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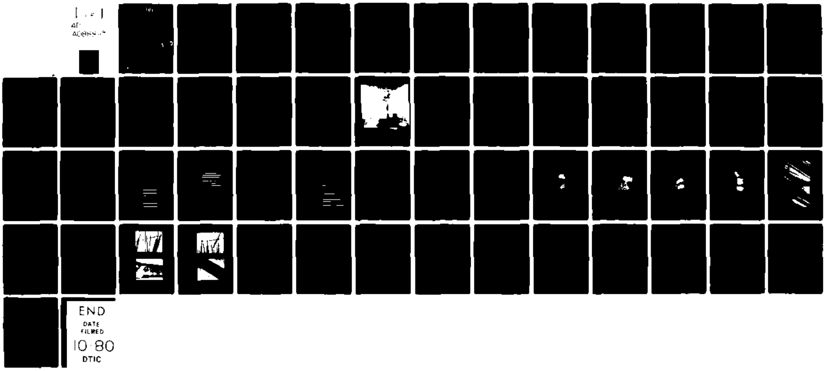

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CATALYTIC INCINERATION OF GRAPHITE FIBERS IN A GRAPHITE/EPOXY COMPOSITE FIRE

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As the use of graphite-reinforced composites grows, so does the risk of electrical interference caused by free graphite fibers which might be liberated from composite fires. Graphite oxidation catalysts or combustion promoters were evaluated to determine the extent to which they would cause graphite fibers to incinerate during a composite fire. Lead catalysts (PbO and Pb_3O_4) proved effective in reducing graphite/epoxy composite specimen weights by more than 90% ~~percent~~ when ignited. However, these results depend heavily on good draft, small conductive and convective heat losses, over

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20. Abstract cont'd

thin specimens and good catalyst distribution. Additional work is underway to find alternatives to the toxic lead oxides.

SUMMARY

Introduction.

This investigation deals with the potential carbon fiber hazard resulting from the burning of graphite composites - the possible release and dissemination of conductive filaments capable of bridging electrical conductors causing electrical interference and/or failure. Specifically, the approach was limited to an evaluation of gasification catalysts applied to the composite system either as a pigment in a paint coating or as a resin modifier prior to fabrication of the composite. In theory, these catalysts should promote the incineration of the fiber, thereby eliminating a large source of potentially hazardous conductive filaments.

The study was performed under Work Request N00019-79-WR 91472, Airtask A3200000/001B/9F54-591-201.

Results.

Two test methods were designed to evaluate the effectiveness of the catalyzed composite specimens: a continuous combustion test using an oxidizing flame and a self-sustaining combustion test using a reducing flame to ignite the specimen and controlled drafts to encourage incineration.

Differences between catalyzed and uncatalyzed specimens tested in the continuous combustion test were relatively small; the largest increase in apparent reaction rate for 6-ply specimens, noted for a paint coating containing Bi_2O_3, was 40 percent. PbO and Sb_2O_3 coatings also exhibited catalytic activity. Apparent reaction rates were found to depend inversely on specimen thickness.

In contrast, differences between catalyzed and uncatalyzed specimens in the self-sustaining combustion test were visually evident when specimens were removed from the reducing flame and placed over a controlled draft. PbO, Bi_2O_3, AgO, and Pb_3O_4 coatings applied to 6-ply specimens caused a uniform orange glow to appear when placed over the draft, coated side down and proved effective in reducing total specimen weight by more than 90 percent when a draft of 8.3 mph or more was used. Without a forced draft (natural convection) none of the catalysts reduced specimen weight by more than about 50 percent. When 10-, 20- and 48-ply specimens were tested with the PbO coating, more than 90 percent of the 10- and 20- ply specimens were consumed when the coated side faced a draft of 4.2 mph or more, whereas no more than about 70 percent of 48-ply specimens were consumed. When the PbO coated side of specimens faced away from the flame, there was an optimum draft interval in which 6-, 10- and 20-ply specimens were 90 percent incinerated.

PbO and Pb_3O_4 were quite effective when dispersed in an Epon 828-Versamid 125 epoxy matrix prior to composite fabrication, both catalyzing specimen incineration to about 90 cercent or more even without a forced draft. But when dispersed in the Hercules 3501-6 resin system, the resultant specimens required an 8.3 mph draft or more to insure about 90 percent incineration.

Virgin "AS" Fabric from which the sizing had been removed by heating in a reducing flame for 30 seconds, separated into easily disseminated individual filaments when cut with a pair of scissors. Unburned fabric and composite specimens from which the resin had been burned away produced relatively few individual filaments.

SUMMARY

The incineration of graphite/epoxy specimens is undoubtedly aided by oxidation catalysts; however, the process was found to be heavily dependent on availability of the reactive gas (draft), conductive and convective heat losses, composite thickness, and catalyst distribution. In a theoretical scale-up of these laboratory studies to actual aircraft fires involving composites, it would be predicted that the availability of reactive gas would be decreased, reducing the incineration rate, while the heat losses would be decreased allowing longer periods of incineration. It is not known, however, whether this catalyzed process would tend to produce more or less free-floating filaments in the heavy turbulence of real fires. If more free filaments are produced, it is not known whether they will survive as such or incinerate in flight. It is also not known whether the fiber remains of a catalyzed fire are more easily dispersed by wind, impact or explosion than uncatalyzed remains. The effect of fibers or vapors from catalyzed composite fires on the health of nearby personnel has not been studied; this problem could be especially serious when lead catalysts are used.

FUTURE WORK

In view of the many unanswered questions regarding the actual performance of catalysts in real composite fires, it is proposed that realistic studies be performed on large composite specimens at the Dahlgren facilities. Concurrent with that effort, the National Aeronautics and Space Administration (NASA) Ames Burn-Bang test should be performed to develop full scale/laboratory correlation regarding fiber release measurements.

It is recommended, however, that a new approach be studied as well: the use of special pigments in composite coatings to cause agglomeration of the free filaments after resin burn-off preventing their release. Such an approach could be considered a "high temperature sizing" formed in-situ. The Jet Propulsion Laboratory at Pasadena has tested such additives as fiber coatings. Elimination of fiber release at the source could obviate the need to study the complex problems of dissemination, filament life, and toxicity.

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INTRODUCTION

Electrical interference caused by the release of graphite fibers from a burned graphite/epoxy composite is of concern to the Navy, a prime user of graphite fiber composites in the F-18 and AV-8B. Although a definitive risk analysis is in progress, it appears prudent to consider means of minimizing any potential problem in this regard. Currently, three general solutions are under investigation: 1) retention of the fibers by a charred matrix or an inorganic binder such as glass, 2) modification of the electrical characteristics of the fibers such that dispersed fibers pose only a minimal threat, and 3) incineration of the fiber during the burn of a composite structure. This report discusses the use of oxidation catalysts to promote the incineration of the graphite fiber during a graphite epoxy composite fire.

Graphite oxidation catalysts have been studied extensively in connection with combustion and gasification since the 1930's. In general, it has been found that graphite combustion is catalyzed to varying degrees by most metals (reference 1). Table I is a collection of the most effective catalysts as determined from the available literature. Because relative effectiveness is difficult to assess due to the variation in test methods, the listing is merely chronological and does not presume to rank catalysts.

A simple demonstration of the catalytic effect on graphite incineration can be made using an aqueous solution of calcium acetate. When a graphite tow is dipped in the solution and dried thoroughly, a film of the compound is retained on the fiber. If held over a lighted match for a few seconds, the tow begins to glow and continues to do so even when the match is removed. When the glow disappears, what remains is an ash which is easily crumbled. If the same effect can be produced in large scale, burning graphite laminates, catastrophic fiber pollution can be avoided.

Catalysts for graphite oxidations can be made available to the substrate by several methods:

1. Composite coating - Cured laminates of graphite/epoxy can be painted with a primer containing the catalyst of interest. Although contact of catalyst and fiber is minimal with this approach, the important load carrying elements of the composite are not compromised.

2. Matrix modification - The epoxy matrix in which graphite fibers are embedded can be modified before prepregging by the addition of solubilized, emulsified, suspended, or dispersed catalyst.

3. Fiber coating - Catalysts can be applied to the solid surface by deposition from an evaporating solution (such as the calcium acetate demonstration) or a condensing vapor.

4. Intercalation - Many inorganic species can form stoichiometric compounds by inserting between the basal planes of graphite crystals. The most intimate mixture of graphite and catalyst is achieved when the catalyst is an intercalant.

Although this report discusses only the first and second approaches, the following analysis of fiber incineration should be applicable to all four approaches.

TABLE I

CATALYSTS

	Reference
Tl	(1)
Pb	(1)(2)
PbO	(1)(2)
Pb_3O_4	(1)(2)
PbO_2	(1)(2)
Sb	(1)
Sb_2O_3	(1)
Bi	(1)
Bi_2O_3	(1)
Se	(1)
SeO_2	(1)
Mn	(2)
Cu	(2)(4)
$Cu(C_2H_3O_2)_2$	(3)
Ag	(4)(6)
Cr	(4)
MoO_3	(5)
V	(7)
V_2O_5	(7)
Li_2CO_3	(8)
Na_2CO_3	(8)

GRAPHITE OXIDATION

Any combustion process is both a chemical and a physical process dependent not only on the chemical reaction mechanism but also on heat and mass transport processes. A discussion of the factors which influence the rate of graphite combustion follows.

<u>Chemical reaction</u>. There are at least four formal reaction equations for the oxidation of graphite by gases present in air or petroleum fuel fires.

CARBON OXIDATION STATE	OVERALL REACTION
$0 \longrightarrow +2$	$C(graphite) + \frac{1}{2}O_2(g) = CO(g)$
	$C(graphite) + H_2O(g) = H_2(g) + CO(g)$
	$C(graphite) + CO_2(g) = 2CO(g)$
$0 \longrightarrow +4$	$C(graphite) + O_2(g) = CO_2(g)$

McKee and Chatterji (references 8 and 11) proposed mechanisms for graphite oxidation catalyzed by alkali metal carbonates and oxides in atmospheres of O_2, H_2O and CO_2. In the graphite - O_2 reaction, an oxidation - reduction cycle involving a higher oxide was proposed:

(Graphite - O_2)

$$M_2O + n/2\ O_2 = M_2O_{n+1}$$

$$M_2O_{n+1} + nC = M_2O + nCO$$

while, for oxidations in H_2O and CO_2, carbonate decomposition by graphite was proposed as the first step in each mechanism:

(Graphite - H_2O)

$$M_2CO_3 + 2C = 2M + 3CO$$

$$2M + 2H_2O = 2MOH + H_2$$

$$2MOH + CO = M_2CO_3 + H_2$$

(Graphite - CO_2)

$$M_2CO_3 + 2C = 2M + 3CO$$

$$2M + CO_2 = M_2O + CO$$

$$M_2O + CO_2 = M_2CO_3$$

As might be expected, catalyst behavior depends on the operative mechanism. For example, Turkdogan and Vinters (reference 4) showed by kinetic studies that while iron-impregnated graphite oxidizes in CO_2 about 10^6 times faster than pure graphite, silver, copper, and chromium have almost no effect on reaction rate. Yet, when similar studies are conducted in air, iron produces less than a 10 - fold rate increase while silver and copper produce as much as a 10^3 - fold increase and chromium almost a 10^2 - fold increase.

Turkdogan and Vinters also report several other important effects:

1. initial rate of weight loss during graphite - CO_2 oxidation depends strongly on the particle size of the iron catalyst;

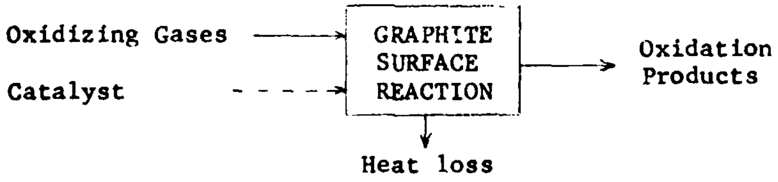
2. graphite - CO_2 oxidation catalyzed by iron, nickel and cobalt exhibit rates which decrease with time (the initial rate can be achieved by purging the reaction system with hydrogen and reintroducing CO_2); and

3. below a specific rate of weight loss, the logarithm of catalyzed and uncatalyzed rates depend linearly on the reciprocal absolute temperature.

