.

[Ref. 64]



Differential thermogravimetric curves for polymer derived from 4,4'-diamino - diphenyl ether and pyromellitic anhydride.

[Ref. 64]

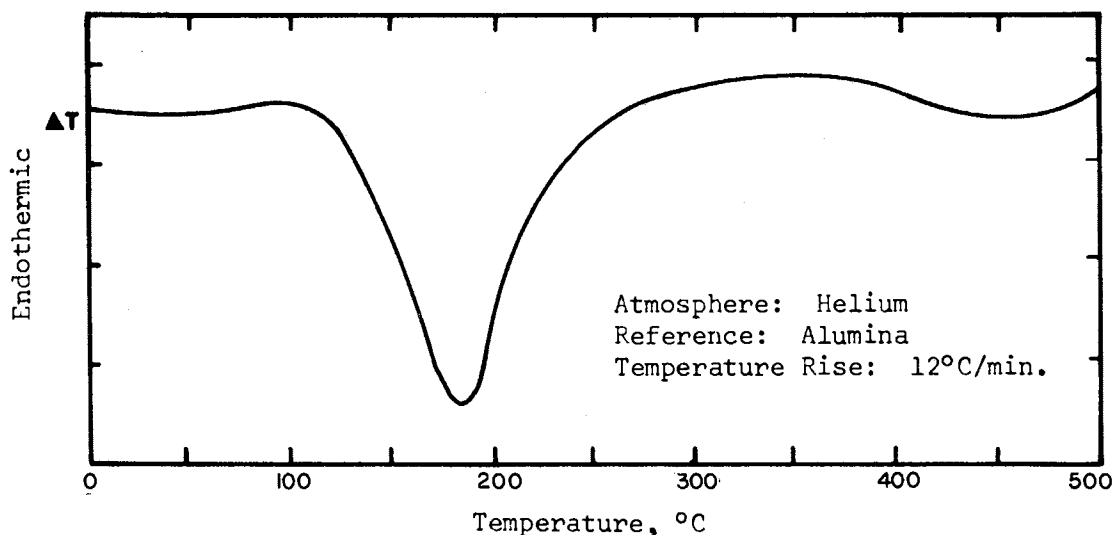
Bower and Frost in their study of aromatic polyimides investigated the elevated temperature-time weight loss characterization of these materials as a function of different types of diamines as shown in the following table:

Diamine	Inherent visc.	Film properties ^a	Weight loss at 325°C., ^b %			
			100 hr.	200 hr.	300 hr.	400 hr.
<i>m</i> -Phenylenediamine	1.19	Brit	3.3	4.3	5.0	5.6
Benzidine	3.22	Flex	2.2	3.6	5.1	6.5
4,4'-Diaminodiphenyl ether	2.45	Flex	3.3	4.0	5.2	6.6
3,4'-Diaminodiphenyl ether	1.31	Flex	3.4	3.8	5.1	7.2
4,4'-Diaminodiphenyl sulfide	1.83	Flex	4.8	5.8	6.8	7.9
4,4'-Methylene dianiline	1.86	Brit	9.4	12.9	14.7	16.8
4,4'-Diaminobenzanilide	2.20	Brit	5.7	8.4	11.9	12.1
4,4'-Isopropylidene dianiline	1.48	Flex	16.1	26.2	31.0	36.0
4,3'-Diaminobenzanilide	1.55	Flex	4.3	7.8	10.8	11.9
3,4'-Diaminobenzanilide	2.19	Flex	2.0	4.2	6.9	9.8
3,3'-Diaminobenzanilide	1.18	Flex	3.2	6.5	9.8	11.2
3,5-Diaminobenzanilide	1.29	No film, just crumbs				
Isophthal(4-aminoanilide)	1.46	Flex	6.9	9.4	14.4	20.4
<i>N,N'</i> - <i>m</i> -Phenylene-bis(4-aminobenzamide)	1.48	Flex	6.0	9.2	12.5	15.6
Isophthal(3-aminoanilide)	1.48	Flex	6.8	8.1	10.5	13.2
<i>N,N'</i> - <i>m</i> -Phenylene-bis(3-aminobenzamide)	1.07	Flex	6.2	8.3	14.0	20.3
<i>N,N'</i> -bis(3-Aminobenzoyl)-2,4-diamino- diphenyl ether	0.54	Flex	24.1	31.5	38.6	44.3
<i>N,O</i> -bis(3-Aminobenzoyl)- <i>p</i> -aminophenol	1.22	Flex	12	17	21	27
bis(4-Aminophenyl)isophthalate	0.93	Flex (shrunk)	3.6	6.7	10.9	15.0
4,4'-Diaminophenyl benzoate	1.21	Brit	3.3	5.5	7.6	9.7
Resorcinol-bis(3-aminobenzoate)	0.78	Brit	9.5	13.3	17.3	22.7
MPD _{7.23} IP _{6.23} ^c	1.16	Flex	6.7	16.5	29.8	
MPD _{7.61} IP _{4.98} TP _{1.66} ^c	1.21	Flex	8.5	22.4	44.4	

^a The samples consisted of films approximately 1 mil thick adhering to aluminum. The film was judged to be flexible if it could be creased without cracking; brittle if it cracked on creasing. Brit = brittle, Flex = flexible. ^b The values at 100 hr. intervals were obtained from plots of weight loss vs. time. ^c MPD = *m*-phenylenediamine units; IP = isophthalic units; TP = terephthalic units.

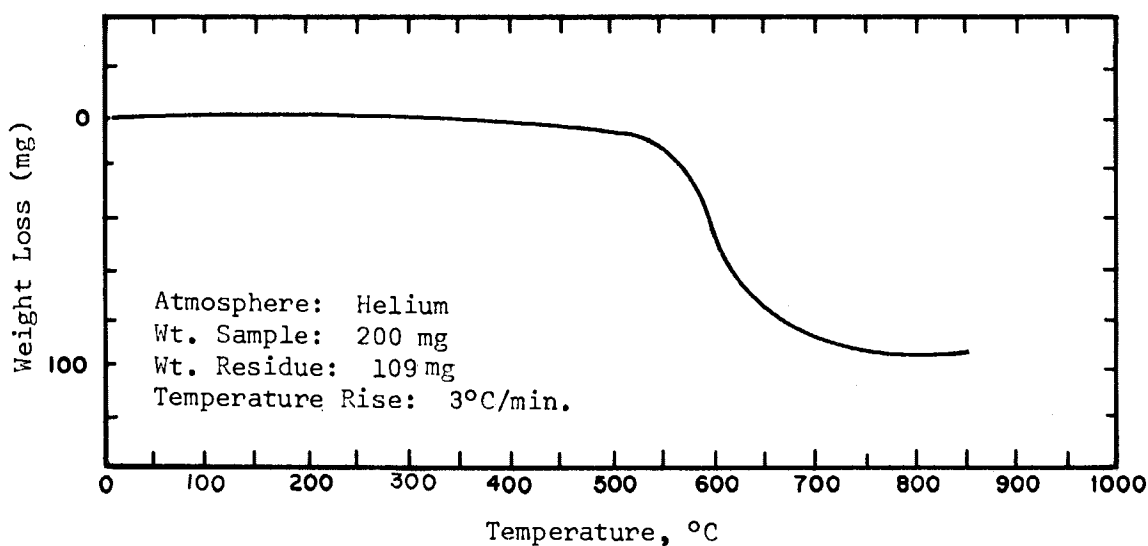
[Ref. 15]

The Monsanto Research Corporation explored a number of high temperature organic and semi-organic coatings under an Air Force Materials Laboratory contract [AF 33(657)-8641, project number 7340, task number 734007]. Included among the heat resistant polymers was the polyimide PMI or poly-4,4'-oxydiphenylpyromellitimide. Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) data were obtained on this polymer in helium and air environments over a wide range of temperatures as follows:



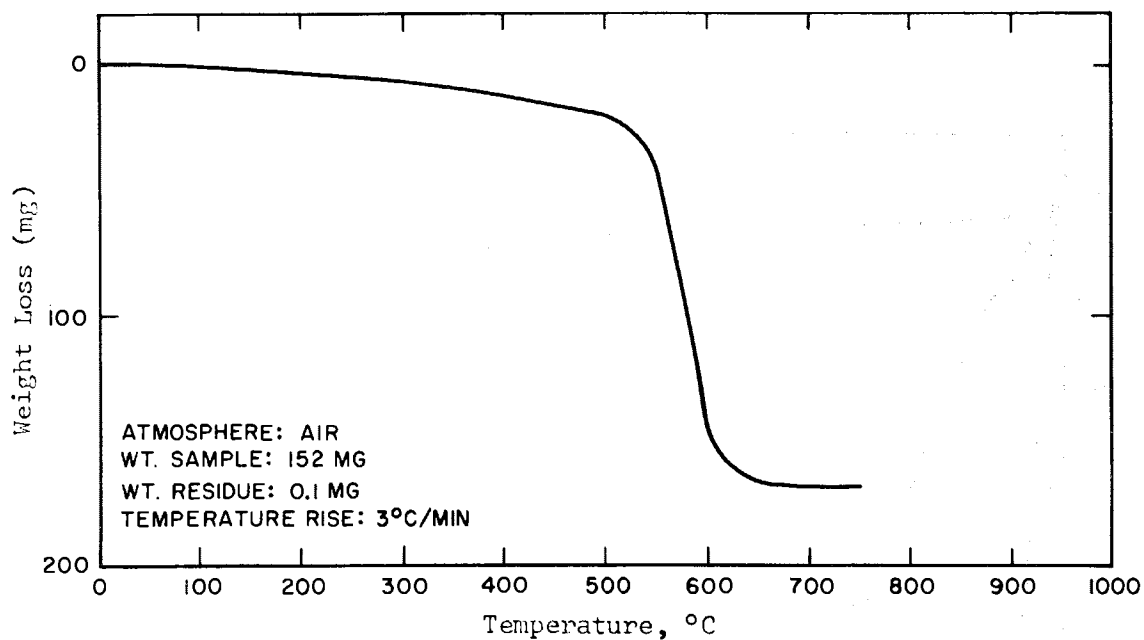
DTA curve for poly-4,4'-oxydiphenylpyromellitimide precursor.

[Ref. 116]



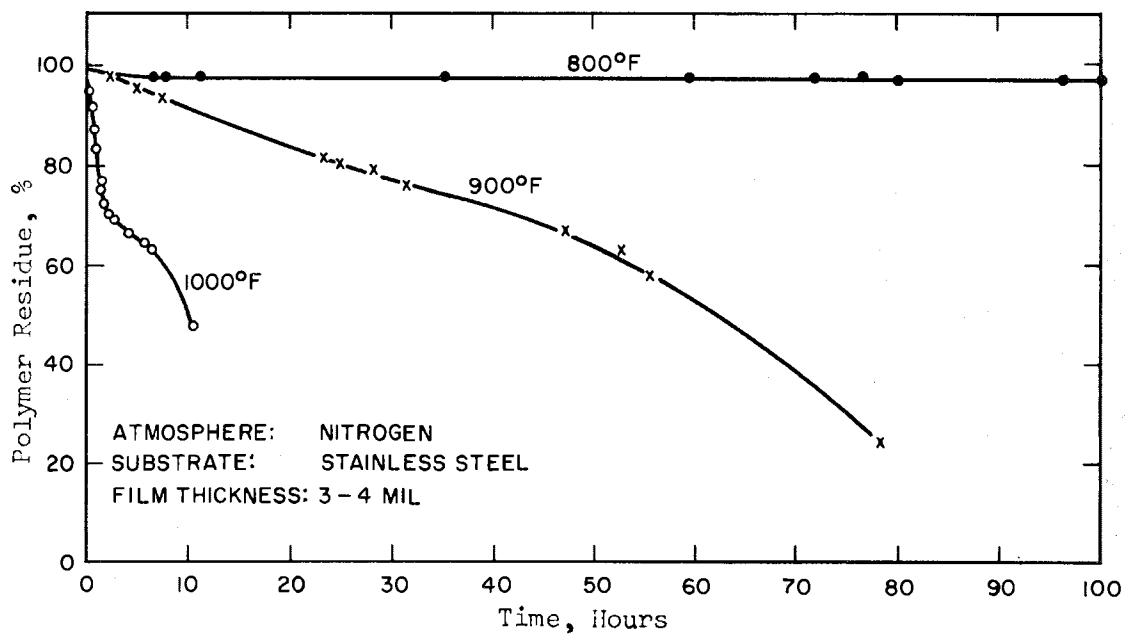
TGA curves for poly-4,4'-oxydiphenylpyromellitimide.

[Ref. 116]



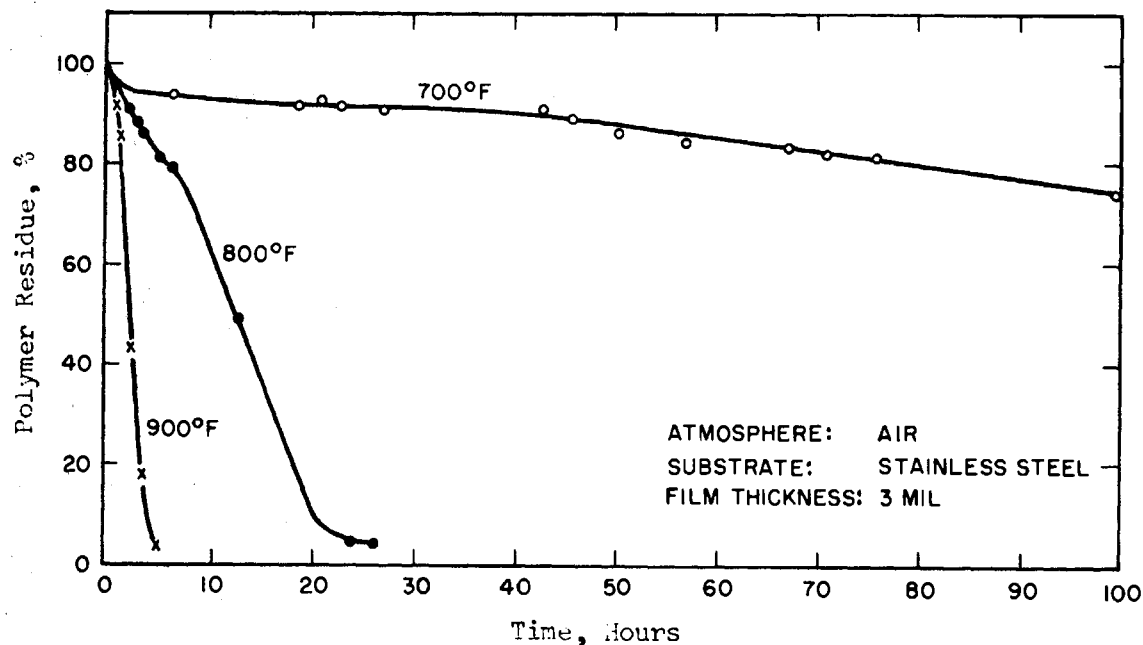
TGA curves for poly-4,4'-oxydiphenylpyromellitimide.

[Ref. 116]



Isothermal TGA curves for poly-4,4'-oxydiphenylpyromellitimide coatings.

[Ref. 116]



Isothermal TGA curves for poly-4,4'-oxydiphenylpyromellitimide coatings.

[Ref. 116]

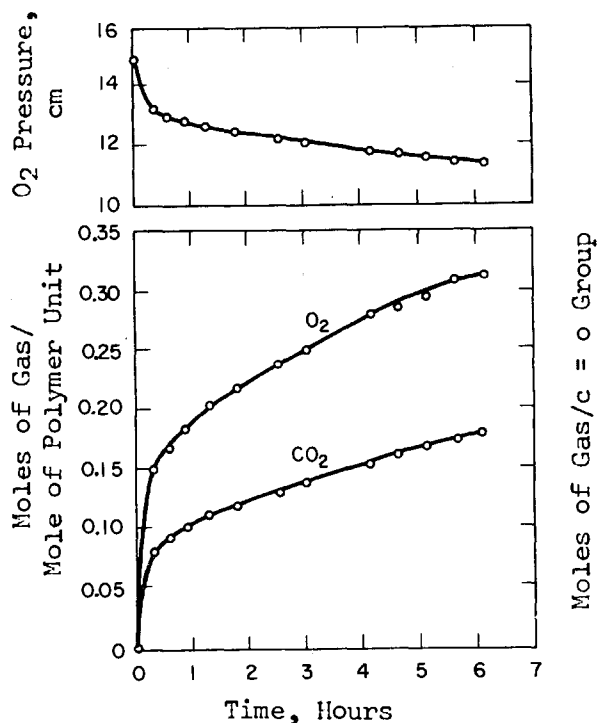
The DTA data showed the amide-imide conversion temperature is about 110 to 120°C. TGA data showed that the maximum rate of decomposition occurred at about 1067°F in both helium and air. This is about 50°C lower than that reported by Jones et al (Ref. 64). A large difference between the rates of decomposition in nitrogen and in air is clearly shown in the above isothermal diagrams. The authors report that their polypyromellitimide compositions exhibited the best oxidation resistance at 800°F of any of the unpigmented organic coatings tested of the series in an oxidizing atmosphere. In appearance, the coating gradually changed from a light-brown to a darker brown over an 8-hour period, with gradual loss of gloss. Coating integrity was lost between 4 and 8 hours, evidenced by wrinkling and flaking loose of coating near the center of the panel. After 12 hours, the degradation was not significantly greater. The authors noted that adhesion of the coating exposed in air was surprisingly low in contrast to the good adhesion of the original specimen and of the polymer which had been heated to 1000°F in an inert atmosphere.

[Ref. 116]

The preceding weight loss data represents the usual amount of screening data necessary to characterize the thermal stability of a polymer. During the developmental stage of polyimide chemistry, it was necessary to evaluate a wide range of dianhydrides, diamines, solvent systems, methods of polymerization, desiccants and curing temperatures in the quest for thermally and oxidatively stable molecular structures. Such data is more of a qualitative nature and not very precise but weeds out many weak and undesirable materials which do not require the expenditure of further costly research effort.

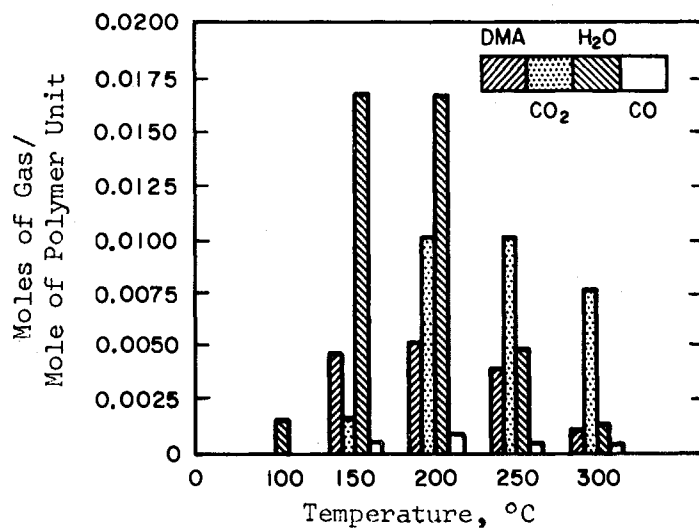
When further definitive or quantitative data are required, the nature of the evaluation methods must become more sophisticated and the precision and accuracy of the methods established on a higher order. Such caliber of work was reported recently (1965) by L.C. Scala and W.M. Hickam of Westinghouse Research and Development Center (Ref. 118). These workers made a very detailed and extensive examination of the thermal and oxidative stability of both polyimides and polyamide-imide polymers in vacuum, in air, in helium, in oxygen-argon atmospheres, and in isotopically-enriched oxygen. Experimental temperatures ranged from 300°C to 400°C.

Their analytical methods encompassed chemical analyses of the gasses evolved (CO , CO_2 , H_2O , O_2) and the remaining residues which were further submitted to infrared spectral analysis (but not reproduced in the paper). A further evaluation of consumed and retained gases (oxygen) by the polymer was made via isotopic oxygen-18 and this compared with the normal oxygen-16. The kinetic reactions which the oxygen O^{16} , CO^{18} , CO_2^{16} , CO_2^{18} . This permitted the postulation of the degradation mechanism to be that of a unit by unit destruction of the polymer and the rate-controlling factor to be the number of available chain ends. Their paper contains numerous weight loss graphs and tables of which only a few typical ones are cited to illustrate the approach taken by these authors in generating meaningful weight loss data:



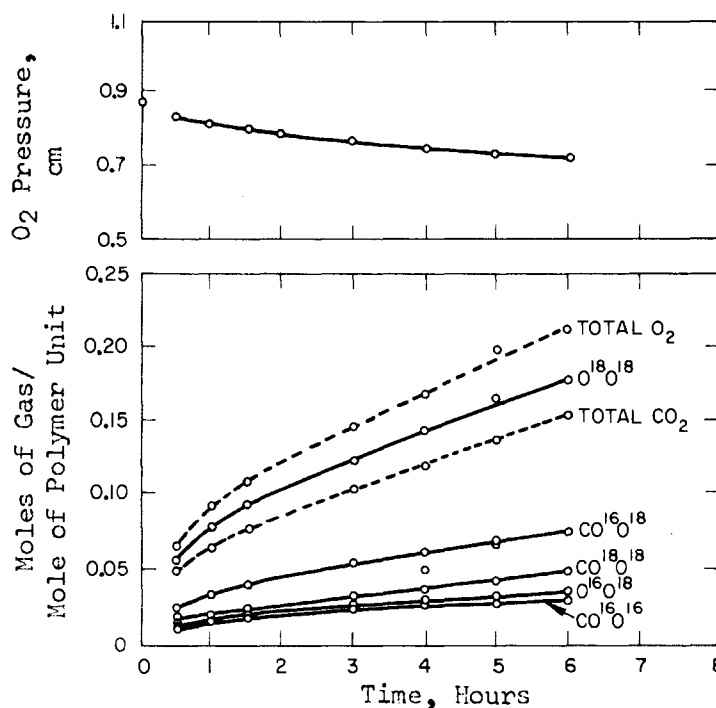
Consumed and evolved gases during oxidation of IM in air at indicated pressure of oxygen using gas circulating system (300°C).

[Ref. 118]



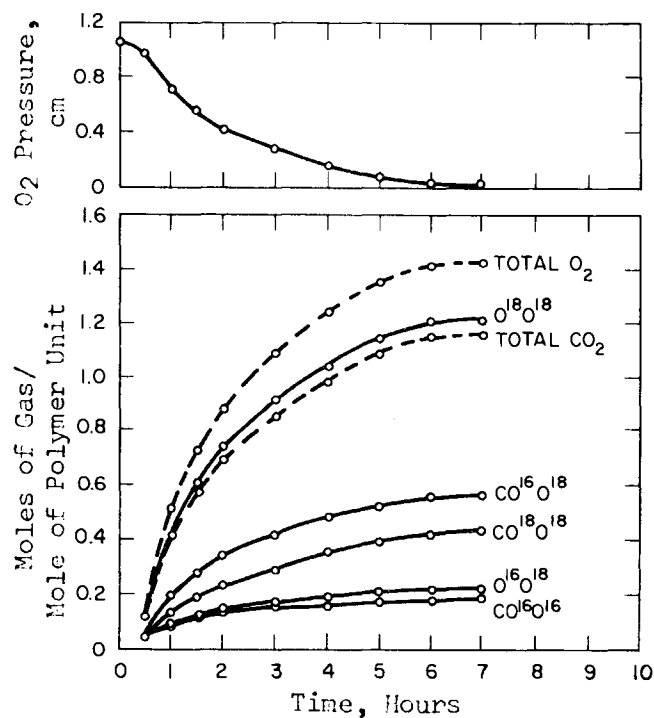
Gas evolution from a single sample of IMAM heated in vacuum for 1 hour at each successive temperature.

[Ref. 118]



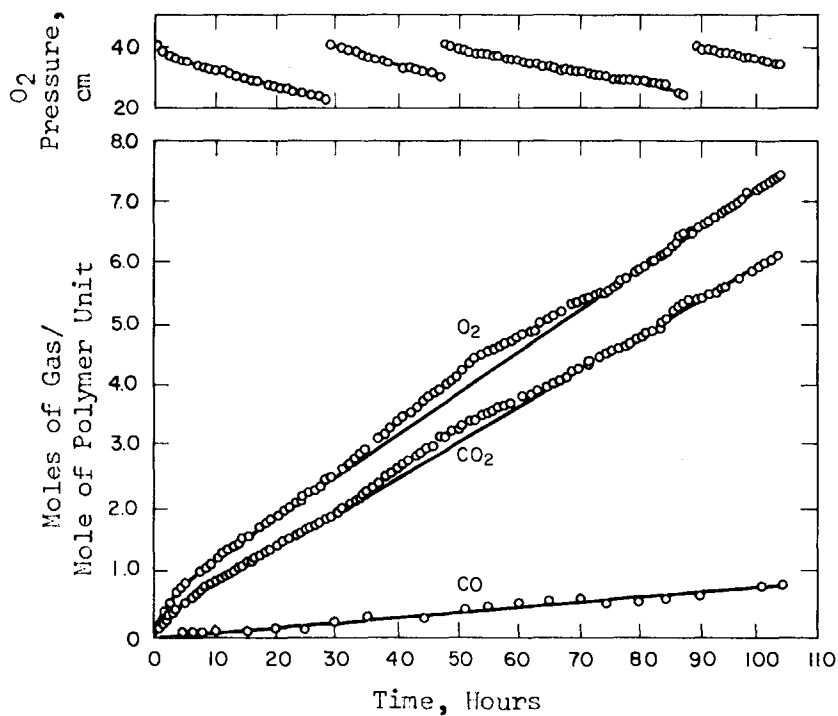
Consumed and evolved gases during oxidation of vacuum-treated IM at indicated pressures of isotopically enriched oxygen (300°C).

[Ref. 118]



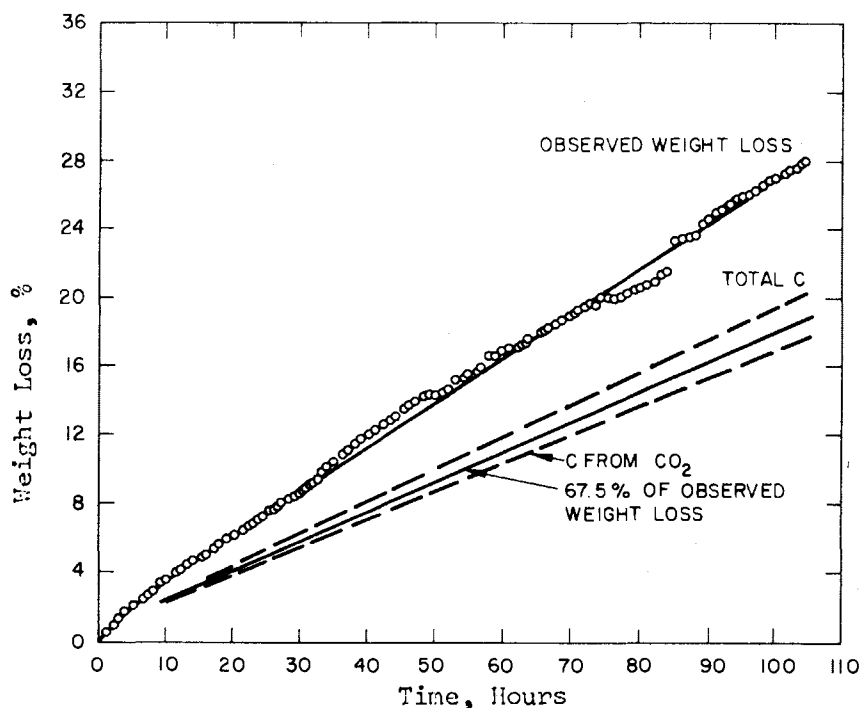
Gases consumed and evolved during oxidation of untreated IMAM at indicated pressure of isotopically enriched oxygen (300°C).

[Ref. 118]



Gases consumed and evolved in gas circulating system during oxidation of IMAM in oxygen-argon atmosphere at 360°C after degassing in He at 400°C.

[Ref. 118]



Observed and computed weight losses ($\Delta W/W_0$) from IMAM at 360°C in oxygen-argon atmosphere after degassing in He at 400°C.

[Ref. 118]

Heacock and Berr of the E.I. du Pont de Nemours Company have conducted thermal stability experiments on "Kapton" Type H-Film and found that the thermal stability and the dependence of the rate of degradation was a function of the availability of oxygen. In air at about 500°C, H-Film decomposes and completely disappears after 12 hours, while in a vacuum or in an inert atmosphere 60-65% of the film remains after prolonged aging at temperatures as high as 1000°C. The remaining residue retains its original shape but has lost all of its mechanical strength.

Pyrolyzing "Kapton" Type H-Film for 2 hours at 540°C in a vacuum yielded the following mass spectrographic analysis data:

Mass Spectrometric Analysis of Gaseous Degradation Products of H-Film
(Vacuum Pyrolysis 540°C-2 hours)

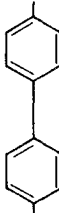
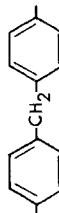
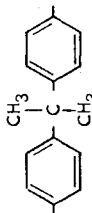
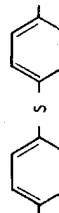
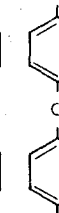
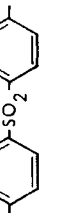
Main components are obviously CO and CO ₂	
Component	Mole, %
Benzene	0.7
Carbon dioxide	35.1
Carbon monoxide	58.7
Water	1.2
Hydrogen	2.6
Ammonia	Trace
Hydrogen cyanide	1.2
Benzonitrile	0.5
Wt. loss on sample	34.6%
Original sample weight	0.4382g

[Ref. 59]

Which indicates that CO and CO₂ are principal products and differ somewhat in percentages from Bruck's analysis reported previously.

7. Thermal Life

Thermal stability has been determined by Scroog and co-workers for polyimide films by weight loss measurements in air and helium atmospheres and by retention of film creasability upon heating in air. The data, summarized below indicate that polyimides as a class possess outstanding thermal stability. When considered along with the retention of mechanical integrity as evidenced by zero strength temperatures in the metallic range, these stabilities become truly significant.

Properties of Polypyromellitimides (R = Diamine Component)					
R	Solubility	Crystallinity	Zero strength temperature, °C	Thermal stability in air*	
				275 °C	300 °C
	Amorphous conc. H ₂ SO ₄ ; crystalline insol.	Crystallizable	900	> 1 yr.	> 1 mo.
	Amorphous conc. H ₂ SO ₄ ; crystalline insol.	Crystallizes readily	900	> 1 yr.	
	Fuming HNO ₃	Highly crystalline	> 900	-	1 mo.
	Conc. H ₂ SO ₄	Slightly crystalline	800		7-10 days
	Conc. H ₂ SO ₄	Crystallizable with difficulty	580		15-20 days
	Fuming HNO ₃	Crystallizable	800	10-12 months (estimated)	6 weeks
	Fuming HNO ₃	Crystallizable	850	1 yr.	> 1 mo.
	Conc. H ₂ SO ₄	-	-	-	> 1 mo.
	Conc. H ₂ SO ₄	-	-	-	> 1 mo.

* As measured by retention of film creasability.

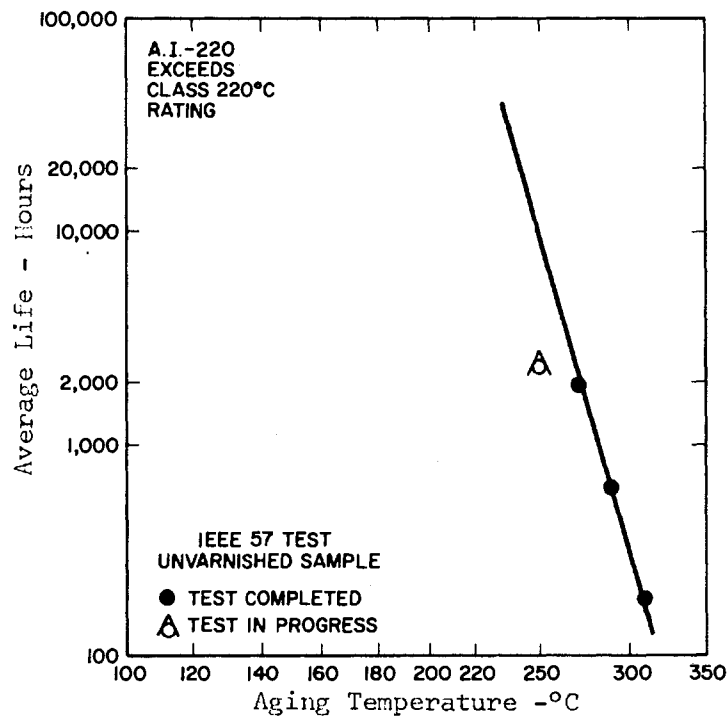
Heacock and Berr of the E.I. du Pont de Nemours Company have predicted life-times in air and in an inert atmosphere for "Kapton" Type H-Film based upon a 1% elongation failure point:

Property Deterioration Rate Parameters In Helium

<u>Property</u>	<u>P_o</u>	<u>E Kcal</u>	<u>K₄₀₀ 1/hour</u>
Elongation (%)	70	55.4 ± 1.7	0.0050
Impact Strength (kg-cm/mil)	6	51.1 ± 2.7	0.0029
Tensile (psi/1000)	23	55.5 ± 1.3	0.0019

[Ref. 59]

AI-220 magnet wire coating:

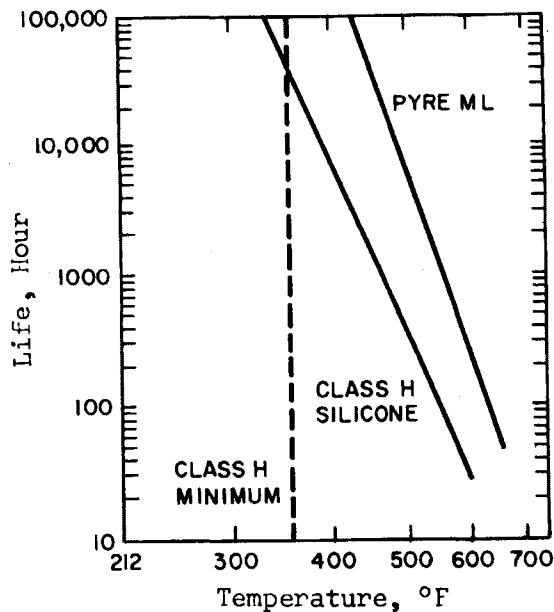


Thermal life curve of AI-220.

