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Part II

DETERMINATION OF THE PERFORMANCE OF PLASTIC LAMINATES AT CRYOGENIC TEMPERATURES

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(Prepared under Contract AF 33(616)-8289 by Narmco Research & Development, A Division of Telecomputing Corporation, San Diego, California; Don W. Chamberlain, B. R. Lloyd and R. L. Tennent, authors)

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FOREWORD

This report was prepared by Narmco Research & Development, A division of Telecomputing Corporation, San Diego, California, under Air Force Contract No. AF 33(616)-8289. This 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. F. H. Bair acting as project engineer.

This report covers work conducted from 15 November 1962 to 15 February 1964.

The authors were assisted in the preparation of this report by Mr. Norman O. Brink and Mr. John F. Brown, Senior Research Engineers.

ABSTRACT

This program is a continuation of the effort begun under this contract for the determination of mechanical properties of various plastic laminate materials at cryogenic temperatures. The results of the first year's endeavor have been published in ASD-TDR-62-794.

This portion of the program was extended to include additional resin systems, some of which have been developed specifically for cryogenic applications. Also investigated were additional reinforcement materials, including stainless steel, nylon, and Fortisan.

Mechanical properties data included tensile, compressive and flexural strength, and modulus of elasticity, as well as bearing strength and tensile fatigue values. Test temperatures used were room temperature, -110°F, -320°F, and -423°F.

Results of this program show several glass reinforced plastic laminate materials to be most promising as structural materials for use in cryogenic environments.

This technical documentary report has been reviewed and is approved.

W. P. CONRARDY, Chief
Materials Engineering Branch
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I. INTRODUCTION

The purpose of this program has been to provide the Research and Technology Division of the United States Air Force with additional data on the mechanical properties of various reinforced plastic laminate materials when subjected to a cryogenic environment. The work described herein is an expansion of that report in ASD-TDR-62-794 (August 1962). Tests under that program were confined to 10 specific resin systems, all of which were qualified to an applicable MIL-specification. All 10 resins were laminated with 181 glass cloth. During the course of the first year's effort, it was brought to the attention of the authors by several manufacturers that a number of new resin systems had been developed specifically for cryogenic applications, which had not been qualified to a MIL-specification. The work described herein includes investigation of these new materials, as well as some additional resin systems which are qualified to a particular procurement specification.

Several additional reinforcement materials were evaluated in addition to the more conventional fiberglass laminates utilizing 181 glass cloth.

Refinements of certain testing techniques and fixtures resulted from experience gained during the performance of this contract. Certain problems peculiar to the field of low-temperature testing and the means of resolving them are described in this report.

> Although some of the materials tested during the course of this program are qualified to a particular procurement specifications, none of the applicable specifications provide for any cryogenic properties data. Those materials which have been developed specifically for low-temperature use may exhibit certain characteristics which are desirable for one application, and not desirable for another. When evaluating the data presented in this report, particular attention must be paid to the application for which a material may be selected.

This program was divided into three phases. Phase I covered the investigation of resin systems which were recommended by their manufacturers as having performance characteristics superior to those resin systems selected for study in the previous portion of this program. All of these materials are readily available commercially, although as previously mentioned they may not be qualified to any particular procurement specification.

Phase II of this program included experimental resins which had shown promise for cryogenic applications during the first part of this program. All of the materials included in Phases I and II were laminated utilizing 181 glass fabric.

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Phase III was an investigation of reinforcement materials other than 181 glass cloth. A limited number of resins were laminated with cloth made from Teflon, Fortisan, and stainless steel.

Test methods and procedures utilized throughout this program are essentially as set forth in Federal Test Method Standard No. 406, "Plastics - Methods of Testing." However, due to the problems peculiar to cryogenic testing, Narmco considered it necessary to modify the standard procedures somewhat. In the section of this report on testing, these deviations and the reasons for such are described in detail.

II. DESCRIPTION OF MATERIALS

Seventeen different materials were investigated during this program: seven resin systems were included in Phase I, five in Phase II, and five special material combinations in Phase III. These materials and their manufacturers are listed in Table 1.