To summarize, the chemical reaction by which graphite is oxidized depends on the following variables:

1. Nature of the oxidizing atmosphere
2. Concentration of the reactive gas
3. Temperature of the substrate
4. Catalyst
5. Catalyst concentration
6. Catalyst poisoning or inhibition

Heat and Mass Transfer. In order to sustain a combustion reaction, a substrate must be able to maintain its temperature, usually well above ambient. This ability depends on a number of dynamic factors which can be visualized as inputs or outputs of a reaction system.



Any one of the input or output factors may cause the reaction to shut down, limit the rate of oxidation by physical means or even prevent it from starting. Transfer rates between the system (a burning collection of filaments) and surroundings are affected by the following:

Oxidizing Gases (transfer to system)

Draft in the ambient atmosphere
Diffusion into spaces between filaments
Diffusion into pores

Diffusion through gaseous or ash boundary layer
Availability of reaction sites: reaction surface area
Adsorption of oxidizing species

Oxidation Products (transfer from system)

Desorption of gaseous product
Diffusion through boundary layer, from pores, into ambient atmosphere

Heat Loss

Radiant losses from the external specimen surfaces (to all surroundings)

- o External area
- o Specimen temperature

Convective losses (to surrounding gas)

- o External area
- o Specimen geometry
- o Ambient draft and direction of draft
- o Specimen temperature
- o Ambient temperature

Conductive losses (by physical contact with surroundings)

- o Contact area
- o Thermal conductivity of the material contacted
- o Temperature distribution in the material contacted

Catalyst (transfer to system)

Type
Particle size
Distribution by gas flow or surface wetting
Poisoning

In short, if the heat losses from the system (burning specimen) cannot be balanced by the heat generated from the oxidation process, the temperature of the system will decrease. Figure 1 shows the dependence of the overall reaction rate constant on temperature and the size of spherical carbon particles. If the oxidation process is diffusion-controlled (that is, if the slow step in the process is the diffusion of reactants to the surface or of products from the surface), a temperature decrease will affect the overall rate constant only a little at first. As the process becomes limited by chemical reaction (straight line), a decrease in temperature reduces oxidation rate considerably, thus reducing the heat generated. However, since much of the heat loss from a burning specimen occurs by radiation, heat loss may decrease faster than the heat generation decreases resulting in steady state combustion (due to the fourth-power temperature dependence of the Stephan-Boltzmann law). When heat loss from the system does not decrease rapidly, system temperature drops reducing reaction rate and further reducing heat generation. System temperature then rapidly drops to ambient.

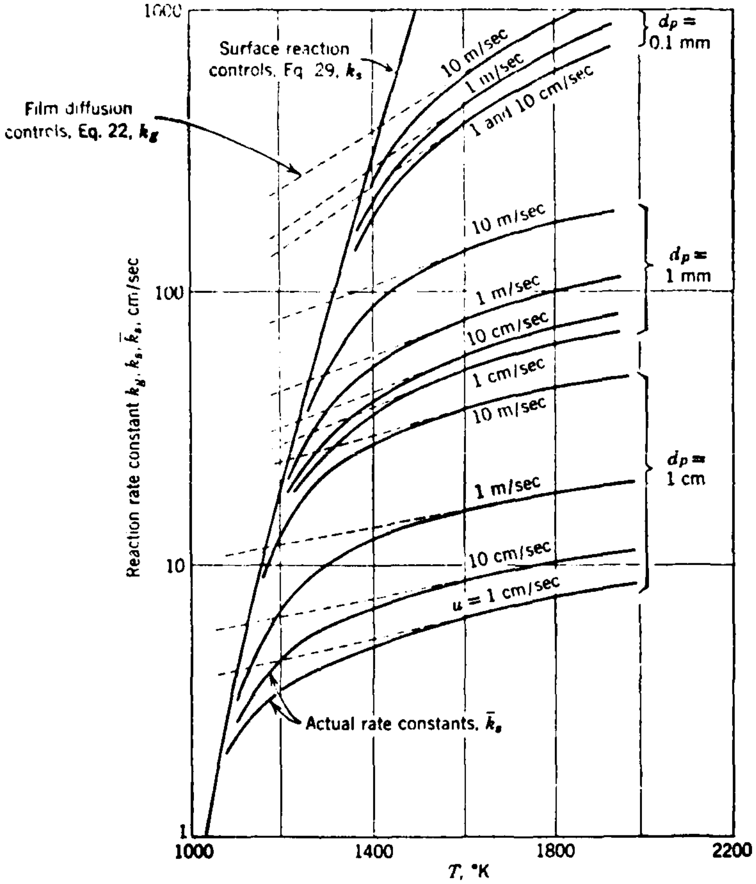


Figure 1 Rate of combustion of pure carbon particles, adapted from Yagi and Kunii (1955).

(Reference 9)

A graphite oxidation catalyst could function in several ways:

(1) A catalyst increases the gasification rate where the reaction is at least partially chemically-controlled by the chemical reaction.

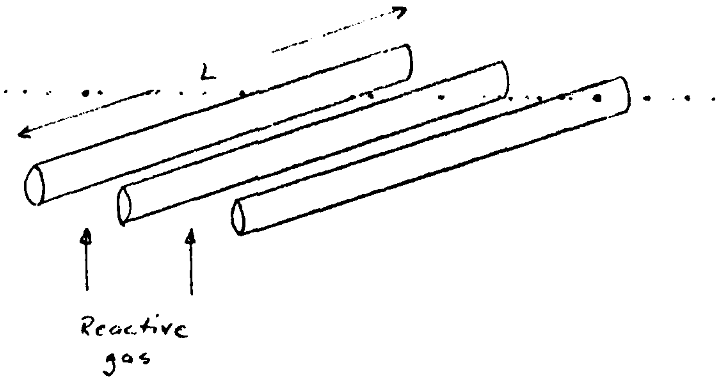
(2) Because of (1), the same amount of heat is generated at lower temperatures; since heat losses are quite temperature dependent, the reaction system may stabilize at these lower temperatures.

(3) The catalyst may accelerate the reaction of graphite with a secondary reactive gas (such as CO_2) which normally occurs at a higher temperature.

While (1) and (2) could not affect the rate of reaction in a diffusion-controlled region, (3) could if there were a sufficient concentration of CO_2.

KINETICS OF FIBER WEIGHT LOSS

When the resin has been burned from the fibers of a graphite composite, graphite oxidation can occur which may be modeled in terms of a single filament. These filaments burn collectively surrounded by a flowing gas which reacts at the fluid-solid interface depleting the graphite and forming a gaseous product which is carried off by the convecting gas stream.



Rate equations for such fluid-solid reactions are usually written as

$$(1)\quad R'' = -\frac{1}{S}\frac{dMs}{dt} = k''\,C^{solid} \qquad \text{(Reference 9)}$$

where R" is the surface reaction rate (moles/cm^2sec), S the instantaneous surface area, M_s the moles of solid, k" the surface reaction rate constant, and C^{solid} the steady-state concentration of reactive gas at the interface. Since the reactive gas is consumed and a product gas generated at the interface, mass transfer occurs which can be described in terms of the reactive gas as

$$(2)\quad \frac{1}{S}\frac{dM_{rg}}{dt} = k_g(C^{gas} - C^{solid})$$

where M_{rg} is the moles of reactive gas, k_g the mass transfer coefficient, and C^{gas} the concentration of reactive gas in the bulk of the gas stream.

Since the rate of consumption of carbon per unit area must be proportional to the net rate of transfer of reactive gas to the solid surface.

$$-\frac{1}{S}\frac{dM_s}{dt} = x\,\frac{1}{S}\frac{dM_{rg}}{dt}$$ where x is a proportionality constant

and therefore,

$$k'' C^{solid} = x\, k_g (C^{gas} - C^{solid})$$

solving for C^{solid} and substituting in equation (1) allows both diffusional and chemical kinetics to be described by a single equation

$$(3) \quad -\frac{1}{S}\frac{dM_s}{dt} = kC^{gas}$$

where

$$k = \frac{1}{\frac{1}{x\,k_g} + \frac{1}{k''}}$$ (an overall rate constant)

Substituting the expression for fiber surface area ($S = 2\pi rL$ where r is the instantaneous fiber radius) and the differential annular loss of solid ($dM_s = 2\pi rL\rho dr$ where ρ is the molar density of the solid) yields, after integration, an equation for the filament radius as a function of time

$$(4) \quad \frac{r}{R} = 1 - \frac{t}{\tau}$$

where $\tau = \frac{\rho R}{kC^{gas}}$, the time required to reduce the filament radius to zero.

Substitution of (4) into the differential annular loss equation yields, after integration, an expression for fractional weight loss as a function of time

$$(5) \quad W = 2\left(\frac{t}{\tau}\right) - \left(\frac{t}{\tau}\right)^2$$

where W is the fractional fiber weight loss. Solving for t gives an equation which plots as a straight line of slope ^{1} :

$$(6) \quad 1 - \sqrt{1 - W} = \frac{t}{\tau}$$

Using linear regression analysis, a "best fit" $\frac{1}{\tau}$ can be determined which is directly proportional to the rate of reaction per unit area as shown in equation (7)

$$(7)\quad R'' = -\frac{1}{S}\frac{dM_g}{dt} = kC^{gas} = \frac{\rho R}{\tau}$$

It should be pointed out that these equations are based on the initial assumption that the graphite fibers remaining after the resin has burned away are surrounded by a flowing gas of relatively uniform composition. Where significant non-homogeneities of gas concentration exist in the collection of filaments, the previous analysis will yield an average rate of reaction as if all the filaments were burning in an average gas concentration. To find the point at which the concentration gradient ceases to be important, specimens of smaller and smaller thicknesses should be tested. When the reaction rate ceases to be a function of specimen size (area), the oxidation is chemically-controlled by the reaction kinetics, in which case, all graphite filaments should decrease in radius uniformly.

COMBUSTION TESTS

When graphite/epoxy laminates are ignited and allowed to burn with no external heat source, the epoxy matrix is rapidly consumed. In a matter of seconds the flame dies out, and the residue cools rapidly. What remains are the graphite fibers and some pyrollyzed epoxy resin which is unable to escape in the short burn time. With an external heat source, such as a fuel fire, the epoxy residue is quickly burned away enabling the graphite fibers to be dispersed by mechanical agitation. Incineration of the graphite fiber can be promoted by incorporating catalysts into the composite system. In order to measure the effectiveness of these catalysts, two burn tests were designed, one in which the specimens were continuously exposed to an oxidizing flame (continuous combustion) and a second in which specimens were heated over a reducing flame then exposed to various drafts of air (self-sustaining combustion).

Continuous Combustion.

A simple burn test was designed using a Meker blast burner, a cylindrical glass column to eliminate horizontal drafts, a nichrome wire specimen support and chromel/alumel thermocouples (see Figure 2). Dimensional data and operating parameters are listed in Table II. When it was discovered that composite specimens tended to buckle away from the wire support, two strands of nichrome wire (0.4 mm diameter) were used to wrap the specimen. This not only maintained the shape and position of the specimen but also prevented the loss of small amounts of fibers to convection currents. Specimens, 1.0 in. (2.5 cm) by 1.0 in. (2.5 cm), were positioned horizontally at a point just above the tip of the flame where thermocouple readings indicated an 870° C flame temperature. After flame exposure for the required time interval, the reaction was quenched by sandwiching the burned specimen between two aluminum weighing dishes, cooling it rapidly to ambient temperature. By weighing specimens before and after exposure to the flame, the rate of fiber consumption was determined.