[Ref. 4, 8]

<u>Specimen</u>	<u>Temperature</u>	<u>Expected Life</u>
DuPont "Pyre-ML" RK-692	220°C	over 100,000 hours
	250°C	over 10,000 hours
	300°C	over 1,000 hours

[Ref. 35]



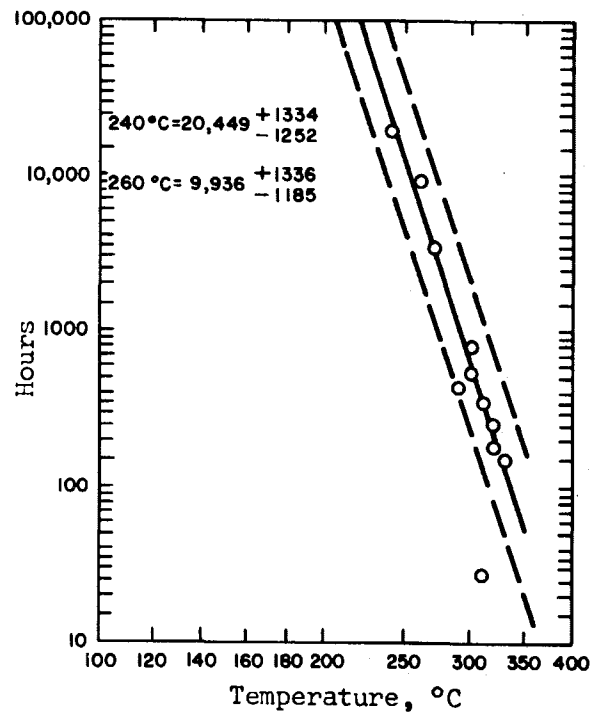
Thermal life comparison (AIEE No.57) of "Pyre-ML" wire enamel to published values for class H silicone shows polyimide superior.

[Ref. 11]

Unvarnished polyimide enameled wire:

Thermal life of unvarnished polyimide enameled wire.

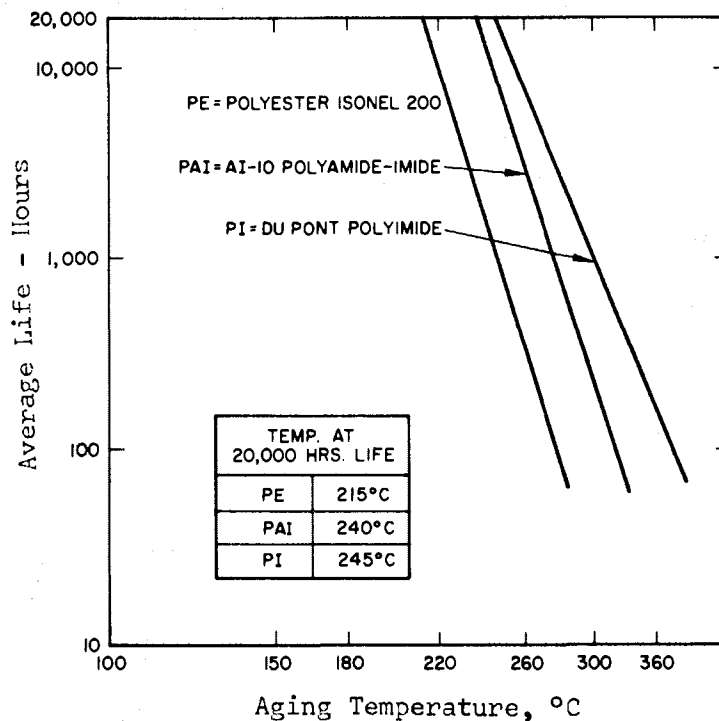
[Ref. 67]



<u>Insulating Varnish</u>	<u>Solid Content</u>	<u>Viscosity (poises)</u>	<u>Temp.</u>	<u>Twist Life of Varnished Wire</u>
DuPont RC-B-24951 (No. 18 HML wire)	45%	2-5	300°C 260°C	1015 hours 5222 hours
DuPont RK-692 (No. 18 HML wire)	13%	6-10	300°C 260°C	780 hours 9936 hours
DuPont RC-5060 (No. 18 HML wire)	19%	5-9	300°C 260°C	1654 hours 7303 hours

[Ref. 33]

Amoco Chemical Company has compared its AI-10 polyamide-imide polymer's life with polyester (Isonel 200) and DuPont polyimide using IEEE-57 test method:



[Ref. 104]

Conductive ML polyimide-coated glass fabric (1):

<u>Watt Density</u> <u>Watt/Ft²</u>	<u>Surface Temp.</u> <u>°C</u>	<u>Life, Hours (2)</u>	
		<u>Uncoated</u>	<u>Coated</u>
500	165	-	-
750	200	25,000	25,000
1000	250	1,700	2,400
1500	300	100	200
2000	350	5	20

Notes: (1) Coating Thickness 1.0 mil
Resistance 90 Ω/sq
Vol. Resistivity 0.22 Ω-cm
Emissivity 0.9

(2) Time to 10%
Resistance Increase

[Ref. 31]

The Amoco Chemical Company has compared its AI-10 polyamide-imide insulation material with polyester Isonel 200 and a DuPont polyimide in a burn out test in which twisted pairs of wires were subjected to 40 amperes with tension of 300 grams:

<u>Insulation Wire</u>	<u>Time to Fail (for short to occur)</u>
Polyester Isonel 200	90 seconds
AI-10 Polyamide-Imide	15 minutes +
DuPont Polyimide	15 minutes +

[Ref. 104]

Isomid (Polyester-imide):

Burnout test - twisted pairs, time in seconds to failure at 120 v.

40 amps	80
45 amps	36

[Ref. 134]

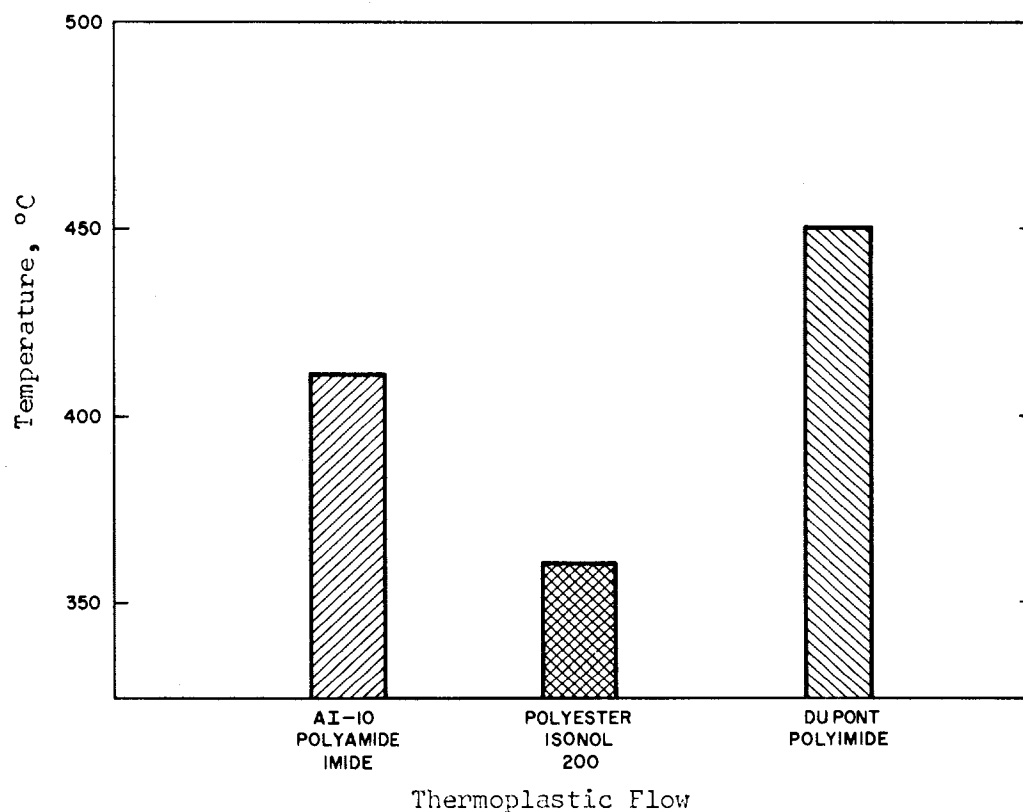
8. Heat Distortion

<u>Specimen</u>	<u>Load (ASTM D-648)</u>	<u>Heat Distortion Temperature</u>
SP Resin	264 psi	470°F [Ref. 10]
SP Polymer	264 psi	473°F [Ref. 97]

<u>Specimen</u>	<u>Load</u>	<u>Zero-Strength Temperature</u>
H-Film	20 psi (5 sec to failure)	1472°F (815°C) [Ref. 29]

<u>Specimen</u>	<u>Cut-Through Temperature</u>
H-Film	815°F (435°C) [Ref. 68]

The Amoco Chemical Company has compared the thermoplastic flow (under a 1000 gram load) of its AI-10 polyamide-imide polymer with a polyester Isonel 200 and a DuPont polyimide wire insulation at different temperatures:



[Ref. 104]

<u>Specimen</u>	<u>Temperature</u>	<u>Load</u>	<u>Deformation</u>
AI-220 Magnet Wire Coating	250°C	5000 gram	< 3%

[Ref. 8]

<u>Specimen</u>	<u>Heat Aging Temperature</u>	<u>Retention of Elongation (hours to 1% elongation)*</u>
"Pyre-ML"	400°C (752°F)	11
	350°C (662°F)	85
	300°C (572°F)	700
	250°C (482°F)	4500

* Initial % E = 25%

[Ref. 36]

<u>Specimen</u>	<u>Temp.</u>	<u>P/B Ratio</u>	<u>Final Sample Condition</u>
Polyimide coating formual #13	800°F	200/100	adhesion good*
DuPont RCW62480 +	900°F		adhesion good*
non-leafing Al pigment (on stainless steel)	1000°F		adhesion fair*
Polyimide coating formula #14	800°F	100/100	powder only after 5 hours
DuPont RCW62480 +	900°F		adhesion good**
non-leafing Al pigment (on stainless steel)	1000°F		adhesion good**
Polyimide coating formula #15	800°F	50/100	powder only after 5 hours
DuPont RCW62480 +	900°F		adhesion good**
non-leafing Al pigment (on stainless steel)	1000°F		adhesion good**
Polyimide coating formula #22	800°F	200/100	adhesion good*
polypyromellitimide +	900°F		adhesion good**
non-leafing Al pigment (on stainless steel)	1000°F		adhesion fair after 25 hours

* Some Al can be scraped off after 25 hours.

** Some Al can be scraped off at 25 hours.

[Ref. 1]

"Amanim" polyamide-imide film:

<u>Aging Temperature</u>	<u>Aging Time</u>	<u>Dimensional Stability (shrinkage)</u>
150°C	134 hours	0.5%

[Ref. 44]

"Kapton" Type H-Film:

<u>Thickness</u>	<u>Temperature</u>	<u>Shrinkage</u>	
		<u>Machine Direction</u>	<u>Transverse Direction</u>
1 mil	250°C	0.23%	0.31%
1 mil	400°C	3.0 - 3.5%	3.0 - 3.5%
2 mil	250°C	0.24%	0.35%
2 mil	400°C	2.0%	2.3%
5 mil	250°C	0.27%	0.37%
5 mil	400°C	1.2%	1.7%

[Ref. 31]

MEASUREMENTS OF SPECIMEN ALONE

Specimen Code *	Before Testing				After Testing		Net Losses	
	Diameter (inch)	Length (inch)	Mass (grams)	Density (grams/cm ³)	Length (inch)	Mass (grams)	Length Loss (inch)	Mass Loss (grams)
I8-35-C-1	.752	.502	4.820	1.32	.501	2.665	.001	2.155
I8-35-C-2	.752	.502	4.830	1.325	.469	2.840	.033	1.990
W-3-C-2	.751	.496	4.745	1.32	.420	2.812	.076	1.933
W-3-C-3	.750	.493	4.15	1.17	--	--	--	--
SP-1	.750	.488	5.035	1.42	--	--	--	--
SP-PT-2	.751	.509	6.135	1.66	.102	1.055	.407	5.080

MEASUREMENTS OF SPECIMEN ASSEMBLY(a)

Specimen Code *	Before Testing		After Testing		Gross Losses	
	Length (inch)	Mass (grams)	Length (inch)	Mass (grams)	Length Loss (inch)	Mass Loss (grams)
I8-35-C-1	1.998	19.35	2.001	16.309	-.003	3.041
I8-35-C-2	2.002	17.51	1.975	15.052	.027	2.458
W-3-C-2	1.994	21.26	1.910	17.177	.084	4.083
W-3-C-3	2.494	25.7	2.381	22.5	.113	3.2
SP-1	2.484	28.500	2.520	24.995	-.036	3.505
SP-PT-2	2.010	21.031	1.632	13.738	.378	7.293

* see following table

(a) Includes specimen, steel disk, Micarta holder, thermocouple and 5 feet of wire (latter on instrumented specimens only).

[Ref. 14]

The Research and Development Division of Avco Corporation in an arc-heater characterization study of ablative plastics made measurements on the dimensional changes of a number of filled polyimide and polyamide-polyimide resins under a variety of molding conditions:

*

Specimen Code	Reinforcing Agent	Resin System Designation	Nominal Resin Content (percent)	Molding Conditions		
				Pressure (psi)	Temperature (°F)	Time (minutes)
I8-35-C-1	carbon cloth	I-8 Polyimide	37	2000	700	60
I8-35-C-2	carbon cloth	I-8 Polyimide	37	2000	700	60
W-3-C-2	carbon cloth	AI-7 Polyamide-polyimide	42	2000	470	60
W-3-C-3	carbon cloth	AI-7 Polyamide-polyimide	35	2000	470	60
SP-1	none	SP Polyimide	100	NA	NA	NA
SP-PT-2	fibrous potassium titanate	SP polyimide	50	NA	NA	NA

[Ref. 14]

SUMMARY of HIGH TEMPERATURE EVALUATIONS OF SINGLE-COAT SYSTEMS

On Stainless Steel

Formula No.	Resin	Pigment	P/B	Appearance After Exposure		
				800°F	900°F	1000°F
13	RCW62480	N.L.A1***	200/100	*1 after 25 hours	*1 at 25 hours	*1 with fair adh** after 25 hours
14	RCW62480	N.L.A1***	100/100	powder only after 5 hours	*1 at 25 hours	*1 at 25 hours
15	RCW62480	N.L.A1***	50/100	powder only after 5 hours	*1 at 25 hours	*1 at 25 hours
22	PPMI	N.L.A1***	200/100	*1 after 25 hours	*1 at 25 hours	**adh fair after 25 hours
24	PPMI	N.L.A1***	50/100	*1 after 25 hours	*1 at 25 hours	failure after 1 hour
25	RCW62480	Leafing A1	200/100	*1 after 25 hours	*1 at 25 hours	coating integrity poor, failure 25 hours
26	RCW62480	Leafing A1	100/100	** adh poor after 25 hours	** adh poor after 25 hours	coating integrity poor, failure 25 hours
27	RCW62480	Leafing A1	50/100	**adh poor after 5 hours	** adh failure after 2 hours	** adh failure after 1 hour
34	PPMI	Leafing A1	200/100	*1 after 25 hours	*1 at 25 hours	*1 at 25 hours
37	RCW62480	SrCrO ₄	200/100	complete failure at 1 hour	complete failure at 1 hour	complete failure at 1 hour
38	RCW62480	SrCrO ₄	100/100	complete failure at 2 hours	complete failure at 1 hour	complete failure at 1 hour
39	RCW62480	SrCrO ₄	50/100	complete failure at 5 hours	complete failure at 1 hour	complete failure at 1 hour

(Table continued on following Page)

The Research and Development Division of Avco Corporation evaluated a wide range of single-coat and double-coat pigmented polyimide systems on stainless steel from 800 - 1000°F:

On Stainless Steel
(continued)

Formula No.	Resin	Pigment	P/B	Appearance After Exposure		
				800°F	900°F	1000°F
48	PPMI	SrCrO ₄	50/100	vehicle gone after 2 hours	failure at 1 hour	failure at 1 hour
49	RCW62480	Black	100/100	complete failure at 1 hour	failure at 1 hour	failure at 1 hour
59	PPMI	Black	50/100	complete failure at 1 hour	failure at 1 hour	failure at 1 hour

On Aluminum

14	RCW62480	N.L.A1***	100/100	**adh poor after 5 hours	discoloration after 25 hours	coating gone after 10 hours
22	PPMI	N.L.A1***	200/100	excellent after 25 hours	discoloration after 25 hours	coating gone after 25 hours
34	PPMI	Leafing A1	200/100	excellent after 25 hours	discoloration after 25 hours	*1 at 25 hours
48	PPMI	SrCrO ₄	50/100	failure at 2 hours	failure at 1 hour	failure at 1 hour
49	RCW62480	Black	100/100	complete failure at 1 hour	complete failure after 1 hour	complete failure at 1 hour
60	PPMI	Black	25/100	complete failure at 2 hours	complete failure after 1 hour	complete failure at 1 hour

* Adhesion good, but some aluminum can be scraped off.

** adh = adhesion

*** N.L. = non-leafing

[Ref. 14]

SUMMARY of HIGH TEMPERATURE EVALUATION of TWO-COAT SYSTEMS
(on Stainless Steel)

Formula No. *	<u>Appearance After Exposure</u>		
	<u>800°F</u>	<u>900°F</u>	<u>1000°F</u>
1 + 13	adhesion failure after 5 hours	coating dark grey, failure at 1 hour	coating white, adhesion failure at 1 hour
1 + 25	adhesion failure after 2 hours	coating dark grey, failure at 1 hour	coating white, adhesion failure at 1 hour
1 + 39	coating gone after 2 hours	coating gone after 1 hour	coating gone after 1 hour
10 + 22	slight browning, adhesion good at 10 hours	moderate browning/white specks at 10 hours	white pigment only, adhesion fair at 10 hours
10 + 34	slight browning, adhesion good at 10 hours	moderate browning/white specks at 10 hours	white pigment only, adhesion fair at 10 hours
10 + 48	no intercoat adhesion	as before	as before
49 + 13	adhesion failure after 2 hours	intercoat adhesion failure after 1 hour	coating integrity gone after 1 hour
49 + 25	adhesion fair after 10 hours	intercoat adhesion failure after 1 hour	coating integrity gone after 1 hour
49 + 39	coating gone after 2 hours	coating gone after 1 hour	coating gone after 1 hour
60 + 22	no intercoat wetting, puddling of top coat	as before	as before
60 + 34	no intercoat wetting, puddling of top coat	as before	as before
60 + 48	no intercoat wetting, puddling of top coat	as before	as before

* see following table

[Ref. 14]

SUMMARY OF TWO-COAT SYSTEMS

<u>Formula No.**</u>	<u>Resin</u>	<u>Top Coat</u>		<u>Primer</u>	
		<u>Pigment</u>	<u>P/B</u>	<u>Pigment</u>	<u>P/B</u>
1 + 13	RCW62480	TiO ₂	200/100	**N.L.Al	200/100
1 + 25	RCW62480	TiO ₂	200/100	Leafing Al	200/100
1 + 39	RCW62480	TiO ₂	200/100	SrCrO ₄	50/100
10 + 22	PPMI	TiO ₂	200/100	**N.L.Al	200/100
10 + 34	PPMI	TiO ₂	200/100	Leafing Al	200/100
10 + 48	PPMI	TiO ₂	200/100	SrCrO ₄	50/100
49 + 13	RCW62480	Black	100/100	**N.L.Al	200/100
49 + 25	RCW62480	Black	100/100	Leafing Al	200/100
49 + 39	RCW62480	Black	100/100	SrCrO ₄	50/100
60 + 22	PPMI	Black	25/100	**N.L.Al	200/100
60 + 34	PPMI	Black	25/100	Leafing Al	200/100
60 + 48	PPMI	Black	25/100	SrCrO ₄	50/100

* Top coat number is listed first.

** N.L. = non-leafing

[Ref. 12]

9. Flame Resistance

"Pyre-ML" coatings:

Flame resistance is excellent as the materials are non-flammable. When tested according to Federal Specification CCC-T-1916, method 5902, "Vertical Flame Test", the coated "Pyre-ML" glass fabrics rated zero (0) for after-flame and zero (0) for after-glow. There was no charring.

"Pyre-ML" varnish:

Flame spread resistance of "Pyre-ML" varnish is excellent; it decomposes slowly without visible burning in a flame. The possibilities of using this coating in retarding flame spread on wood surfaces was determined by using the ASTM-E-162-61 Radiant Panel Test. The ratings found by the DuPont Company were:

<u>Panel</u>	<u>Flame Spread Index</u>
Uncoated plywood	112
Plywood coated with "Pyre-ML"	20

Similar tests on cedar shingles, impregnated with "Pyre-ML" enamels, further demonstrated the effectiveness of the enamels in these uses.

[Ref. 67]

DuPont "Kapton" Film

"Kapton" film begins to char above 800°C.

Lewis and Stabler (Ref. 91) report that "Kapton" Type H polyimide film is self extinguishing for its flammability characteristic. The DuPont Company ran some exploratory tests using "Kapton" Type H as a flame retardent coating on plywood. A considerable reduction in flame spread was noted as measured by the Radiant Panel Flame Spread Test (ASTM-E-162):

<u>Material</u>	<u>Flame Spread Index</u>
1/4" exterior plywood control	115
1 mil "Kapton" polyimide film on plywood	5

[Ref. 31]

The DuPont Company reports the following flash point (open cup) test results for three of their insulating varnishes:

<u>Varnish</u>	<u>Solid Content</u>	<u>Viscosity (poises)</u>	<u>Solvent System</u>	<u>Open Cup Flash Point</u>
DuPont RC-B-24951	45%	2-5	NMP/Xylene	37°C
DuPont RK-692	13%	6-10	NMP/Aromatic Hydrocarbon	37°C
DuPont RC-5060	19%	5-9	NMP/Xylene	37°C

[Ref. 33]

10. Heats of Ablation

The Research and Advanced Development Division of Avco Corporation in their study of arc-heater characterization of ablatative plastics made some heats of ablation measurements on several polyimide reinforced resins:

Specimen Code *	Heats of Ablation		Initial Thickness of Specimen (in.)	Time for Back-Face Temperature Rise		Pseudodiffusivity		Insulative Cold-Wall Heat of Ablation	
	Cold-Wall (10 ³ Btu/lb.)	Thermochemical (10 ³ Btu/lb.)		T = 50°F (sec.)	T = 200°F (sec.)	T = 50°F (ft ² /hr.)	T = 200°F (ft ² /hr.)	T = 50°F (Btu/lb.)	T = 200°F (Btu/lb.)
18-35-C-1	44.5	-3.40	.502	10.4	23.2	.609	.272	2940	6570
18-35-C-2	29.5	-1.63	.502	No T.C.	No. T.C.	No T.C.	No T.C.	No T.C.	No T.C.
W-3-C-2	31.6	-1.81	.496	(a)	11.1	--	.554	--	3190
W-3-C-2	31.6	-1.81	.496	(a)	11.1	--	.554	--	3190
W-3-C-3	31.7	1.83	.493	10.6	19.0	.572	.318	3550	6360
D-A-1-2	15.0	6.06	.471	20.1	47.5	.275	.116	5310	12560
18-35-R-1	12.2	7.42	.502	19.5	40.7	.323	.155	4500	9390
18-35-R-2	13.8	8.59	.502	No T.C.	No. T.C.	No T.C.	No T.C.	No T.C.	No T.C.
W-3-R-4	9.6	7.15	.484	25.1	43.9	.232	.132	6720	11760
SP-1	37.7	4.95	.488	22.5	48.5	.264	.121	6250	13470
SP-PT-2	6.4	2.97	.509	8.6	(i)	.747	--	1960	--

*See following table

(a) Excessive electrical noise in date; no valid reading obtained.

[Ref. 14]

Specimen Code	Reinforcing Agent	Resin System Designation	Nominal Resin Content (percent)	Molding Conditions		
				Pressure (psi)	Temperature (°F)	Time (minutes)
I8-35-C-1	carbon cloth	I-8 Polyimide	37	2000	700	60
I8-35-C-2	carbon cloth	I-8 Polyimide	37	2000	700	60
W-3-C-2	carbon cloth	AI-7 Polyamide-polyimide	42	2000	470	60
W-3-C-3	carbon cloth	AI-7 Polyamide-polyimide	35	2000	470	60
SP-1	none	SP Polyimide	100	NA	NA	NA
SP-PT-2	fibrous potassium titanate	SP Polyimide	50	NA	NA	NA
D-A-1-2	refrasil	SP Polyimide	50	NA	NA	NA
18-35-R-1	refrasil	I-8 Polyimide	31	2000	700	60
18-35-R-2	refrasil	I-8 Polyimide	31	2000	700	60
W-#-R-4	refrasil	AI-7 Polyamide-polyimide	35	2000	470	60

[Ref. 14]

Specimen *	Gas Enthalpy		Heat Flux				Density (lb/ft ³)	Steady-State Loss Rates			Heat of Ablation	
	Free Steam (Btu/lb)	Surface (Btu/lb)	Cold-Wall (Btu/ft ² - sec)	Hot Wall (Btu/ft ² - sec)	Surface Radiation (Btu/ft ² - sec)	Net (Btu/ft ² - sec)		Length		Mass (10 ⁻² lb/ft ² - sec)	Cold-Wall (10 ³ Btu/lb)	Thermo- chemical (10 ³ Btu/lb)
								(10 ⁻³ in/sec)	(10 ⁻⁴ ft/sec)			
18-35-C-1	9,400	2,940	978	623	698	- 75	82.5	3.2	2.7	2.2	44.5	-3.40
18-35-C-2	9,300	2,940	978	620	674	- 54	82.8	4.8	4.0	3.3	29.5	-1.63
W-3-C-2	9,300	2,970	978	618	674	- 56	82.5	4.5	3.75	3.1	31.6	-1.81
W-3-C-3	8,520	2,860	1,005	624	566	58	73.0	5.2	4.3	3.2	31.7	1.83
D-A-1-2 (4)	8,580	2,060/1,630	1,004	713/763	417/247	296/516	96.9	8.3	6.9	6.7	15.0	6.06
18-35-R-1	8,930	1,650	993	759	154	605	103.0	9.5	7.9	8.1	12.2	7.42
18-35-R-2	8,900	1,520	978	762	154	608	100.0	8.5	7.1	7.1	13.8	8.59
W-3-R-4	7,280	1,620	982	719	123	596	91.0	11.0	9.2	8.4	9.6	7.15
SP-1	8,640	2,350	1,004	681	550	131	88.8	3.6	3.0	2.7	37.7	4.95
SP-PT-2	8,700	1,870	993	729	266	463	103.5	18.1	15.1	15.6	6.4	2.97
HF-1	8,610	2,450	977	665	437	228	209.0	8.9	7.4	15.5	6.4	1.47

*See following table

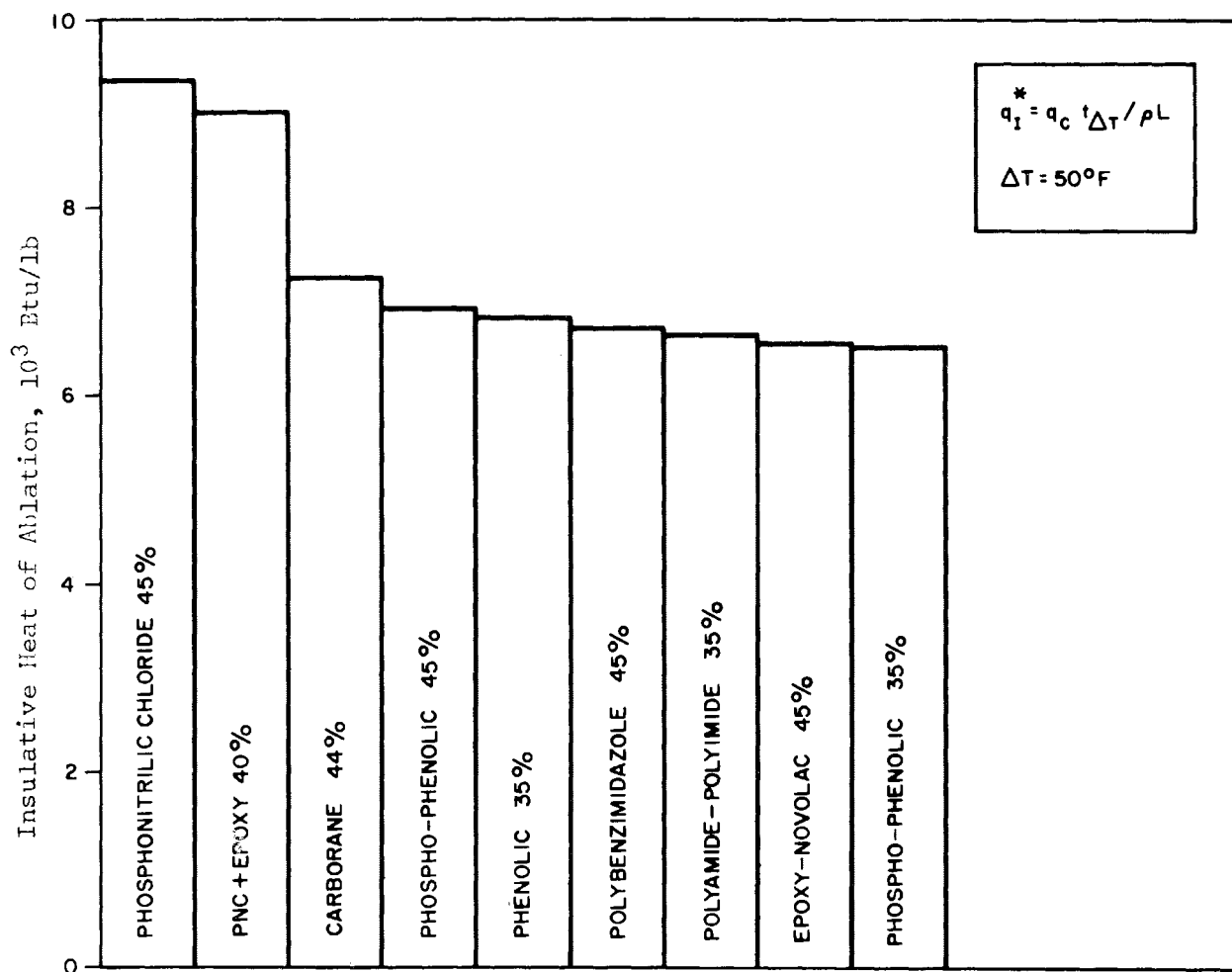
[Ref. 14]

Specimen Code	Reinforcing Agent	Resin System Designation	Nominal Resin Content (percent)	Molding Conditions		
				Pressure (psi)	Temperature (°F)	Time (minutes)
I8-35-C-1	carbon cloth	I-8 Polyimide	37	2000	700	60
I8-35-C-2	carbon cloth	I-8 Polyimide	37	2000	700	60
W-3-C-2	carbon cloth	AI-7 Polyamide-polyimide	42	2000	470	60
W-3-C-3	carbon cloth	AI-7 Polyamide-polyimide	35	2000	470	60
SP-1	none	SP Polyimide	100	NA	NA	NA
SP-PT-2	fibrous potassium titanate	SP Polyimide	50	NA	NA	NA
D-A-1-2	refrasil	SP Polyimide	50	NA	NA	NA
18-35-R-1	refrasil	I-8 Polyimide	31	2000	700	60
18-35-R-2	refrasil	I-8 Polyimide	31	2000	700	60
W-#-R-4	refrasil	AI-7 Polyamide-polyimide	35	2000	470	60

[Ref. 14]

According to the Avco researchers, the pure SP-type polyimide resin had a tendency to spall, expand, and delaminate. When reinforced with fibrous potassium titanate, this resin still exhibited below average ablation behavior. However, the abalative performance of type I-8 polyimide was satisfactory with Refrasil or carbon cloth reinforcement.