The Epon 826 NMA, Epon 826 DDS, Trevarno F-131, Hetron H-31, and Scotchply Type 1002 materials were all fabricated into laminate form by their respective manufacturers. All the other materials included in this program were laminated at Narmco Research & Development. Laminating and postcure data are given for each material in Table 2.

Table 3 gives specific gravity and resin content figures for each panel of each material included in this program. These data were determined in accordance with FTM 406, "Plastics - Methods of Testing," Methods Nos. 7061-1 and 5011, except that resin content determination by Method 7061-1 is not applicable to silicone resin systems, nylon, or Fortisan fabrics. Resin content for these materials is an estimated value given by the manufacturer, or is based on known fabric weight and content, or is estimated from previous experience with similar materials.

III. PREPARATION OF SPECIMENS

The dimensions of the finished laminate panels were as follows:

Supplied by manufacturer:	1/8 in. x 15 in. x 20 in.
	1/2 in. x 6 in. x 8 in.
Fabricated by Narmco:	1/8 in. x 18 in. x 18 in.
	1/4 in. x 6 in. x 8 in.

In both cases, six of the 1/8-in. panels and one of the 1/2-in. or 1/4-in. thick panels were fabricated for each material being evaluated. The 1/8-in. panels provided all of the test specimens except for compression, these being obtained from the thicker panels.

The compression specimen configuration chosen for this program was the block or prism type specified by FTM 406. The recommended size is 1/2 in. x 1/2 in. x 1.00 in; however, in the preceding portion of this program, specimens this size sometimes could not be failed due to the tremendous increase in strength at low temperatures. For this reason, a smaller specimen was considered necessary and a size of 1/4 in. x 1/4 in. x 1/2 in. was chosen. The panels for compression specimens which were fabricated at Narmco were therefore only 1/4-in. instead of 1/2-in. thick. In the case of some particularly stong materials, the compression specimen size had to be reduced further in order to assure specimen failure within the capacity of the test machine. Even though it was necessary to reduce the compression specimen size from that which is recommended by FTM 406, a smaller specimen does comply with the requirements set forth in that document.

Upon receipt from the manufacturer or the Narmco experimental fabrication department, each individual panel was assigned an identification number. After the panels were marked for cutting, each specimen blank was coded so that the parent panel and its exact position in that panel could be readily determined. Figure 1 shows the manner in which both types of 1/8-in. panels were divided in preparation for cutting into blanks. The cutting was accomplished utilizing a radial arm saw equipped with a diamond-tipped blade (see Figure 2). The thicker laminate panels were cut in the same manner and the individual specimen blanks identified.

The tensile test specimen configuration is shown in Figure 3. The specimens were machined from their blanks in two operations, the first of which is illustrated in Figure 4. An air-driven router with a diamond bit was used to cut the specimens to shape. The specimen was installed in the combination routing template and holding fixture during this operation. Two diamond bits were utilized; a coarse-grade bit for roughing the specimen to shape, and a fine-grade bit for the finish cut. The surface finish left by the fine diamond bit was such that the only additional finishing required was a very light sanding with No. 600 paper. The second machining operation was to place three or four specimens in the fixture shown in Figure 5 and grind the gripping areas to their finished tapered configuration.

The tapered end tensile specimen was developed to alleviate some of the problems encountered with other configurations. The original type was a more conventional "dog-bone" specimen with a 1/2-in. pin hole in each end. The pin holes necessitated the addition of doublers, which was time-consuming and expensive. Also, the specimen would often fail in interlaminar shear immediately under the doublers, which wedged the specimen tightly into the clevis, resulting in an additional time loss.

To improve the situation, a set of serrated clamp-type grips was made, with a 1/2-in. bolt passing through the pin hole. These grips were most effective, but required additional setup time and a special fixture to assure proper alignment.

A tapered end specimen with the titanium grips (described later) eliminates all the problems mentioned. The new design is narrower (0.400 in.) than previous configurations (0.500 in.) because of the high tensile strengths expected and the fact that some laminates were delivered significantly thicker than the nominal 1/8-in. specified. It was felt that the capacity of the test machine might be exceeded should a very strong, unusually thick laminate be encountered.