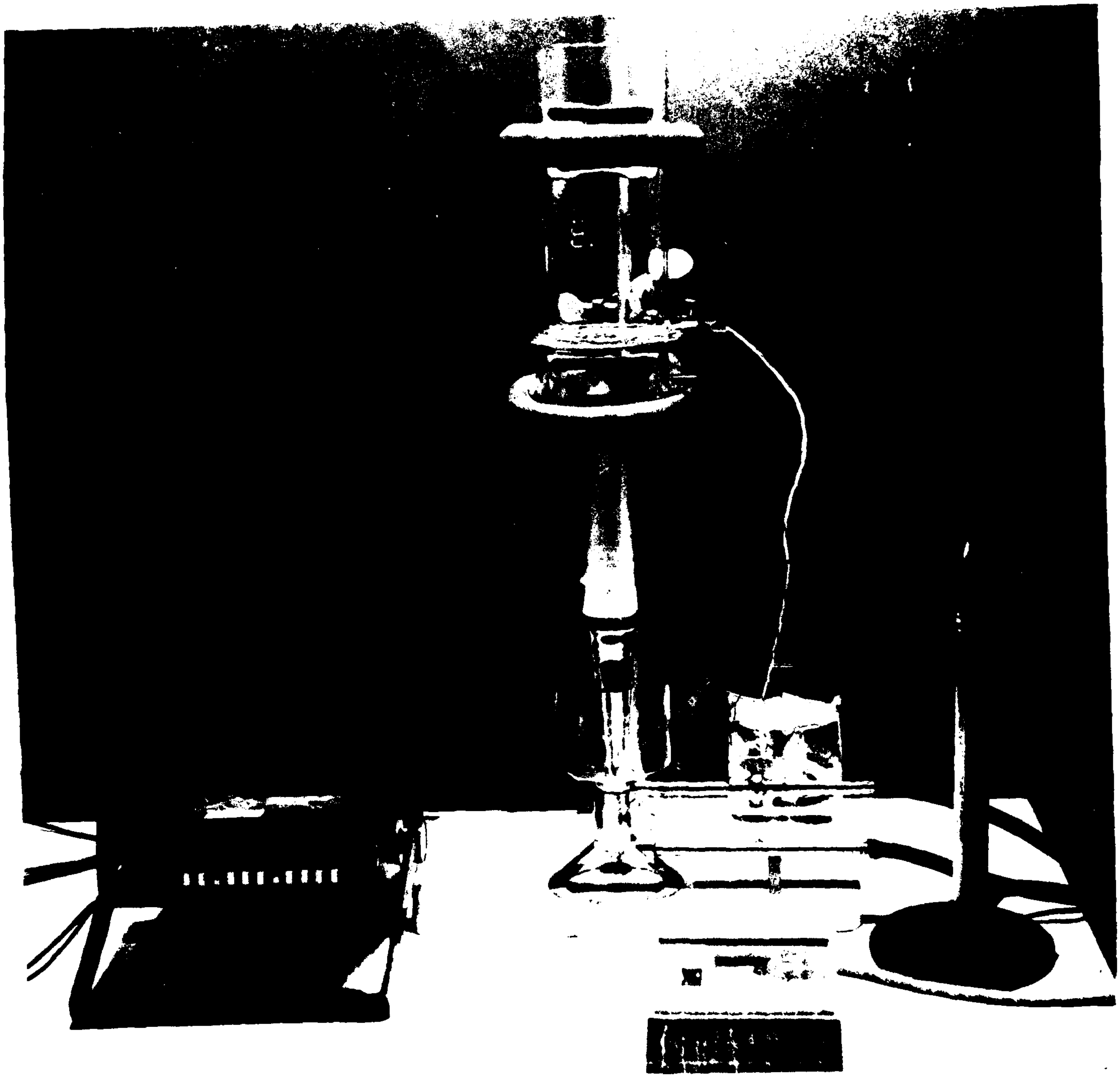


Figure 2. Continuous burn apparatus

TABLE II

CONTINUOUS BURN TEST PARAMETERS

Dimensions:	Fisher Blast Burner (1977 Fisher Cat. No. 3-910-5)	HT 20.5 cm
	Glass Tubing	ID 8.5 cm
	Lower section	HT 30 cm
	Upper section	Ht 19 cm
	Separation between sections	3 mm
	Distance from base of burner	10.7 mm
	Thermocouple Wire	
	Chromel/Alumel	Diam. 0.8 mm
Burner Operation:		
	Air Supply	5.6 liters/min
	Propane	1.0 liters/min
	Adjusting Barrel	Holes full open
	Temperature at Tubing Separation	870°C
Specimen Support:		
	Nichrome wire mesh	
	Opening	0.6 cm square
	Wire diam	0.39 mm

Self-Sustaining Combustion.

A second type of burn test was designed using a Fisher blast burner (no compressed air supply). With the same nichrome wire support as previously used, specimens, 1.0 in. (2.5 cm) by 1.0 in. (2.5 cm), mounted horizontally, were heated 0.25 in. (0.64 cm) above the burner top where thermocouple readings averaged 1030° C. After a prescribed ignition period the specimen and support were quickly transferred to a ring stand with support ring beneath which a glass tube 0.16 in. (9.4 mm) in inside diameter was clamped 1.75 in. (4.5 cm) from the specimen to provide an upward stream of air.

The following air flow rates (expressed as air velocity at the tip of the glass tube) were used in this combustion test:

Flowmeter Reading	Velocity	
	m/sec	mph
0	0	0
2	1.9	4.2
4	3.7	8.3
6	5.6	12.5
8	7.4	16.6

There are two basic differences between these combustion tests. First, composite specimens burned in the continuous test were eventually completely consumed by the oxidizing flame while specimens exposed to the reducing flame of the Fisher burner for as much as 20 minutes lost almost no graphite mass, although the resin was completely burned away. Second the rate at which specimens were consumed in the continuous combustion test varied with thickness and catalyst but always burned completely if exposed to the flame long enough. Composite specimens ignited in the self-sustaining combustion test either continued to burn to 90-95 percent weight loss or stopped burning well short of this level, depending on the catalyst present.

APPROACH #1

COMPOSITE PRIMER COATING

To test the feasibility of using an exterior finish containing a catalyst on cured graphite/epoxy, fifteen experimental primers were formulated with various catalysts. As an example, Formulation #1 is listed in Table III. Using the binder for the Navy aircraft primer (MIL-P-23377), catalyst has been substituted for strontium chromate and titanium dioxide on a volume basis in an effort to preserve coating mechanical properties.

Composite specimens (one-inch squares) were spray-coated on one side with 1 to 2 mils (25 to 50 μm) of an experimental primer and baked for 4 hours at 180° F (82° C).

Since coated specimens produce a residue when burned which contains not only fibers but also coating pigment, the actual fractional fiber loss is calculated from equation (8) for the continuous combustion test.

$$(8)\quad W = 1 - \frac{w_r - w_c X_{pig}}{x_f w_o}$$

where w_r is the residue, w_c the weight of the coating, X_{pig} the pigment weight fraction in the dried film, x_f the fiber weight fraction of the uncoated specimen, and w_o the initial weight of the specimen.

TABLE III

FORMULATION #1

	Weight
Part A	
Epon 1001-T75	19.1
$CaCO_3$ (CATALYST)	10.1
Magnesium Silicate	5.8
Silica	3.0
Methyl isobutyl ketone	13.2
Toluene	6.8
Grind twice for 3 minutes with ultrasonic mixer.	
Part B	
Versamid 115	7.9
Toluene	13.5
Isopropanol	8.6
n - Butanol	8.6
Xylene	3.4
	100

Results.

1. Continuous combustion.

1.1 Uncoated specimens. In order to determine the base rate of reaction, the first series of weight loss measurements were made with uncoated unidirectional Hercules AS/3501-6 specimens. Total weight loss for 6 ply laminate as a function of flame exposure time is shown in Figure 3. An inflection point occurs when all of the epoxy matrix has been burned away (at about 25 percent). At that point, the specimen temperature rises to the graphite ignition temperature where oxidation of the fiber begins. In Figure 4, fiber weight loss data as calculated from total weight loss using equation (8) is replotted as $1 - \sqrt{1-W}$ vs. time as suggested by equation (6). Only data points for times greater than two minutes were used since fiber loss is almost negligible prior to this time.

Linear regression was used to determine a best fit straight line, the slope of which is $1/\tau = 0.1304$ (see equation (6)). Since the base rate of reaction, using $R = 4\,\mu m$ and $\rho = 0.15$ g moles/cm^3, is calculated to be 1.3×10^{-7} g-moles C/cm^2 sec.