Avco compared the insulative heat of ablation of polyimide-Refrasil specimens with other plastic-Refrasil specimens in the following graph:



II. PROPERTIES

G. IRRADIATION EFFECTS ON PROPERTIES

Because the polyimide plastics exhibited such good high temperature resistance characteristics, they were soon tested for radiation resistance to determine their potential for nuclear and space applications. Hence, a number of organizations have conducted gamma ray, neutron (fast neutron and thermal), electron irradiation combination tests for various time exposure periods, in air and in vacuum, at ambient and cryotemperature levels, and mainly on H-Film and SP Polymer materials. The resulting effects on the tensile, elongation, and electrical properties are detailed below.

1. H-Film

General Dynamics (Fort Worth) irradiated DuPont H-Film with gamma rays (up to 8.8×10^9 ergs/gm(C)), thermal neutrons (up to 1.52×10^{14} n/cm²), E > 2.9 Mev neutrons (up to 7.5×10^{15} n/cm²), E > 8.1 Mev neutrons (5.6×10^{13} n/cm²) either in air or in a vacuum of 3×10^{-7} torr. Their results are summarized in the following tables:

Trade Name	Gamma Dose [ergs/gm(C)]	Vacuum (torr)	Specimen Configuration	Measured Property	Percent Change in Measured Property
DuPont H-Film	0 Vacuum Controls	1×10^{-6}	Straight tensile specimens	Tensile strength @ 25%	- 2.2
				Tensile strength @ 50%	- 3.2
				Ult. tensile strength	- 8.3
				Ult. elongation	- 1.4
	8.8×10^9	3×10^{-7}	Straight tensile specimens	Tensile strength @ 25%	0
				Tensile strength @ 50%	0
				Ult. tensile strength	0
				Ult. elongation	0

[Ref. 53]

Test Environment and Results for Low-Force Dynamic-Test Materials: Electrical Insulation

Material and Test Equipment	Radiation Exposure				Machine Crosshead Speed (in./min)	Tensile Strength (psi)				Ultimate Elongation (%)	Temperature		Pressure
	Gamma [ergs/gm (C)]	Neutrons (n/cm ²)				@ 25% Elongation	@ 50% Elongation	@ 100% Elongation	Ultimate		Avg (°F)	Avg (torr)	
		Thermal	E>2.9 Mev	E>8.1 Mev									
H-Film Instron Tester	0	0 (control specimens) January 1962	0	0	5	15110/200/3 ^a	15960/200/3 ^a	-	20990/470/3 ^a	151/8.9/3 ^a	77	760	
Instron Tester	1.58(10) ^c	0 (air irradiation) January 1962	4.8(15) ^c	0	5	15138/363/8	16101/376/7	-	19795/714/8	118/10.5/8	-	-	
Instron Tester	4.04(10)	0 (air irradiation) January 1962	7.5(15) ^c	0	5	15045/360/6	16186/234/6	-	17983/647/5	81.6/7.3/5	-	-	
Instron Tester	0	0 (control specimens) December 1962	0	0	5	14700/340/4	15362/243/4	17772/180/4	20520/923/4	155/16.5/4	77	760	
Instron Tester	0	0 (control specimen) October 1962	0	0	0.5	14220 14160 14400 14580 14650 14402/211	15000 14580 15230 15500 15500 15162/396	17200 16950 17020 17620 17110 17192/335	17380 18230 17370 19740 17110 17996/1131	105 130 101 138 - 119/21.9	77	760	
Instron Tester	8.75(9)	1.52(14) ^c (vacuum irradiation) September 1962	2.47(15) ^c	5.6(13) ^c	0.5	14150 14580 13800 14177/461	15420 15500 14830 15250/396	b	16600 20600 18400 16950 18138/1943	85 149 120 94 112/28.3	132	3(-7)	
Instron Tester		(control specimens) February 1963			0.5	14492 14153 14746 14407 14450/288	15254 14746 15593 15085 15195/411	17203 16780 17966 17288 17309/576	18475 16780 17966 17288 17627/823	118 100 100 100 105/8.7	-	-	
Instron Tester		(vacuum controls) February 1963			0.5	14831 14915 14746 14576 14767/165	15932 15848 15509 15509 15700/205	18220 18136 17712 17881 17987/247	19831 19273 19322 18475 19216/659	127 118 130 113 122/8.3	-	-	

^aAverage value/standard deviation based on an individual basis/number of specimens.

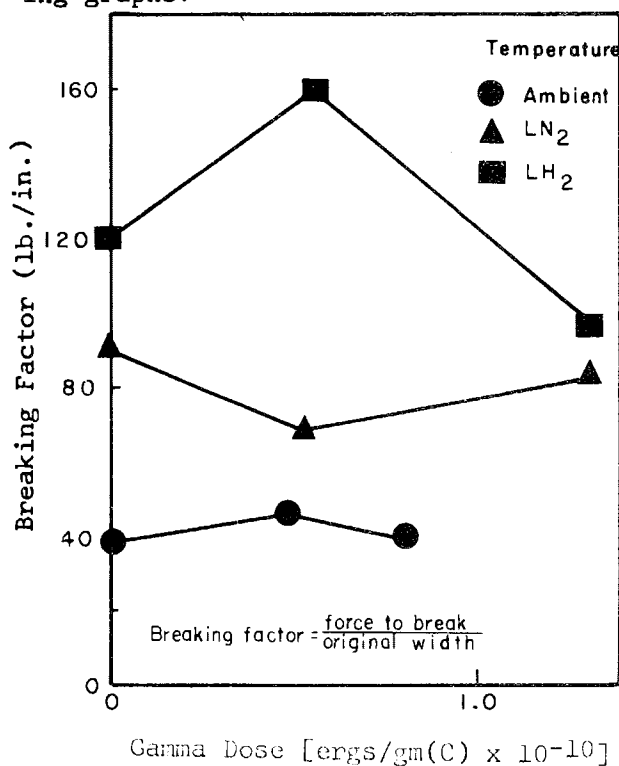
^bBroke in jaws.

^cNumbers in parentheses are exponents of base 10.

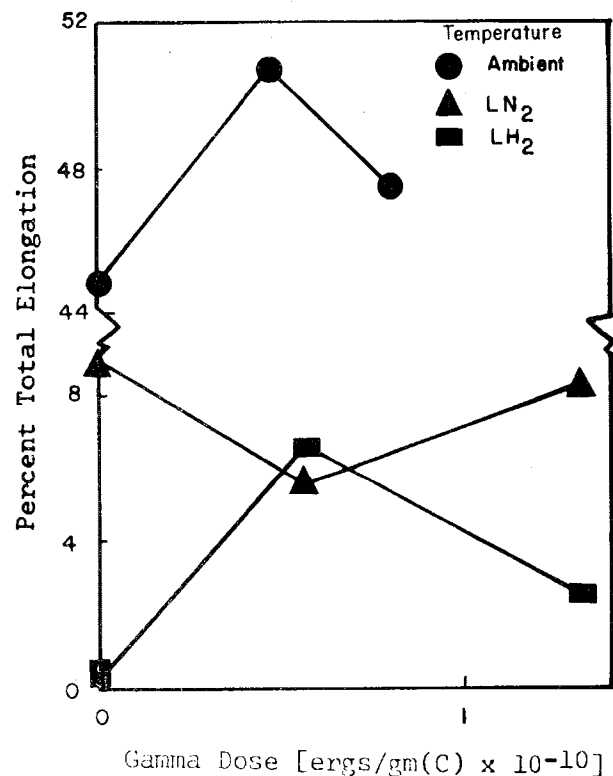
[Ref. 53]

The film was tested according to ASTM D-882-56T with the Low-Force Instron Tester and only the results of the static samples of the vacuum-irradiation test reported. After a dose of 4.0×10^{10} ergs/gm(C) in air, the ultimate elongation was reduced by 46% of the original value and the ultimate tensile strength decreased by 15% of the original value. General Dynamics felt that there was no significant change in ultimate tensile strength and ultimate elongation due to the high-vacuum and radiation-dose environment. They noted that the difference in averages between the vacuum-irradiation static samples and the control samples is less than the statistical variation within the sample batch.

General Dynamics (Fort Worth) also conducted H-Film gamma irradiations at three temperatures (ambient, liquid nitrogen, liquid hydrogen) and tested the effects on breaking factor and also on elongation as shown in the following graphs:



Breaking Factor vs Gamma Dose for
Three Different Temperatures:
Material G (H-Film)



Percent Total Elongation vs Gamma Dose
for Three Different Temperatures:
Material G (H-Film) [Ref. 52]

It will be noted that the ultimate strength of the H-Film is significantly higher at the lower temperatures and also at the higher doses the strength at liquid hydrogen temperatures decreased sharply. The percent elongation followed the same trends at the three temperatures.

The Redstone Scientific Information Center (1964) reported similar data (from General Dynamics, Fort Worth sources) in a survey on space environmental effects on materials. Gamma ray and neutron irradiation effects on the tensile strength and ultimate elongation properties of 2 mil H-Film are shown in the following table:

Material Trade Name	Gamma [ergs/ gm(C)]	Radiation Exposure			Time Until Test, days	Sample Weights		Tensile Strength*(psi)				Ultimate Elongation, percent	Temperature	Pressure
		Neutron (n/cm ²)				Original, gm	Change, gm	at 25% Elongation	at 50% Elongation	at 100% Elongation	Ultimate		Avg. , °F	Avg. , torr
		Thermal	E>2.9 Mev	E>8.1 Mev										
H-film (2 mil)	0	0 (control specimen)	0	0		-	-	14661	15593	18475	20678	140	-	-
								14237	15339	17627	19068	120		
								14407	15678	18305	19407	125		
								15678	16610	19322	20508	140		
								14492	15254	17542	18644	140		
								15339	16186	19068	20339	130		
								-	15085	17966	17966	100		
								-	15085	15847	19153	128		
								14802/569	15604/536	18019/1221	19470/953	128/14		
	6.0(9)	1.26(15) (air irradiation)	0	16	-	-	15169	15678	-	16356	-	100	-	
							14831	15254	-	17797	110			
							14661	15678	-	18729	115			
							15254	16102	-	20508	135			
							15424	16102	-	20508	130			
							15068/328	15763/365	-	18780/1785	123/12			
	3.9(10)	2.8(14)	6.1(15) (air irradiation)	2.6(14)	16	-	-	15678	16610	-	17458	65	100	-
								15678	16525	-	17288	60		
								15085	15932	-	18136	105		
								15424	16441	18729	18729	100		
								15466/288	16377/329	18729	17903/700	83/22		
	1.05(10)	1.32(14)	1.65(15) (vacuum irradiation)	5.90(13)	11	-	-	14831	16017	-	16949	90	150	1(-3)
								14831	15847	-	20339	135		
								14831	15593	-	19492	125		
								14254	16102	-	21102	138		
								14937/205	15890/247	-	19871/2017	122/23		
	2.85(10)	3.88(14)	6.62(15) (vacuum irradiation)	2.09(14)	10	0.1352	0.0000	15169	15932	-	15932	50	200	6(-7)
						0.1464	-0.0015	-	-	-	-	-		
						0.1420	-0.0003	-	-	-	19492	120		
						0.1401	-0.0009	15000	16016	-	18898	105		
						0.1373	-0.0001	14576	16355	-	21186	135		
						14915/350	16101/250	-	18877/2552	103/41				

Note: numbers in parenthesis are exponents of base 10.

[Ref. 82]

Tatum et al. have acclaimed that H-Film has the best resistance to ionizing radiation of any other available organic film. After 10^8 roentgens (10^6 R/hour rate) exposure to gamma rays from a Cobalt 60 source at Oak Ridge, there was no significant change in the mechanical or electrical properties of H-Film. For the same exposure, however, Mylar retains less than 50% of its original tensile strength and elongation, while Teflon TFE retains less than 10% of its original value. A Plastics Technology Staff report (December 1962) noted that the radiation resistance of H-Film appears quite good. Exposed to gamma radiation, it remains flexible (180° bend) although it darkens slightly after exposure to 5×10^9 rep (for comparison, polyester film fails at levels of 10^5 rep). When the H-Film was exposed to high energy electrons from a Van De Graaff accelerator in air at 10 watt-sec/sq. cm., the elongation decreased. Extrapolating the data obtained after 100-minute exposure gives exposure times of 150 and 250 minutes required to produce a drop in 25% of original elongation in two specimens of the H-Film. Exposure to mixed neutrons and gamma rays in the Brookhaven pile, at 350°F , they report, resulted only in darkening and toughening of the film after exposures equivalent to 0.55 pile years (one pile year is 10^{13} n/sec for 365 days).

Harrison & Proulx at the Sandia Corp. (1962) irradiated three H-Films (0.024 cm thickness, $d_4^{20} = 1.39$) with gamma ray (range from 1.8×10^{-3} to 6.0×10^3 rads) (H_2O)/sec at 38°C and measured the photoconductivity with an applied field of 2.4×10^3 V/cm. For the photoconductivity constants " δ " and " A " given by the equation:

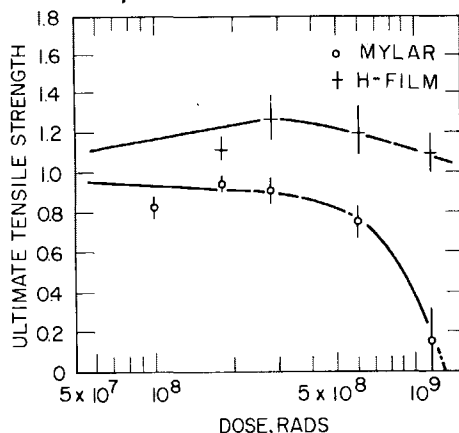
$$(\sigma - \sigma_0) = A\gamma^\delta$$

they found the following values:

$$\delta = 1.1$$

$$A = 5.8 \times 10^{-18}$$

Koehler and associates at Harvard University [Ref. 106], irradiated by means of a 160 MeV external proton beam of the Harvard Synchrocyclotron 1 mil Mylar and 1 mil H-Film (polyimide) to an irradiation dose of 10^{11} ergs $\text{g}^{-1}(\text{C})$. The specimens were tested in an Instron tensile tester using a 5 kilogram load cell. Jaw separation was 0.25 inches and jaw travel was 0.1 inch per minute, that is 40% elongation per minute. The results are plotted in the following graph:



[Ref. 106]

EFFECT OF GAMMA RAY EXPOSURE 1 MIL "KAPTON" POLYIMIDE FILM

Source: Cobalt 60 at Oak Ridge, Tenn.

<u>Property</u>	<u>Control</u>	<u>10⁶R</u>	<u>10⁷R</u>	<u>10⁸R</u>	<u>10⁹R</u>	<u>2.25x10⁹R</u>
Tensile Strength (psi x 10 ⁻³)	30	30	31	31	22	24.9
Elongation, (%)	80	78	78	79	42	51.7
Initial Tensile Modulus (psi x 10 ⁻³)	460	475	490	475	421	458
Volume Resistivity (Ω-cm) 200°C	4.8x10 ¹³	6.6x10 ¹³	5.2x10 ¹³	1.7x10 ¹³	1.6x10 ¹³	1.5x10 ¹³
Dielectric Constant 23°C						
10 ³ CPS	3.46	3.54	3.63	3.71	3.50	3.89
10 ⁴ CPS	3.44	3.54	3.62	3.69	3.48	3.86
10 ⁵ CPS	3.42	3.51	3.59	3.66	3.38	3.81
Dissipation Factor 23°C						
10 ³ CPS	0.0020	0.0023	0.0024	0.0037	0.0029	0.00424
10 ⁴ CPS	0.0060	0.0046	0.0046	0.0060	0.0057	0.00671
10 ⁵ CPS	0.0081	0.0086	0.0083	0.0089	0.0094	0.01040
Dielectric Constant 200°C						
10 ³ CPS	2.91	2.99	3.02	3.06		
10 ⁴ CPS	2.90	2.97	3.01	3.15		
10 ⁵ CPS	2.89	2.97	3.00	3.14		
Dissipation Factor 200°C						
10 ³ CPS	0.0008	0.0012	0.0007	0.0013		
10 ⁴ CPS	0.0011	0.0013	0.0004	0.0015		
10 ⁵ CPS	0.0013	0.0034	0.0016	0.0031		
Dielectric Strength (V/mil)	6500	5660	5540	5700	6460	[Ref. 31]

The DuPont Co. exposed 1 mil "Kapton" Type H-Film to cobalt 60 (gamma ray) irradiation at Oak Ridge, Tenn., and measured the degradation effects on both the mechanical and electrical properties. The results were privately communicated to the author in the following table:

The polyimide film became stronger with exposure up to 5×10^{10} ergs $\text{g}^{-1}(\text{C})$. Samples were irradiated at a dose rate of 3.5×10^7 ergs $\text{g}^{-1}(\text{C})/\text{min}$. which is equivalent to a 160 MeV proton flux of 6.5×10^{10} protons/ cm^2/sec . In these tests, the polyimide film appeared to be 10 times less sensitive to radiation damage than Mylar. This was confirmed by gamma irradiation to 10^{11} ergs $\text{g}^{-1}(\text{C})$ and an electron irradiation to 6×10^{11} ergs $\text{g}^{-1}(\text{C})$. These exposures showed that H-Film was 50 times less sensitive than Mylar. It was concluded in this study that proton irradiation was neither more or less efficient at causing radiation damage in Mylar or H-Film than electron or gamma irradiation.

Lockheed [Ref. 107] found that Polyimide H-Film remained relatively flexible and easy to handle at liquid nitrogen temperatures. Only the yield tensile strength increased when the temperature was lowered. No radiation effects were noted at an exposure dose of 8.8×10^8 ergs $\text{g}^{-1}(\text{C})$.

2. "Kapton" Type HF

The DuPont Company has tested "Kapton" polyimide film Type HF (composite of polyimide "Kapton" Type H and Teflon FEP) insulation wire by exposing it to 10^9 rads in a Van De Graaff generator. The wire was still flexible and passed dielectric breakdown tests after this exposure.

The Tensolite Insulated Wire Company contracted with the Radiation Dynamics, Inc. to conduct irradiation tests of the same "Kapton" Type H-Film-Teflon FEP wire insulations in their DYNAMITRON at 200 megarad and 2000 megarad exposure. The results are summarized in the following two tables:

Wire Gauge	Insulation	Wall Thickness	Dielectric Strength (volts)	
			Unirradiated	After 200 megarads
24	TFE	.010	9,600	—0— (1)
24	Kynar	.010	12,000	—0— (1, 2)
24	FEP-ML	.0065	7,700	—0— (1)
20	Polyethylene	.025	15,000	5,500
24	H-Film FEP	.010	16,000	16,000

(1) Sample cracked on bending.

(2) By slow and careful manipulation, one sample was wrapped around the mandrel carefully without cracking and gave 10,000 volts.

Wire Gauge	Insulation	Wall Thickness	Dielectric Strength (volts)	
			Unirradiated	After 2,000 megarads
16	H/FEP	.0025	5,000	2,000
16	H/FEP	.0045	11,000	6,200
16	H/FEP	.007	13,000	13,000
22	H/FEP	.003	10,000	7,850
22	H/FEP	.005	14,000	9,900
22	H/FEP	.0075	16,400	13,450
26	H/FEP	.003	9,800	7,400
26	H/FEP	.0045	9,900	5,500
26	H/FEP	.008	15,000	13,000
16	All-Polyimide	.004	12,000	12,000

[Ref. 95]

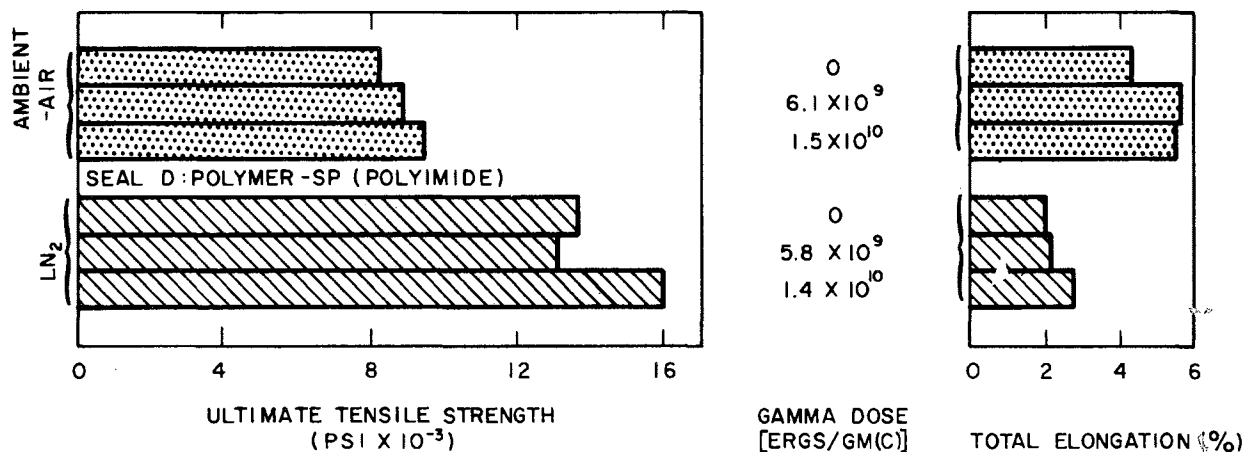
The samples were tested by a wet hipot after five turns on a 1/4-inch mandrel. Measurements were made consecutively on unirradiated and irradiated samples as soon after exposure as was feasible. Analysis by the Tensolite people indicated that the "Kapton" Type H-Film was superior in radiation resistance and was unaffected, appearance-wise, by the irradiation. In the 2000 megarad exposures, an average of all samples indicated that 73% of the dielectric strength had been retained: the heavier wall samples stood up somewhat better, retaining over 80%; and the thinner wall samples - poorer, somewhat less than 70%.

Several laboratories have conducted tests on the radiation resistance of Teflon FEP-"PYRE M.L." insulation system. It was found that the "PYRE M.L." film, while it does not seem to have a radiation shielding effect, will reduce the damage due to radiation on the FEP, since the "PYRE M.L." has a relatively low oxygen permeability. The reported threshold dosage for FEP-"PYRE M.L." wire at which deterioration of the physical properties (tensile strength and elongation) become noticeable is approximately 10^8 rads compared to 5×10^5 rads for uncoated Reflon in a 10^{-5} mm Hg vacuum at 250°C.

The threshold dosage for "PYRE M.L." alone is about 10^9 rads and the 50% value for retention of physical characteristics is 10^{11} rads.

3. SP Polymer

General Dynamics (Fort Worth) (1964) conducted combined (gamma ray and neutron) radiation-cryotemperature experiments on a Polymer-SP-1 (Seal D) polyimide material at ambient-air and liquid nitrogen temperatures. Their resulting data on the irradiation effects on the ultimate tensile strength and total elongation are summarized in the following:



[Ref. 54]

Campbell and Chenny (1965) noted that Polymer SP encounters threshold damage when exposed to a radiation level of 7×10^9 rads in a 2 MeV Van De Graaff beam. Another reference reports that a sample of unfilled polyimide

SP-Polymer irradiated in a Van De Graaff generator at a rate of 10 watts/sq. cm. (equivalent to 4×10^9 rads) showed no significant change in tensile strength or elongation, or change in appearance.

4. "PYRE M.L."

Scarborough [Ref. 108] found that DuPont "PYRE M.L.", a polyimide resin wire enamel insulation, did not exhibit noticeable degradation of dielectric properties after an exposure dose of 10^{10} ergs $\text{g}^{-1}(\text{C})$. The insulation material was used on the stator of a generator being tested for use for electrical power systems on vehicles which are to be propelled by nuclear power.

In tests on coated glass fabrics and on electrical insulation systems, "PYRE M.L." has shown excellent radiation resistance according to the DuPont Co. Exposure to 3000 megarads of either 1.33 and 1.17 MeV gamma radiation or 2 MeV electrons produced essentially no change in the electrical properties or general appearance of the "PYRE M.L.". Further, thermal stability appears to improve under exposure to combined heat and radiation. Leppla and Carryer (1963), and Schweitzer et al. yield the following table of data for polyimide ("PYRE M.L.") coated fabrics and insulation systems exposed to ionizing radiation:

Radiation Resistance

Property	Dosage (Megarads)			
	0	1000	2000	3000
Polyimide coated fabrics have shown good resistance to ionizing radiation. This table shows the effect of various dosages of 2 MeV electrons on the electrical properties of the 4-mil coated product (6500-004).				
Dissipation Factor @ 10^3 cps	0.0062	0.0310	0.0259	0.0388
Dielectric Constant @ 10^3 cps	3.5	3.4	3.9	4.2
Volume Resistivity, ohm-cm	6.5×10^{14}	5.8×10^{14}	5.3×10^{14}	3.1×10^{14}
Electric Strength, Vpm	1700	1610	1720	1695

Motorettes which were made with a complete polyimide insulation system appeared unchanged after exposure to similar dosages of gamma ray at 1.33 and 1.17 MeV in a cobalt garden. About 500 hours exposure were required for 3000 megarads dosage. Voltage breakdowns of these motorettes unexposed and after exposure were as follows:

Dielectric Strength, Vpm	1460	1280	900	1350

[Ref. 67, 89 & 34]

The DuPont Company states that exposure of its RK-692 polyimide varnish to 3000 megarads of electrons caused only minor changes in electrical and physical properties. The DuPont Company also carefully studied the effects of radiation on POP-PI film (poly[N,N'-(p,p'-oxydiphenylene)pyromellitimide]) which is closely related to H-Film. The POP-PI film keeps its strength after 40 days exposure to a high neutron flux of 0.4×10^{13} neutrons/sq.cm./sec.

The data in the following table indicate that films of the aromatic polypyromellitimides are outstandingly resistant to irradiation from high energy electrons and from thermal neutrons. Thus films of the polypyromellitimide

from bis(4-aminophenyl)methane exhibit good mechanical and electrical properties after high energy electron exposure of over 10,000 Mrad in the Van de Graaff generator while films of polystyrene and poly(ethylene terephthalate) become embrittled after 500 - 600 Mrad. Films of the polypyromellitimide from bis(4-aminophenyl)ether remain creasable after 40 days exposure to thermal neutrons at 175°C in the Brookhaven pile.

Effect of Radiation on Polypyromellitimide Films

(Exposure Van de Graaff 2 M.e.v. Electrons)

<u>Polymer Film</u>	<u>Thickness, mils</u>	<u>Number passes</u>	<u>Dose, Mrad</u>	<u>Remarks</u>
DDM-PI (a)	2.0	8000	10,000	Retains toughness, good electrical properties
Mylar (b)	2.0 - 3.0	200	240	Creasable brittle, yellow yellow, brittle
	2.0 - 3.0	500	600	
Polystyrene	1.2	500	600	Yellow, extremely brittle
Polyethylene (branched)	6 - 10	200	240	Very weak sticky gum
	6 - 10			

a - Polypyromellitimide from bis(4-aminophenyl)methane

b - DuPont's registered trademark for its polyester film

(Thermal Neutron Degradation (a))

<u>Polymer Film</u>	<u>Thickness, mils</u>	<u>Exposure, days</u>	<u>Temp. °C</u>	<u>Flux 10¹³ neutrons/ cm²/sec</u>	<u>Remarks</u>
POP-PI (b)	2 - 2.7	40	50 - 75	0.4	Slightly darkened, brittle in spots
		40	175	0.5	Darkened, tough
		80	175	0.5	Darkened, brittle
Polystyrene	1.2	10	50 - 75	0.4	Yellow, very brittle
Polyethylene (branched)	3.0	10	50 - 75		Sticky, rubbery
	3.0	40	50 - 75		Brown Varnish
Mylar (c)	3.0	10	50 - 75		Failed
	3.0	20	175		Yellow, brittle

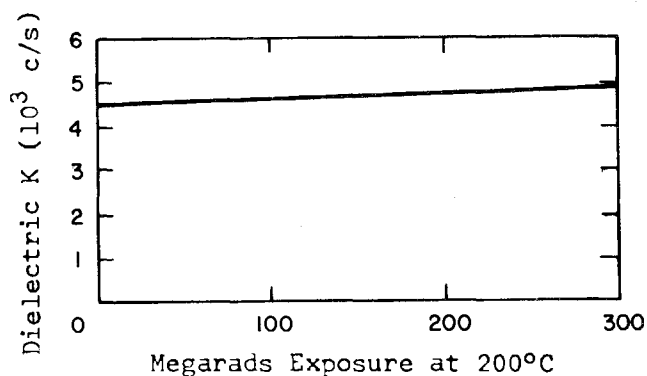
a - Courtesy of Brookhaven National Laboratory

b - Polyimide from bis(4-aminophenyl)ether and pyromellitic dianhydride

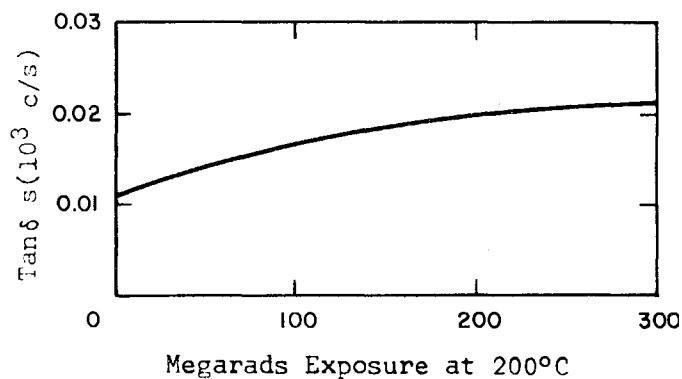
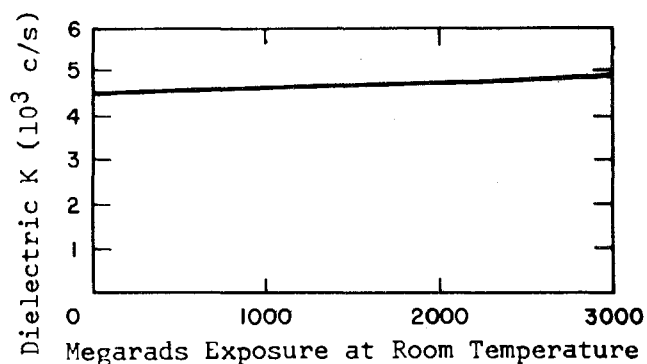
c - DuPont registered trademark for its polyester film

[Ref. 123]

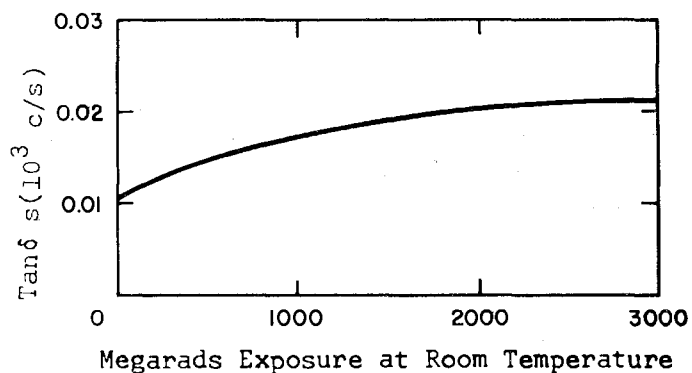
R.V. Einstein reported on an experimental polyimide MK polymer coating on woven glass fabrics and the effects of irradiation exposure:



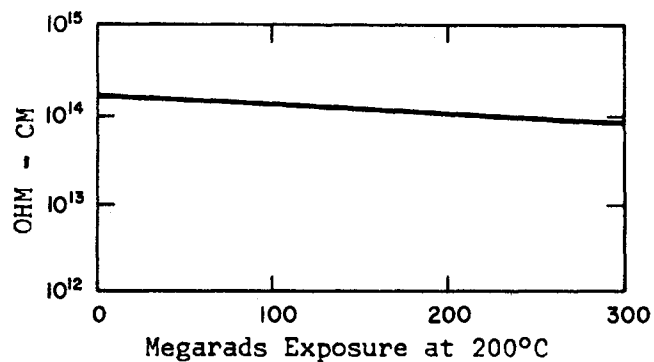
MK coated glass fabric dielectric constant as a function of radiation dosage.



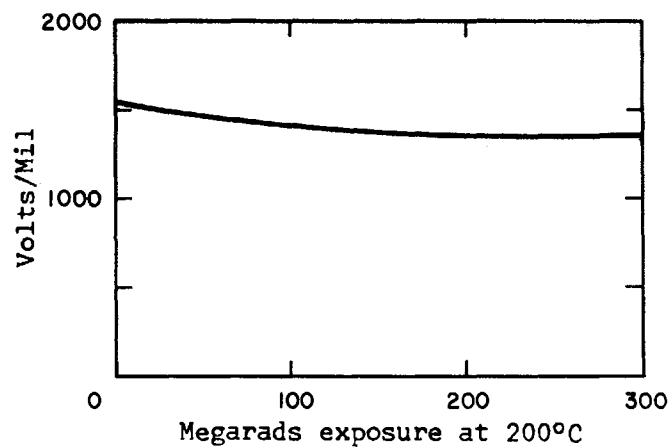
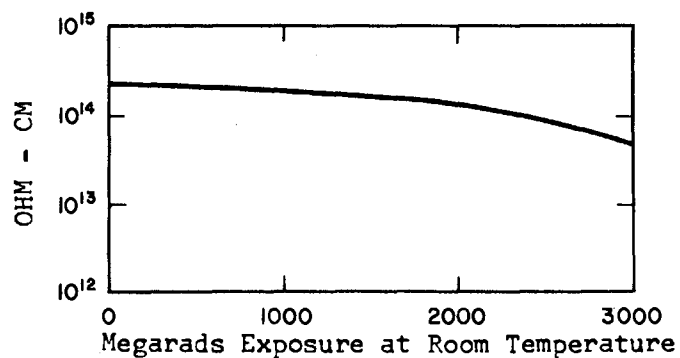
MK coated glass fabric Tan δ as a function of radiation dosage.