The configuration of the tensile fatigue specimen is shown in Figure 6. This shape remains unchanged from the previous program, and has proven to be entirely satisfactory. The fatigue specimens are produced from blanks in two operations. First, the blank is drilled and reamed as illustrated in Figure 7. Several drilled blanks are then installed in the grinding fixture (Figure 8) and the surface grinder is utilized to produce the finished shape.

Flexural and bolt bearing specimens were ground to size (1/8 in. x 1 in. x 4 in. and 1/8 in. x 0.923 in. x 4 in. respectively) then the holes in the bearing specimens were drilled and reamed (see Figure 9). The flexural test specimen is as described in FTM 406 (Method 1031, Table 1). The bearing strength specimen is the same as that specified in FTM 406 (Method 1051) for 1/8-in. materials, with two exceptions: The length was reduced to 4.00 in. from 4.75 in., and the bearing hole diameter was increased to 0.187 in. from 0.126 in. The first deviation was to conserve material, to reduce the size of the test specimen and fixtures in the cryostat, and to simplify the cutting of specimens from the large panels, since the flexural and bearing specimen blanks were now the same length. The increased hole diameter was necessary due to the fact that at cryogenic temperatures, the hardened steel pin would fail in shear before the test part would fail.

All specimens cut from the 1/8-in. thick panels were randomized as much as possible. Specimens adjacent to one another when cut from a panel were not tested under the same conditions. This randomization was further enhanced by the similarity in sizes of blanks for tensile and fatigue, and flexure and bearing specimens.

Compression specimens were prepared (after cutting into blanks with the diamond saw) by grinding to the desired size utilizing the surface grinder. Several specimens were accommodated at one time in a simple clamp support on the bed of the grinder. In most cases, this method of preparation produced accurate and well-finished specimens. However, some of the less rigid materials (e.g., Teflon fabric laminated with Adiprene resin) do not lend themselves to grinding or machining of any kind. Extreme care in machining and some hand-finishing was necessary with these materials.

IV. TEST EQUIPMENT AND PROCEDURES

A. EQUIPMENT DESCRIPTION

As in the previous portion of this program, two basic test machines were utilized for all mechanical testing throughout the program. The general arrangement of the Narmco cryogenic laboratory and the location of these test machines is shown in Figure 10.

All of the tests with the exception of tensile fatigue were performed with a type TTC Instron Universal test machine equipped with a specially designed cryostat for tests at low temperatures. The cryostat incorporates all the fixtures and adapters necessary for static tests performed under this program. These accessories will later be described in detail. The Instron test machine with the cryostat in place ready for a test with liquid hydrogen is shown in Figure 11.

A Tatnall-Krause direct stress fatigue machine was employed for all tensile fatigue tests. During the original portion of this program a special cryostat had been designed and built, and the machine suitably modified for safe operation in a hydrogen atmosphere. The fatigue testing equipment remains essentially unchanged for this continuation, except for the addition of a dynamic load cell and associated readout equipment. This improvement permits the operator to maintain a constant check of the load pattern imposed upon the fatigue specimen without interruption of the test. If minute adjustments are required, they may be made by the manual operation of the machine's integral hydraulic load-maintaining system. The fatigue machine with its cryostat and the new load cell is shown in Figure 12.

Extensometers were used for deflection measurements in the performance of all static tests. Depending on which was more suitable for a particular test, either a strain gage type or linear variable differential transformer (LVDT) type of extensometer was utilized. All of the extensometers were designed and built specifically for this program, and some are integral with the test fixture itself. A typical tensile test specimen is shown in Figure 13 with the tensile extensometer (which is of the strain gage type) installed. The light weight and the resulting minimum effect on the specimen dictated the use of a strain gage type extensometer for tensile tests.

B. CALIBRATION OF EQUIPMENT

Accurate calibration of all extensometers employed during the course of this program was considered of paramount importance. Due to the change in electrical characteristics with changes in temperature, a means for calibrating all the extensometers at the test temperature was devised.