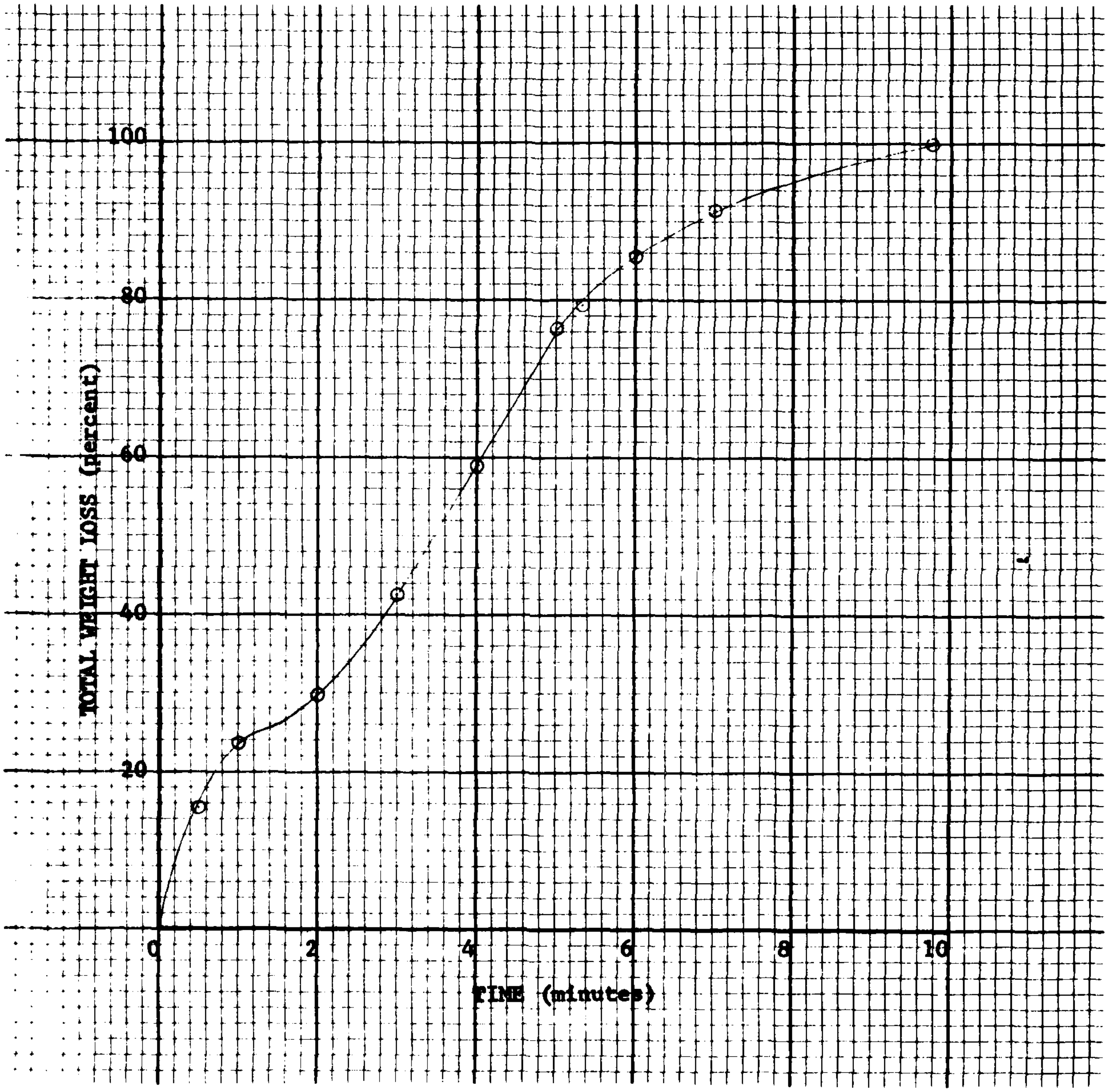


Figure 3. Total weight loss as a function of time for uncoated specimens

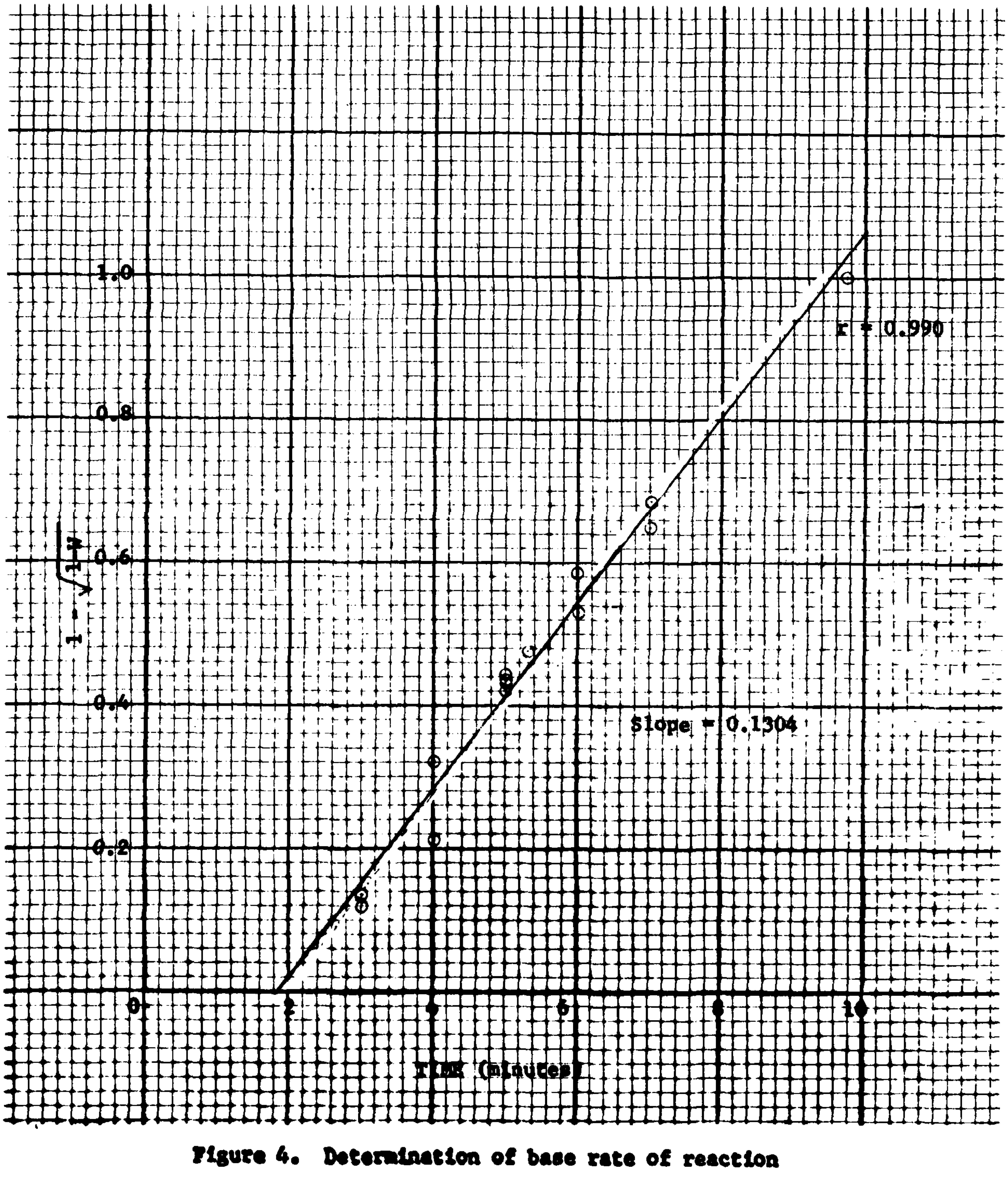


Figure 4. Determination of base rate of reaction

Other experiments with 3-, 10-, 20-, and 48-ply specimens (also one-inch square) showed that the apparent reaction rate ($1/\tau$) is inversely proportional to the number of prepreg plies in the specimen as shown in Figure 5. Further investigation of the data indicates that the rate of fiber weight loss is fairly constant during the first half of the burning process. In addition, this initial rate of weight loss is nearly the same for 48- and 20-ply specimens, slightly less for 10- and 6-ply specimens and much less for 3-ply specimens. Figure 5 shows that there appears to be a critical specimen thickness (at about 6 plies) where apparent reaction rate increases rapidly while the rate of fiber weight loss begins to drop.

1.2 Coated specimens. Six-ply unidirectional Hercules AS/3501-6 specimens coated with the fifteen catalytic primers listed in Table IV were measured for weight loss in the continuous combustion test in the same manner as uncoated specimens. It was decided that for maximum effectiveness, all coated specimens would first be tested with the coated surface down (i.e., toward the flame). Table IV shows clearly that, of those tested, only three catalysts significantly affected the rate of fiber consumption; lead monoxide, antimony trioxide, and bismuth trioxide. Almost all of the other catalysts inhibited the graphite oxidation, probably by partially blocking the flow of reactive gas into the laminate remains.

Three other specimen variables were considered with and without lead monoxide primer coating: bias, thickness and coating orientation. Results, summarized in Table V, show that specimen thickness affects the apparent reaction rate ($1/\tau$) to a greater degree when the PbO primer is present, but that it does not matter whether the coated side faces toward or faces away from the flame or whether the composite is unidirectional or crossplied. It has been observed that when a specimen is burned coated side to the flame, showers of sparks are generated by the PbO coating during resin burn-off.

2. Self-sustaining combustion.

2.1 Uncoated specimens. Initial work with a self-sustaining combustion test made use of the Meker burner and glass cylinders as shown in Figure 1. After igniting specimens for 2 minutes over the 870° C oxidizing flame, the fuel and air supplies to the burner were cut off. Specimens continued to burn (glow) regardless of the type or existence of a catalyst present in a coating (with the exception of $CaCO_3$) as shown below:

Self-Sustaining Combustion

(Warm tube)

	Fiber Weight Loss (%)
Uncoated	66
$CaCO_3$	8
PbO	62
CuO	86
$PbO/Ca(Ac)_2$	81

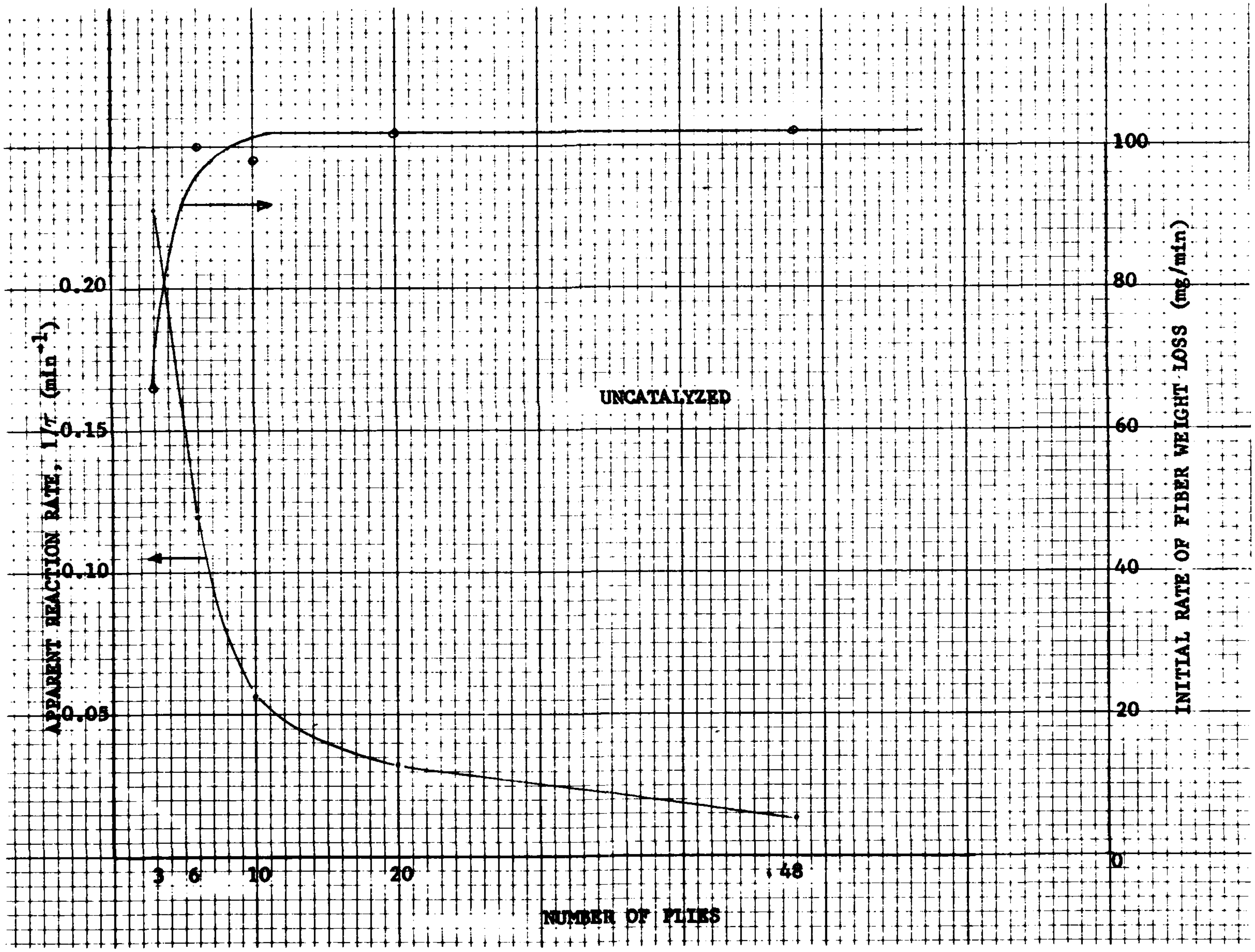


Figure 5. Apparent reaction rate ($1/\tau$) and initial rate of fiber weight loss as functions of the number of specimen plies

TABLE IV

APPARENT REACTION RATE, $1/\tau$ ($\min^{-1}$)

*	SrO	0.095
*	$Ca(C_2H_3O_2)_2\ H_2O$	0.095
	V_2O_5	0.103
*	MIL P 23377	0.104
*	$CaCO_3$	0.107
	Mn_2O_3	0.108
	Li_2CO_3	0.110
	SeO_2	0.111
	MoO_3	0.111
*	CuO	0.114
	Na_2CO_3	0.115
	Uncoated	0.120, 0.130
	AgO	0.127
	PbO	0.151
	Sb_2O_3	0.152
*	PbO	0.158
	Bi_2O_3	0.168

*Primer prepared with silaceous extenders (Mg silicate and silica)

TABLE V

EFFECT OF BIAS, THICKNESS, AND COATING ORIENTATION ON APPARENT REACTION RATE, $1/\tau$ (min^{-1})

	Bare	PbO Primer
BIAS - 6 ply		
Unidirectional	0.120	0.151
(+45,-45,+45,-45,+45,-45)	0.115	0.156
THICKNESS - unidirectional		
3 ply	0.228	0.374
6 ply	0.120	0.151
COATING ORIENTATION - 6 ply		
Toward flame (down)	-	0.151
Away from flame (up)	-	0.148

Subsequent studies were performed as described under COMBUSTION TESTS/self-sustaining combustion. Without catalysts, 6-, 10- and 20-ply (unidirectional) and 48-ply (crossplied) one-inch square specimens exhibited almost no oxidation of the graphite fiber after the specimens were removed from the reducing flame (see Table VI).