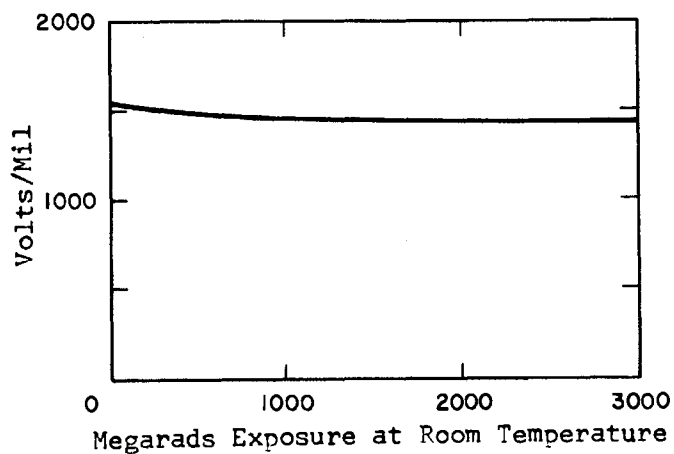
[Ref. 42]



MK coated glass fabric volume resistivity as a function of radiation dosage.



MK coated glass fabric electric strength as a function of radiation dosage.



[Ref. 42]

III. MOLDING AND FABRICATION

Fabrication of polyimides, in general, is difficult because of their high viscosity and thermal stability, thus intricate parts cannot be produced. The DuPont Company claims it uses a heat-pressure process similar to that used in powder metallurgy (Ref. 135)

In the polyimide patent literature it is often noted that the shaped articles should be composed of at least 50% polyamide-acid which is then converted to the respective polyimide shaped articles. It also applies to compositions containing at least 50% of the salt derivatives of polyamide-acids, e.g., the triethyl ammonium salt of the polyamide-acids. The polyimide patent literature also describes a second process step of treating a polyamide-acid shaped article with an acetic anhydride-pyridine mixture; the ratio of acetic anhydride to pyridine varying from just above zero to infinite mixture. The pyridine probably functions as a catalyst for the action of the cyclizing agent, acetic anhydride. A similar chemical treatment involves the use of a carbodiimide (e.g., dicyclohexylcarbodiimide).

Practically all the polyimide patents treat the film casting of the resulting polyimide compositions patented. The films are obtained by spreading a uniform film of solution on a glass or metal plate, baking at 150°C for an hour or more, and stripping off the cured film. In other cases, more satisfactory results can be obtained by curing slowly in a vacuum. Bower and Frost (Ref. 15) report on a number of cures and the resulting films obtained thereby. In one series of comparative tests, films were cast in 55mm diameter aluminum dishes, using sufficient solution to give 0.1 gram of resin (film thickness = ca 1 mil). The curing schedule they used was as follows:

24 hours at 50°C in vacuum
1 hour at 100°C in air
1 hour at 150°C in air
1 hour at 200°C in air
2 hour at 250°C in air
2 hour at 300°C in air

Edwards and Robinson (U.S. 2,710,853) in their patent claims reported that three polypyromellitimides were especially useful and valuable in the production of shaped articles by injection molding or extrusions. These were prepared with the following diamine components:

Injection Molding Properties of Polyimides

Diamine Component of Polypyromellitimide	Toughness	Oxidative Stability Hours	Melt Viscosity 360°C, 20.4 psi	Glassy State Transition Temperature, °C
Nonamethylene diamine	3+	20 - 25	3×10^6	110
4,4-Dimethylheptameth- ylene diamine	3+	20 - 30	8.6×10^3	135
3 - Methylheptameth- ylene diamine	3+	8 - 10	less than 6×10^4	135

The DuPont SP-1 polymers are designed as **prepreg molding compounds**. Two types of compound are used: an unfilled compound and a compound containing 15% graphite weight. Polymer SP-1 does not melt and cannot be fabricated by conventional injection molding or extrusion methods. Proprietary techniques have been developed for fabricating parts from this material. Because of the unusual type of equipment required and the exacting nature of the process, SP-1 is marketed only in the form of fabricated parts or of sample shapes for prototype preparation and testing; the resin form is not sold. Molded shapes can be machined to close tolerance on standard metalworking equipment and precision parts can be punched from sheets.

The September 1, 1965 issue of Design News carried a two-page advertisement announcement that DuPont will make polyimide "Vespel" precision parts to outside specifications. Pictures of "Vespel" gears and a font yoke showed typical designed and fabricated parts.

Although the cured polyimides do not melt in the usual sense, they can be molded and bonded under pressure. Westinghouse workers showed that the curing temperature is approximately that indicated by the power factor curve. Filled and unfilled moldings and flat tubular laminates have been made. Laminates made from 181 glass cloth with different finishes have been made by various companies and property data reported in the property sections of this report. Practically all fabricators of polyimide laminates have experienced problems of voids and porosity with polyimide laminates. Westinghouse Electric Company has conducted a very extensive study of laminating conditions and systems for reinforced plastic laminates for long time high temperature use. A wide range of resin systems were evaluated and their thermal stability, bonding characteristics, T_g (second order transition point) and handling properties measured. Various reinforcements were tested and pressure-time temperatures varied to obtain high quality boards.

According to Reference 135, the development of polyimides for laminating has been slow because of their very short gel time, which requires temperatures and pressure too high for conventional laminating techniques. Thus, they do not lend themselves readily to "B" staging, or partial curing, a necessary step in lamination. The problem has been to develop polyimides with a better balance between gel time and laminating temperature and pressure.

Monsanto, however, says its Skygard 700 can undergo "B" staging. The resin was developed especially for use in glass-cloth laminates for structural, electrical (radar domes, printed circuit boards, slot liners), and specialty applications where resistance to extended exposure to temperatures up to 370°C is required.

Amoco's AI (Type 10) material can be used for lamination by conventional techniques.

The Narmco Research and Development Division of the Whittaker Corporation has conducted a study program to develop or fabricate high density 14-ply poly (m-phenylene-3,3', 4,4' benzophenone-tetracarboxylimide) laminates with high initial strengths. Various strength and thermal data has been reported to date and are included in the mechanical property section of this survey. The polymers have exhibited excellent thermal stability under isothermal conditions. 14-ply laminates were found to exhibit porosity in the resin matrix, however,

experiments were being continued to vary the prepreg drying cycle and cure conditions in an attempt to obtain high-density laminates according to their latest report documents. The Hughes Aircraft Company has also made a study of laminating conditions and found void formation to be a problem; data on their laminate tests are found in the mechanical properties section of this report.

The DuPont Company (Ref. 32) suggests the following fabrication conditions for their PI-2501 "B" staged polyimide fiberglass prepreg: "Lay up composite in desired configuration on a vacuum table which has been covered with polyvinyl acetate or polyester sheeting and silicone (DC-550) release agent. Use Osnaberg cloth edge bleeders and cover composite with perforated polyvinyl acetate or polyester sheeting coated with silicone release agent. Cover with more Osnaberg cloth or dry fiberglass bleeder, then cover with polyvinyl acetate or polyester sheeting. Zinc chromate or equivalent sealer material is suitable for sealing edges. Apply vacuum (10 - 14 psi) to the table and place unit in a circulating air oven or autoclave at 100 - 125°F. Bring oven or autoclave temperature to 350°F over one hour period and hold 350°F at pressure (10 - 14 psi) for 3 - 5 hours. Additional pressure (25 - 200 psi) can be applied after reaching 350°F if an autoclave is used and higher density structures are desired. Cool to 100 - 125°F under pressure and remove the article from the vacuum bag. A post-cure of 10 hours at 600°F in a circulating air oven is recommended when high temperature properties are desired.

Alternatively, the composite can be laid up between caul plates which have been "Teflon" coated or if uncoated use a silicone release agent. The unit is then placed in a press having platens heated to 350°F. The composite is pressed 4 hours at 350°F under 15 - 25 psi and removed from the press while hot. Greater pressure (25 - 2000 psi) can be applied after the first 3 hours at 350°F and 15 - 25 psi if a more dense structure is desired. A post-cure of 10 hours at 600°F in a circulating air oven is recommended where high temperature properties are desired.

The directions given are not necessarily optimum, but they represent conditions which have been used successfully. Deviations from these conditions may be at the discretion of the fabricator to fit a particular application."

The DuPont Company has a polyimide "B"-staged prepreg roving for filament winding purposes and is designated as PI-3301FCR. Their laboratory results indicate that this polyimide system offers a filament wound structure of high strength at elevated temperatures for extended periods of time and more uniform electrical properties throughout the temperature range than conventional binders. They announce that 20 end E-Glass (801 Roving), coated with PI-3301 Binder and "B"-staged and having the following characteristics, is available in spools carrying from 2 - 10 pounds of roving:

<u>Loading</u>	<u>Volatiles</u>	<u>Cured Resin</u>
25 - 30%	5 - 8%	17 - 22%

A winding tension of 0.8 - 1.2 pounds per end is recommended. Heating of the prepreg roving while winding softens the resin to give tack flow and better fiber compaction. The temperature of the mandrel should not exceed 150°C or non-

uniform resin distribution throughout the structure will result. A cure of 4 hours at 350°F followed by 10 hours at 600°F is recommended for NOL ring test specimens. For flat laminates and shaped articles, DuPont recommends that a vacuum bag be employed with a cure of 4 hours at 350°F at 10 - 15 psi. The vacuum bag should then be removed and the article cured 10 hours at 600°F in a circulating air oven. Other DuPont products suitable for laminating purposes are PI-2101 and PI-2501 binders. For fiberglass composite structures, the composite of 181 Style E glass (A-110 finish) plus polyimide PI-2501 is placed within a properly prepared vacuum bag on a vacuum table (using Osney cloth edge bleeders and polyester or PVA sheeting coated with silicone release agent). Suitable sealing and a vacuum (10 - 14 psi) are applied before placing unit in an autoclave or an air oven at 100 - 125°F under pressure and then removed from the vacuum bag. A post cure of 10 hours at 600°F in a circulating air oven is recommended when high temperature properties are desired.

Freeman (Ref. 49) states two methods may be used to prepare polyimide laminates. The fabric is first impregnated in a conventional treater equipped with metering rolls to control varnish pick up and an oven for evaporation of the solvent. The materials remain in the oven twenty to forty minutes at 100°C. The ratio (treated to bare cloth weight) is 1.9. Volatiles (10 minutes at 150°C) measured 9%. Flow measured, measured on a six-ply, 3 x 3-inch sample at 1000 psi and 200°C was about 30%. The molding technique used involved an incremental rise of pressure from 25 - 250 psi at 180°C during the first hour followed by two additional hours at 200°C and 250 psi. The sample was allowed to cool in the press.

Alternatively, the impregnated material could be "B" staged carefully to prevent blister formation, then treated at 200°C to fully cure the polymer. The treated fabric was then recoated with a varnish solution which had been diluted to about one-half the original solids content and the solvent evaporated at 100°C for about one-half hour. A pressure of 1000 - 1500 psi was then applied to the stack for two hours at 200°C. The laminates were post cured in circulating air oven at a programmed rate of rise of 2 - 3° per hour from 150 - 300°C.

The following conditions are recommended by Monsanto for the processing of their Skygard 700 in the stages of impregnating glass cloth, pressing of laminations and post-curing of the laminate. Post-curing of the laminate is essential to achieve maximum heat resistance:

Impregnated Cloth

	<u>250 psi</u>
Flow (measured at pressing conditions)	18 - 21%
Volatile Content	10 - 11%
Resin solids content (not to include volatiles)	34 - 38%
Number of plies to make 1/8-inch laminate	12

Pressing Conditions

<u>Time</u>	<u>Temperature</u>	<u>Pressure</u>
30 minutes	600°F	250 psi

Pressure of lamination should be applied gradually to avoid rupture of the reinforcement fibers of the cloth which is laid up in a uniform manner. It has been laboratory practice to introduce laminate into a press heated to 600°F. A contact pressure of 25 psi has been maintained for 3 minutes before applying the ultimate laminating pressure of 250 psi. Pressure build-up from 25 - 250 psi should be carried out at a steady rate in about 8 seconds.

Direction of the work should be the same for each layer of cloth. The type of treatment or finish on the reinforcement cloth can affect the adhesion and curing characteristics of the resin.

The pressed laminate is air cooled to 500°F and then water cooled in the press. The highest pressure reached during the press cycle is maintained during the cooling period.

Post-Cure

Total post-cure of 16 hours is recommended:

4 hours	at 390°F
↓	435°F
	480°F
↓	575°F

Post-cure conditions cited are for 1/8-inch laminates. It is likely that for thicker laminates the post-cure cycle would have to be extended.

Melvin and Parish (Ref. 133) report that laminates of polyimide film with "Nomex" high temperature nylon paper available from commercial laminators for use where extra protection against puncture by burrs and sharp protrusions, sometimes present in electrical slots, is desired.

DuPont's "Kapton" H-Film can be laminated, metalized, formed, punched, or adhesive coated. In addition, a heat-sealable modification ("Kapton" HF) is also available which consists of Teflon FEP resin bonded to either one of both sides of the "Kapton" H-Film. Applications of these "Kapton" H- and HF-laminations are discussed in Section IV of this report; electrical, mechanical, and thermal properties are reported in Section II.

Armature coils, comprising thin film, pre-insulated magnet wires (both round and rectangular), have been constructed with 150 gauge "Kapton" Type HF film spirally wrapped with universal taping equipment. Melvin and Parish (Ref. 133) noted that "using a 50% overlap, a 3-mil wall (6 mils with dual taping heads) can be applied at fast taping speeds and in one continuous operation pass directly into heat-sealing equipment." These authors also state"Although heat-sealing Type HF polyimide film can be achieved over a broad temperature range, economics favor the use of high temperatures and short dwell times." Bond strength data are reported in the mechanical property section of this report.

The DuPont Company reports that "Kapton" polyimide Type HF may be spiral wrapped and sealed on wire to give a tough, non-peeling bond. Preliminary tests at their Chestnut Tun Laboratory indicate sealing will take place under a wide range of conditions depending on the size of the wire, oven temperature and speed:

<u>Wire Gauge</u>	<u>Minimum Sealing Time (seconds)</u>		
	<u>600°F</u>	<u>700°F</u>	<u>800°F</u>
#22	60	30	30
#16	120	60	60
#22 Twisted Pair Shield and Jacketed	-	90	60

"Kapton" Type H-Film can be bonded to itself or to metallic and non-metallic surfaces by using a polyimide high temperature binder solution such as the DuPont PI-1200. The bond is made by bringing the two coated surfaces together and curing 1 - 5 minutes at 700°F while under pressure of from 20 - 200 psi depending upon the substrate to which H-Film is being bonded. Some difficulty can be experienced when making bonds to copper due to the formation of an oxide on the metal surfaces in an oxidizing atmosphere. Other binder solutions have different cure temperatures, time and pressures, hence, one must consult the manufacturer's data sheets for suggested curing conditions. A number of firms have explored polyimides for adhesive bonding purposes, e.g., metal-to-metal, film-to-film, and film-to-metal. Mechanical property data on various polyimide adhesives are reported in the mechanical properties section of this report. Polyimide parts can also be cemented to themselves or other plastics, metals and rubber, using a variety of other adhesives. No special surface preparation is required other than a normal roughening and cleaning. Both epoxy and phenolic adhesives work well, and offer service up to 400 - 500°F. For higher temperature use, the polyimide adhesives can be used.

The Permacel Company (Ref. 113) supplies a pressure-sensitive H-Film (EE-6379) in which 1-mil thick DuPont "Kapton" Type H-Film is bonded with a pressure-sensitive silicone adhesive.

A number of polyimide binder solutions (e.g., DuPont PI-1100, PI-1101, and PI-1200) are currently available commercially in solids contents ranging from 18 - 53% and viscosities from 15 - 90 poises that can be applied by brush, knife, or roller coat. Various diluents (e.g., DMF/toluene, xylene) may be required for optimum effects in some applications. For surface preparation, the usual commercial procedures are followed and a wide range of film thicknesses are possible. No attempt will be made to detail completely each binder solution's curing and fabrication capabilities hence recourse must be made to the supplier's bulletins or data sheets. A few typical guidelines, moreover, are given below.

DuPont recommends the coated substrate to be baked 15 - 20 minutes at 300°F and then 5 minutes at 600°F for their PI-1200 solutions: and 1 hour at 200°F plus 2 hours at 400°F for their PI-1100. Other binder solutions have different baking temperatures and times.

According to Schweitzer (Ref. 90), "Polyimide binder solutions can be applied by spray, dip, or roller coating. Required baking conditions vary with size, shape, and heat capacity of the coated object. Preliminary bakes in the 250 to 350°F range are usually required to remove most of the solvent,

followed by a final bake of 2 minutes at 750°F to cure the resin. Some formulations can be cured at lower temperatures, such as 16 hours at 300°F. Adhesion is good on practically all types of metal, including copper, brass, untreated automobile-body steel, electrical-grade aluminum, and chemically treated steel and aluminum. Best adhesion is attained at the higher baking temperatures."

The E.I. du Pont de Nemours Company reports that their "Pyre-ML" varnish may be successfully applied by spray, brush, vacuum impregnation, total immersion, end turn dipping and roll over methods depending on the size of equipment being varnished and customer preferences.

For spray application DuPont recommends that RC-675, RC-5034, or RK-692 be reduced to 11-13% solids with dimethyl formamide, or a 50/50 mixture of dimethyl formamide and toluene. Approximately 0.2 mil dry film thickness can conveniently be applied per coat on vertical surfaces. If more than one coat is required, a short bake (3 minutes at 150°C - 302°F) is recommended between coats.

For dip application it is suggested that the varnish be reduced with dimethyl acetamide, T-8557 Thinner, or xylene to obtain the desired viscosity. The dip tank and other coating equipment which comes in contact with the enamel should be stainless steel or polyethylene. For best results the unit to be varnished should be heated to 150°C (302°F) and dipped hot into the varnish. The unit should be removed from the varnish while still hot and allowed to flash 15 minutes or until no condensed solvent vapors can be seen around the unit. It should then be placed in 150°C (302°F) oven and prebaked to remove the solvent before either the final bake or a second dip coat. Cold dipping and vacuum impregnation have been used successfully. The time required in the prebake oven will depend on the size and shape of the unit being varnished.

"Pyre-ML" varnish can be applied by roller coating. Care should be taken to obtain rollers which are resistant to the solvents used. One supplier of rollers has recommended butyl or natural rubber rollers. Either RC-675 or RC-5034 may be used. If reduction is necessary, use T-8557 or dimethyl acetamide. Approximate strip coating oven zone temperatures should be:

#1 Zone	200-300°F
#2 Zone	250-400°F
#3 Zone	600-700°F

The line should be operated so that the residence time in the maximum temperature zone is a minimum of one minute.

The baking conditions for "Pyre-ML" varnishes are dependent on the size, shape, and weight of the varnished substrate. The cure of this varnish occurs by the loss of solvents and water from the film. The circulation in the oven will, therefore, affect the rate of cure. Generally a prebake is used to remove most of the solvent and prevent blistering during the final high temperature bake. A suitable prebake is from 5 to 60 minutes at 150°C (302°F) depending on the type of material being varnished. Hot dipping

will sometimes eliminate the need for prebaking.

The final bake should approximate one of the following: (The temperatures referred to are the temperature of the metal substrate.)

- 1 - 2 minutes at 400°C (752°F)
- 3 - 10 minutes at 300°C (572°F)
- 30 - 100 minutes at 200°C (392°F)
- 16 hours at 150°C (302°F)

For maximum flexibility, moisture resistance, solvent resistance, and adhesion, a bake of 2 minutes at 400°C (752°F) is recommended. The optimum baking schedule should be determined for each end use. The completeness of cure can be determined by the hardness, solvent resistance or flexibility, or by measuring the weight lost at elevated temperatures, or the electrical dissipation factor. Well-cured films will lose less than 2% of their weight in 5 minutes at 300°C (572°F), or will have a dissipation factor of 0.004 or less at 25°C (77°F) and 1,000 cps.

Monsanto researchers (Ref. 116) report that...."Polyimide coatings were prepared readily from the soluble carboxylated polyamide at 20% solids in dimethylacetamide. Conditions for solvent removal were found to be a function of coating thickness.

Generally, the solvent was removed by heating at 50 to 90°C to yield a tack-free film in less than an hour. The film was then heated at 140 - 145°C for an hour to effect conversion to the imide. Attempts to remove solvent from a wet film several mils thick at temperatures sufficient for the amide-imide conversion caused precipitation of the polyimide before the solvent was removed and yielded a powdery, noncontinuous residue.

Final curing for an additional hour at 300°C under nitrogen increased film toughness and removed any residual solvent which could cause bubble formation upon rapid heating to test temperatures approaching 1000°F.

The preparation of 3- and 4-mil coatings required a special technique of multiple coating in order to obtain satisfactory adhesion to the metal. Good adhesion resulted when solvent was removed from the first coating at a temperature not in excess of 150°C. In contrast, recoat adhesion to a first coat that had been heated to 300°C was nil. Adhesive failure between the two coats is attributable to too complete conversion to polyimide at the higher temperature."

Polyamide-imide AI Polymer (Type 10) is available from the Amoco Chemical Company as a brown powder but applied as a solution using proper solvents. In most cases, this material is applied from solutions that are easily obtained by stirring the polymer into the desired solvent (e.g., dimethylacetamide or N-methyl pyrrolidone) at room temperature. The Physical Property Section of this report should be consulted for solvent-viscosity data on this material. Total addition time is about 15 - 30 minutes depending on the desired concentration. The dispersion is then stirred 1 - 2 hours to obtain a clear solution which should be stored 15 - 20 hours and stirred again before it is used.

The viscosity will increase 10 - 20% in this time. The curing temperature (250 - 600°F) and times (2 - 10 minutes) can be varied to obtain optimum chemical, electrical, mechanical, and thermal properties as well as end use application.

The Shawinigan Resins Corporation report (Ref. 112) that their cross-linked polyimide resins, RS-5303 and RS-5305 can produce coatings which can be partially cured or "B"-staged. For initial evaluations, the wet coatings can be heated for 60 - 90 minutes at 120°C to produce a "B"-staged condition. To produce a final cure, the "B"-staged resin can be heated at 310 - 320°C for 1 - 3 minutes and then placed under 250 psi pressure for 15 minutes to 1 hour at 310 - 320°C. For applications where "B"-staging with subsequent cure under pressure is impractical, the following cure cycle is suggested:

2 hours at 180°C
4 hours at 200°C
2 hours at 225°C

The Sterling Varnish Company (Ref. 92) recommends that its Amide-imide varnishes (AI-220 and Alvar 220) should be cured a minimum of 4 hours at 200°C. However, lower temperatures for longer cure times are satisfactory. The length of the cure is dependent upon the application and physical properties desired.

The Schenectady Chemical Incorporated Company report that their new "Isomid" polyester-imide enamel runs easily on existing equipment, with existing applicators and ovens, at normal and non-critical heat patterns. The company also says that the enamel has a broad cure temperature range -- good thermal characteristics have been obtained at relatively low as well as relatively high baking temperatures. The following table lists some properties of interest to enamellers:

Enameling Properties

Typical Properties

Viscosity, 25°C, cps	300 - 700
Specific Gravity, 25°C	1.00 - 1.10
Solids, %	30 ± 1

Application Parameters

Wire Size, awg	18
Bare Wire, inches	0.0402
Coated Wire, inches	0.0432
Build, mils	3.0
Speed, ft/min (C-E lab type 1-M, 15 ft)	30
Tower Temperature, Top, °F	775
Tower Temperature, Center, °F	750

[Ref. 134]

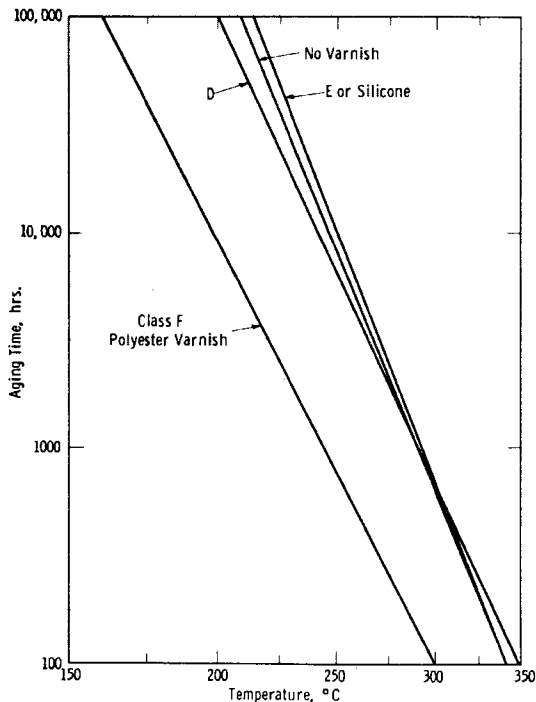
IV. APPLICATIONS

A. ELECTRICAL INSULATION APPLICATIONS

The combination of outstanding thermal and chemical resistance with excellent electrical and mechanical properties make the polyimide plastics especially attractive for a wide variety of high temperature electrical applications. Since the polyimides are a relatively new family of resins and their properties only cursorily explored, most vendors or suppliers have suggested applications to offer. Also, their availability in a variety of resin forms: binder solutions, moldings, film, prepregs, laminates, make them contenders for a wide range of applications. Research on the various applications is continuing at a rapid pace.

1. Magnet Wire Enamels

Amoco Chemicals Corp. granted Anaconda Wire & Cable Co. (in June 1964) an exclusive license for its trademarked A.I. 220 polyimide polymer for magnet wire applications. The enamel is prepared by dissolving AMOCO AI Polymer



Dielectric twist tests on polyimide Enamel D with various varnishes.

[Ref. 51]

itself (D) caused a slight drop, and a silicone varnish or a different polyimide (E) caused a slight improvement: Leppla & Carryer (1963) report in one typical test: the generator was made to vibrate at its resonant frequency, accelerating the type of breakdown of materials that might take place from vibration in a missile or plane. In previous tests, other enamels had shaken right off the wire, but the polyimide wire enamel showed no degradation." They state that

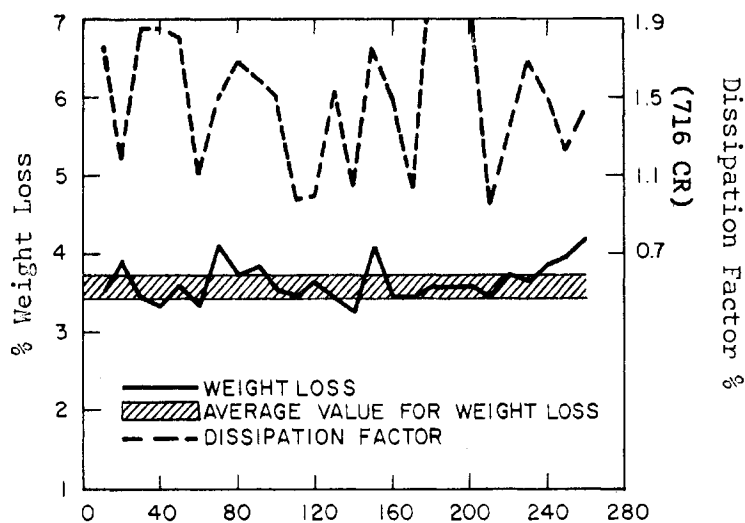
(Type 10) in a suitable solvent blend. The wire coating is dried and cured at high temperatures to form a tough, heat resistant film. Mechanical and electrical properties of this A.I. 220 polymer are reported in the electrical and mechanical properties section of this report. Frost and Bower (1963) screened nearly 100 aromatic polyimides as wire enamels. They reported that several were found to possess suitable physical properties. One of the best gave the following typical test results on #17 copper wire: quick snap O.K.; 15 to 20% elongation + 1X mandrel O.K.; heat shock (1X) >400°C; cut through (1000g.) >500°C; dielectric strength 9 KV; abrasion (GESA, 740 g.) >200; good resistance to all solvents and Freon 12 and 22; dielectric twist extrapolation to 210°C at 100,000 hours (unvarnished specimens). The graph shows results of dielectric twist tests with various varnishes over good polyimide enamel. These authors report that . . . "compared with the unvarnished specimen, a polyester varnish caused a large drop in thermal stability, the enamel over

other tests confirmed data already available from wire manufactures.

Lavin and Hunt in their patent (U.S. 3,105,775) elaborated on the composition of polyimide enamels for magnet wire coatings. They also gave considerable one-kilovolt life, abrasion resistance and resistance to hydrolysis data.

Saums (Ref. 136) in a state-of-the-art survey of magnet wire materials discussed the status and comparison of the polyimides and polyamide-imide enamels to other coatings. A brief mention is made of the European polyester-polyimide magnet wire development.

M.W. Jawitz of the Kearfott Division, General Precision Aerospace Company has become concerned about the various cure tests for polyimide enamel coated magnet wire and reported on the problems encountered by his company in establishing acceptable methods for determining the degree of cure of polyimide enamel on most sizes of wire. He hopes to stimulate further work by the technical societies to eventually produce a universally acceptable test for degree of cure of this type of magnet wire since polyimide enamel is an excellent coating for magnet wire. His investigations concerned four test methods: a) Dissipation Factor at 1000 cycles (measured with a General Radio 716 Bridge and 650A impedance bridge). b) Dissipation Factor at 4.6 Megacycles (measured with a Boonton Q meter). c) Weight Loss. d) Solvent Shock (Dimethylacetamide). Jawitz concluded that there is no direct correlation between any of the four above specified tests. As regards dissipation factor at 1000 cps and 4.6 Mc, there was some correlation between these tests and the weight loss test for the heavier wire sizes (AWG 18 through AWG 30). However, when wire size ranged from AWG 30 and finer, the tests were extremely erratic. Test data were so non-reproducible that if the wire was moved three or four inches in the mercury, he obtained altogether different results. The weight loss test was found to be reproducible to the extent that within given spool of wire some values were repeated. However, there was no correlation of data between the weight loss test and the electrical tests in the finer sizes (AWG 30 and finer). Typical data from his tests are shown in the following graph.



Consistency of degree of cure within a given spool of wire.

2. Insulation Varnish Coatings

Leppela & Carryer (1963) reported that Bendix conducted temperature cycling tests on the new polyimide varnish which showed that the film maintained a hard, smooth surface through 300°C. Weight loss at 300°C was substantially less. Other Bendix tests for resistance versus temperature showed excellent results. In thermal life tests at 300°C for bare copper field coils coated with the new varnish, the results were also outstanding.

The DuPont Co. (Tech. Bull. #25) reports that their PYRE-M.L. varnishes have been used for making conductive heating panels with carbon as the pigment material. These have a resistance in the range of 60-70 ohms/square. They state further . . . "Because of the outstanding stability of the 'PYRE-M.L.' binder, these uniform heating elements can be used for 3,000-6,000 hours at surface temperatures of 200°C (412°F), with no more than 10% change in resistance."