The calibration fixture depicted in Figure 14 is shown with a strain gage extensometer installed, but with suitable adaptors can be used to calibrate any of the extensometers utilized in this program at any of the test temperatures. The heart of the fixture is a 4-in. diameter micrometer graduated in 1 x 10^{-4}-in. divisions. A Vernier scale facilitates accurate readings to 1 x 10^{-5} in. The device consists basically of two concentric stainless steel tubes; in operation, the extensometer is clamped to the lower ends of the tubes, and rotation of the micrometer head causes the inner tube to move in relation to the outer tube. This motion is sensed by the extensometer and is transmitted to the Instron chart drive system. A calibration chart is produced by turning the micrometer head in 0.001-in. increments, (causing the chart to move) and "blipping" the chart pen. The result is an accurate, easily interpreted chart for translating chart travel into specimen deflection as measured by the extensometer.

The outer tube diameter of the calibration device is 0.500 in., the same as the cryostat load rods. For low-temperature calibration, the device is inserted in the cryostat in place of the load rod and the extensometer submerged in the applicable cryogen.

Calibration of the Instron machine load cell was accomplished in accordance with the manufacturer's standard procedures utilizing certified weights.

The fatigue machine load cell was calibrated by installing it in the Instron machine and certifying it against the calibrated Instron load cell.

C. TESTING PROCEDURES

In general, the procedures set forth in FTM 406 were adhered to as closely as possible. Since that document makes no provisions for testing at cryogenic temperatures (nor does any other recognized standard test method), certain deviations were necessary. However, Narmco feels that these deviations did not significantly affect the test results.

1. Tensile Tests

As mentioned previously, the tensile test specimen designed for the continued portion of this program is different from that used in the earlier portion (see Section II). A new specimen and the new grips are shown in Figure 15. These grips were machined from 6 Al-4V titanium alloy, and with the titanium clevises provide perfect axial alignment of the specimen. (Figure 13 shows a view of the specimen ready for testing.) This new specimen configuration and the new grips completely eliminate the difficulties experienced with earlier types.

To perform a low-temperature tensile test, the assembled specimen, grips, and extensometer were installed in the test machine, and the cryostat load cylinder and cup were then put in place (see Figure 16). The cryostat assembly was completed and filled with the appropriate cryogen.

The sample was loaded at the rate of 0.05 in./min, and a stress-strain diagram plotted on the Instron chart. The initial, secondary, and ultimate stress as well as the initial and secondary modulus was determined from the chart.

As in the previous portion of this program, it was necessary to establish some ground rules for the determination of the values just mentioned; the ground rules remain unchanged and are as follows:

a. Initial Modulus — This value is defined as the modulus of elasticity as determined by the initial slope of the stress-strain curve.

b. Secondary Modulus — Since most materials tested exhibited a definite, easily detected change in slope of the stress-strain curve, the second portion of this curve is used to determine the secondary modulus.

c. Initial Stress — The value for initial stress was chosen to be the point of intersection between the portions of the stress-strain curve representing initial and secondary modulus.

d. Secondary Stress — Although not typical in every case, many materials exhibited a definite "third slope" portion of the stress-strain curve. When this third slope was readily apparent, the intersection point between the third slope and the slope defining secondary modulus was identified as the secondary stress level.

e. Ultimate Stress — This was the load in psi at which the test specimen failed.

2. Flexural Tests

A test fixture designed to accommodate a standard flexural test specimen as described by FTM 406 is illustrated in Figure 17. The extensometer chosen for these tests is the LVDT type, due to the relatively large deflections to be measured and the lack of extensometer effect on the specimen.

The actual test procedure and determination of test results is quite similar to that described for tensile tests.

3. Compression Tests

The compression test fixture is depicted in Figure 18. This fixture is a refinement of the one utilized in the preceding portion of this program and, as a result of findings therein, incorporates two significant improvements. Due to the very small size of some compression test specimens, deflection measurements became quite critical. To eliminate unwanted deflections in the test fixture itself, tungsten carbide blocks were utilized for the portion of the fixture in contact with the ends of the specimen. This material, with a modulus on the order of 90 x 10^6, has deflections under load so insignificant they may be considered negligible.