TABLE VI

SELF-SUSTAINING COMBUSTION OF BARE SPECIMENS (Percent Loss of Specimen)

		Draft Velocity (mph)				
Laminate	Resin Content (weight %)	16.6	12.5	8.3	4.2	0
6-ply (1)	25.3	31.0	29.5	27.8	23.9	23.4
10-ply (1)	25.7	22.2	39.6	41.0	24.4	22.6
20-ply (2)	25.3	27.2	17.2	30.9	22.4	21.9
48-ply (3)	25.3	19.8	20.2	19.3	19.9	19.5

Ignition Times: (1) = 60 seconds
(2) = 90 seconds
(3) = 300 seconds

2.2 Coated specimens. As shown in Table VII, several primers exhibited a remarkable ability to produce sustained combustion in 6-ply laminates, notably lead monoxide, lead tetroxide, bismuth trioxide and silver oxide. Of these catalysts, none consumed more than 96 percent of the coated specimen.

Residues were composed of coating pigments, partially burned fibers and a gray ash. It should be noted that the first six primers were prepared with magnesium silicate and silica extenders, as used in the control formulation for MIL-P-23377.

The effect of thickness and coating orientation can be seen in Table VIII. Each point in the table represents one experiment with one specimen, and therefore, some scatter in the data is expected. Nevertheless, it is apparent that orientation of the coating, either toward or away from the flame, significantly affects the quantity of fiber incinerated where thick laminates (20- or 48-ply) are concerned.

TABLE VII

SELF-SUSTAINING COMBUSTION OF COATED SPECIMENS
(6 ply - 60 sec ignition time)

Bar indicates greater than 90 percent loss of sample at the draft velocity shown; losses less than 40 percent not shown.

	Draft Velocity (mph)				
Coating catalyst	16.6	12.5	8.3	4.2	0
* $CaCO_3$					
* PbO					
* $Ca(C_2H_3O_2)_2 \cdot H_2O$					
* CuO					
* SrO					
* $PbO/Ca(C_2H_3O_2)_2 \cdot H_2O$	▬	▬	▬	67.9	41.0
Sb_2O_3					
Bi_2O_3	▬	▬	▬	57.0	50.2
SeO_2					
AgO	▬	▬	▬	54.1	
MoO_3					
V_2O_5					
Li_2CO_3					
Na_2CO_3					
PbO	▬	▬	▬	75.8	
Mn_2O_3					
Pb_3O_4	▬	▬	▬	47.2	

*Prepared with magnesium silicate and silica extenders.

TABLE VIII

EFFECT OF THICKNESS AND COATING ORIENTATION ON SELF-SUSTAINING COMBUSTION (PbO coating)

Bar indicates greater than 90 percent loss of sample at the draft velocity shown; losses less than 40 percent not shown.

		Draft Velocity (mph)				
Coating Orientation	No. of Plies	16.6	12.5	8.3	4.2	0
DOWN (toward flame)	6 (1)	█	█	█	75.8	
	10 (1)	█	█	█	█	48.4
	20 (2)	█	█	█	█	
	48 (3)	67.6	71.5	67.7		
UP (away from flame)	6 (1)	█	█	█	59.6	52.4
	10 (1)	69.9	█	█	58.6	50.4
	20 (2)	56.8	65.7	█	█	
	48 (3)					

Ignition times:
(1) = 60 seconds
(2) = 90 seconds
(3) = 300 seconds

Thin laminates tend to glow uniformly when a good catalyst (i.e., PbO, Pb_3O_4, Bi_2O_3, AgO or $PbO/Ca(Ac)_2$) has been applied as a coating. When the catalytic coating faces down (i.e., toward the airstream), thick laminates (20- or 48-ply) burn from the bottom up leaving a small ash residue. However, when the coated side faces away from the air stream, the glow due to combustion starts at the edges parallel to the graphite fiber and moves toward the center of the specimen.

There is an optimum draft range in which the specimen weight is reduced by more than 90 percent. The range for PbO-coated 10-ply specimens corresponds well with that found for Pb_3O_4-coated 10-ply specimens (data not shown).

Even with the lead catalysts and under the optimum draft, both thin and thick specimens stop short of complete ashing with approximately 5-10% of fiber weight remaining. It was also noted that glowing regions could easily be extinguished by touching the area with a metal object.

APPROACH #2

RESIN MODIFICATION

Previous work at the Naval Research Laboratory (NRL) showed that lead catalysts incorporated into uncured epoxy resin (Epon 828 cured with Versamid 125) caused finished composites (prepared with Hercules AS Fabric) to lose about 97 percent of its weight after a 60 second ignition in a 700° C furnace followed by exposure to ambient air. Evaluations of these NRL composites using the continuous combustion test and the early version of the self-sustaining combustion test (Meker burner instead of Fisher burner) are summarized in Table IX.

The success of this approach was tested at the Naval Air Development Center (NAVAIRDEVCEN) by preparing and evaluating Epon 828/Versamid 125 and Hercules 3501-6 composites using AS Fabric and catalysts which proved effective in composite primer studies. Procedures for resin modification and composite preparation are contained in Appendices A and B. One-inch square specimens were evaluated using the self-sustaining combustion test.

TABLE IX

EVALUATION OF NRL SPECIMENS
(4 Fabric Plies)

Fabric	Catalyst	Apparent Reaction Rate, $1/\tau\,(\text{min}^{-1})$	(Warm tube test) Weight Loss at Draft Velocity=0 (percent)
AS	-	0.078	34.5
AS	Pb_3O_4	0.089	97.0
T300	-	0.105	48.0
T300	Bi	0.112	88.9
T300	CuO	0.120	86.4
T300	$CaCO_3$	0.104	70.0
T300	"Pb-half ester"	0.121	97.6
T300	Pb_3O_4	0.103	96.6

Results.

As shown in Table X, the best catalyst was Pb_3O_4 followed closely by PbO. Bi_2O_3 was much less effective while CuO and Sb_2O_3 did not cause any significant glow or combustion promotion. In addition, the influence of the resin used was significant; while the Hercules 3501-6 resin appeared to inhibit the catalysis of the lead additives when compared to the Epon 828/Versamid 125 resin, its use eliminated the effect of the Bi_2O_3 catalyst.

The importance of ignition time in the self-sustaining combustion test was studied by exposing specimens to the Meker burner oxidizing flame for progressively longer ignition times. After the ignition period the specimen and support were transferred to the ring stand (no draft was applied). The weight percent remaining is plotted as a function of the time in the flame for Hercules AS Fabric/3501-6 in Figure 6 and for AS Fabric/Epon 828 - Versamid 125 in Figure 7. As in the continuous combustion test, specimen weight decreased slowly with increasing ignition time, although more rapidly than the uncatalyzed specimen. Only PbO and Pb_3O_4 exhibited any self-sustaining combustion promotion as evidenced by their continued glow after the relatively short ignition time of 60 seconds. Longer ignition times did <u>not</u> cause enough loss of catalyst to prevent the specimen from burning to near completion (see Figure 7 at 300 seconds).

TABLE X

SELF-SUSTAINING COMBUSTION OF RESIN-MODIFIED COMPOSITES*
(AS Fabric, 4 Plies)

Bar indicates greater than 90 percent loss of sample at the draft velocity shown; losses less than 40 percent not shown.

		Draft Velocity (mph)				
Resin	Combustion Catalyst	16.6	12.5	8.3	4.2	0
Hercules 3501-6	None					
	PbO	█	89.2	85.9		
	Bi_2O_3					
	Sb_2O_3					
	CuO					
	Pb_3O_4	█	█	█		
Epon 828/ Versamid 125	None					
	PbO	█	█	█	█	89.5
	Bi_2O_3	█	█	56.4**	60.0**	55.2**
	Sb_2O_3					
	Pb_3O_4	█	█	89.9	█	█

* 60 sec ignition time

** These high weight losses are due to an error in fabrication which resulted in excessive resin content.

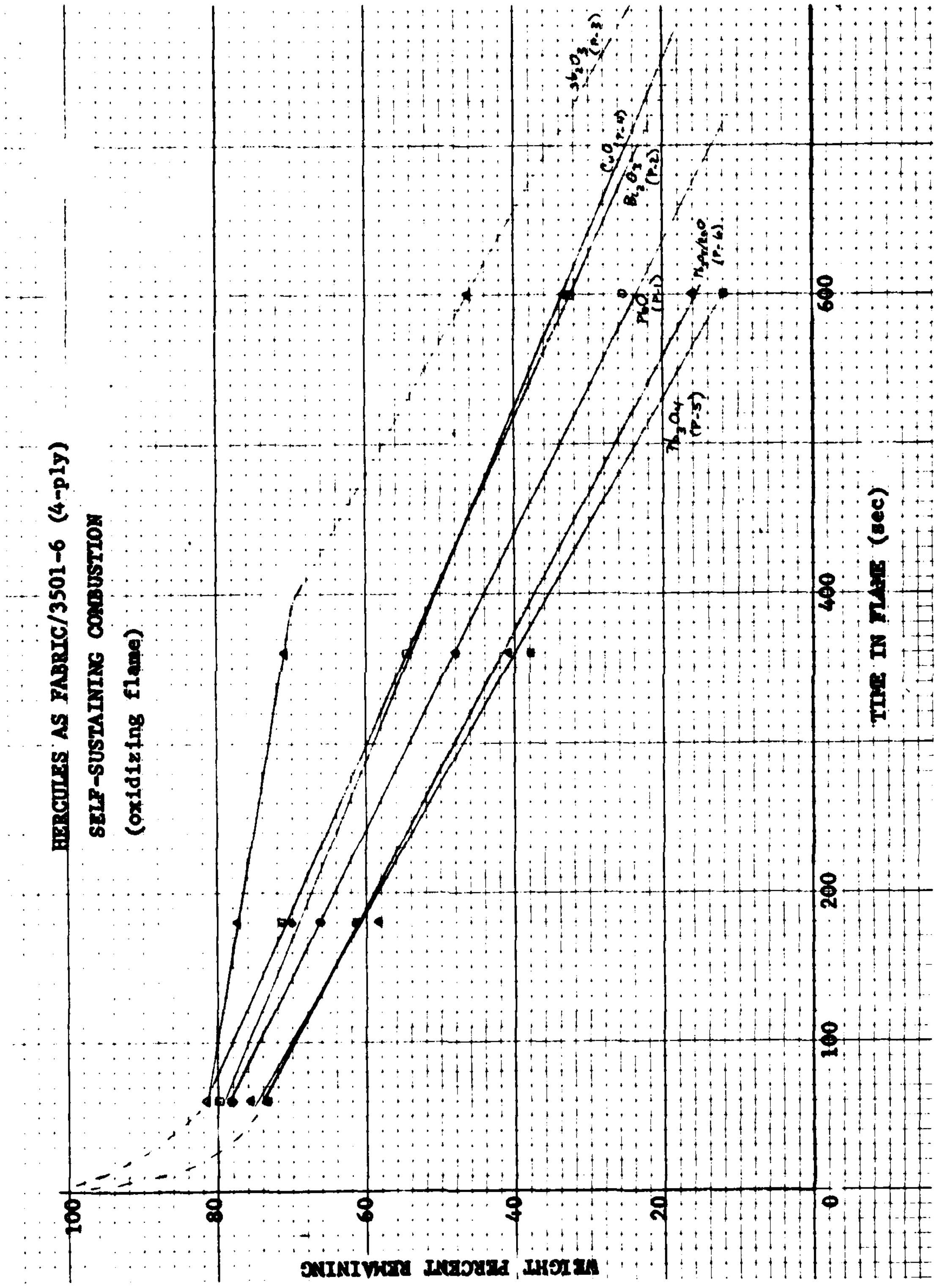


Figure 6. Hercules AS Fabric/3501-6 (4-ply) - Effect of ignition time in oxidizing flame on self-sustaining combustion