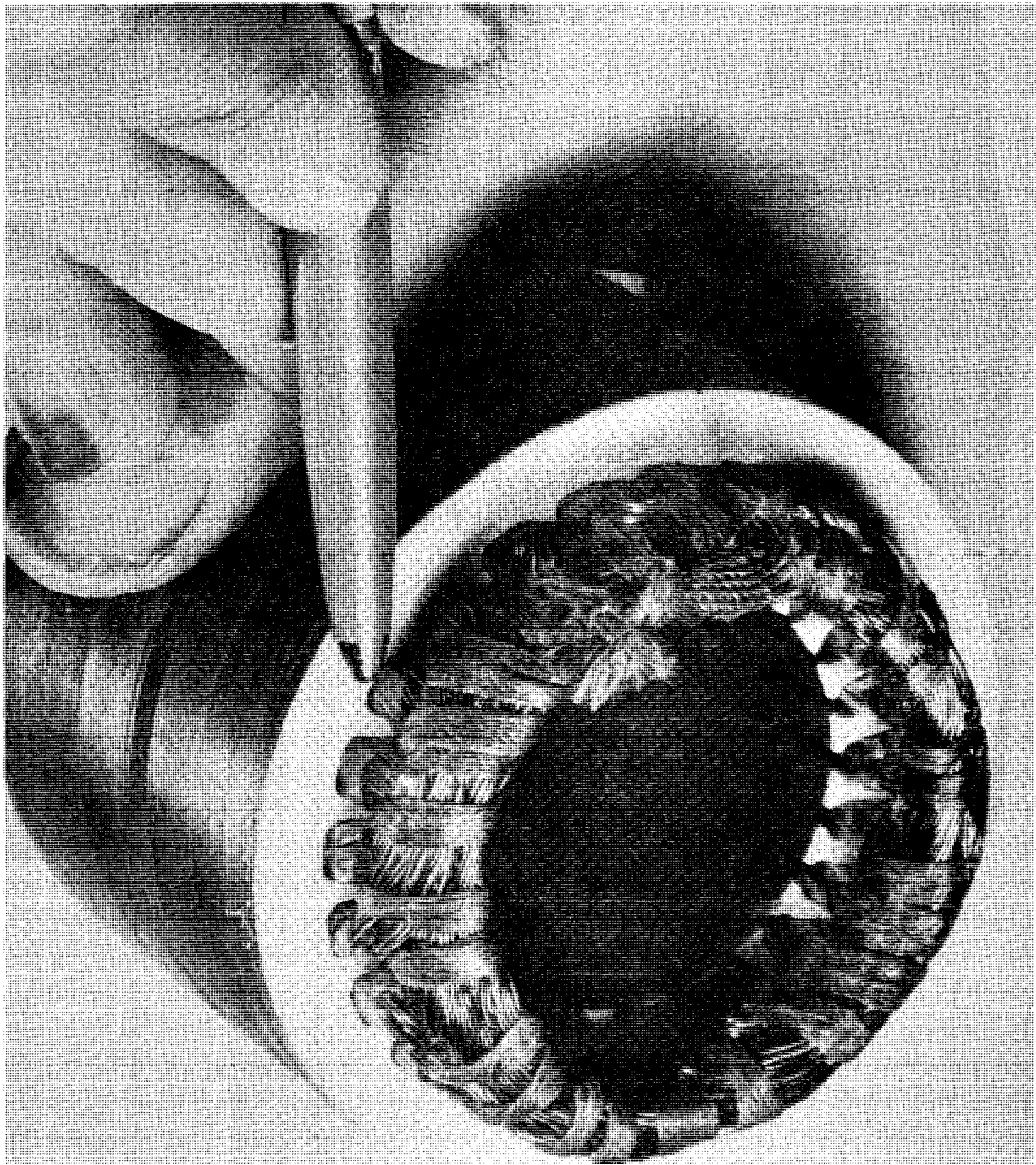
Amoco Chemicals Corp. have suggested applications such as capacitors and other electronic components for their AMOCO AI Polymer (Type 10) poly (amide-imide) coatings.

3. Electric Motors

F. E. Schweitzer (1964) reports . . . "in electric motors, the use of polyimide insulating varnishes and polyimide-impregnated slot liners may permit increasing the horsepower of a motor as much as 100% with no increase in frame size." Further . . . "High heat resistance of polyimide coatings is of interest from an insulation standpoint in electrical equipment requiring high-load starts such as in traction equipment drives. The use of polyimide insulation could impart burn-out resistance and extend the overload limits." In a second paper, Schweitzer et al. reported extensive "PYRE-M.L." system motorette evaluation tests.

Mallouk (1964) reports . . . "In motors, where size is important, the high dielectric strength and toughness of H-Film permit the use of thinner insulation and a corresponding increase in use of copper, allowing greater horsepower without increasing frame size. The reduced thickness of the film would also improve heat dissipation." The high dielectric strength and toughness of the film also make it useful for insulating formed coils and for phase insulation.

Leppela & Carryer (1963) described the Bendix Corporation's Red Bank Division evaluation of polyimide slot cell insulation, (a mica type of material laminated to glass fabric with a tacky resin) . . . "This material was borderline in its retention of electric strength after creasing. However, in folding and creasing tests that were run on the new polyimide coated glass fabric, Bendix found that more than 80% of the original dielectric was retained after 180-degree creasing. Since the original electric strength averaged more than 700 volts per mil for the 5.5-mil material and 600 volts



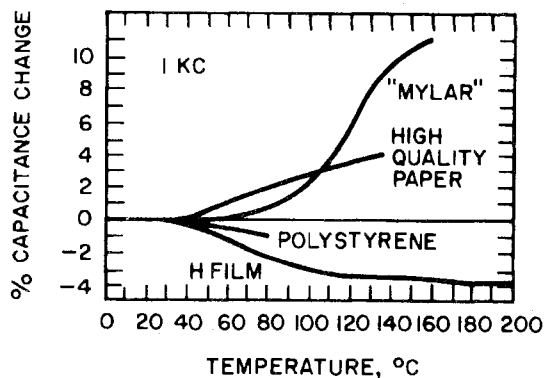
per mil for the 10.5-mil material (two-inch electrodes), this meant a substantial factor of safety. A disadvantage noted by Bendix during winding tests of the polyimide fabric under production conditions was a slight loss in tear strength. But when the slot cells were cut on the bias, a degree of stretch was permitted and the fabric's resistance to tear was increased satisfactorily. Thermal aging tests by Bendix confirmed DuPont's data showing that more than 50% of the original dielectric was retained after 400 hours exposure at 300°C for the 5.5-mil material." These same authors also detailed a cooperative study to develop polyimide insulations for fractional motors with the Lamb Electric Division of Ametek, Inc. Prototype motors (1/4 HP, D-C in 4-inch frame) with the complete polyimide insulation system - wire coated with polyimide wire enamel, slot liners made of polyimide coated glass fabric, and the entire assembly being immersed in polyimide insulating varnish - were fabricated by the Bendix Corp. Evaluation studies by the Lamb Electric Division showed that the homogeneous polyimide system provide, finally, the desired balance of properties necessary for this unusually compact motor. It was used for that application and has been used repeatedly since by Lamb to answer high-temperature needs, particularly under conditions where minimum space requirements must be met.

Bendix reports that the polyimide insulation system has also been used on prototype units for helicopters, and also on the starter motors and the prime electrical supply units in Boeing's experimental "Hydrofoil" water craft. The polyimide system has also been approved for use in the reactor firing motors of both the firing and non-firing types of Polaris submarines. These motors raise and lower the firing rods into the nuclear reactor and must be guaranteed for a minimum of 14 years.

4. Capacitor Applications

H-Film exhibits a negative temperature coefficient of capacitance, suggesting uses in capacitor applications. The relatively constant loss factor ensures against failure due to thermal runaway.

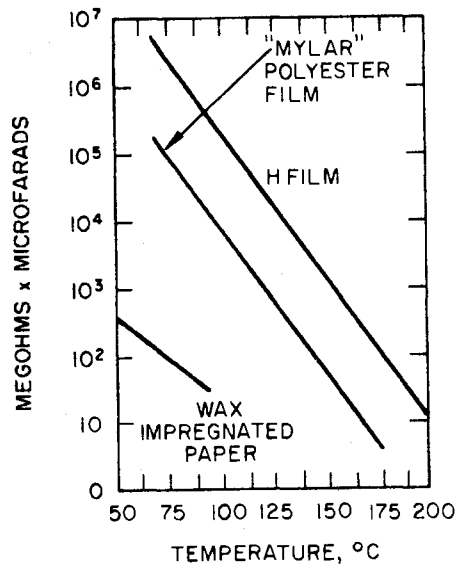
Amborski has made an extensive comparative evaluation of H-Film with other plastic films for capacitor dielectric applications. Measurements made on single sheets of 1-mil polyimide film between aluminum foil electrodes to a size of 0.06 μ f show that the temperature coefficient of capacitance is negative:



Capacitance change vs. temperature.

[Ref. 1 & 94]

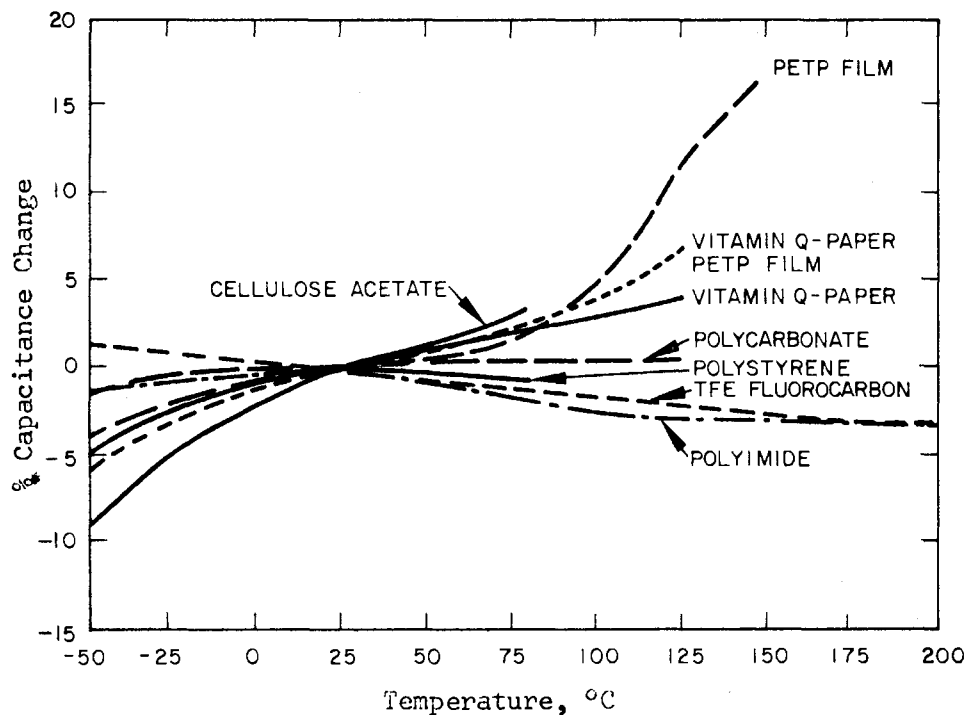
The insulation resistance of 0.006 μ f capacitors wound from 1-mil polyimide film bears out the superior electrical resistance over a wide temperature range. A well-known figure of merit is the product of megohms x microfarads of capacitor materials. Ambroski offers the following graph comparing H-Film with other dielectric film materials:



Insulation resistance of capacitors as a function of temperature. Capacitors wound with 0.001-inch H-Film, 100 Mylar C polyester film, and paper.

[Ref. 1 & 94]

W.C. Lamphier (Sprague Electric Company) reviewed recent developments in capacitor dielectric films and reported test data on polyimide "Kapton" Type H-Film:

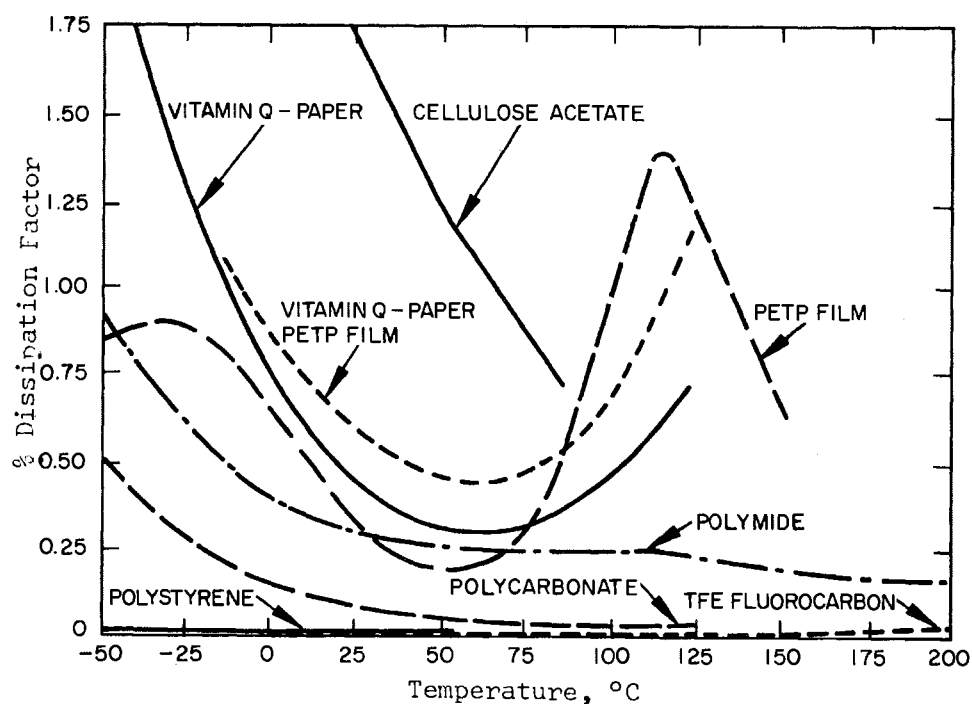
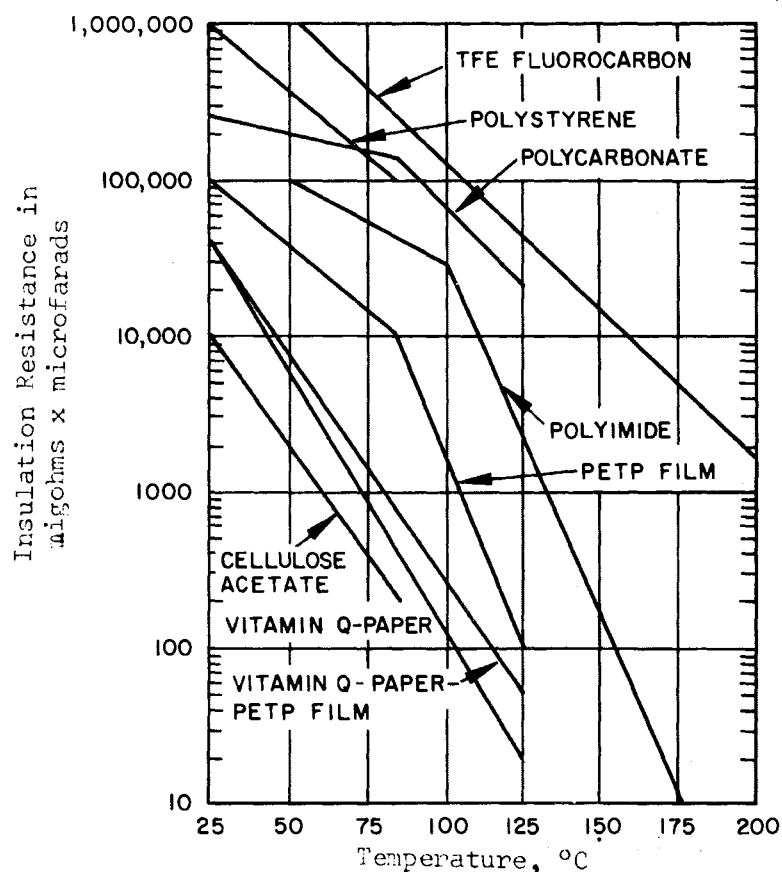


Capacitance change characteristics at different temperatures for various dielectric materials.

[Ref. 115]

Dielectric loss characteristics. Polystyrene is not now commercially available as a metalized film.

[Ref. 115]



Insulation resistance is important for low leakage current.

[Ref. 115]

In this author's opinion, polyimide film is a very promising prospect because of its high temperature capability and find applications when thinner gauges are available for metallizing.

5. Cable and Wire

A number of companies are marketing composite cable and wire constructions composed of polyimide resins (e.g. "Kapton" Type H or "Pyre-ML") with Teflon TFE or Teflon FEP. The DuPont company designates their various constructions as "Kapton" Type HF and has currently nine different constructions of "Kapton" Type H-Film with Teflon FEP on one or both sides. The polyimide and FEP films can vary from 1/2 - 2 mils in thickness. The Haveg/Super - Temperature Wires Company also supplies Teflon wire construction with jackets of "Pyre-ML" polyimide. They report the following data on their Teflon-"Pyre-ML" insulations:

Comparison of Properties - only as a function of FEP/ML

<u>Test</u>	<u>Method</u>	<u>Results</u>	
		<u>FEP only</u>	<u>FEP/ML</u>
Abrasion Resistance	MIL-T-5438 1/2 lb load, 400 grit	29.7"	45.8"
Room Temperature Cut-Through	10 kg load, 1/16" radius	<one minute	>24 hours
High Temperature Cut-Through	1 kg load, .003" radius, 200°C	<one minute	>24 hours
Solder Iron Heat Resistance	1 lb load, 45° tip, 800°F	<15 seconds	>5 minutes

[Ref. 129]

K.N. Mathes of the Missile and Space Vehicle Department, General Electric Company, has made an extensive evaluation study of both polyimide "Kapton" Type H-Film and ML enamel dielectrics for electrical conductors (standard round cable and ribbon cable) for cryogenic applications. His conclusions were that ML enamel possesses relatively good dielectric characteristics at room temperature and at liquid helium temperature; its dielectric loss is as low or lower than Teflon at room temperature. ML provides very much less insulation build than conventional cable insulation and where it can be used it will permit significant savings in space and weight, particularly with small wires. He states further . . . "Without question, properly-cured ML enamel is the most flexible wire insulation at cryogenic temperatures. It seems almost impossible to crack an ML standard construction by flexing it even in liquid helium, although conventional insulation is shatter brittle at very low temperatures. In final conclusion, ML enamel alone is considered to provide adequate, low voltage insulation on solid conductors for use over a wide range of temperatures, if properly controlled in manufacture and selected in use."

Mathes found that a lapped layer of 0.001 inch H-Film breaks down at a surprisingly high voltage if it is sufficiently overlapped. In the cited reference [Ref. 72] work, the approximate 2/3 overlap on the #26 cable is very adequate, while the 1/2 lap on the #16 cable is marginal. With a very tight wrap, the "Kapton" type H-Film clings closely to the wire - so tightly on the #26 cable that it could be unwrapped only with considerable difficulty. The dielectric loss is reasonably low at room temperature and in liquid helium is as low as Teflon at room temperature. The flexibility of the H-Film wrapping at cryogenic temperatures is difficult to analyze. The first samples of #16 cable evaluated exhibited remarkable flexibility in liquid helium.

The cryogenic flexibility was apparently damaged by moisture exposure. Unfortunately, most recent samples of H-Film taped #16 cable have cracked badly even on a 1 3/4 inch mandrel in liquid helium. The #26 H-Film wrapped cable seemed to be a little better in cryogenic flexibility than extruded Teflon. Mathes concluded that it is possible (and perhaps likely) that, at the present stage of development (1964), H-Film is not a uniform and well-controlled product. Like ML enamel, H-Film may be sensitive to undercure and perhaps to moisture, particularly in the undercured state. The only justification for using H-Film, with the inherent disadvantages of taping, lies in the potential for improved flexibility at cryogenic temperatures.

Samples of 25-mil diameter magnet wire (coated with "PYRE-M.L." enamel) were cooled by the DuPont Co. to 4°K in liquid helium and subjected to a mandrel bend. The radius at which failure occurred on forward and reverse bends is shown in the following table. The superiority of the "PYRE-M.L." to the other materials tested is clearly evident:

	<u>Failure Diameter in Inches</u>	
	<u>Forward Bend</u>	<u>Reverse Bend</u>
Formvar	3/4	1
Polyester	1/2	3/4
"PYRE-M.L."	OK 1/8	OK 1/8
Filled "Teflon" TFE-fluorocarbon	1/2	1/2
Asbestos	1/2	1/2
Polyvinyl Chloride	1-3/4	1-3/4

[Ref. 36 & 71]

Mathes also studied ribbon cable constructed from H-Film bonded by hot pressing with layers of Teflon FEP adhered to one or both surfaces of the H-Film as needed. He concluded that all of the six samples of H-Film evaluated in his program possessed remarkable flexibility even in liquid helium if the test specimens are not twisted or bowed in bending. The sensitivity to twisting at low temperatures, however, does constitute a limitation in use. Reference should be made to the original reports for more detail on the voltage breakdown, flexibility, resistance and capacitance measurements on the various types of cable and wire constructions.

Martz (Ref. 138) reported recently at the Fifth Annual Bureau of Weapons symposium on the techniques for fabricating light weight electronic harnesses utilizing ribbon cables by the W.L. Gore and Associates, Inc. The cable consisted of laminated H-Film with Teflon TFE insulations in which the helically-wound H-Film takes up the stress loads, increases the cut-through loads and increases the abrasion resistance by significant amounts.

Parish and Melvin (Ref. 79) of the E.I. du Pont de Nemours Company reviewed the merits of "Kapton" Types H and HF for manufacturers and end-users of electrical equipment, especially to designers of rotating machinery. Problem areas involving bonding, shrinkage, adherence, enamel and varnish coatings versus film wrap processing encountered by wire processors were discussed extensively by these authors at the September 1965 Electrical Insulation Conference in New York City.

Melvin and Parish (Ref. 133) reported that...."The use of polyimide film in base and cover sheets for flat cable and printed circuits makes possible the design of thin structures having outstanding dimensional stability, solder resistance, and high temperature operating capability. Light in weight and very flexible, these structures offer the additional advantages of good resistance to cut-through and abrasion, and high dielectric strength.

For single layer circuits, continuous bonding of oxide-treated copper to base sheets of one-side coated type HF film can be done with conventional hot roll laminating equipment. The FEP surface should be next to the copper and temperature at the film/copper interface should exceed 280°C to insure adequate flow of the FEP. Nominal pressures of 250 to 350 psi give intimate contact of the two surfaces and improve bond uniformity. After conventional layout and etching of the film/copper laminate, pre-punched or pre-cut cover sheets of one-side coated type HF film are bonded to the etched surface by heat sealing in a flat press.

For multi-layered circuits, the same process is used with pre-punched or pre-cut sheets of two-side coated type HF film sandwiched between successive circuit layers. Similar structures can be prepared using type H film with appropriate high-temperature binders instead of type HF film."

James H. Freeman (Westinghouse Electric Corp.) reported [Ref. 44] on a new concept in flat cable systems using an aromatic amide-imide polymer (called "AMANIM") insulation. It involved the following cable construction steps: 1) the continuous coating on one side of a metal foil with the amide-imide polymer, 2) printing the conductor pattern with a resist on the metal surface, 3) removing the unwanted metal and resist, and finally 4) laminating

a film onto the open face of the conductors with an adhesive. Properties of a typical flat cable are shown in the following table:

<u>Pattern</u>	<u>Conductor Dimension</u>	<u>Center Spacing</u>	<u>Square mils</u>	<u>Circular mils</u>	<u>Resistance ohms/M ft. at 20°C</u>	<u>Current 30° Rise amps</u>
A	0.0027 x 0.075	0.100"	202	258	41	4.0
B	0.0027 x 0.100	0.125"	270	342	31	5.8

Nominal Overall Thickness:	0.008 inch
Tensile Strength:	15 lbs/conductor x no. conductors
Conductor Peel Strength:	4-6 lbs/inch-width
Dielectric Withstand Voltage: (conductor to ground)	6000 volts rms
Weight/1000 conductor feet:	1.05 lbs.

Weight/1000 conductor feet, AWG 26 hook up wire > 2 lbs.
AWG 22 hook up wire > 4 lbs.

[Ref. 44]

Terminals are molded in place at any desired point on the cable and in any numbers. Properties of a typical molded terminal are cited by Freeman:

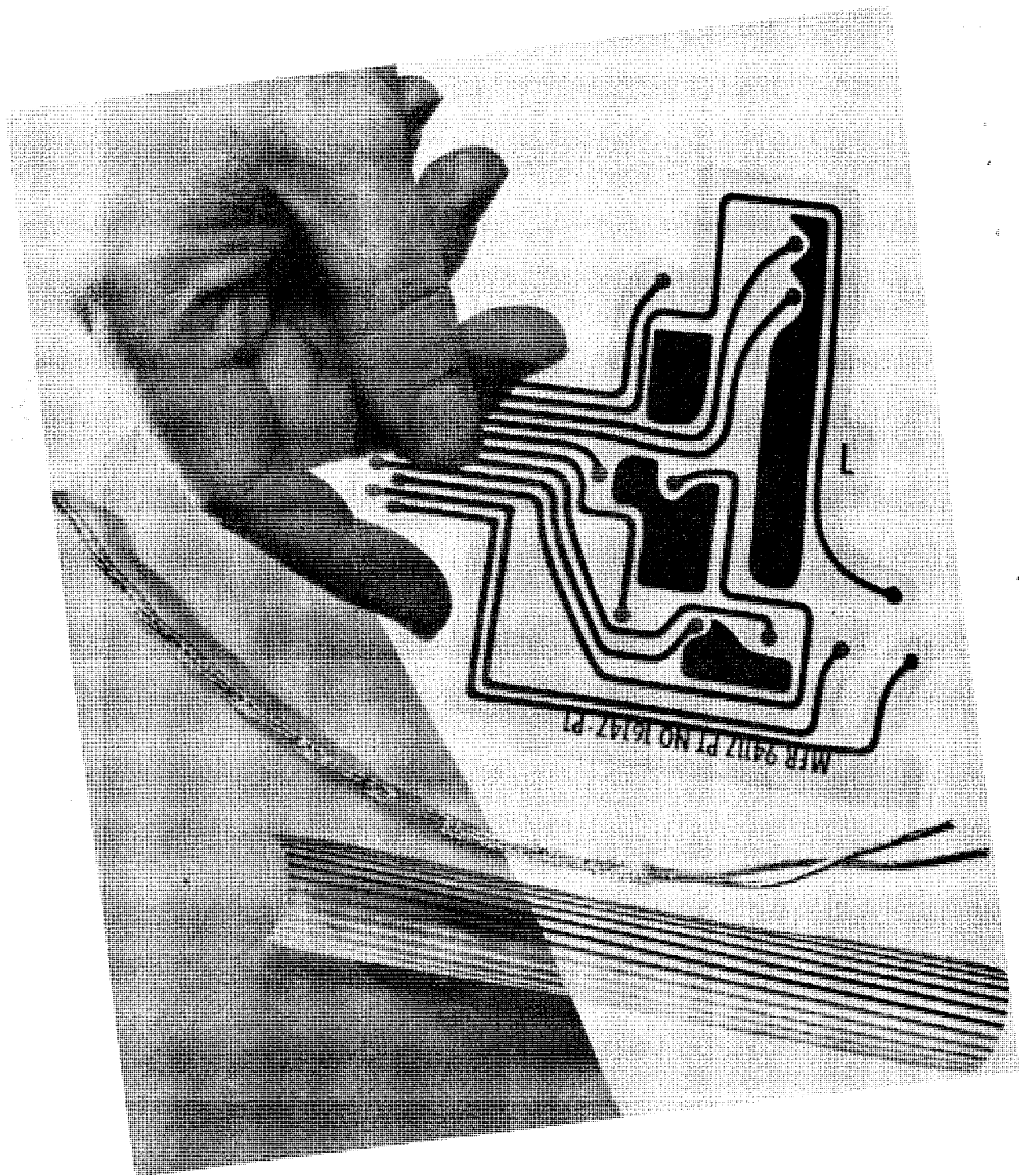
Properties of Typical Molded Terminal*

Dielectric Withdtand Voltage ¹	3-4000 volts rms
Insulation Resistance ²	60-100,000 megohms
Humidity ³ , Steady State (95% RH)	2-3000 megohms
Immersion ⁴	14-16000 megohms
Contacting Resistance (one amp current)	3.0-4.0 millivolts
Thermal Shock ⁵ (5 cycles, -55 to +125°C)	No effect
Durability ⁵ (500 cycles, insertion-withdrawal)	No change in contact resistance

^{1,2,3,4} Tested according to Mil. Std. 202C, Methods 301, 302, 103B, Cond. B, 104 Cond. C, respectively.

⁵ Tested according to Mil. -C-26482C.

* Cable Pattern A, contacts gold plated, epoxy mold compound. [Ref. 44]



A line of contour cable fabrication utilizing "Kapton" Type H-Film has been available from the Contour Cable Division of the Hughes Aircraft Co. for several years. Samples of typical cable construction with "Kapton" Type H-Film are shown at the end of this report. Recently, the Hughes Aircraft Co. has begun a NASA study contract to evaluate thin wall electrical wiring for spacecraft applications. Twenty-four tests comprise the spectrum of electrical, mechanical, and thermal testing involved in this program. Some of the preliminary test data on polyimide ("PYRE-M.L." and "Kapton" Type H-Film)-Teflon FEP wire insulation are shown in the following table:

Thin-Wall Candidates

Wire Type	FEP - H-Film	FEP - Polyimide PYRE ML Coating
Insulation thickness, inch (nom.)	0.006	0.007
O.D. of no. 24 AWG wire, inch	0.036	0.038
Mandrel wrap (1 x diam.)	wrinkles, minor delamination	slight coating wrinkling on inside
Notch sensitivity .002 inch nick	nick did not propagate	nick did not propagate
Soldering minimum heating	no insulation damage	no insulation damage
excessive heating	"	"
Solder Iron Contact (5 sec.)	no damage	no damage
Insulation Cold Flow, room temp. .015 inch radius edge, grams withstand 24 hours	exceeds 15,000	2400
Insulation Cold Flow, 160°F .015 inch radius edge, gram withstand 24 hours	exceeds 10,000	1700
Low temp. bend (-68°F)	satisfactory	satisfactory
Flexing	14108	2260
Cycles to first 2 failures	21618	3344
Mandrel heat aging 225°F, 1/8 in. diam., 1 lb. weight	satisfactory	satisfactory
Thermal strippability (Rheostat setting)	conductor strands fray when insulation re- moved (high heat 9, 10)	satisfactory (medium to high 5,6, 7,8)
Marking, Kingsley	satisfactory	satisfactory
Bondability	18.6	8.6
average pull in lbs., 5 samples		

The Hughes Aircraft Co. has just prepared a Company Specification HMS-2-1293 (for copy see Specification Section of this report) for the procurement of a modified polyimide-coated copper alloy insulated electrical wire for 200°C service and 250 volts RMS. It covers thin-wall, hook-up wire insulated with Teflon (FEP and TFE) having a copper alloy conductor and an outer coating of modified polyimide (ML).

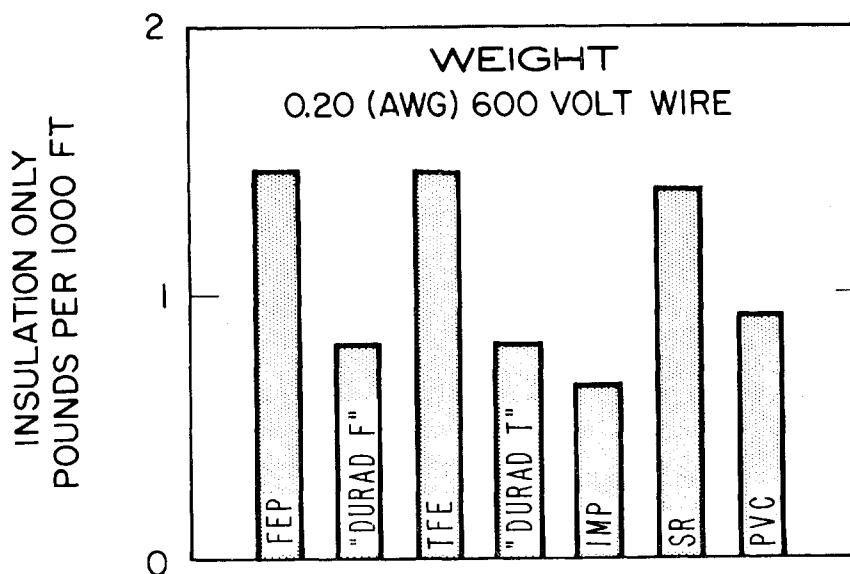
Numerous other cable and wire companies have perfected techniques for applying polyimide and modified polyimide resin coatings, varnishes, enamels

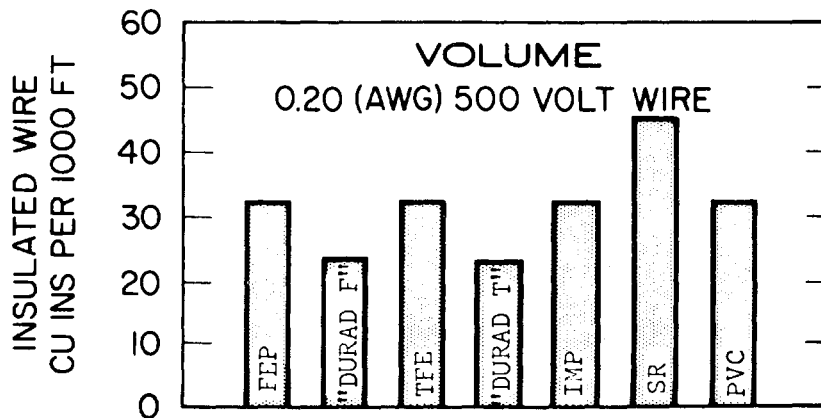
to Teflon TFE and FEP surfaces to provide improved construction as well as enhanced mechanical and electrical properties of cables and wires. Another useful jacket combination involves composites of polyimide films with Teflon TFE and FEP for cable and wire construction. Such polyimide-Teflon combinations give improved cut-through resistance, increased abrasion resistance, and permit valuable weight and space savings without sacrificing the excellent electrical and thermal properties afforded by Teflon TFE and FEP (potting is also accomplished more easily than with insulations of TFE or FEP alone). Other advantages to cable and wire applications include: improved radiation resistance, non-flammability, zero flame travel in the Underwriter Laboratories vertical flame test. The abrasion resistance characteristic is important when wires are drawn over rough surfaces and sharp edges during installation (measured values depend upon method of testing and variables within the test procedures used).

The Teflon-"PYRE-M.L." system can be made in colors including red, orange, yellow, green, gray, tan, brown and black which can be manufactured within the range of specification MIL-STD-104 according to Learn & Seeger of Hi-Temp Wires Co. [Ref. 66]. Polyimide film insulation is widely used in hook-up wire (in sizes 30 AWG to 16 AWG) meeting the test requirements of MIL-W-16878 for 300 V (tested to types ET and KT), 600 V (types E and K) and 1000 V (types EE and KK) and available from various wire and cable companies:

- a) Tensolite Insulated Wire Co., Inc.
- b) Circuit Materials Co.
- c) Philadelphia Insulated Wire Co.
- d) Anaconda Wire & Cable Co.
- e) Hi-Temp Wires Co.
- f) American Super-Temperature Wires, Inc.