The second change involved placing the LVDT extensometer on the same axis as the fixture and its load rods. The armature was spring-loaded against one of the tungsten carbide bearing blocks, and the coil against the other block. This axial arrangement eliminated any erroneous data resulting from possible wobble in the fixture, and the tungsten carbide blocks effectively eliminated any deflection in the fixture which might be seen by the extensometer.

An effective demonstration of the accuracy and efficiency of this improved fixture is the straight horizontal line on the Instron chart (indicating zero deflection) when the fixture is loaded with no specimen in place.

Test procedures for compression testing are essentially the same for tensile tests.

4. Bolt Bearing Strength Tests

The bolt bearing test specimen is essentially the same as that specified in FTM 406 Method 1051 except for the bearing hole diameter and specimen length. As mentioned previously, with the standard 0.126-in. diameter hole, the pin failed in shear during preliminary tests in liquid nitrogen. The pin diameter was increased to 0.187 in. and no further difficulties were encountered. The specimen length was reduced from 4.75 in. to 4.00 in. to conserve material and reduce space requirements inside the cryostat.

The test procedure was similar to that described in Method 1051 except that a vastly improved means of determining bearing strength and deformation offset values was utilized. Instead of a dial gage (which obviously could not be used inside a cryostat) and the resulting hand-plotted curve, an extensometer was developed to permit continuous automatic recording of the bearing strength-deformation curve.

The extensometer originally built for this purpose was of the LVDT variety, but proved to be too heavy and was sensitive to the slightest fixture misalignment. A strain gage extensometer was then constructed which eliminated both of the objections mentioned. A typical test specimen with this extensometer installed is illustrated in Figure 19.

A template was made as suggested by Method 1051 for determining bearing strength at 4% hole elongation. Figure 20 shows this template in use with a typical bearing stress-deformation curve.

5. Tensile Fatigue Tests

The procedure for tensile fatigue tests performed for this program was mutually agreed upon by the Air Force and Narmco since no standard test method exists for tensile fatigue testing of plastic laminate materials. In the original portion of the program, 15 specimens were tested at each of the four specified test temperatures. Based on the experience gained in that part of the program, and to expedite the many tests to be performed, it was decided to test fewer specimens in this continuation. Results from the earlier portion of the program showed that an accurate diagram could be produced with fewer specimens than 15. For the continuation of the program, 40 specimens of each material were prepared for testing, 10 at each temperature.

It was also decided at the beginning of this part of the program that those materials exhibiting low fatigue life at room temperature, or the two intermediate cryogenic temperatures would be eliminated from further testing, or that only a few specimens would be subjected to further testing. Therefore, the 40 specimens prepared for testing were not necessarily all used.

The procedure for tensile fatigue testing was as follows:

a. The average static tensile strength of the material was determined for the applicable temperature.

b. The test specimen cross-sectional area was recorded and the test load determined. (This usually was 60% or 70% of ultimate for the first test.)

c. The assembled specimen and grips were installed in the cryostat, which was then filled with the appropriate cryogen.

d. After thermal stabilization was reached, the predetermined load was applied. In all cases, the maximum load was that determined in b. above, and the minimum load was approximately 5% of the maximum. This 5% minimum load assured that the specimen was always subjected to a tensile load and could not go into compression.

e. After the preparation was completed, the machine counters were zeroed and the test begun.

After testing several specimens and plotting the results, the load levels at which additional tests were necessary to round out the S-N diagram became apparent.

The fatigue machine with the cryostat being installed is shown in Figure 21, and the disassembled components of the cryostat are illustrated in Figure 22.

The test machine incorporates an electronically controlled, hydraulically actuated load maintaining system. This system insures that a uniform load pattern will be applied to a test specimen even though it may elongate or creep during the test.

The tensile fatigue testing equipment utilized throughout this program was developed specifically for the cryogenic testing of plastic and adhesive materials in tensile fatigue. It is the first such equipment for tests utilizing liquid hydrogen known to exist in this country.