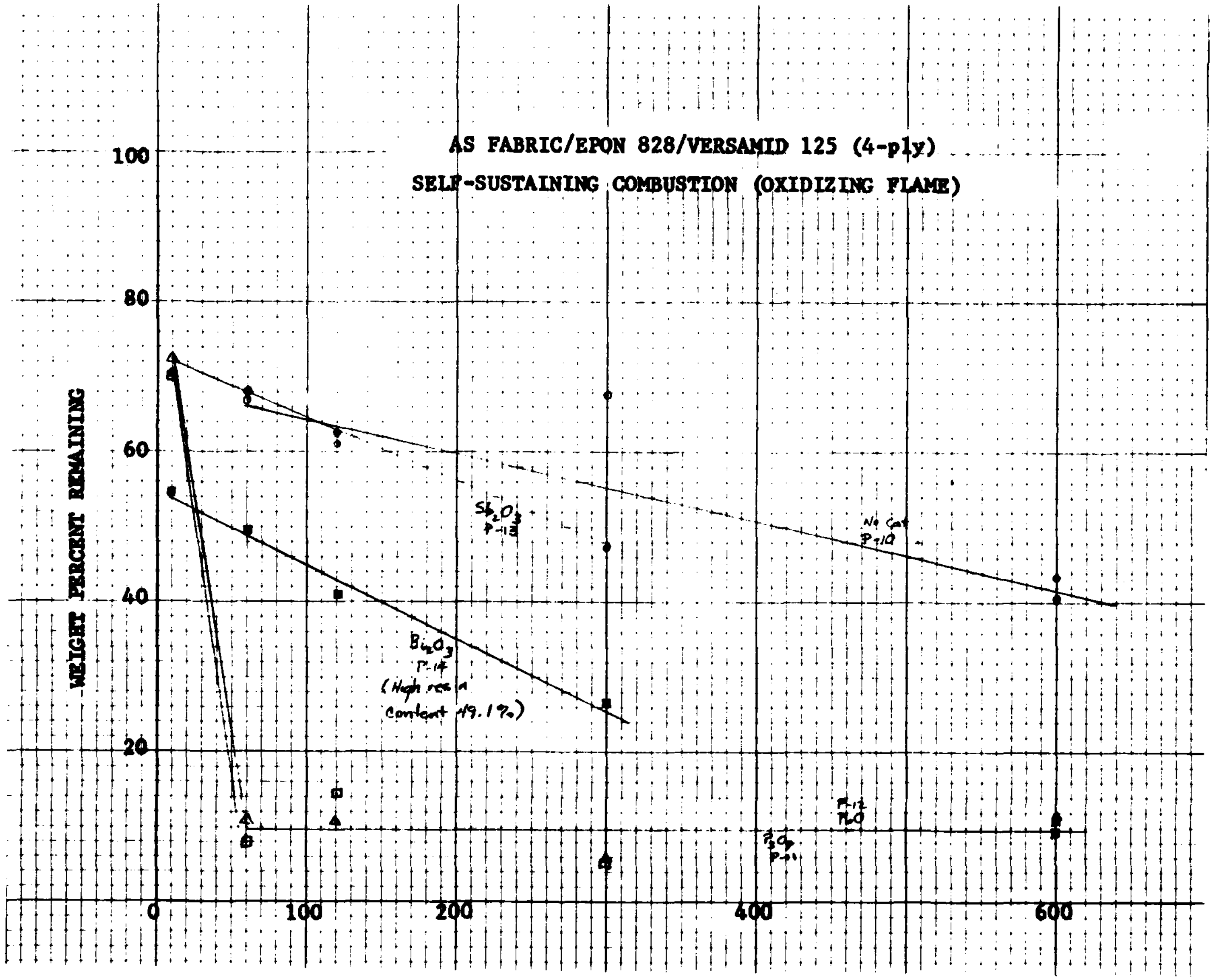


Figure 7. AS Fabric/Epon 828-Versamid 125 (4 ply) - Effect of ignition time in oxidizing flame on self-sustaining combustion.

ADDITIONAL OBSERVATIONS

Fiber Release.

Cutting fiber and composite specimens for the scanning electron microscope (SEM) allowed a qualitative study of some striking fiber release characteristics. When a one-inch square of virgin Hercules AS Fabric was cut in half with a pair of scissors, some of the tows tended to distort and pull out of the weave (see Figure 8), but only a very few individual filaments or groups of filaments were freed from the fabric. In contrast, a one-inch square of AS Fabric, which had been exposed to a 1000° C reducing flame for 30 seconds and subsequently cut in half with the same scissors, exhibited extensive separation into single filaments about 0.5 inches (10 mm) to 1.0 inches (25 mm) in length (see Figure 9).

For comparison, two one-inch squares of Hercules AS Fabric/3501-6 graphite/epoxy composite (4 ply) were burned to remove the resin and then cut in half with the same scissors. One specimen was burned 10 minutes in an oxidizing flame at about 870° C prior to cutting (see Figure 10), while the other was burned for 10 minutes in a reducing flame at about 1000° C prior to cutting (see Figure 11). While the specimen exposed to the oxidizing flame produced some individual filaments (about 0.1 to 0.5 inches (2 to 10 mm) in length) when cut and the specimen exposed to the reducing flame produced some individual filaments (about 0.1 inches (2 mm) and less in length) when cut, the number and length of specimens generated by the burned and cut virgin AS Fabric was much greater.

Scanning Electron Microscopy (SEM).

In order to confirm that burning graphite filaments actually decrease rather uniformly in diameter while maintaining a cylindrical shape, several SEM photomicrographs were made. Following the fiber release observations made above, virgin "AS" Fabric (shown in Figure 12) was compared with "AS" Fabric from which "sizing" had been removed by a 30 second exposure in the 1000° C reducing flame (shown in Figure 13). It was apparent that the filaments in "sized" fabric were held together in bundles while those where "sizing" had been burned away were loose filaments with little or no attraction for each other. That some types of organic filament coating exists was confirmed by thermogravimetric analysis (see Figures 14 and 15). Approximately 5 percent by weight of finished fabric is "sizing," burning off in pure oxygen at 290° C and in pure carbon dioxide in two discrete reactions loosing 3 percent up to 300° C and an additional 2 percent up to 430° C.

Figure 16 shows some of the remains from the bottom 2 plies of a Hercules AS Fabric/3501-6 composite (resin modified with Pb_3O_4) after a self-sustaining combustion test with an air flow of 8.3 mph. To the naked eye these plies appeared to have been reduced to an ash. Yet they were peeled in tact as one layer from the top two layers. In this figure (1000 X magnification) and Figure 17 (5000 X magnification, different location on the specimen) it appears that burned filaments have coalesced or been bound together by some type

Figure 8. Virgin "AS" fabric cut with scissors

Figure 9. "AS" fabric exposed to reducing flame 30 sec. cut with scissors

Figure 10. Hercules AS/3501-6 exposed to oxidizing flame 10 min. cut with scissors

Figure 11. Hercules AS/3501-6 exposed to reducing flame 10 min. cut with scissors

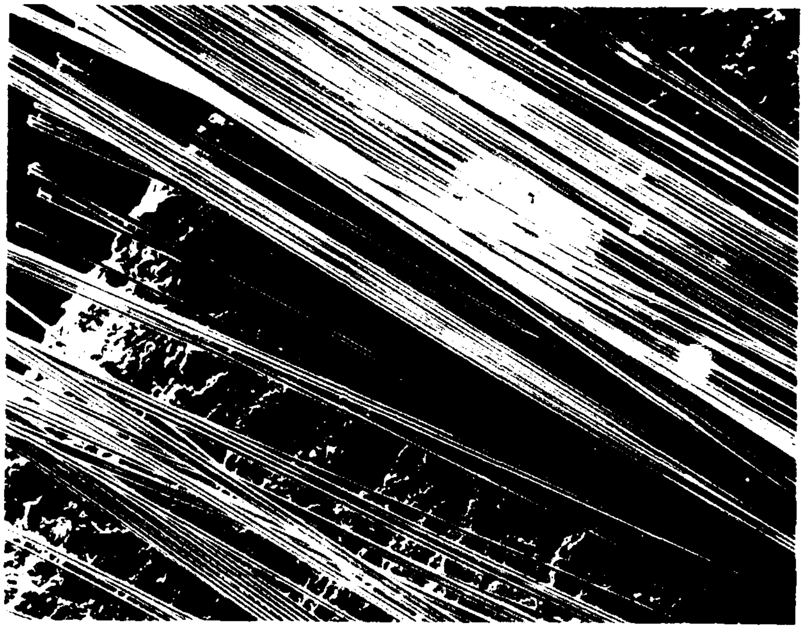


Figure 12. Tow of virgin "AS" Fabric (SEM photo micrograph, 100 X magnification

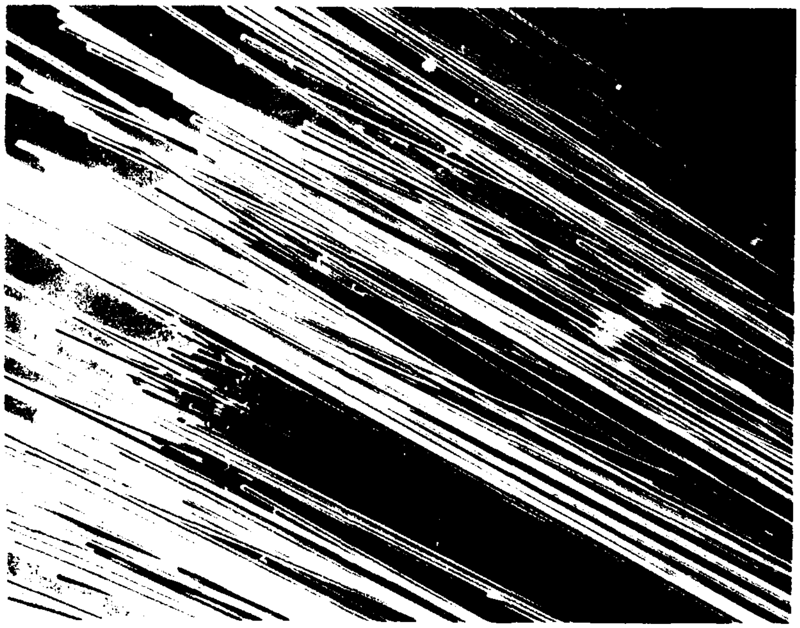
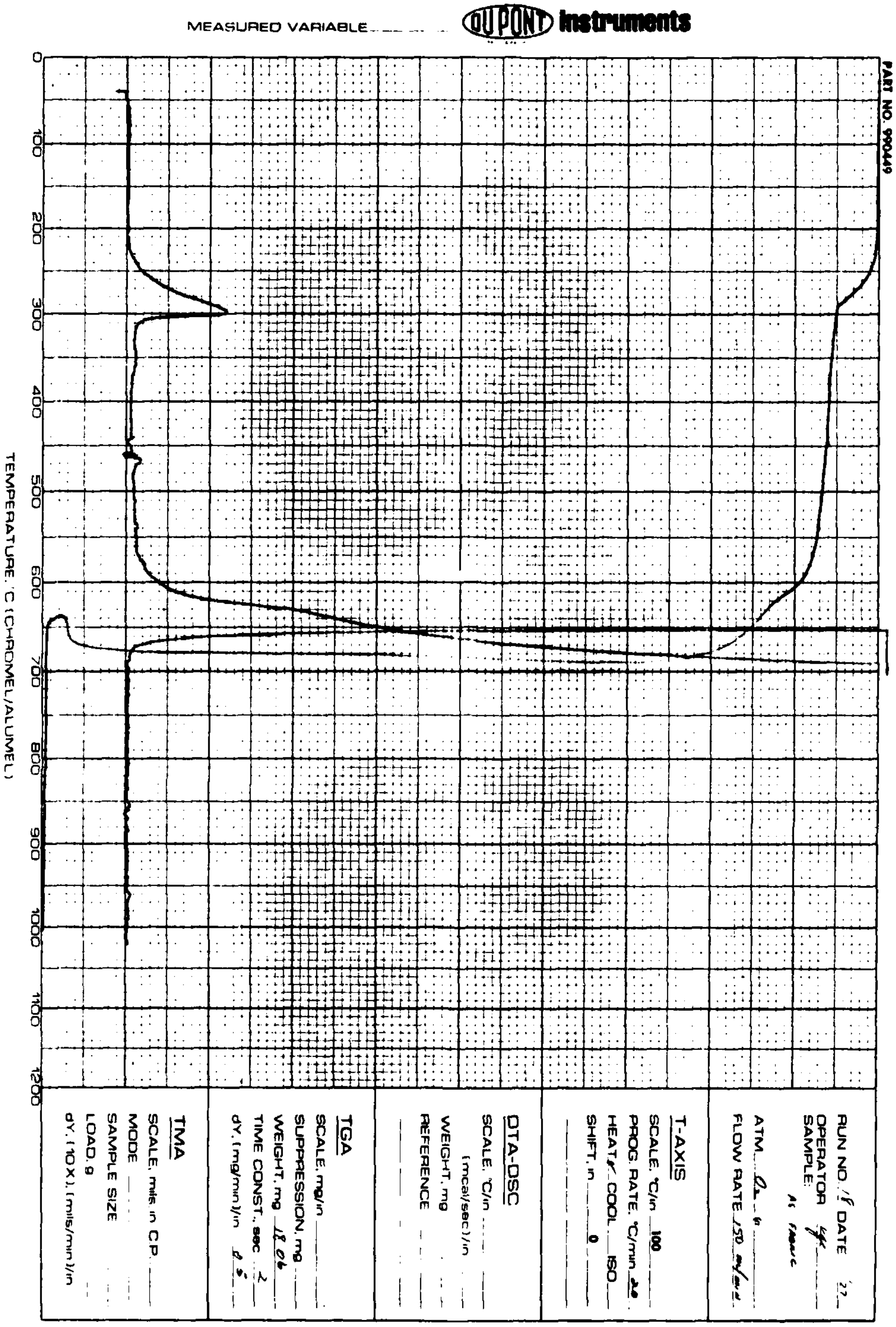