The American Super-Temperature Wires, Inc. reports the following comparative table of polyimide insulations against other standard insulations with regard to the weight and volume savings possible:





Polyimide insulating films (e.g., DuPont "PYRE-M.L.", Anaconda A.I.-220, or Amoco A.I. Polymer, Type 10) have the following definite advantages over other similar wire insulations:

- Unusually high dielectric strength
- Tougher, smooth slippery surface highly resistant to abrasion damage
- High dielectric strength at elevated temperatures under humid conditions and after long-term aging
- Extreme resistance to film deformation under heat and pressure (this property assures prevention of cut-thru failures at wire crossovers)
- Higher thermal stability and longer thermal life
- Excellent windability (magnet wire and coil applications)
- High chemical resistance
- Provides higher overload resistance thus permitting heavier duty operations

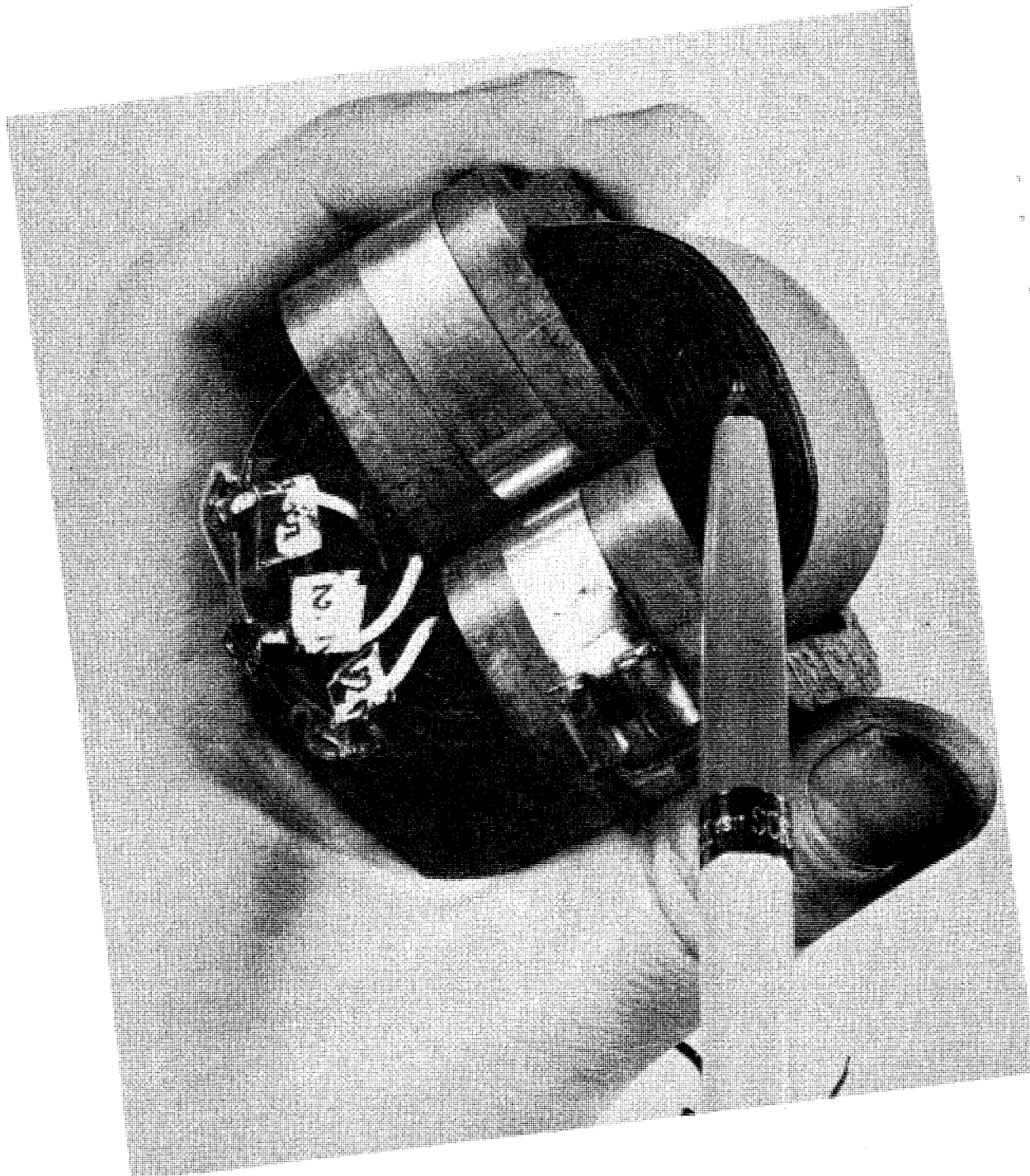
The Amoco Chemical Company has recently released overload resistance data on their polyamide-imide AI-10 polymer for different overload amperages:

Varnish	Wire*	Overload Current		
		22 amps	24 amps	25 amps
Phenolic	PE	7 cyc.-fails		
	PAI	10 cyc.-OK	2 cyc.-OK	3 cyc.-fails
	PI	10 cyc.-OK	2 cyc.-OK	4 cyc.-fails
Polyester	PE	4 cyc.-fails		
	PAI	10 cyc.-OK	2 cyc.-OK	1 cyc.-fails
	PI	10 cyc.-OK	2 cyc.-OK	1 cyc.-fails

* PE = Polyester Isonel 200
PAI = AI-10 Polyamide-imide
PI = DuPont Polyimide

[Ref. 104]

The Molecu-Wire Corporation has announced recently the development of a new nickel-base, non-magnetic alloy wire insulated with polyimide resins for continuous operation at temperatures up to 480°F and for short exposures to 840°F. The 800-ohm alloy wire is claimed to offer unprecedented resistance stability over a broad temperature range. It has high resistivity and a low temperature co-efficient of resistance. The wire, in light, regular, or a heavy insulation film thickness, is available in diameters from 0.004 to 0.01 inch.



6. Computers

In computers, polyimide-Teflon (TFE and FEP) wire and cable composites offer the following advantages: thin-wall construction, resistance to cut-through, and non-flammability.

7. Miscellaneous Electrical Applications

Todd & Wolff (1964) mention the following electrical applications for SP Polymer: relay components such as bobbins and actuators, connector inserts, stand-off terminals, and rigid printed-circuit boards. In many of these applications, the nonelectrical properties such as abrasion resistance or creep resistance were important reasons for using polyimides.

Applications for H-Film include: wire and cable wrap, motor slot liners, formed coil wrap, transformers, capacitors, flexible printed circuits, magnetic and pressure sensitive tapes, and hose and tubing. Many of these applications are based on the fact that its electrical properties, such as dielectric strength and dissipation factor, are excellent and remain nearly constant over a wide range of temperature and frequency. DuPont reports that printed circuits and flat cable constructions have been made from H-Film because of its dimensional stability at high temperature and its solder resistance. In transformer layer insulations, H-Film permits the design of smaller units which operate at higher temperatures with no reduction in rating.

Mallouk (1964) reported that the dimensional stability of H-Film at high temperatures (shrinkage is 0.3% at 392°F and 3.5% at 752°F) should prove important in uses such as magnetic tape and printed circuits, where shrinkage could distort the signals.

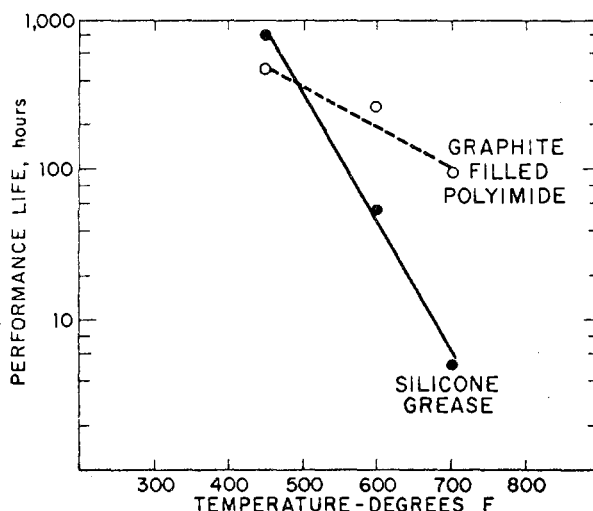
B. MECHANICAL

Mechanical applications for SP Polymer include sleeve bearings, ball bearing cages, gears, O-rings, vanes for rotary compressors, seals, valve seats and piston rings. Todd & Wolff (1964) note that the self-lubricating properties of Polymer SP were required in the cages which must function in severe environments of high temperature and/or vacuum and where lubricants cannot be used. SP Polymer offered greater strength and better wear properties than carbon graphite in the compressor vanes exposed to dry gases. These authors also revealed that one of the first applications for SP Polymer was in diamond abrasive wheels, where it replaced a phenolic binder for the diamond particles. Advantages of molded polyimide "Vespel" wheels over conventional ones are: better wear, greater cutting rate, and production of a finer surface on the ground part. Further, they noted . . . "Resistance to creep and solvent resistance were essential properties in fuel seals used in jet engines where temperatures exceed 450°F at 3000 psi pressure. A thermocouple plug in a plastics extruder required the strength and creep resistance as well as the thermal stability of polyimide materials. At the opposite temperature extreme, the new polymer offered the needed toughness and dimensional stability for a liquid nitrogen valve component".

G. R. Bower of DuPont has been an advocate of self-lubricating plastics on the basis of various data on plastic materials including graphite-filled

polyimide (see section of Wear & Frictional Properties). He was able to obtain a critical temperature of 600°F at the bearing surface with polyimide.

Devine and Kroll have also studied the self-contained lubricating characteristics of polyimide compositions (unfilled and graphite-filled) and report the following performance life as a function of temperature for polyimide lubricated ball bearings:



Performance Life vs Temperature for Silicone Grease and Polyimide Lubricated Ball Bearings [Ref. 22]

These authors found the unlubricated dynamic coefficient of friction of unfilled polyimide against steel is low and the graphite-filled material is even lower. When run in contact with polished SAE 1025 mild steel at a relative velocity of 834 fpm (30 psi) the coefficient of friction for unfilled polyimide varies from 0.08 to 0.15 and for filled material under the same conditions the value varies from 0.05 to 0.08.

The same authors explored experiments for providing additional sources of lubricants to the bearing by substituting polyimide balls for some of the steel balls in addition to polyimide bearing retainers. Duplicate determinations for performance life were 303 and 312 hours, respectively. Other test experiments utilized one-piece machined polyimide-graphite retainers designed for thrust bearings. Initial tests were conducted at 600°F and 10,000 rpm for the one-piece outer land riding retainer having pockets to accommodate eight balls. Failure occurred after 130 hours of operation. Three additional tests provided running times of 133, 151 and 93 hours. It was observed that failure was associated with wear of the low retainer cross-sections separating the balls. Increasing this cross-section by reducing the number of ball pockets to 6 resulted in performance life of 228 hours. The following table compares the bearing performance life of the various combinations tested in this program:

Comparison of Bearing Performance Life

Apparatus: High Temperature Performance Test Apparatus
 Specimen: 20 mm Radial Ball Bearing
 Load: 5 lb Thrust, 3 lb Radial
 Speed: 10,000 rpm
 Lubricant: Self Lubricating Aromatic Polyimide Retainer

Bearing	Retainer	Temp. °F	Performance Life (Hrs)
AISI-M10	Polyimide	450	151, 153, 159
AISI-M10	Polyimide-Graphite	450	473
AISI-M10	Polyimide-Graphite	600	183, 205, 341
AISI-440C	Polyimide-Graphite	600	200, 238
AISI-M10	Polyimide-WSe ₂	600	79
AISI-M10	Polyimide-MoS ₂ /Graphite	600	90
AISI-M10	Polyimide-MoS ₂	600	122
AISI-M10	Polyimide-Graphite	650	180, 155
AISI-M10	Polyimide-Graphite	700	95, 81

[Ref. 22]

The stability of polyimides at high temperatures has led to studies of the material for use as compressor seals and bearings in hermetic refrigerent systems. Test bars of polyimides, along with test coupons of copper and steel, were sealed in a refrigerant and refrigeration oil. These tests showed that the polyimide has no significant effect on the decomposition of a typical fluorocarbon refrigerant (DuPont's "Freon 12") after 30 days at 250°F; and no effect with another fluorocarbon ("Freon 22") after 30 days at 300°F. Likewise, only very slight traces of permanent gases could be detected in the sealed tubes containing the polyimides.

The DuPont Co. has suggested their PI-1101 Polyimide experimental binder solution for metal-to-metal, non-metal to non-metal, and non-metal to metal bonding applications where high bond strength (1000-2000 psi) at elevated temperatures (400-700°F) for prolonged periods (e.g., >1000 hours at 550°F) is desired. For their PI-1100 Polyimide binder solution, they suggest that PI-1100 adhesives would have a service life on the order of 2 to 5 years at temperatures up to 500°F. Ruffing & Traynor (1964) demonstrated some steel washers bonded with DuPont H-Film and Polymer Film heat-bonded to stainless steel.

Schweitzer (1964) reports uses for the polyimides as high temperature adhesives for bonding metals to metals, film to film, and film to metal are being investigated at the DuPont Co. One such adhesive, he claims, retains a tensile shear strength of 1100 psi after 60 hours at 700°F.

Various shear strength property data of polyimide adhesive formulations are reported in the Mechanical Properties section of this report.

C. AIRCRAFT, MISSILE & SPACE APPLICATIONS

Schweitzer (1964) of the DuPont Co. has suggested that the polyimides may provide a key to better ablative coatings on future satellites and missiles. Because they are useful at temperatures in the range of 550 to 700°F, polyimides might be used as protective coatings on leading edges of aircraft wings, where temperatures at supersonic speeds can exceed the limits of currently used coatings which disintegrate at 400 to 500°F. Polyimides could also be used in radar domes or covers on aircraft. Here, the polyimides would be the binder for glass fiber or other high-temperature-resistant materials.

The excellent thermal stability, weatherability and adhesive properties suggest "PYRE-M.L." enamels as good candidates for binding resins in filament wound constructions and laminates, particularly in the aerospace industries, according to the DuPont Co. Other uses suggested by the excellent heat resistance and high temperature toughness of "PYRE-M.L." enamels would be coatings for molds, for internal gas turbine parts, and for aircraft and missile parts.

Mallouk (1964) states . . . "A variety of aerospace applications are suggested by H-Film's resistance to ionizing radiation, which is greater than that of any other organic film."

Leppla & Carryer [Ref. 67], in discussing Bendix's experience with polyimide insulation systems, noted that the new insulation system has also proved quite satisfactory for the electrical supply units on missiles carried by military aircraft. During firing, these units must withstand extremely high temperatures for short periods of time. An increase in reliability has also been noted in production of equipment for high altitude military aircraft. These units well exceed the requirements of a 50% overload for two minutes at an altitude of 35,000 feet. This level of performance was difficult to achieve with the old insulation system. The new units have recently been approved also for use in civilian commercial aircraft.

The Defense Products Division of the Brunswick Corporation has been investigating fiberglass reinforced polyimide resin laminating systems for use in radomes, heat shields and related structures subject to high temperature exposures. Their tests to date indicate that their current commercial polyimide system can be used under continuous service temperatures up to 700°F and intermittent service temperatures up to 1000°F.

The Tensolite Insulated Wire Co., Inc. have reported that their "Tufflite" wire (insulated with DuPont's "Kapton" polyimide film Type HF) has been supplied to provide lightweight, chemically resistant harnesses for Titan III missiles. By using the "Kapton" insulation, a weight saving of 216 pounds was effected.

The Lunar Excursion Module (LEM) made by Grumman is using "Kapton" polyimide insulation for the major portion of the wiring.

The SPRINT anti-missile missile is utilizing "Kapton" insulation exclusively for both engine and capsule wiring.

While no space applications have been reported in the literature, some preliminary vacuum testing has been accomplished on "SP-Polymer" and "Kapton" Type H-Film in order to gain some insight into the possible usefulness of these materials as space lubricants. Buckley and Johnson at NASA conducted some evaporation studies on "SP-Polymer" in vacuum (down to 10^{-8} mm Hg) and at ambient temperatures to 875°F. The results obtained in these experiments are presented in the section on THERMAL PROPERTIES, Thermal Degradation & Weight Loss. The evaporation rate for the polyimide was less than 10^{-10} (g) (cm^{-2}) (sec^{-1}) at ambient temperatures to 500°F. The same authors conducted some friction and wear studies in a high vacuum (10^{-9} mm Hg) with the polyimide sliding on both metals and itself. The data are presented in the MECHANICAL PROPERTIES, Friction & Wear section of this report.

Haveg Super/Temp. Division of American Super-Temperature Wires, Inc. ran a series of outgassing tests on "Kapton" Type H-Film wire insulation in a vacuum. Samples of "Kapton" Type H-Film insulated #20 AWG wire, weighing between 700 and 800 mgs., were placed in a high vacuum chamber equipped to continuously record weight changes to a sensitivity of 5 micrograms. The samples were heated to $111^{\circ} \pm 2^{\circ}\text{C}$ and maintained at that temperature while pressures from 1 to 10^{-7} torr with a precision of $\pm 50\%$ of the value read were recorded. The entire test run was 200 hours. The total weight loss was 1.12×10^{-4} gm/cm². The actual weight loss rate at various times was as follows:

<u>Time</u>	<u>Weight Loss Rate</u>
5 hours	2.72×10^{-6} gm/cm ² /hr
40	3.66×10^{-7}
168	1.24×10^{-7}

[Ref. 110]

For spacecraft, polyimide-Teflon (TFE and FEP) wire and cable constructions offer an unmatched combination of resistance to mechanical abuse, electrical reliability, resistance to radiation and space savings.

For wiring in conventional aircraft, the advantages of extreme toughness and weight and space savings are afforded by polyimide-Teflon (both TFE and FEP) wire and cable constructions along with complete non-flammability.

P. M. Cook of the Raychem Corp., in a comparative study of intermediate-temperature, light-weight, small-size, abrasion-resistant wire for use on high-speed modern aircraft, compared various insulations with current military specifications. Two polyimide composites for airframe wires were included in summary charts and excerpted as follows:

	<u>FEP/ML</u>	<u>TFE/ML</u>
Outside Diameter (Average - inches)	0.062	0.062
Weight (Average - lbs/M ft.)	4.60	4.56
Insulation Resistance (Megohms/M ft.)	157,000	133,000

table (continued)

	<u>FEP/ML</u>	<u>TFE/ML</u>
Dielectric Breakdown (Average Volts)	> 21,700	> 21,700
Heat Aging - Bend Test	Fail-Note 1	Pass
Heat Aging - Dielectric (Average Volts)	Fail	5,280
Cold Bend - Dielectric (Average Volts)	> 21,700	> 21,700
Abrasion, Tape (Average - inches)	12.3	9.7
Abrasion, Tape, Temperature (Average - inches)	12.4	12.7
Bundle Flammability		
Part a	Pass	Pass
Part b	Fail	Fail
Part c	Fail	Fail
Vertical Flammability (inches)	3.3	2.4
Dynamic Cut-Through (Pounds)	26.1	27.3
Static Cut-Through (Ounces)	39	32
Notch Sensitivity (Visual)	Jacket Split	Jacket Split
Notch Sensitivity - Dielectric (Average Kilovolts)	17.7 (arcing)	12.2
Short-time Overload (Seconds)	60	47
Insulation Shrinkage (inches)	0.01	0.01

Note 1: All specimens flowed

DuPont reports that work is under way to assess the possibilities of "PYRE-M.L." varnish as a heat transfer coating for the aerospace environment. It showed desirable high absorption (0.92-0.93) in the visible and near infrared and emissivity of about 0.9 from 40°C. (104°F) to 350°C. (662°F).

D. PRESSURE VESSEL APPLICATION

Schweitzer (1964) states that high-temperature pressure vessels, constructed from filament-wound glass impregnated with polyimides, are another potential application. The DuPont Company recommends their PI-3301 polyimide binder solution for filament wound structures. DuPont also recommend their "PYRE-M.L." enamels as good candidates for binding resins in filament wound construction and laminates, particularly in the aerospace industries.

V. PRICES AND AVAILABILITY

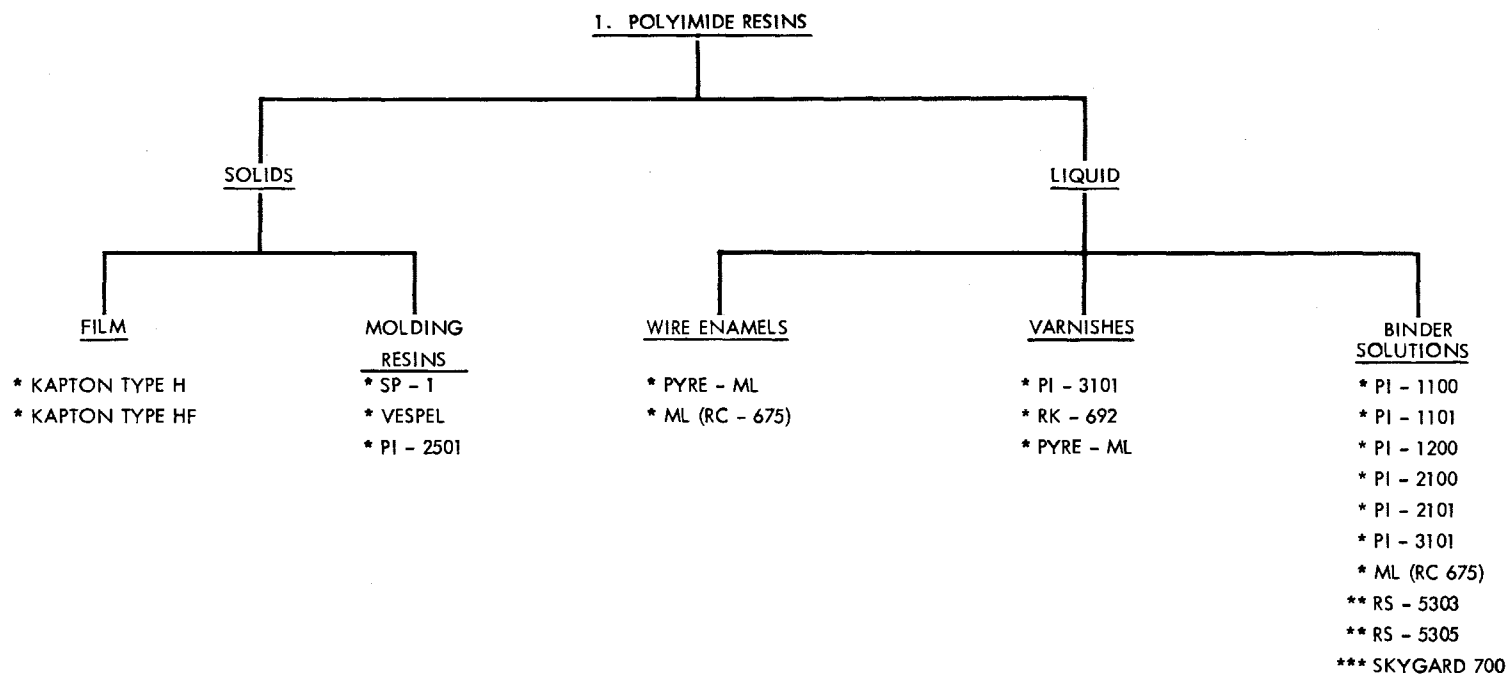
Since the polyimide polymers have been a relatively newcomer to the plastics field and involved in some patent controversies as well as problems of a proprietary nature, there has been no release of official information on the production of each company. For 100% solids, one reliable source quotes (unofficially) the following polyimide production statistics:

1962: 70,000 to 80,000 lbs.
1965: 250,000 lbs.
1970: 8 to 10 million lbs. (estimated)
1972: 10 million lbs. (wire enamel polyimide market)

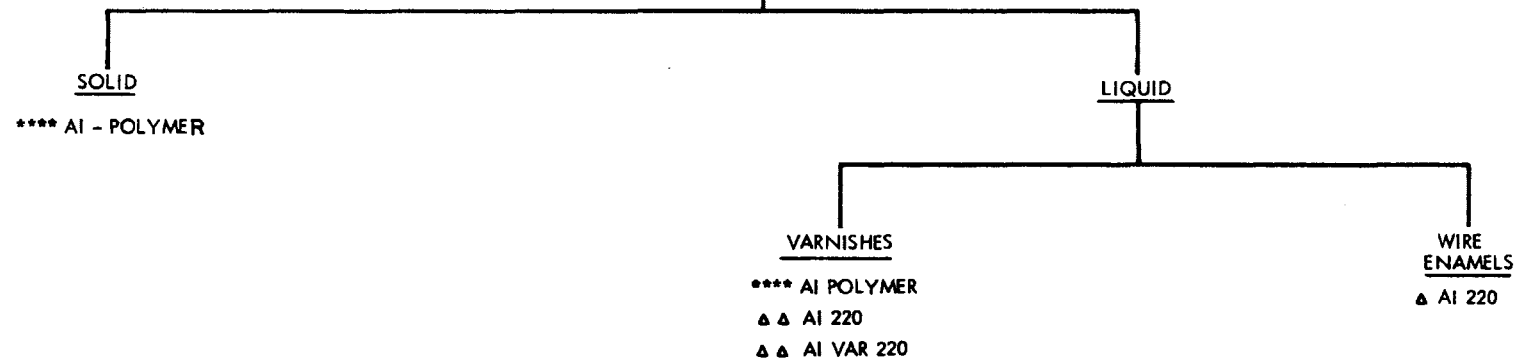
The following companies are suppliers of polyimide or polyamide-imide in one form or another:

- 1) E.I. du Pont de Nemours and Company, Wilmington, Delaware
 - "Kapton" Type H Film
 - "Kapton" Type HF Film (in 9 different constructions)
 - "SP-1" Polymer
 - "Vespel" Fabricated Polyimide Parts
 - "Pyre-M.L." Varnish
 - "ML (RC-675)" Binder Solution
 - PI-1100 Binder Solution
 - PI-1101 Binder Solution
 - PI-1200 Binder Solution
 - PI-2100 Binder Solution
 - PI-2101 Binder Solution
 - PI-2501 "B-Staged" Fiberglass Prepreg
 - PI-3101 Binder Solution
 - PI-3301FGR Prepreg Fiber Glass Roving
- 2) Amoco Chemicals Company, Chicago, Illinois
 - A.I. Polymer (Type 10) Polyamide-imide
- 3) Shawinigan Resins Corporation, Springfield, Massachusetts
 - RS-5303 Binder Solution
 - RS-5305 Binder Solution
- 4) Sterling Varnish Company, Sewickley, Pennsylvania
 - "AI-200" Varnish
 - "Aivar 220" Varnish
- 5) Monsanto Company, Plastics Division, St. Louis, Missouri
 - "Skygard 700"
- 6) Anaconda Wire and Cable Company, 605 Third Avenue, New York, New York. 10016
 - "A.I. 220"
- 7) Schenectady Chemicals, Inc., Schenectady, New York 12301
 - Isomid

Manufactured resins currently available on the market fall into the following categories:



II. POLYAMIDE - IMIDE RESINS



III, POLYESTER - POLYIMIDE RESINS

LIQUID

WIRE ENAMELS

- ◆ ISOMID
- ◆ TEREPEC FN
- ◆ TEREPEC FH

Δ ANACONDA WIRE AND CABLE

Δ Δ STERLING VARNISH CO

* E.I. du PONT de NEMOURS

** SHAWINIGAN

*** MONSANTO

**** AMOCO CHEMICAL CORP

◆ DR. BECK & CO.

There are a number of fabricators or converters who take "Kapton" Type H Film and the various binder solutions or varnishes from the above suppliers and convert them into composites of one type or another, e.g. tapes, adhesives, cable, wire, etc:

- 1) W.L. Gore and Associates
555 Paper Mill Road, Newark, Delaware
- 2) Tensolite Insulated Wire Company
West Main Street, Tarrytown, New York
- 3) Circuit Materials Company
812 Hamilton Street, Somerset, New Jersey
- 4) Hughes Aircraft Company Contour Cable Division
Bldg. 121/MS-9, P.O. Box 90515, Los Angeles, California 90009
- 5) Philadelphia Insulated Wire Company
Moorestown, New Jersey
- 6) Hi-Temperature Wires Company
Westbury, New York
- 7) American Super-Temperature Wires, Inc.
50 West Canal Street, Winooski, Vermont
- 8) Molecu-Wire Corporation
Eatontown-Freehold Pike, Scobeyville, New Jersey
- 9) Haveg/Super-Temperature Wires
4530 Brazil Street, Los Angeles, California 90039
- 10) Brand Rex, Division of American ENKA Corporation
817 West Beverly Boulevard, Montebello, California
- 11) Brunswick Corporation, Defense Products Division
Marion, Virginia
- 12) Niemand Brothers
45-10 94th Street, Elmhurst, Long Island, New York 11373

Towards the end of 1965, the E.I. du Pont de Nemours and Company expects to have in operation a full-scale plant in Circleville, Ohio to manufacture its "Kapton" Type H film in any width up to 36 inches. At the present time (July 1965) the film is available in any width from 3/16" in 1/16" increments. The price for 1,2,3, and 5-mil "Kapton" Type H film is \$25 per pound; for the 1/2"-mil "Kapton" Type H, it is \$40 per pound. Developmental price for "Kapton" Type HF Film (a composite of Type H and Teflon FEP fluorocarbon film) is \$30 per pound in all constructions:

Code for Type HF Films	Construction	Thickness (mils)	Area Factor (sq.ft./lb.)
019	1H/ 1/2FEP	1 1/2	77
919	1/2FEP/ 1H/ 1/2FEP	2	54
011	1H/ 1FEP	2	54
111	1FEP/ 1H/ 1FEP	3	34
929	1/2FEP/ 2H/ 1/2FEP	3	39
021	2H/ 1FEP	3	39
022	2H/ 2FEP	4	27
031	3H/ 1FEP	4	30
051	5H/ 1FEP	6	21

The Fabrics and Finishes Department of the E.I. du Pont de Nemours and Company reported effective as of February 1, 1965 the following price schedules will be in effect for all the polyimide high temperatures binder solutions:

	<u>Container</u>	<u>Per Pound Polyimide Solids</u>
Less than 5 gallons	1's or qts.	\$25.00
5 gals. but less than 25 gals.	1's or 5's	22.50
25 " " " " 50 "	1's or 5's	20.00
50 " " " " 100 "	5's	17.50
100 " " " " 150 "	5's	15.00
150 " and over	5's	12.50

The above prices, which cover individual releases of a given code, are F.O.B. our Philadelphia, Pennsylvania plant, with freight charges prepaid and allowed, and subject to the terms Net 15th Prox.

Amoco Chemicals Corporation quote the following prices for their AI Polymer (Type 10):

<u>DRUMS</u>	<u>QUANTITY, POUNDS</u>	<u>PRICE, \$/LB.*</u>
T/L** or C/L**		3.00
40 - T/L	5,000 - T/L	3.05
10 - 39	1,250 - 4,875	3.10
2 - 9	250 - 1,125	3.25
1	125	3.50
1	10	5.00

*FOB Plant (Joliet, Illinois)

**Minimum Tariff Weight

MINIMUM ORDER: 10 Pounds

TERMS: Net 30 Days

Shawinigan Resins Corporation report (as of November 1964 Bulletin)
the following price schedule for their RS 5303 and RS 5305 binder solutions:

<u>Quantity</u>	<u>Price/Lb of Solution</u>	<u>Terms</u>
Less than 10 lbs	\$6.00	Net 30 days, FOB point of origin.
10 lb. Containers	4.20	
50 lb. Lined Steel Cans	3.65	
Lined Steel Drums (550 lb. net)	3.20	

- - - - -
"Kapton" Type H-Film



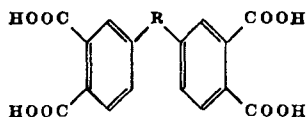
VI. PATENTS AND TRADENAMES

A. PATENT LITERATURE REVIEW

The developmental chemistry and method of processing polyimide resins have had an interesting patent history and have been only recently (April 20, 1965) resolved. In the following discussion only the major highlights of each patent will be treated and the important details stressed as they appeared to the author (who incidently is not versed in patent legal practices).

Polyimides were recorded in the patent literature as early as 1945, e.g., British Patent 570,858 to the E.I. du Pont de Nemours and Company "Linear fiber-forming polymers." These same patent claims eventually became U.S. Patent 2,710,853 "Polyimides of Pyromellitic Acid," (June 14, 1955). This patent discloses that linear fiber-forming polyimides could be obtained by a process of heating a mixture of substantially equimolar amounts of a diprimary diamine having a radical length of at least four and a tetracarboxylic acid, such as pyromellitic acid (having two pairs of carboxylic acid groups), the members of each pair being attached to adjacent carbon atoms. It also disclosed that equivalent amide-forming derivatives of such acids, such as the esters, anhydrides, acid chlorides, amides, or nitriles, may be used instead of the acids themselves.