6. Testing Unidirectional Fiber Materials

The procedures for testing unidirectional fiber materials were essentially the same as used for tensile tests. However, due to the nature of the material, it was necessary to utilize doublers bonded to the gripping areas of the specimens and to use the earlier separated clamp-type grips.

Again due to the nature of a unidirectional material, extreme care was necessary when installing the extensometer on these specimens.

D. CRYOGENIC FLUIDS

The three low test temperatures, -110°F, -320°F, and -423°F, were obtained with dry ice in alcohol, liquid nitrogen, and liquid hydrogen respectively.

The most desirable and convenient of these cryogens is liquid nitrogen. It presents no fire or explosion hazards as does liquid hydrogen, nor does it require the constant attention of the dry ice/alcohol mixture to assure the desired temperature. Liquid hydrogen is the next most desirable of the three cryogens. The hazards associated with liquid hydrogen were minimized by the attention to safety in the design of the laboratory and the thorough training of all operating personnel. With the hazardous aspects of liquid hydrogen reduced, the largest objection to its use is the additional time required for tests. This time is necessary for proper transfer procedures, and for ensuring that all hydrogen has been removed from the cryostat upon completion of a test.

The least desirable low-temperature fluid is dry ice in alcohol, which is messy and time-consuming in its preparation, and must be closely observed, with additional dry ice being added periodically in order to assure that the temperature is at the desired level. In addition, the temperature of -110°F is not truly "cryogenic" in the accepted sense of the term.

V. PRESENTATION OF TEST RESULTS

Results of the tests performed during this program are presented in tabular form, and graphically in the form of curves and S-N diagrams. Due to the large number of specimens, the static test data reflect the average values obtained from several specimens together with the estimated standard deviation.

The standard deviation applied throughout this program was obtained by the use of an estimating procedure for a small number of samples.*

Sample Size	σ_{Est}	Efficiency
2	0.886 w	1.000
3	0.591 w	0.992
4	0.486 w	0.975
5	0.430 w	0.955
6	0.395 w	0.933

For a normal population, $\sigma_{Est.}$ can be determined from the range w. Because of the ease and speed with which computations may be made, this method is particularly useful in programs such as this where there is a large number of small groups of data.

Comments on the tables and figures of this report are given below. Tables 1 through 3, as mentioned earlier in this report, give the list of materials and manufacturers, laminating and postcure data, and specific gravity and resin content. Table 4 lists the materials tested in tension during Phase I, and the average results therefrom. The ultimate tensile strengths and initial moduli of these materials are graphically depicted in Figures 23 and 24.

Average compression strength and modulus for Phase I materials are given in Table 5, with the same values being shown graphically in Figures 25 and 26.

Flexural strength values are tabulated and shown in graph form in Table 6 and in Figures 27 and 28 respectively for materials tested during Phase I.

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*Wilfred J. Dixen and Frank J. Massey, Jr., Introduction to Statistical Analysis, McGraw-Hill Book Co., 1957

Table 7 gives average bearing strength values for Phase I, and the results are plotted in Figure 29. At the request of the Air Force, bearing strengths of most of the materials included in the original portion of this program were determined. Sufficient material was not available to perform bearing tests on the Vibrin 135 high-temperature polyester, nor on the Epon 1001 epoxy. Due to the limited amount of Narmco 506 and 513 materials, bearing tests at -320°F only were conducted. Bearing strength values for the remaining materials at room temperature and -320°F are given in Table 8. Since material was limited for all these resin systems, it was agreed that no tests at -110°F or -423°F would be performed.

Table 9 gives the results of all tensile fatigue tests performed during Phase I. The S-N diagrams produced from these results are shown in Figures 30 through 36.

Average tensile strength and modulus values for the Phase II materials are given in Table 10. Figures 37 and 38 show graphically the results of Phase II tensile tests.

Phase II compressive strength and modulus is shown in Table 11, and in Figures 39 and 40.

Table 12, and Figures 41 and 42, convey flexural strength data for Phase II.

Bearing strength for Phase II is tabulated in Table 13, and shown in Figure 43.