Figure 13. Tow of "AS" Fabric exposed to reducing flame 30 seconds (SEM photomicrograph, 100 X magnification)



RUN NO. 18 DATE 27
OPERATOR
SAMPLE: AS fabric
ATM. O2
FLOW RATE 150 ml/min

T-AXIS
SCALE, °C/in 100
PROG. RATE, °C/min 20
HEAT/COOL ISO
SHIFT, in 0

DTA-DSC
SCALE, °C/in
(mcal/sec)/in
WEIGHT, mg
REFERENCE

TGA
SCALE, mg/in
SUPPRESSION, mg
WEIGHT, mg 18.06
TIME CONST., sec 2
dY, (mg/min)/in 0.5

TMA
SCALE, mils in C.P.
MODE
SAMPLE SIZE
LOAD, g
dY, (10X), (mils/min)/in

Figure 14. Thermogravimetric analysis of virgin "AS" Fabric (Atmosphere = pure O_2)

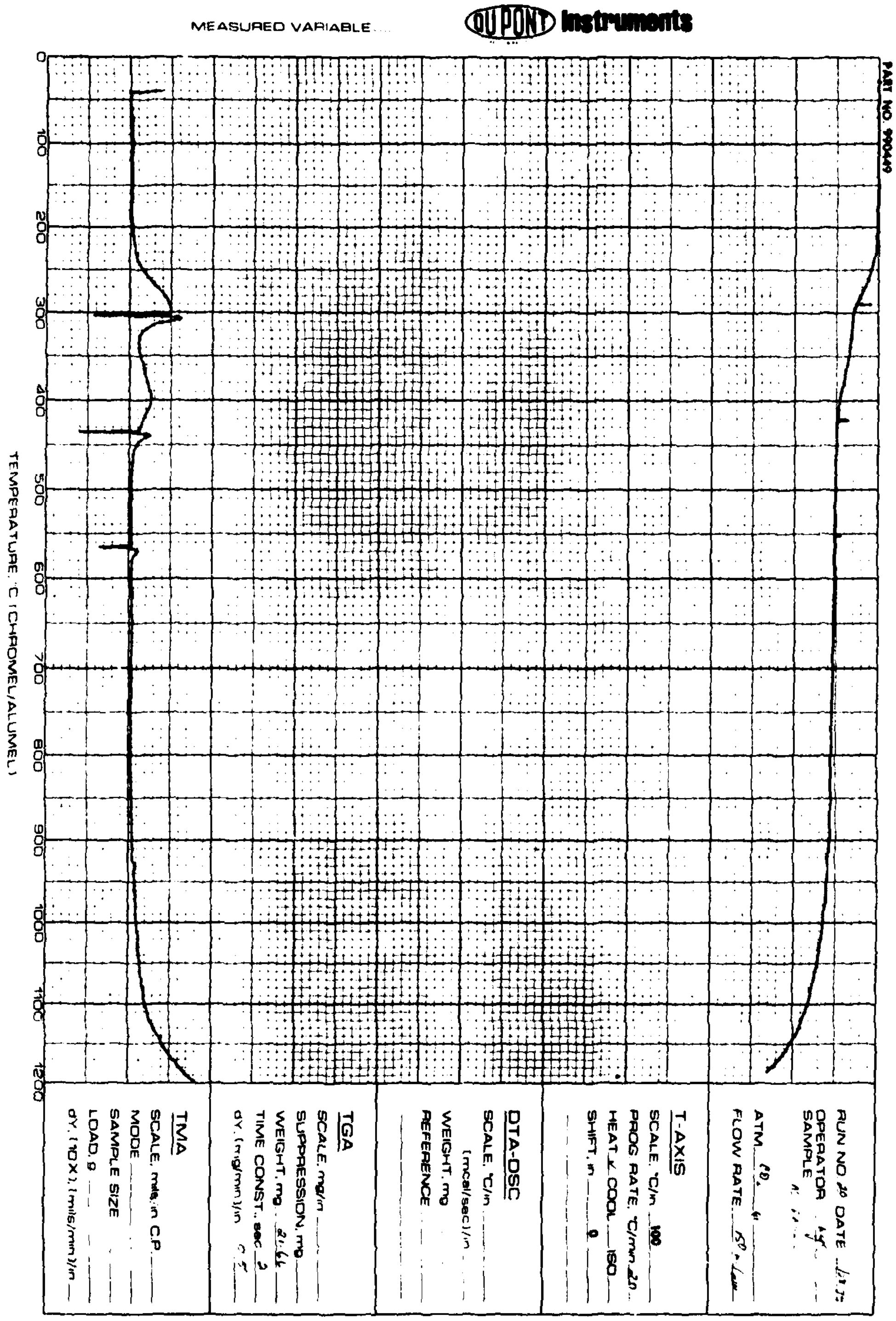


Figure 15. Thermogravimetric analysis of virgin "AS" Fabric (Atmosphere - pure CO_2)

Figure 16. Section of fiber remains from bottom 2 plies of Hercules AS/3501-6 (resin modified with Pb_3O_4) after self-sustaining combustion test; Air flow = 8.3 mph (SEM photomicrograph, 1000 X magnification)

Figure 17. Fused filaments from specimen shown in Figure 16; different location (SEM photomicrograph, 5000 X magnification)

Figure 18. Partially oxidized fibers in the third ply from the bottom (SEM photomicrograph, 1000 X magnification)

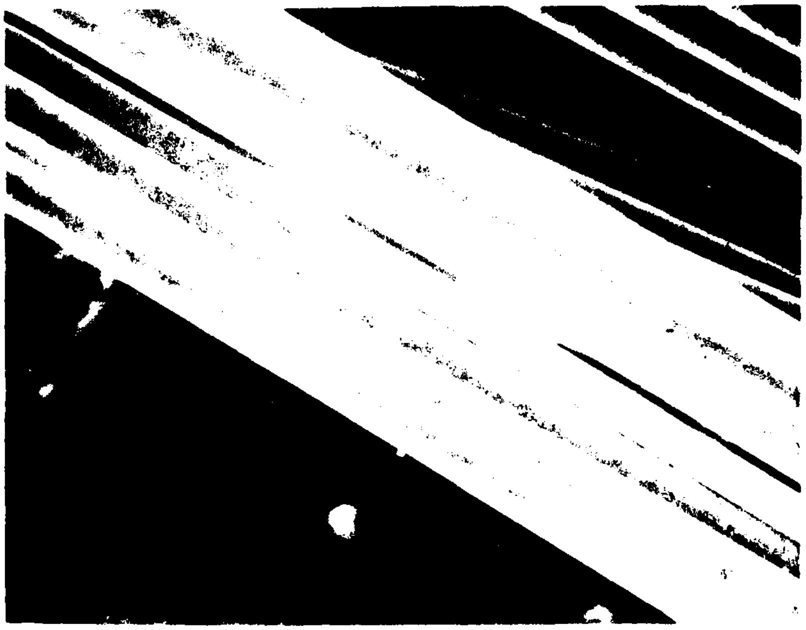


FIGURE 19. Virgin "AS" Fabric (SEM photomicrograph, 1000 X magnification)

of residue. X-ray fluorescence spectroscopy could detect only small amounts of sodium and potassium. The lamellar structures which make up the surface of the bundle seem to be too large for graphite crystalline regions.

For comparison Figure 18 shows some of the remains of the third ply of the same specimen (also at 1000 X magnification). Visually the third ply did not appear to be ash coated as in the first two plies but did seem to be much thinner than virgin fabric. The figure shows that, indeed, filaments are cylindrical with smaller diameters after being burned (compare with virgin filaments in Figure 19). The dented appearance of most of the filaments probably reflects the action of small currents of oxidizing gases as they pass upward through the filament collection.

SUMMARY AND DISCUSSION OF RESULTS

The method of obtaining apparent rates of reaction in the continuous combustion test (plotting $1 - \sqrt{1-W}$ versus time) works well for thin and thick graphite/epoxy specimens: correlation coefficients for all plots were greater than 0.96 and in most cases greater than 0.985. In addition, it was shown that graphite filaments shrink in diameter (see Figures 18 and 19) as required by the model.

The burning of graphite filaments in the continuous combustion test does not occur uniformly. Bottom plies oriented toward the draft of the oxidizing flame "see" the highest concentrations of reactive gas (O_2 and H_2O; CO_2 does not react with graphite significantly below 1000° C), and therefore, oxidize more rapidly than the upper plies. The variation of initial rate of fiber weight loss and apparent reaction rate with the number of specimen plies (see Figure 5) is explained on this basis. Figure 20 depicts a relative concentration profile as a function of distance into the collection of filaments as represented by the number of plies. The concentration drops rapidly due to consumption of the reactive gas by the lower plies. In 48-ply specimens, the weight loss due to oxidation in the upper plies is negligible. In 6- and 10-ply specimens, upper plies which contributed to small weight losses in 20-ply specimens are not present. As a result, the initial rate of weight loss is smaller. In 3-ply specimens, upper plies which accounted for significant oxidation in the 6-ply specimens are absent, lowering the initial rate of fiber weight loss even more. However, the apparent reaction rate always increases as the number of plies are reduced, since this eliminates the regions of slowest fiber consumption. In other words, because the 3-ply specimens burn with the highest average concentration of reactive gas, as shown in Figure 18, the apparent reaction rate is also greatest.