Polyimide intermediates (2,2-bis-3,4-dicarboxyphenyl)propane were patented in U.S. 2,712,543 (July 5, 1955). The DuPont Company was granted U.S. 2,731,447 (January 17, 1956) "Novel polyimides" in which part of the linear polyimide polymers is derived from tetracarboxylic acids of the formula:



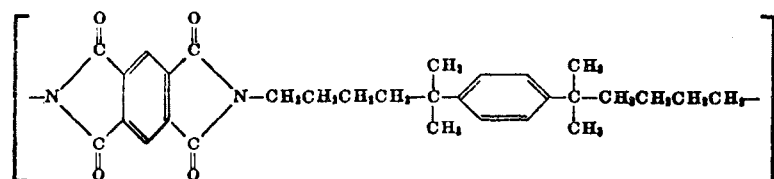
and contained the novel -R- which could be an -O- ether radical or a bivalent aliphatic saturated hydrocarbon (2,2-propane) radical. This had not been described previously in the chemical literature.

U.S. 2,811,548 (Oct. 29, 1957) "Method of purifying aromatic dicarboxylic acids" extended the -R- radical to -O-R"- and -O-R"'-O- radicals where the R"" and R"' are alkyl groups containing from 1 to 6 carbon atoms. The acids were extended to terephthalic acid plus several others and included two solvent recrystallization medias (DMF and DMA).

The next patent to appear, U.S. 2,867,609 (Jan. 6, 1959) (to the DuPont Company) covered the preparation of high molecular weight polypyromellitimides by heating finely divided polypyromellitimide-forming salts in the solid state to remove volatiles until a solid-stage condensation product having an inherent viscosity of at least 0.04 is obtained, and further heating the resulting product until a polypyromellitimide-forming salt results and which consists essentially of substantially equimolar amounts of (a) a difunctional diprimary diamine component and (b) an acidic component such as a pyromellitic diacid diester in which the esterified carboxyl groups are attached to non-adjacent carbon atoms. A companion patent, U.S. 2,880,230 is a continuation-in-part of U.S. 2,710,853 issued June 14, 1955 which disclosed that high molecular weight polypyromellitimides, derived from a diamine which disclosed that high

molecular weight polypyromellitimides, derived from a diamine of the group consisting of 4,4-dimethylheptamethylene diamine, nonamethylene diamine, and 3-methylheptamethylene diamine, have a unique combination of desirable physical properties. The novelty of the continuation patent lies in the preparation of poly-pyromellitimides of high tractability, from novel organic salts consisting of substantially equimolar amounts of (a) a basic difunctional diprimary diamine and (b) temporarily difunctional pyromellitic diacid diesters. Considerable attention is given to the various groups of diamines.

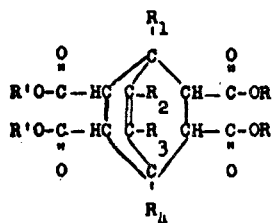
In U.S. 2,900,369 (August 18, 1959) Edwards and Robinson of the E.I. du Pont de Nemours and Company patented a polyimide composition (PDAPI) having the formula:



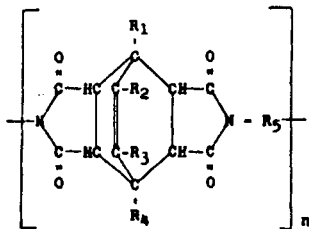
and reported various physical, mechanical, and electrical properties. PDAPI refers to poly-para-phenylene-bis-(1,1-dimethyl pentamethylene) pyromellitimide.

Several years later, A.L. Endrey also of the DuPont Company patented U.S. 3,073,784 an electrically conductive polymeric composition in which a polyimide is the structure (tape, tile, film, fiber filaments, tubing) for containing silver particles (of less than 1 micron size). This patent was followed up by a similar electrically conductive polymeric polyimide composition containing either metallic iron, cobalt, nickel, or copper by R.J. Angelo in U.S. 3,073,785. The process of polymerization and reactants were similar to the previous. Major differences resided in the metal organic salts used (acetylacetonates).

Chow and Whelan (Union Carbide Corporation) in the U.S. 3,037,966 claimed a method for producing thermoplastic polyimides from diacid diester derivatives of bicyclic tetracarboxylic acid dianhydrides and diprimary diamines. The diacid diester dianhydride having the general formula:



wherein one R and one R' are hydrogen atoms and the other R and R' are alkyl radicals having up to 12 carbon atoms. R₁, R₂, R₃, and R₄ are each members selected from the group consisting of hydrogen, alkyl, aryl, and halogen groups. The diprimary diamine recommended by the authors is H₂N-R₅-NH₂ wherein R₅ is a divalent hydrocarbon free of aliphatic unsaturation. The final polyimide structure resulting from the imidization step is:



On October 1, 1963, Lavin, Markhart and Hunt of Shawinigan Resins Corporation received their patent (U.S. 3,105,775) on combination coatings in which wires are coated with multiple layers of cured polyvinyl acetate resin composition and polyimide resin compositions. A commercial "ML" polyamide enamel is included in the comparative property measurements (1 kilovolt-life test, scrape abrasion test, hydrolysis resistance test). The polyimide layers, whether as an inner or outer layer, extended the life of the wires by a large factor. The polyamide formulation results from a combination of pyromellitic dianhydride (tetracarboxylic aromatic acids) with a diamine (e.g. ether group of 4,4'-oxydianiline) dissolved in equal volumes of dimethyl acetamide and N-methylpyrrolidone.

A milestone in polyimide patent disclosures was reached on April 20, 1965 with the release of six basic patents to the E.I. du Pont de Nemours and Company as follows:

- 1) U.S. 3,179,614
- 2) 3,179,630
- 3) 3,179,631
- 4) 3,179,632
- 5) 3,179,633
- 6) 3,179,634

and one to Westinghouse Electric Corporation: U.S. 3,179,635.

U.S. 3,179,614 covers polyamide-acid compositions and their methods of preparation. The author of the patent (W.E. Edwards) detailed the preferred process involving premixing equimolar amounts of the diamine and the dianhydride as dry solids and dissolving them in an organic solvent (at least 60% of the final polymeric solution). Since the reaction is exothermic, it is necessary to regulate the temperature and also to agitate the system to achieve maximum viscosity which denotes maximum polymerization. Another preferred process described, comprised dissolving the diamine reagent in one portion of a solvent and the dianhydride reagent in a second portion of the same or another solvent, and mixing the two solutions. He notes further that the use of equal molar amounts of the reactants under the prescribed conditions provides polyamide-acids of very high molecular weight. The use of either reactant in large excess limits the extent of polymerization. Although for some purposes, it may be desirable to use 1-3% excess of either reactant, preferable the dianhydride. Besides using an excess of the polyamide-acid, a chain terminating agent such as phthalic anhydride may be used to "cap" the ends of the polymer chains. For the preparation of shapeable compositions, it is essential that the molecular weight be such that the inherent viscosity of the polymer is at least 0.1, preferably 0.3 - 5.0. The patentee, Edwards, discusses the utility and effectiveness of the primary diamines and lists 40 diamine compositions which can be used to prepare polyimide polymers. In similar fashion, he discusses preferred and suitable dianhydrides and lists 13 of them.

Since solvents play an important role in the polymerization process, the author devotes some discussion to their functional behaviour and preferred types, particularly, N,N-dimethylformamide and N,N-dimethylacetamide. Twenty other solvents are also listed as candidates. Additionally, this author presents a table with 36 diamine-dianhydride-solvent system combinations for the preparation of polyamide-acid film plastics. Finally, a number of polyimide film methods are cited and are obtained by baking the polyamide-acids anywhere from 150 to 375°C. The tensile properties of some of the resultant polyamide-acid films are quoted in the patent.

In U.S. 3,179,630, Endrey patented a process for preparing polyimides by treating polyamide-acids with lower fatty monocarboxylic acid anhydrides, preferably acetic anhydride. Also, he prefers to have some tertiary amine (e.g. pyridine) present as well. The ratio of the tertiary amine to anhydride may vary from zero to almost infinite mixtures with a 1:1 ratio being the most commonly used. The amine functions as a catalyst for the action of the cyclizing agent, the anhydride. He lists a number of other possible lower fatty acid anhydrides and aliphatic ketenes as well as tertiary amines. To make shaped polyimide polymer structures, Endrey found that a composition containing a polymeric component made up of at least 50% of the polyamide-acid will suffice for most combinations of diamine/dianhydride reaction products. He stresses further that the maximum permissible temperature of the reaction process will depend on the diamine used, the dianhydride used, the particular solvent, the percentage of polyamide-acid desired in the final composition and the minimum period of time that one desires for the reaction. He notes, that temperatures up to 175°C. may be tolerated, however, to obtain the maximum inherent viscosity (i.e. maximum degree of polymerization), for any particular combination of diamine, dianhydride, solvent, etc. and thus produce shaped articles such as films and filaments of optimum toughness, it has been found that temperature throughout the reaction should be maintained below 60°C, preferably 50°C. A total of 55 combinations of reactants-solvents-conversion step chemicals to produce polyimide films are cited in the patent. Tensile strength data on various compositions and methods of processing are also reported therein. For other details (order of mixing reactants, viscosity, terminating agents, solvent quantities, pigment additions, application, etc.), the original patent should be consulted.

In U.S. 3,179,631, Endrey (E.I. du Pont de Nemours and Company) reviews the basic polyimide chemistry and reactants, i.e., the diamines, the tetracarboxylic acid dianhydrides, solvent systems, and the lower fatty monobasic acid anhydrides/tertiary amine conversion systems as detailed in his previous sequenced patent. The novelty in this new patent is the precipitation process using the lower fatty monobasic acid anhydride/tertiary amine as the precipitant. The end product of this precipitation process is a polymeric powder that is predominantly polyimide powder. However, Endrey prefers to complete the conversion process by heating the reaction mixture to an elevated temperature (preferably above 200°C) but below the crystalline melting point of the polyimide. Some emphasis in this patent is given to specific temperatures and specific times to complete the polyimide conversion and is dependent on the diamine used, the dianhydride used, the particular solvent, the percentage of polyamide-acid desired in the final composition and the minimum period of time desired for the reaction. A discussion of coatings (with and without pigments) and the substrates is also given since the novelty of this patent involves particulate solid particle formation or powders.

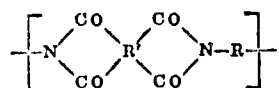
The patent's author treats the resulting mechanical and electrical property data obtained by incorporating additives in some cases to the polyimides and compacting (by pressure) the powders into a chip or disc with the further application of heat. A wide variety of possible applications are also discussed in view of the novel processes or methods of preparation of the polyimide powders.

In U.S. 3,179,632, "Process for preparing polyimides by treating polyamide-acids with aromatic monocarboxylic acid anhydrides," Hendrix reviews the general chemistry of reactants, solvent systems, conversion agents (listing numerous anhydrides and tertiary amines) cited in the above previous patents. He notes the greater ease of forming shaped articles with the polyamide-acid compositions (minimum of 50%) rather than the polyimides and claims that it is better to provide a composition containing enough polyamide-acid so that it can be shaped into useful objects prior to conversion of polyamide to polyimide. Pigment additions, substrates, coatings, chemical conversion methods are also discussed. The novelty in this patent is the use of an aromatic monocarboxylic acid anhydride to form the insoluble solid polyimide. Some of the previously reviewed patents used lower fatty acid anhydrides in the conversion processing phase.

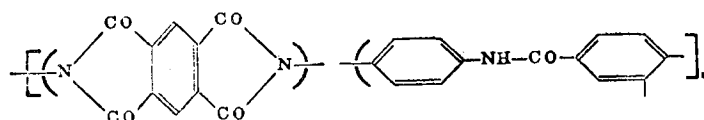
Endrey also reviewed (in U.S. 3,179,633) the same general chemistry of polyimides as did Hendrix above. His novelty claims lie in a composition prepared from a combination of two diamines with a dianhydride. The two diamines being meta-phenylene diamine and para-phenylene diamine. The resulting polyamide-acids prepared from this combination having a composition content of at least 50% for producing an easily shaped object as described above by Hendrix. Further, Endrey reviewed previous polyamide-acid → polyimide conversion processes of (a) heating 300° - 500°C, (b) chemical treating with a dehydrating agent alone or in combination with a tertiary amine (e.g. acetic anhydride-pyridine and added two new ones: (c) treatment with a carbodiimide (e.g. dicyclohexylcarbodiimide) and (d) partial chemical treatment as in (c) plus a subsequent heat treatment. He cites various mechanical, thermal, electrical and physical property data for these new two diamine combination formulations as well as for the two new polyimide conversion methods.

In the final patent (U.S. 3,179,634) of the series of six granted to the E.I. du Pont de Nemours and Company on April 20, 1965, Edwards reviews briefly the general chemistry of polyimides and their insolubility in solvents such as concentrated 96% sulfuric acid, fuming nitric acid, the monohydrate of sym-dichlorotetrafluoroacetone and the hydrate of monochloropentafluoroacetone. He discusses the preference of a composition containing enough polyamide-acid (at least 50%) to make useful shaped articles and their modification with pigments, dyes, inorganic and organic fillers as reported in the previous DuPont patents above. Also stressed are the specific time and temperature, method of mixing and reacting, solvents for forming polyimides from a specific diamine and a specified dianhydride as in the other patents. Edwards cites 35 examples of diamine-dianhydride-solvent reactant-solvent combinations to produce polyimide compositions and gives various electrical, mechanical, thermal, and physical property data on these combinations. A wide variety of potential electrical and thermal applications are cited by Edwards for the polyimide films and fillers produced by following the formulations noted in the various claims. These applications appear to be the only novelty in the patent.

Frost and Bower (Westinghouse Electric Corporation) received U.S. 3,179,635 on April 20, 1965 to cover the preparation of "Linear polymeric amide-modified polyimides and process of making same." The novelty of this patent lies in the amide-modification groups (-R-) attached to the imide (>N) radical. These authors cite a wide range of molecular amide linkages or configurations which can be incorporated into the polyimide structure--such materials were not cited in any of the previously discussed patents. In a similar manner, a wide range of molecular groupings are depicted for the R' or tetravalent radical in the polyimide structure:



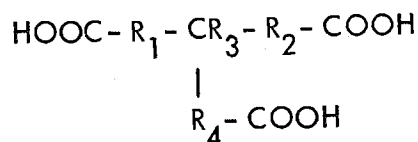
A number of different organic oxidation-reduction reactions are cited for the preparation of the amide linkages in the diamine. The cured amide-modified polyimide has the following typical molecular structure:



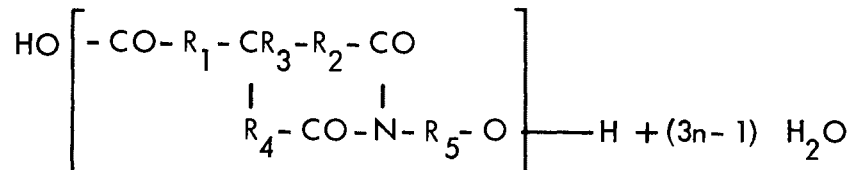
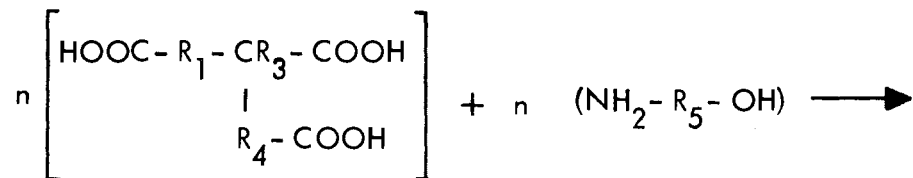
where n is an integer of at least 5.

Frost and Bower utilize the novel diamine compound m-amino-benzoyl-p-aminoanilide as a starting reactant with various dianhydrides to form a soluble polymeric intermediate. Another novel feature is the use of a phenylene diacyl halide (isophthaloyl chloride and terephthaloyl chloride) in amounts sufficient to react with substantially all of the remaining amino groups resulting from prior reaction of the aromatic dianhydride and excess aromatic diamine.

Frosch of the Bell Telephone Laboratories obtained U.S. 2,421,024 (May 27, 1947) for linear polymers having cyclic imide groups in their molecular chains. The reactants consist of a tricarboxylic acid of the formula:

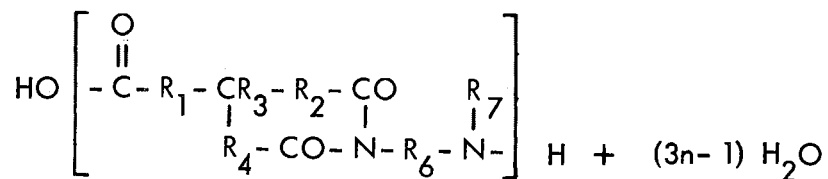
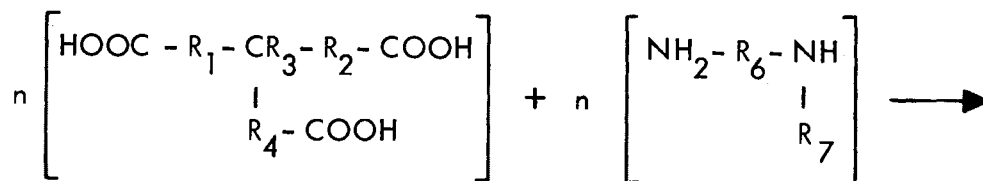


where R₁ and R₂ each is a divalent organic radical of chain or cyclic structure, R₃ is hydrogen or a monovalent organic radical of chain or cyclic structure. R₄ can be a divalent hydrocarbon radical of chain or cyclic structures with either a diamine (NH₂-R₅-NHR₆) or alcohol-amine (NH₂-R₇-OH). R₅ and R₆ are divalent organic radical or chain or cyclic structure, R₇ is hydrogen or a monovalent hydrocarbon group. The reaction involved with these alcohol-amine reactants yields:



in which R_1 and R_5 are divalent radicals connected together by alternate ester and cyclic imide groups to form the linear polyimide-ester.

The reaction with a diamine reactant yields:



in which R_1 and R_6 are divalent radicals connected together by alternate amide and cyclic imide groups to form a linear polyimide-amide.

The patent mentions applications such as adhesives, coatings or impregnating materials and preparation possibilities of polyimide fibers, films, sheets, coatings, molded articles, and impregnated materials.

Tricarballic acid is cited as a typical reactant tricarboxylic acid material. Diamine reactants mentioned include decamethylene diamine; non-amethylene diamine; 1,10 decanolamine; ethylene diamine; and propylene diamine.

In addition to the above detailedly discussed patents were found a number of other U.S. and foreign polyimide patents as follow:

- 1) U.S. 2,421,024 (C.J. Frosch: to Bell Telephone Laboratories)
"Linear polymer containing cyclic imide groups" (May 27, 1947).
- 2) U.S. 2,502,576 (J. Lincoln and J.G.N. Drewitt: to Celanese Corporation of America)
"Polyamides containing glutarimide rings" (April 4, 1950).
- 3) Australian 58,424 (E.I. du Pont de Nemours and Company)
"Polyimides and methods of making" (1960).
- 4) Belgian 614,941 (W.M. Edwards: to E.I. du Pont de Nemours and Company)
"Polyamide acids" (September 10, 1962).
- 5) Belgian 615,937 (E. Lavin et al: to Shawinigan Resins Corporation)
"Composite insulation for electrical wires." (October 3, 1962).
- 6) Belgian 617,901 (E. Lavin et al: to Shawinigan Resins Corporation)
"Enamels for insulating electrical wires" (November 21, 1962).
- 7) Belgian 627,623 (W.R. Hendrix: to E.I. du Pont de Nemours and Company)
"Polyimides" (July 25, 1963).
- 8) Belgian 627,625 (W.M. Edwards and A.L. Endrey: to E.I. du Pont de Nemours and Company)
"Powdered Resins" (July 25, 1963).
- 9) Belgian 627,626 (W.M. Edwards and A.L. Endrey: to E.I. du Pont de Nemours and Company)
"Moldable polyimides" (July 25, 1963).
- 10) Belgian 630,749 (E.E. Berr et al: to E.I. du Pont de Nemours and Company)
"Carbon-filled polyimide resins". (November 4, 1963).
- 11) Belgian 635,549 (J.R. Chalmers: to E.I. du Pont de Nemours and Company)
"Coating composition consisting of a polyamino acid and a solvent"
(January 29, 1964).
- 12) Belgian 638,688 (L.E. Amborski and W.P. Weisenberger: to E.I. du Pont de Nemours and Company)
"Expanded polyimides" (March 18, 1963).

- 13) Belgian 638,689 (W.R. Hendrix: to E.I. du Pont de Nemours and Company)
"Expanded polyimides" (March 18, 1963)
- 14) British 570,858 (E.I. du Pont de Nemours and Company)
"Linear fiber-forming polymers" (July 25, 1945)
- 15) British 762,152 (E.I. du Pont de Nemours and Company)
"Polyimides of pyromellitic acid" (November 21, 1956).
- 16) British 815,821 (Monsanto Chemical Company)
"Thermoplastic interpolymers derived from maleic anhydride" (July 1, 1959)
- 17) British 898,651 (W.M. Edwards: to E.I. du Pont de Nemours and Company)
"Polymers containing polyamide-acid recurring units" (June 14, 1962).
- 18) British 903,271 (W.M. Edwards and A.L. Endrey: to E.I. du Pont de Nemours and Company)
"Polyimides" (August 15, 1962).
- 19) British 903,272 (E.I. du Pont de Nemours and Company)
"Polyimides for films and coatings" (April 1, 1959).
- 20) British 904,559 (M.W. Thompson: to Imperial Chemical Industries Limited)
"Polyamide-imide resins" (August 29, 1962).
- 21) British 935,388 (Westinghouse Electric Corporation)
"Linear polymeric amide-modified polyimides" (August 28, 1963).
- 22) British 945,673 (A.L. Endrey and R.J. Angelo: to E.I. du Pont de Nemours and Company)
"Polyimide films" (January 8, 1964).
- 23) French 1,239,491 (E.I. du Pont de Nemours and Company)
(1960)
- 24) German 1,111,635 (F. Reicheneder et al: to Badische Anilin- and Soda-Fabrik)
"Polycyclic dicarboximides" (July 27, 1961)
- 25) German 1,151,120 (H. Seifert et al: to Farbenfabriken Bayer)
"Ion-exchange resins" (July 4, 1963).
- 26) German 927,292 (Thuringische Zellwolle)
"Mixed linear polyamides" (May 5, 1955).
- 27) Spanish 288,912 (A.H. Lopez)
"Terephthalimide polyester for insulation of electrical conductors"
(June 21, 1963)

B. TRADENAMES

- AI Polymer (Amoco Type 10) - a single system poly(amide-imide) resin based on trimellitic anhydride of the Amoco Chemicals Corporation. It is not considered a copolymer.
- AI-200 - an impregnating varnish offered by Sterling Varnish Company based upon unmodified poly(amide-imide) polymers developed by Amoco Chemicals Corporation in conjunction with Anaconda Wire and Cable Company. It is the trademark of the Anaconda Wire and Cable Company for its poly(amide-imide) magnet wire enamel.
- AIvar-220 - an impregnating varnish offered by Sterling Varnish Company based upon modified poly(amide-imide) polymers developed by Amoco Chemicals Corporation in conjunction with Anaconda Wire and Cable Company.
- Aramidyl - tradename for an aromatic amide-imide polymer produced by Westinghouse Electric Corporation.
- Durad - designation of a modified polyimide wire insulation of the American Super-Temperature Wires, Inc.
- Durad F - designation of Teflon FEP-coated wire with a "Durad" polyimide jacket of the American Super-Temperature Wires, Inc.
- Durad T - designation of Teflon TFE-coated wire with a "Durad" polyimide jacket of the American Super-Temperature Wires, Inc.
- EE-6379 - a pressure-sensitive "H-Film" tape product composed of DuPont "H-Film", 1.0 mil thick backing with a high-temperature pressure-sensitive silicone adhesive offered by the Permacel Company (New Brunswick, N. J.).
- FXM-34-B-25 - an experimental polyimide adhesive for high temperature application with 112 glass cloth and Volan "A" finish of the Bloomingdale department, American Cyanamid Company.
- H-Film - original tradename for a polyimide film material marketed by the E.I. du Pont de Nemours and Company which is now designated as "Kapton" H. or "Kapton" Type H.
- Isomid - a polyester-polyimide polymer manufactured by Schenectady Chemicals, Inc. Schenectady, New York 12301.
- "Kapton" Type H - trademark for a polyimide film material marketed by the E.I. du Pont de Nemours and Company. It was formerly called H-Film.
- "Kapton" Type HB - an experimental E.I. du Pont de Nemours and Company laboratory film material containing Teflon TFE dispersed in a "Kapton" Type H-Film.
- "Kapton" Type HF - a composite of polyimide film ("Kapton" Type H) and Teflon FEP marketed by the E.I. du Pont de Nemours and Company.
- MQ - a polyimide resin in solution form developed by the E.I. du Pont de Nemours and Company.

- MRE-66 - a polyimide in solution form at 40% solids content developed by the E.I. du Pont de Nemours and Company.
- PI-1100 - an E.I. du Pont de Nemours and Company polyimide thermoplastic high temperature binder solution containing 18.6% solids with a viscosity of 35-55 poises at 77°F.
- PI-1101 - an E.I. du Pont de Nemours and Company polyimide thermosetting high temperature binder solution containing 18.3% solids with a viscosity of 10-20 poises at 77°F.
- PI-1200 - an E.I. du Pont de Nemours and Company polyimide thermoplastic high temperature binder solution containing 18.4% solids and a viscosity 13-35 poises at 77°F.
- PI-2501 - an E.I. du Pont de Nemours and Company "B-Staged" polyimide fiber-glass prepreg with 181E Glass-A1100 finish.
- PI-3301 - an E.I. du Pont de Nemours and Company polyimide high temperature thermosetting binder solution containing 53.0% solids and a viscosity of 60-90 poises at 77°F.
- PI-3301FGR - an experimental E.I. du Pont de Nemours and Company polyimide prepreg fiberglass roving for filament wound constructions.
- PY 37 - a polyimide research material developed by Dr. Beck and Company GmbH, Hamburg, Germany.
- "Pyre-ML" - trademark of E.I. du Pont de Nemours and Company polyimide binder solution resin compositions.
- RC-5060 - an experimental E.I. du Pont de Nemours and Company polyimide insulating varnish containing 19% solids and a viscosity of 5-9 poises at 25°C.
- RC-B-24951 - an experimental E.I. du Pont de Nemours and Company polyimide insulating varnish containing 45% solids and a viscosity of 2-5 poises at 25°C.
- RK-692 - an E.I. du Pont de Nemours and Company insulating varnish with a solids content of 13% and a viscosity of 6-10 poises at 25°C.
- RS-5303 - a Shawinigan Resins Corporation high temperature polyimide solution with a solids content of 60-64% and a viscosity of 4,000 to 10,000 cps (Brookfield) at 25°C. It permits a more rigid type of bond.
- RS-5305 - a Shawinigan Resins Corporation high temperature polyimide solution with a solids content of 60-64% and a viscosity of 4,000 to 10,000 cps (Brookfield) at 25°C. It permits a more flexible type of bond.
- SP-1-Polymer - trademark of the E.I. du Pont de Nemours and Company for their molding grade of polyimide polymer.

Terebec FN - a polyester-polyimide polymer produced by Dr. Beck and Company GmbH, Hamburg, Germany.

Terebec FH - a polyester-polyimide polymer produced by Dr. Beck and Company GmbH, Hamburg, Germany.

Tufflite - trademark of Tensolite Insulated Wire Company, Inc. for their "Kapton" Type HF polyimide film insulated wire construction.

VespeI - tradename for parts made by the E.I. du Pont de Nemours and Company from polyimide polymer type SP-1 molding.

VII. SPECIFICATIONS

A number of specification documents have been prepared by the military, NASA, and the Hughes Aircraft Company to cover polyimide insulated wires. These are reproduced in toto to indicate the state-of-the-art of the utilization and consideration of polyimides for design purposes and procurement by these organizations.

Polyimide magnet wire is classed as a Class 220 insulation in MIL specification document MIL-W-583C and can be used for 220°C maximum temperature service. Various types of wire either with single and multiple insulation or combination insulations are listed in this specification. This document is not reproduced or included below.

Several enquiries among other electronic companies indicated that they were preparing company specification documents on polyimide insulations but they were not available for external distribution.

SPEC-40M39513/1
September 18, 1964

GEORGE C. MARSHALL SPACE FLIGHT CENTER
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
HUNTSVILLE, ALABAMA

SPECIFICATION SHEET

Wire, Electrical, Type C, 200°C., 250 Volts

1. Scope
 - 1.1 This specification covers the detail requirements for type C, 200°C., volt insulated wire.
2. Applicable Document
 - 2.1 The following document, of the issue in effect on date of invitation for bids, forms a part of this specification to the extent specified herein.

SPECIFICATION

George C. Marshall Space Flight Center

Specification 40M39513

Wire, Electrical, Hookup

(Copies of specifications, standards, drawings, and publications required by contractors in connections with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

3. Requirements
 - 3.1 General requirements. - The type C2 wire shall be in accordance with specified herein.
 - 3.2 Construction. - The type C2 wire shall be constructed in either of the two classes of conductor, a primary insulation, and an outer coating as follows:
 - (a) Class A, stranded alloy conductor, silver-coated.
Class S, stranded copper conductor, silver-coated.
 - (b) A thin wall of primary insulation of fluorinated ethylene propylene.
 - (c) An outer coating of polyimide bonded to the primary insulation.

- 3.3 Temperature and potential rating. - The type C2 wire shall have a maximum temperature rating of 200°C. The maximum potential rating shall be 250 volts (r.m.s.).
- 3.4 Part numbers, dimensions and weight. - The type C2 wire shall conform to the details of Table I of Specification 40M39513 and to the part number, dimensions, and weight of Table I below.

TABLE I
Part Number, Dimensions, and Weight

Part No.	Length of lay	Diam. over Primary Insulation		Diam. over outer covering		Cold Bend Test Mandrel Diam. (max)	Std. Pkg.
	(max)	(min)	(max)	(min)	(max)		(nom)
	inch	inch	inch	inch	inch	inch	ft.
(1)							
C2S32N	0.25	.019	.023	.0195	.024	1	5000
C2S30N	0.25	.022	.026	.0225	.027		5000
C2S28N	0.25	.025	.029	.0255	.030		5000
C2S26N	0.25	.029	.033	.0295	.034		4500
C2S24N	0.33	.034	.038	.0345	.039		3000
C2S22N	0.33	.040	.044	.0405	.045		3000
C2S20N	0.50	.048	.052	.0485	.053		2000

- (1) Insert the letter of the conductor class in column (1) of the part number in accordance with para. 3.2. Stranded alloy conductors are to be used only in AWG sizes 32 through 22.
- 3.5 Physical and electrical requirements. - The FEP, with modified polyimide, 250 volt wire shall conform to the physical and electrical requirements shown in Table II below.

TABLE II
PHYSICAL AND ELECTRICAL REQUIREMENTS OF WIRE OF INSULATION C2

REQUIREMENTS		TEST CONDITIONING
Conductor resistance	See Table I Spec. 40M39513	As received, no special conditioning.
Spark Test	2.5 kv	As received, no special Conditioning.
Finished wire weight	See Table I Spec. 40M39513	Test to para. 4.8.6 of Spec. 40M39513.
Conductor Tensile strength		
Class A	52,000 psi	Test to 4.8.8 of Spec. 40M39513.
Class S	Para. 4.8 of Spec. 40M39513	Test to para. 4.8 of Spec. 40M39513.

REQUIREMENTSTEST CONDITIONING

Dielectric strength	0.5 kv (min)	Immersion in water with .5% aerosol or other wetting agent at 25°C ±5°C.
Insulation resistance	20,000 megohms/1,000	Immersion in water with .5% aerosol or other wetting agent at 25°C ±5°C.
Heat resistance	1.5 kv (min) dielectric strength 1/8 inch (max) shrinkage.	Temperature conditioning, 96 hrs. at 250°C.
Cold Bend (See Table I for mandrel diam.)	1.5 kv (min)	Temperature conditioning, 4 hrs. at -65°C.
Soldering	1/8 inch (max)	As received, no special conditioning.
Dielectric constant	2.2 (max)	As received, no special conditioning.
Power Factor	0.005 (max) (or 0.5 per cent)	As received, no special conditioning.
Surface resistance	5 megohms (min) surface resistance ±50 (max) change when applicable.	Humidity conditioning, 96 hrs. at 25°C at 95% humidity.
Outer coating bend test, 10 turns on 2X mandrel.	No cracking or flaking	Immersion in solvent of two parts NMP and one part DMAC, 2 hrs. at 25°C ±5°C.

3.6 Manufacturer's identification. - Manufacturer's identification shall not be required in type C2 wire.

4. Quality Assurance Provisions

4.1 Inspection shall be in accordance with Specification 40M39513.

5. Preparation for Delivery

5.1 Preparation for delivery shall be in accordance with Specification 40M39513 and as specified herein.

5.2 Packaging

HMS

2-1293

MATERIAL SPECIFICATION**HUGHES AIRCRAFT COMPANY**
CULVER CITY, CALIFORNIAREV.
H.