Table 14 summarizes tensile fatigue test results for Phase II, and Figures 44 through 48 show S-N diagrams for the same materials.

Phase III tests were conducted at one temperature only, -320°F. The results of all Phase III static tests are shown in Table 15, and in Figures 49 and 50.

Fatigue tests conducted during Phase III are tabulated in Table 16, and a combined S-N diagram for all Phase III materials is presented in Figure 51.

VI. DISCUSSION OF TEST RESULTS

The results determined from the tests in the continued portion of this program have further substantiated the trends established during the original part of the program.

The most obvious trend, of course, is that at subnormal temperatures all materials tested displayed significant increases both in strength and modulus over room temperature values. The large increase in strength (with a moderate increase in modulus) indicates that most materials should retain their desirable toughness characteristics at low temperatures. This general trend was not followed by all the materials tested: the F-131 and Narmco 527 exhibited a decrease in modulus from room temperature to -110°F, and although the F-131 then showed a higher than room temperature value at -320°F and -423°F, the 527 modulus value remained below that of room temperature for all the low-temperature tests. Observation of the ultimate strength values shows that both materials exhibited only moderate increases in strength compared with others in the same group, and the F-131 strength actually decreased slightly from -320°F to -423°F.

A more detailed examination of the individual types of materials in Phase I further confirms the general trends.

A. EPOXIES

The three epoxy materials — Epon 826/NMA, Epon 826/DDS and Narmco 522 — displayed the highest strength and modulus values for all static tests. These results further confirm the already accepted fact that epoxy laminate materials show excellent promise for cryogenic structural applications. The fatigue test results indicate that these materials show good fatigue strength as well as ultimate strength values.

B. POLYESTERS

The second category of resin systems in Phase I indicated two polyester materials: Narmco 527 and Hetron H-31. Both of these materials were suggested by their respective manufacturers during the earlier part of the program as having superior performance characteristics at low temperatures. The fact that both materials lived up to these expectations, to some extent, is most clearly shown in Figure 23 where it can be seen that the tensile strength of these two polyester resin materials is only slightly below that of the epoxy systems. Modulus values also follow this trend, except that the 527 material shows a sharp drop from -320°F to -423°F. Compressive strength and modulus is not good for the H-31 and is only moderate for the 527 material. Flexural strength and modulus values are good for both materials, as is bearing strength, although neither is up to the values shown in the tensile tests.

Fatigue strengths do not reach the values one might expect based on static tensile tests. The 527 material shows slightly more fatigue resistance than the H-31, but neither can compare with the epoxy materials in this respect.

All things considered, the polyester materials definitely have a place as structural cryogenic materials, especially when material cost and manufacturing time are significant factors.

C. SILICONE SYSTEM

Only one silicone resin system was evaluated during Phase I, and one additional system in Phase II. Although tested in different phases of the program, they are compared together here with the other materials in Phase I. The F-131 material had the lowest initial strength in all tests but compression, and although the strength increase with the accompanying temperature decrease was good, the low room temperature values render it a less attractive material than either the epoxies or polyesters. The DC 7146 silicone system from Phase II followed the pattern set by the F-131 material.

D. PHENYL-SILANE

The Narmco 534 resin system was developed as a high-temperature system, and since other high-temperature systems had shown indications of good cryogenic properties, this material was included in Phase I. As can be seen from the figures, the 534 material was peculiar in that it displayed some of the highest modulus values but only average strength, and at room temperature the strength is below average. This material may find use in applications where the relatively high modulus is a factor, but for general structural considerations, the epoxy and polyester materials appear to be more desirable.

The materials evaluated in Phase II of this program included a nylon-epoxy system, Metlbond 406; a flexible polyurethane, Adiprene L-100; a proprietary resin system, IMIDITE; the previously discussed silicone resin system, DC 7146; and the Teflon FEP resin system.

The silicone system with the other silicone resin system evaluated under this program (F-131) is not one of the more promising materials with respect to possible cryogenic applications.