PbO, Sb_2O_3 and Bi_2O_3 significantly catalyze fiber oxidation in the continuous combustion test when applied to 6-ply Hercules AS/3501-6 composite specimens as a pigment in a paint coating. It must be concluded from these tests that there are regions in which the gasification is chemically controlled. It was noted in Table V that the increase in apparent reaction rate for 6-ply PbO-coated specimens over that for bare specimens is 26 percent, while the

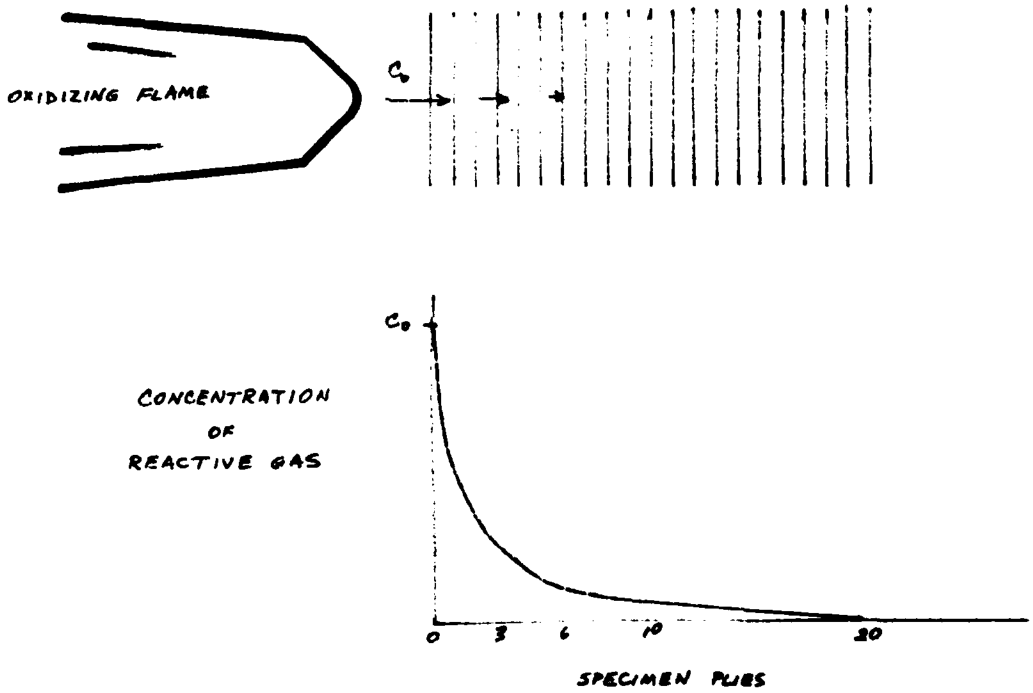


Figure 20. Reactive gas concentration in a collection of burning filaments

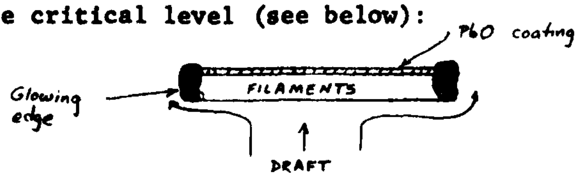
increase for 3-ply PbO-coated specimens is 64 percent. This probably reflects the higher average concentration of reactive gas in the 3-ply specimen. Since gasification catalysts are known to increase reaction rate constants by factors of 10^2 to 10^6, the availability of reactive gas at high reaction rates must be a limiting factor, thus, the higher the average gas concentration, the higher the rate of fiber consumption.

Calcium acetate, reportedly a good catalyst, did not catalyze fiber oxidation. Other non-catalysts, as determined by the continuous burn test, were SrO, V_2O_5, $CaCO_3$, Mn_2O_3, Li_2CO_3, SeO_2, MoO_3, CuO, Na_2CO_3, AgO.

The burning of graphite filaments in the self-sustaining combustion test cannot occur uniformly in thick specimens due to rapid heat generation on the draft side of the specimen and rapid heat loss by convection on the upper surface. While the draft causes an injection of O_2 into the filament collection, increasing the O_2 concentration and therefore the oxidation rate, heat losses by conduction and convection prevent the combustion process from going to completion. When the rate of heat generation begins to decrease due to the decreasing mass of the specimen, conduction losses to the specimen support become significant and quench the reaction. Because the means of specimen support is a wire mesh for studies at NAVAIRDEVCEN while at NRL the support is a single wire, resulting in lower conduction losses, NRL's data can be expected to show more complete combustion.

PbO, Pb_3O_4, AgO and Bi_2O_3 proved to be excellent catalysts when used in coatings for composite specimens tested in the self-sustaining combustion test; Sb_2O_3, however, was not a good catalyst. It is noted that AgO and Sb_2O_3 have opposite behaviors: while AgO proved not to be a catalyst in the continuous test, Sb_2O_3 did show catalytic activity. It is also interesting to note that a PbO coating prepared with silica and magnesium silicate extenders did not promote self-sustaining combustion, but that, when calcium acetate was also present in the coating, the result was similar to that obtained with the PbO coating minus the extenders. It is believed that the extenders coat many of the filaments during the test forming a physical barrier to the reactive gas.

The PbO coating (without extenders), will cause nearly complete incineration (greater than 90% by weight) of the graphite/epoxy composite up to a 20-ply thickness and at drafts of 8.3 mph or better for 6-ply specimens and 4.2 mph or better for 10- and 20-ply specimens. With 48-ply specimens, the long ignition time required to burn away the resin probably caused excessive loss of the catalyst. When the PbO coating is oriented away from the flame, there is an optimum draft for the 6-, 10- and 20-ply specimens: too great a draft causes excessive heat loss by convection from the non-burning bottom areas of the specimen while too small a draft reduces the rate of oxidation at the edges of the specimen below the critical level (see below):



PbO and Pb_3O_4 catalyzed fiber oxidation when used as resin modifiers in both the AS Fabric/3501-6 and the AS Fabric/828-125 composites. However, the modified AS/3501-6 system could sustain combustion only at an 8.3 mph or greater draft while the AS/828-125 system could sustain combustion without a draft. This is probably due to the presence of BF_3 in the 3501-6 resin. While the BF_3 acts as an accelerator in the cure of the epoxy resin, boron and fluorine are known as inhibitors to graphite oxidation. Bi_2O_3 produced sustained combustion only in the AS/828-125 system at drafts of 12.5 mph or more. At zero draft no significant sustained combustion could be generated in PbO modified AS/3501-6 specimen even with 10 minute ignition times in the oxidizing flame.

The burning of virgin graphite fabric or cloth appears to be a very serious problem, but one which could not affect fleet operations unless the unprepregged material is stocked for repair.

CONCLUSIONS

While this investigation was undertaken to find the means through which graphite/epoxy composites would safely incinerate in the event of aircraft composite fires, it has been found that, even using good oxidation catalysts, the extent to which the graphite burns depends heavily on reactive gas availability, distribution of the catalyst, conductive and convective heat losses, and time. Although it appears that a PbO primer coating on the Hercules AS/3501-6 composite would succeed in burning at least 90 percent of the composite if at least an 8.3 mph wind was blowing, the remaining fibers might still be a hazard.

FUTURE WORK

It is anticipated that the NASA Ames burn-bang test will be performed to determine the dispersability of fibers generated by catalyzed fires. It is recommended, however, that a new approach be studied as well: the use of pigments in paint coatings which would cause agglomeration of free filaments after resin burn-off and prevent their dispersal. Although, the use of an additive to become a "high temperature sizing," has been investigated as a fiber coating by the Jet Propulsion Laboratory at Pasadena, its use has not been attempted in coatings.

ACKNOWLEDGMENTS

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APPENDIX A

RESIN MODIFICATION

System No. 1 - Hercules AS Fabric/3501-6

Dissolve 50 grams of Hercules 3501-6 resin (manufactured in accordance with Hercules specification HS-SG-560A) in 20 grams MEK. Add 0.5 grams of catalyst and mix thoroughly with a spatula.

A three-roll paint mill is adjusted so that a 0.004 inch (0.10 mm) clearance exists between the center roll and the feed and apron rolls. The resin - MEK - catalyst mixture is milled for 2 passes and the dispersion collected and placed in a freezer at 32° F (0° C) until required for lamination.

System No. 2 - Hercules AS Fabric/Epon 828-Versamid 125

Mix 50 grams of Epon 828 with 33.3 grams of Versamid 125 by stirring with a spatula. Add 0.83 grams of catalyst and stir until the mixture is reasonably homogeneous. This mixture is milled as above and stored in a freezer at 32° F (0° C) until required.

APPENDIX B

COMPOSITE FABRICATION

Layup

Cut 4 pieces of Hercules AS Fabric (A-370-8H), each 6 inches square. Spread a thin layer of the modified resin on a non-porous Armalon release cloth and lay the first fabric ply. Spread additional resin into the fabric with the spatula and lay the second ply. Continue in the same manner, spreading resin and laying the third and fourth plies.

System No. 1 - Hercules AS Fabric/3501-6

Evaporate the solvent from the layup by heating in a vacuum oven at 120° F (49° C) and less than 20 mm Hg (2.7 KPa) for 2 hours. Cool to room temperature and complete the layup design as shown in Figure 21. Cure the laminate in an autoclave as follows:

1. Place layup on the autoclave tool, attach thermocouples and cover with 120 E-glass breather cloth and a Mylar bag sealed to the tool at the edges.

2. Evacuate the Mylar bag and raise the autoclave pressure to 85 psi (0.59 MPa).

3. Raise the temperature to 240° F (116° C) in 30 to 85 minutes and hold 60 to 70 minutes.

4. Raise the autoclave pressure to 100 psi (0.69 MPa) and vent the vacuum bag.

5. Raise the part temperature to 350° F (177° C) in 20 to 55 minutes and hold for 120 minutes.

6. Cool to 200° F (93° C) in not less than 30 minutes with at least 8 psi (0.056 MPa) autoclave pressure.

7. Laminates were not post cured.

System No. 2 - Hercules AS Fabric/Epon 828-Versamid 125

Complete the layup as shown in Figure 21 and cure the laminate by pressing at 30 psi (0.21 MPa) between the plattens of a hydraulic press preheated to 200° F (90° C). After 1 hour at 200° F (90° C), the temperature is raised to 250° F (120° C) for the final cure, while maintaining the same initial pressure. Laminates were not postcured.

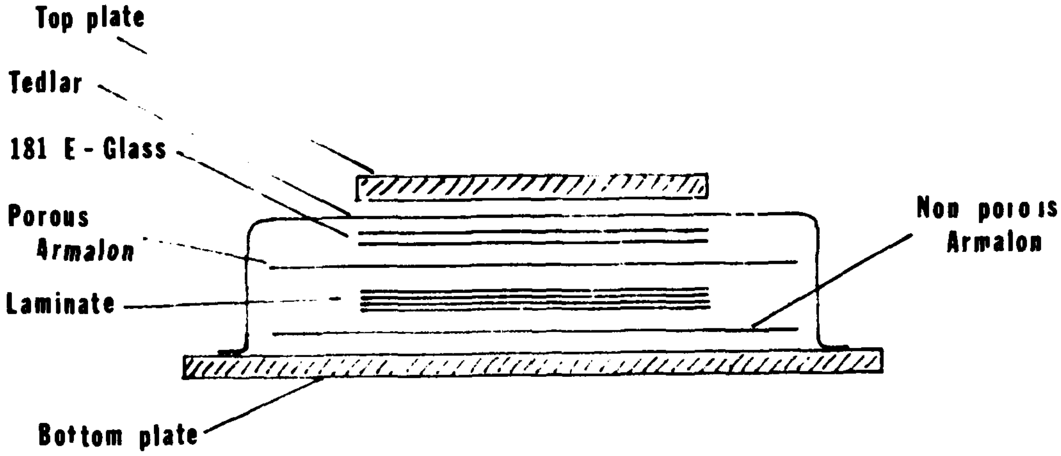

Figure 21. Layup design for resin-modified composites

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