PAGE 4 OF 7

4.3 Acceptance Tests.- Acceptance tests shall consist of the following plus those tests deemed necessary to assure that the wire ~~meets~~ the requirements of this specification:

- (a) High temperature resistance
- (b) Outer coating bend test

4.4 Test Methods.- Except for the following, tests shall be conducted in accordance with MIL-W-16878 as applicable to the requirements stated herein.

4.4.1 Breaking Strength and Elongation.- Breaking strength and elongation tests shall be conducted on a conductor specimen that has been taken from the completed wire and has had the insulation removed. Care shall be used in the removal of the insulation to insure that there is no nicking or damage to the strands. Test procedure shall be in accordance with QQ-W-343 using a 10-inch long specimen and a pull rate of 12 inches per minute. The jaws used for gripping the wire shall be the non-crushing type. The ultimate elongation shall be determined at the point where the first strand snaps.

4.4.2 High Temperature Resistance.-

4.4.2.1 Apparatus.- The test apparatus shall consist of a solder iron fastened to an upright frame by a rigid hinge located on the solder iron handle. The solder iron tip shall be a nominal 45° and form a flat surface with the asbestos test disk (see figure 1). The solder iron tip shall have a bearing surface of 1/2 inch $\pm$ 1/8 inch. The iron shall be weighted at the handle sufficiently to provide a 1/2 pound ($\pm$ 10%) tip pressure on the specimen. The apparatus shall include equipment sufficient to control the temperature at the tip of the solder iron to within $\pm$ 10°F. The apparatus shall have a 30 to 50 volt electric circuit arranged to indicate a burn thru failure when the solder iron tip contacts the conductor.

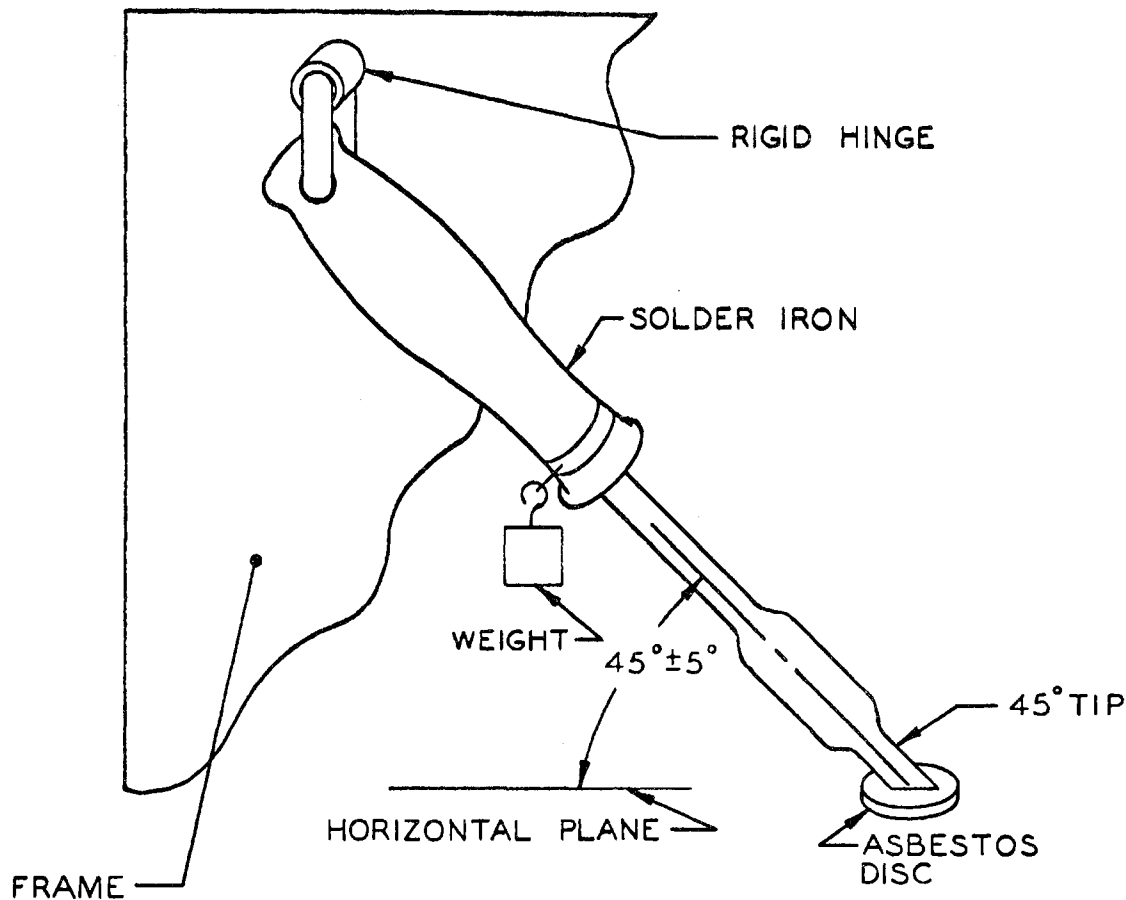


FIGURE 1 -- BURN-THROUGH APPARATUS

- 4.4.2.2 Procedure.-- One specimen six inches long shall be tested. Test procedure shall be as follows:

With the solder iron in a horizontal position, stabilize the tip temperature at $675 \pm 10^\circ \text{ F}$. Energize the test circuit. Place the specimen on the asbestos disc. Carefully lower the iron so that the flat part of the tip comes in contact with the specimen.

Specimens resisting burn-through for over 30 seconds shall be considered to have passed the test.

- 4.4.3 Outer Coating Bend Test.-- An unshielded wire specimen that has not been previously tested shall be wrapped a minimum of 10 complete turns around a smooth surfaced mandrel. The diameter of the mandrel shall be within the range of two times the outside diameter limits of the wire being tested (see Table II). Remove the specimen from the mandrel and expand the loops enough to thoroughly inspect the wire. Inspection shall be made using no less than 7 x magnification.

5.2.1 Wire shall be delivered on nonreturnable spools, each having an appropriate diameter in accordance with normal commercial practice. In no case shall the barrel of the spool be less than 1 3/4 inches in diameter. Unless otherwise specified in the order, the nominal length shall be 200 feet and the minimum acceptance length shall not be less than 50 feet.

5.2.2 No less than 75% of the total quantity of each size of wire ordered shall be in lengths equal to or greater than the nominal length. The maximum quantity of wire in a single length shall be limited only to the manufacturing and handling facilities.

6. Notes

6.1 The notes specified in SPEC-40M39513 shall apply including the note below.

6.2 Use. - The C2 wire specified herein is comparatively light weight and is suitable for high frequency use. It is resistant to ionization, radiation, and to crushing during installation. The outer covering resists hot soldering irons, does not support combustion, and is resistant to certain solvents, fuels, hydraulic fluids, and acids. The outer covering also provides a bondable surface for adherence to potting and encapsulating resins.

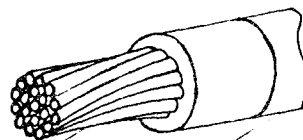
Notice. - When MSFC drawings, specifications, or other data are used for any purpose other than in connection with a definitely related MSFC procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever and the fact that MSFC may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian:

George C. Marshall
Space Flight Center

Preparing activity:

George C. Marshall
Space Flight Center



NICKEL-COATED COPPER CONDUCTOR

POLYIMIDE, DIP-COATED, 0.0007 ± 0.0002 in.

EXTRUDED POLYTETRAFLUOROETHYLENE (TFE)

CONSTRUCTION DETAILS

MS PART NO.	WIRE SIZE	STRANDING	MAX. DIA. STRANDED COND. (INCH)	FINISHED WIRE		
				MAX. RESISTANCE AT 20°C (68°F)-CLASS II (OHMS/1000 FT.)	DIAMETER (INCH)	MAX. WEIGHT LBS./1000 FT.
MS18105-28-*	28	7 X 36	0.016	68.3	0.035±.002	1.30
MS18105-26-*	26	19 X 38	0.021	40.1	0.039±.002	1.90
MS18105-24-*	24	19 X 36	0.026	25.1	0.043±.002	2.60
MS18105-22-*	22	19 X 34	0.033	15.5	0.050±.002	3.60
MS18105-20-*	20	19 X 32	0.041	9.79	0.058±.002	5.40
MS18105-18-*	18	19 X 30	0.052	6.08	0.070±.002	7.90
MS18105-16-*	16	19 X 29	0.060	4.76	0.080±.002	10.30
MS18105-14-*	14	19 X 27	0.074	3.00	0.100±.003	17.30
MS18105-12-*	12	19 X 25	0.093	1.88	0.120±.004	26.00

PERFORMANCE DETAILS

MS PART NO.	MINIMUM INCHES OF TAPE	WEIGHT SUPPORT BRACKET	WEIGHT (LBS.)	TENSION LOAD (LBS.)	BEND TESTING				
					MANDREL DIAMETER (INCHES-MAX.)			TEST LOAD (POUNDS)	
					LIFE CYCLE TEST	COLD BEND TEST	WRAP TEST	LIFE CYCLE TEST	COLD BEND TEST
MS18105-28-*	15	A	0.50	1.00	1/8	1/8	1/4	0.50	0.50
MS18105-26-*	21	A	0.50	1.00	1/8	1/8	1/4	0.50	0.50
MS18105-24-*	21	A	0.50	1.00	1/8	1/8	1/4	0.50	0.50
MS18105-22-*	30	A	0.50	1.00	1/4	1/4	1/4	0.75	0.75
MS18105-20-*	30	A	0.50	1.00	1/4	1/4	1/4	0.75	0.75
MS18105-18-*	24	B	0.75	1.00	1/4	1/4	3/8	2.00	2.00
MS18105-16-*	24	B	0.75	2.00	1/4	1/4	3/8	2.00	2.00
MS18105-14-*	36	B	1.25	2.00	3/4	3/4	3/4	3.00	3.00
MS18105-12-*	36	B	1.25	2.00	3/4	3/4	3/4	4.00	4.00

TEMPERATURE RATING: 260°C (500°F) Maximum conductor temperature
 VOLTAGE RATING: Sizes 28-16 inclusive, 600 volts; Sizes 14-12, 1000 volts.
 INSULATION FLAWS: 60 CY RMS-5000 Volts (Minimum)
 SHRINKAGE: 0.03 Inch (Max.) in 12 Inches @275°C
 BLOCKING: 260°C (500°F)
 SMOKE: 275°C (527°F)
 COLD BEND: 2 Turns (720°)

DIELECTRIC: 3000 Volts - 60 CY RMS

DIELECTRIC TANK TEST: 2200 Volts - 60 CY RMS, Sizes 14-12, 3000 volts 60 CY RMS

INSULATION RESISTANCE: 50,000 Megohms Per 1000 Ft. (Minimum) 100% test performed during Dielectric Tank Test

AIR OVEN: 275°C ± 3°C (527°F ± 5°F)

SURFACE RESISTANCE: 500 Megohm-In. Min.

COLOR: Yellow preferred. Available also in the following solid colors: Red, Orange, Green, Black, and Brown.

All Colors shall be in accordance with MIL-Std-104, class 1. White not available because of the nature of the polyimide coating.

IDENTIFICATION OF PRODUCT: Required (#28-#22 Print Vertical) (Sizes 20-12, print vertical or horizontal). (B)

MS PART NO.: The * shown in the tables above shall be replaced by a color code designator as shown below:

YELLOW	RED	ORANGE	GREEN	BROWN	BLACK
4	2	3	5	1	0

EXAMPLE: Size 20, Yellow: MS18105-20-4
Red: MS18105-20-2.

THIS STANDARD TAKES PRECEDENCE OVER DOCUMENTS REFERENCED HEREIN.
 REFERENCED DOCUMENTS SHALL BE OF THE ISSUE IN EFFECT ON DATE OF INVITATIONS FOR BID.

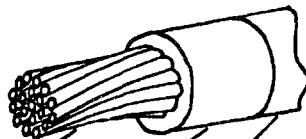
This standard has been approved by the Bureau of Naval Weapons Department of the Navy, and is mandatory for use by their activity. All other military activities are required to employ this standard where suitable.

P.A. NAVY-WP Other Cust	TITLE WIRE, ELECTRIC, EXTRUDED-TFE-FLUOROCARBON INSULATED, POLYIMIDE-COATED, NICKEL-COATED COPPER.	MILITARY STANDARD MS18105(wp)
PROCUREMENT SPECIFICATION MIL-W-22759(WEP)	SUPERSEDES:	SHEET OF

DD FORM 672-1 (Limited coordination)

PREVIOUS EDITIONS OF THIS FORM ARE OBSOLETE.

APPROVED 10 March 1964 REVISED 21 September 1964 7 December 1964



A SILVER-COATED COPPER CONDUCTOR

POLYIMIDE, DIP-COATED, 0.0007 ± .0002 in.

A
EXTRUDED POLYTETRAFLUOROETHYLENE (TFE)

CONSTRUCTION DETAILS

MS PART NO.	WIRE SIZE	STRANDING	MAX. DIA. STRANDED COND. (INCH)	MAX. RESISTANCE AT 20°C (68°F)-CLASS I (OHMS/1000 FT.)	FINISHED WIRE	
					DIAMETER (INCH)	MAX. WEIGHT LBS./1000 FT.
MS18104-28*	28	7 X 36	0.016	62.5	0.035±.002	1.30
MS18104-26*	26	19 X 38	0.021	36.2	0.039±.002	1.80
MS18104-24*	24	19 X 36	0.026	23.2	0.043±.002	2.60
MS18104-22*	22	19 X 34	0.033	14.6	0.050±.002	3.60
MS18104-20*	20	19 X 32	0.041	9.05	0.058±.002	5.40
MS18104-18*	18	19 X 30	0.052	5.80	0.070±.002	7.90
MS18104-16*	16	19 X 29	0.060	4.54	0.080±.002	10.30
MS18104-14*	14	19 X 27	0.074	2.37	0.100±.003	17.38
MS18104-12*	12	19 X 25	0.093	1.81	0.120±.004	26.00

PERFORMANCE DETAILS

MS PART NO.	ABRASION RESISTANCE (PROCEDURE 11)				BEND TESTING			
	MINIMUM INCHES OF TAPE	WEIGHT SUPPORT BRACKET	WEIGHT (LBS.)	TENSION LOAD (LBS.)	MANDREL DIAMETER (INCHES-MAX.)			TEST LOAD (POUNDS)
					LIFE CYCLE TEST	COLD BEND	WRAP TEST	
MS18104-28*	15	A	0.50	1.00	1/8	1/8	1/4	0.50
MS18104-26*	21	A	0.50	1.00	1/8	1/8	1/4	0.50
MS18104-24*	21	A	0.50	1.00	1/8	1/8	1/4	0.50
MS18104-22*	30	A	0.50	1.00	1/4	1/4	1/4	0.75
MS18104-20*	30	A	0.50	1.00	1/4	1/4	1/4	0.75
MS18104-18*	24	B	0.75	1.00	1/4	1/4	3/8	2.00
MS18104-16*	24	B	0.75	2.00	1/4	1/4	3/8	2.00
MS18104-14*	36	B	1.25	2.00	3/4	3/4	3/4	3.00
MS18104-12*	36	B	1.25	2.00	3/4	3/4	3/4	4.00

TEMPERATURE RATING: 200°C (392°F) Maximum conductor temperature

VOLTAGE RATING: Sizes 28-16 inclusive, 500 volts; Sizes 14-12, 1000 volts.

INSULATION FLAYS: 60 CY RMS-5000 Volts (Minimum)

SHRINKAGE: 0.03 Inch (Max.) in 12 inches @275°C

BLACKING: 260°C (500°F)

SMOKE: 275°C (527°F)

COLD BEND: 2 Turns (720°)

DIELECTRIC: 3000 Volts - 60 CY RMS

B DIELECTRIC TANK TEST: 2200 Volts - 60 CY RMS, Sizes 14-12, 3000 volts 60 CY RMS

INSULATION RESISTANCE: 50,000 Megohms Per 1000 Ft. (Minimum) 100% test performed during Diel. Tank Test

AIR OVEN: 275°C ± 3°C (527°F ± 5°F)

SURFACE RESISTANCE: 500 Megohm - In. Min.

COLOR: Yellow preferred. Available also in the following solid colors: Red, Orange, Green, Black, and Brown.

All Colors shall be in accordance with Mil-Std-104, class 1. White not available because of the nature of the polyimide coating.

IDENTIFICATION OF PRODUCT: Required (#29-#22 Print Vertical) (Sizes 20-12, print vertical or horizontal). B

MS PART NO.: The * shown in the tables above shall be replaced by a color code designator as shown below:

YELLOW	RED	ORANGE	GREEN	BROWN	BLACK
4	2	3	5	1	0

EXAMPLE: Size 20, Yellow: MS18104-20-4
Red: MS18104-20-2.

THIS STANDARD TAKES PRECEDENCE OVER DOCUMENTS REFERENCED HEREIN.

REFERENCED DOCUMENTS SHALL BE OF THE ISSUE IN EFFECT ON DATE OF INVITATIONS FOR BID.

This standard has been approved by the Bureau of Naval Weapons Department of the Navy and is mandatory for use by that activity. All other military activities are required to employ this standard where suitable.

P.A. NAVY- WP Other Cust	TITLE WIRE, ELECTRIC, EXTRUDED-TFE-FLUOROCARBON INSULATED, POLYIMIDE-COATED, SILVER-COATED COPPER	MILITARY STANDARD MS 18104(WP)
PROCUREMENT SPECIFICATION MIL-W-22759 (WEP)	SUPERSEDES:	SHEET OF

DD FORM 672-1 (Limited coordination)

PREVIOUS EDITIONS OF THIS FORM ARE OBSOLETE.

APPROVED 10 March 1964 REVISED 1 21 September 1964 2 7 December 1964

PREPARED BY <i>R.O. McLaughlin</i>	HUGHES AIRCRAFT COMPANY CULVER CITY, CALIFORNIA	HMS 2-1293	
APPROVED <i>D.L. Robinson</i>	MATERIAL SPECIFICATION	PAGE 1 OF 7	
APPROVED		ISSUE DATE 11 March 1962	
APPROVED		REVISION DATE 20 October 64	REV. H

1. SCOPE

- ① 1.1 Scope.- This specification covers thin-wall, hook-up wire insulated with fluorocarbon resins (Teflon FEP and TFE) having a copper alloy conductor and an outer coating of modified polyimide (ML). This wire is rated for 250 volts rms and is suitable for continuous operation within the temperature range of -65°C to 200°C (see 6.1).

- 1.2 Classification.- Wire covered by this specification shall be classified as follows:

Types

I	--	Unshielded
II	--	Shielded and Jacketed

Classes

1	--	Silver Coated Conductors
2	--	Nickel Coated Conductors

- 1.3 Superseding Information.- Where documents do not specify a Class, Class 1 shall be furnished.

2. APPLICABLE DOCUMENTS

- 2.1 The following document forms a part of this specification to the extent specified herein:

SPECIFICATIONS

Federal

QQ-W-343	Wire, Electrical and Nonelectrical, Copper (Uninsulated)

Military

MIL-W-16878	Wire, Electrical, Insulated, High Temperature

3. REQUIREMENTS

- 3.1 Qualification.- Wire covered by this specification shall be a product which has been tested and has passed the qualification tests specified herein. Qualified products are listed in Section 7 of this specification.

MMS	2-1293	MATERIAL SPECIFICATION	HUGHES AIRCRAFT COMPANY CULVER CITY, CALIFORNIA
REV. H	PAGE 2 OF 7		

Ⓜ 3.2 Materials and Construction.--

(a) Type I Wire.-- Type I wire shall consist of (1) a coated and stranded copper alloy conductor and (2) extruded primary insulation and (3) a thin modified polyimide outer coating.

(b) Type II Wire.-- Type II wire shall consist of (1) Type I wire and (2) silver coated copper shielding braid and (3) an extruded fluorinated ethylene propylene outer jacket.

3.2.1 Conductors.-- Conductors shall be silver coated or nickel coated strands of a copper alloy that shall retain its physical properties during manufacture. Conductor coatings shall meet the requirements of MIL-W-16878 and the conductor shall otherwise meet the requirements of Table I. Testing for conductor breaking strength and elongation shall be as specified in 4.4.1.

TABLE I -- CONDUCTOR REQUIREMENTS

Conductor Size, AWG	Minimum No. of Strands	Size of Strands, AWG	Conductor Dia., Inch			Max. Conductor Resistance (DC) per 1,000 ft. at 25° C (ohms)	Stranded Conductor	
			Min.	Nom.	Max.		Breaking Strength, lbs., Min.	Elongation % Min.
28	7	36	.014	.015	.016	70.0	6.5	6
26	19	38	.019	.020	.022	40.2	11	6
24	19	36	.024	.025	.027	25.8	18	6

Ⓜ 3.2.2 Primary Insulation.-- The wire shall be insulated with either natural or clear fluorinated ethylene propylene (FEP) containing no color pigmentation or white pigmented polytetrafluoroethylene (TFE) (see 6.5). In addition, the surface shall be prepared for bonding of the outer coating.

3.2.3 Outer coating.-- An outer coating of modified polyimide shall be bonded directly to the primary insulation. The coating shall have the natural yellowish-gold color of the resin and shall contain no foreign color pigmentation. The outer coating shall be not less than .0005 inch thick and at any cross section along the length of the completed conductor, the minimum measured thickness of the polyimide coating shall be not less than 50 percent of the maximum measured thickness at the same cross section.

3.2.4 Shielding Braid.-- Type II wire shall have a woven braid of 40 AWG coated copper strands applied immediately over the insulation coating. The shielding braid shall provide a coverage of 80 to 90 percent. The angle of the braid with the longitudinal axis of the wire shall be 18-28° and the braid shall have good push back ability.

3.2.5 Outer Jacket.-- Type II wire shall have an outer jacket of natural or clear extruded fluorinated ethylene propylene, containing no color pigmentation, applied directly over the shielding braid.

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Ⓜ 3.3 Finished Wire.- Wire covered by this specification shall meet all the requirements of MIL-W-16878 for Type KT or ET wire, as applicable, and except as modified herein: Testing for finished wire requirements shall be as specified in 4.4. In addition, the manufacturer shall be responsible for all testing that will satisfy the requirements herein. (See 6.5)

3.3.1 High Temperature Resistance.- The modified polyimide outer coating shall withstand for not less than 30 seconds the solder iron penetration test specified in 4.4.2.

3.3.2 Outer Coating Bend.- The outer coating of modified polyimide shall withstand the bend test specified in 4.4.3 without fracturing, splitting or peeling.

3.3.3 Jacket Flaws.- In addition to the dielectric requirements of MIL-W-16878, the outer jacket of Type II wire shall not break down when subjected to the spark test specified in 4.4.4.

3.3.4 Dimensions.- The finished wire shall conform to the dimensional requirements specified in Table II.

TABLE II -- FINISHED WIRE DIMENSIONS

Unshielded Type I			Shielded Type II					
Conductor Size, AWG	OD Over Insulation Including Coating, Inch		OD Over Shielding Braid, Inch		Outer Jacket Wall Thickness, Inch		Overall Diameter, Inch	
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
28	.027	.031	.041	.045	.008	.012	.057	.069
26	.031	.035	.045	.049	.008	.012	.061	.073
24	.036	.040	.050	.054	.008	.012	.066	.078

4. QUALITY ASSURANCE PROVISIONS

4.1 Classification of Tests.- The inspection and testing of the wire shall be classified as follows:

- (a) Qualification Tests: Qualification tests are tests performed on samples submitted for approval as qualified products.
- (b) Acceptance Tests: Acceptance tests are tests performed on individual lots that have been submitted for acceptance against a purchase order.

4.2 Qualification Tests.- Qualification tests shall consist of all the tests specified for Type KT wire per MIL-W-16878 plus those tests contained herein.

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- 4.4.4 Spark Test.- All Type II wire shall be subjected to a 1500 volt rms spark test in accordance with MIL-W-16878 by the manufacturer.

5. PREPARATION FOR DELIVERY

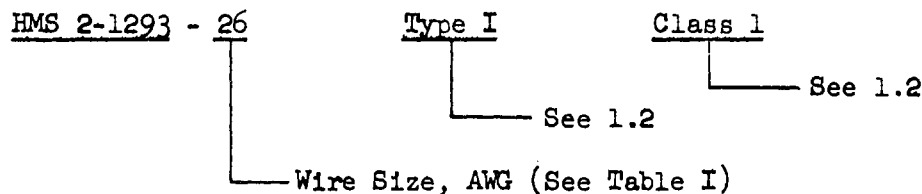
- 5.1 Packaging.- Unless otherwise specified, the minimum continuous length of wire shall be 100 feet. The wire shall be packaged on 6-1/2 inch spools to suitably fill the spool. Wire ends shall be brought out for testing. Packaging shall be done in a manner assuring protection of the wire during shipping, receiving, and storage.
- 5.2 Lot Identification.- For purposes of acceptance testing, all completed wire of the same size, type, and class, produced using the same conductor material and on the same machines without change in the machine set-ups shall be considered a lot. The manufacturer shall adequately identify this lot number on their shipping spools and papers.

- 5.2 Lot Identification.- For purposes of acceptance testing, all completed wire of the same size, type, and class, produced using the same conductor material and on the same machines without change in the machine set-ups shall be considered a lot. The manufacturer shall adequately identify this lot number on their shipping spools and papers.

6. NOTES

- 6.1 Intended Use.- This wire is intended for use in hook-up wire applications that require a reliable, light weight, 250 volts (rms), thin wall insulation. The wire provides a stronger construction than comparable copper conductor wires at a slight sacrifice in conductivity. The nickel coating is subject to less solder wicking than a silver coated conductor.

- 6.2 Method of Designation.- Wire covered by this specification is identified in accordance with the following example.



See 1.2

See 1.2

-Wire Size, AWG (See Table I)

- 6.3 Current Carrying Capacity.- Current carrying capacities are shown in Table III.

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TABLE III -- CURRENT CARRYING CAPACITY FOR GROUPS OF CONDUCTORS,
HARNESSED TOGETHER, 50% LOADED¹

Copper Alloy Conductor Size AWG	Maximum Conductor Temperature					
	Free Air, 105° F Ambient, Ground Support Equipment		Vacuum 130° F Ambient, Space Equipment		Vacuum 260° F Ambient Space Equipment	
	300° F	392° F	300° F	392° F	300° F	392° F
28	2.0 amps	2.3 amps	1.4 amps	1.8 amps	.8 amps	1.5 amps
26	2.0 amps	3.5 amps	2.0 amps	2.7 amps	1.3 amps	2.1 amps
24	3.8 amps	4.7 amps	3.0 amps	3.7 amps	1.7 amps	3.0 amps

- 1/ Under normal conditions, wire will withstand 392° F (200° C) continuously. Where heat sensitive electronic components such as diodes, transistors, etc. are subject to receiving heat conduction from hook-up wire, it may be desirable to limit the current.

6.4 Weights.- Approximate weights are shown in Table IV.

TABLE IV -- WIRE WEIGHTS

Type (See 1.2)	Conductor Size, AWG	Approximate Weight M ft., Lbs
I	28	.92
	26	1.37
	24	2.03
II	28	4.50
	26	5.06
	24	5.85

- ⑥ 6.5 Ordering Data.- The purchase orders should specify this specification number and revision, and in addition may request the furnishing of test data described in paragraph 3.3. The purchase order should specify either fluorinated ethylene propylene (FEP) or polytetrafluoroethylene (TFE) as the primary insulation (see 3.2.2).

7. QUALIFIED PRODUCTS

- ⑥ 7.1 Procurement under this specification is limited to the product furnished by the following manufacturers:

Manufacturer's Designation	Manufacturer's Name and Address
	Surprenant Mfg., Co. Clinton, Mass.

VIII HEALTH HAZARDS AND HANDLING PRECAUTIONS

The Du Pont Company has released various bulletins detailing handling precautions, health hazards, and safety considerations with each of their polyimide products. The following information is reproduced in toto from the Company and would apply to other polyimide materials from other companies as well.

A. SAFETY CONSIDERATIONS FOR "KAPTON" POLYIMIDE FILM

Animal toxicity studies have been conducted at Du Pont's Haskell Laboratory for Toxicology and Industrial Medicine on polyimide resins of the type of "Kapton" polyimide film.

Air-micronized dust of the polyimide resin powder has a low order of acute toxicity by inhalation. Exposure of rats to dust levels as high as 15.6 mg/l (nominal) for four hours caused only transient minor discomfort. No rats died as a result of these exposures. The powder had a low order of toxicity by ingestion. Rats fed a diet containing 25 per cent (w/w) of the polyimide powder survived a fourteen-day feeding period. No effects attributable to the compound were found by microscopic examination of tissues from rats inhaling the dust nor from rats consuming the polymer in their diet.

The air-micronized polymer powder was tested for primary irritation and sensitization by applying a 25 per cent suspension on guinea pig skin. It did not cause allergic contact dermatitis. In most of the test situations there was no evidence of skin irritation caused by the polymer. A few mild, transient cases of erythema developed in old animals which may have resulted from causes other than chemical irritation.

Rats were exposed to atmospheres containing pyrolysis products from approximately 5 grams of polymer powder heated to various temperatures in an air stream. Carbon monoxide was detected in the pyrolysis effluent beginning at 300°C. The CO concentration increased with higher temperatures. Exposures to products from heating to 450°C and above were lethal in 20-140 minutes. Four-hour exposures to products from heating at 400°C and below were not lethal. There was evidence of pulmonary irritation in the animals that died from exposures in the higher temperature range. Histopathological examination of lungs, livers, and kidneys of animals exposed to products of heating the polymer at any of the lower test temperatures showed no effects.

The results of these animal experiments forecast no hazard to health in the handling and use of "Kapton" polyimide film at ordinary temperatures. Ventilation must be provided when "Kapton" polyimide film is handled at elevated temperatures. Small amounts of dimethyl acetamide vapor may be released from film on heating slightly and at 300°C (572°F) emission of carbon monoxide begins. They must be removed by exhaust ventilation or diluted to safe levels which are respectively 10 parts per million by volume for DMAC and 100 parts per million for CO.

B. DU PONT POLYIMIDE BINDER SOLUTIONS

The toxicity hazards associated with the polyimide high temperature binder solutions experimental resin solutions have not been fully investigated. They contain nitrogenous solvents like Dimethylacetamide, Dimethylformamide, and N-Methylpyrrolidone. The following precautions should be observed:

Keep container closed when material is not being used. Avoid breathing vapors given off during application of binder solutions or fumes during force drying, baking or laminating of parts, as they may contain amines; use only with adequate ventilation.

Avoid contact with skin, eyes and clothing. In case of contact, flush skin or eyes with plenty of clean water for at least 15 minutes; for eyes, get medical attention. Remove and wash contaminated clothing before re-use.

After using these materials, wash hands before eating or smoking.

It is suggested that solvent resistant rubber apron and gloves be worn when using these materials and that safety glasses or monogoggles be used whenever there is a chance that the product might be splashed.

Store in tightly closed containers. Do not store outside. Avoid contact with water. Material is stable for the times shown by the following temperatures:

32° to 40°F - Approximately 1 year	Not over 100°F - 1 month
Not over 77°F - 3 months	Not over 120°F - 7 to 10 days

Protect from moisture of condensation, which has a deleterious effect on binder solution, by not opening cold container in an area of high humidity.

Viscosity of cold material can best be reduced by heating to room temperature rather than be adding diluent, which will reduce solids level. If it is necessary to reduce the viscosity of a product, this can be done by adding, with thorough and constant stirring, the diluent suggested on the data sheet covering the product involved. Use only the diluent shown and do not exceed the amount specified. Reduction of 50% by volume means 100 parts of the material being reduced and 50 parts of the diluent.

C. DU PONT "PYRE-M.L." ENAMEL

Toxicity studies by the Du Pont Company on animals indicate that single exposures to the solvents in "PYRE-M.L." enamels are not particularly hazardous. Both solvents possess a low order of acute toxicity. On the other hand, repeated animal exposures have disclosed that dimethylacetamide is capable of producing systemic injury when inhaled or absorbed through the skin in sufficient quantities over a period of time.

Adequate ventilation should be provided. Skin contact should be avoided. Exposed areas should be flushed with water immediately. Du Pont's T-8557 Thinner or other mixtures containing dimethylacetamide should not be used for cleaning equipment without adequate ventilation or without protective apparel.

D. DU PONT "RK-692" VARNISH

Use in a well-ventilated area. The solvents in this varnish consist of N-Methylpyrrolidone/Dimethylacetamide/Toluol (6/3/4). Solvent vapors should not be inhaled at length. Concentrations in the atmosphere should be kept below 20 parts per million to avoid toxic effects if eight hour daily exposure is involved. This can be accomplished with adequate ventilation. Solvent fumes introduced during the varnish cure operation should be exhausted to the outside atmosphere.

Skin contact should also be avoided. Areas exposed accidentally should be flushed with water immediately. Protection of the eyes is very important. Safety glasses or monogoggles should be worn whenever splashing is a possibility. If some of this varnish does get in the eyes, flood with clear water and obtain medical attention promptly.

Pressure Sensitive H-Tape Permacel

Gore & Associates
"Kapton" Polyimide Ribbon Cable

"Kapton" H-Film
E.I. du Pont de Nemours

Hughes Aircraft Company, Contour Cable Division

Polyimide Etched
Cable

Polyimide Flat-
Ribbon Cable

Super - Temperature FEP/H-Film Insulation

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