E. NYLON EPOXY

The nylon-epoxy resin system (Meltbond 406) was originally developed as a cryogenic adhesive, and performed quite well in that capacity. As a laminating resin, it exhibits better-than-average, but not outstanding, properties, both in static tests and in tensile fatigue.

F. FLEXIBLE POLYURETHANE

The Adiprene L-100 material presented a problem: it was not only one of the most difficult materials to machine, but one of the most difficult to test. The tensile test results show that it has the lowest strength at room temperature of any of the Phase II materials, and although the strength increased tremendously at the lower temperatures, the modulus remained low. The fatigue tests of this material are not particularly encouraging at any test temperature other than -320°F. This material should undoubtedly find many low-temperature applications where flexibility and low modulus are desirable; however, it does not appear to be one of the more promising structural materials.

G. IMIDITE

IMIDITE shows only average strength, but exhibits high modulus values in tension and compression, and exceptionally high strength and modulus in flexure when compared with other materials in Phase II. The S-N diagram for IMIDITE shows that the fatigue life is also above average for this group. This material has exhibited very high values as a cryogenic adhesive, and with the better-than-average properties as a laminating material is one of the more interesting materials investigated.

H. PERFLUORINATED HYDROCARBON

The remaining resin system included in Phase II was Teflon FEP. This material is puzzling, as it showed the greatest increase in tensile strength with temperature decrease, and the highest tensile strength recorded for Phase II, but only moderate performance in the remainder of the static tests. The fatigue characteristics of Teflon FEP were not outstanding, but the LOX compatibility of this material together with reasonable physical properties makes it one of the more attractive materials for specialized applications.

I. SPECIAL REINFORCEMENTS

In Phase II of this program, five additional materials based on reinforcements other than type 181 glass cloth were tested at -320°F only. The first of these, Adiprene L-100 resin and Fortisan fabric, displayed only average strength and modulus as compared to other materials in the group. Fatigue life was slightly better than average.

The second material in Phase III was a laminate made from Teflon fabric and Adiprene L-100 resin. This material was so flexible at room temperature that it was impossible to set up a flexural test. In the other static tests, this material did not perform particularly well, nor did the fatigue tests prove encouraging.

The next Phase III material to be considered was Scotchply 1002. This material exhibited one of the highest tensile strength values and by far the highest flexural strength of any material in the entire program. Compressive and bearing strength values were also very good. Based on these tests, a unidirectional fiber material such as this is one of the most encouraging materials yet developed for cryogenic structural purposes. Fatigue test results were also outstanding, and serve to further substantiate the choice of this material as one of the best considered in this program.

A laminate made from Type 994 HTS glass cloth and Epon 828 epoxy resin was the next type considered in Phase III. Tensile and compressive strength was greater than the other Phase III materials with the exception of Scotchply, but flexural and bearing strength values were not particularly encouraging. Tensile fatigue life was below average for this group of materials.

The remaining material in Phase III was a laminate of stainless steel wire mesh and Epon 828 epoxy resin. This material showed only average strength in the static tests and the lowest tensile fatigue life of any material in Phase III. The fabrication difficulties, greater weight, uninteresting performance, and expected high thermal conductivity when compared with other materials in Phase III would tend to dismiss this material from further consideration as a possible cryogenic structural material.

VII. SUMMARY OF TEST RESULTS

A. CONCLUSIONS

Results of this program would indicate that, in general, the epoxy materials are the most desirable materials evaluated for cryogenic uses, with the polyesters running a close second. The materials evaluated under phases II and III in general did not show the potential of the Phase I materials, with the exception of Scotchply 1002. The flexible resin systems, Teflon and Adiprene, undoubtedly are suitable for specialized usage where their flexibility is an asset. The silicone resin systems and the stainless steel, Teflon fabric, and Fortisan reinforcement materials showed so little encouragement that they may be considered unsuitable for any but the most specialized cryogenic applications. The nylon-epoxy system, which has shown such good cryogenic adhesive properties, and the phenyl-silane material, which is excellent at high temperatures, can be considered only average as cryogenic laminating materials. IMIDITE, another high-temperature system with very good cryogenic adhesive properties, is also only average as a laminating material.

B. RECOMMENDATIONS

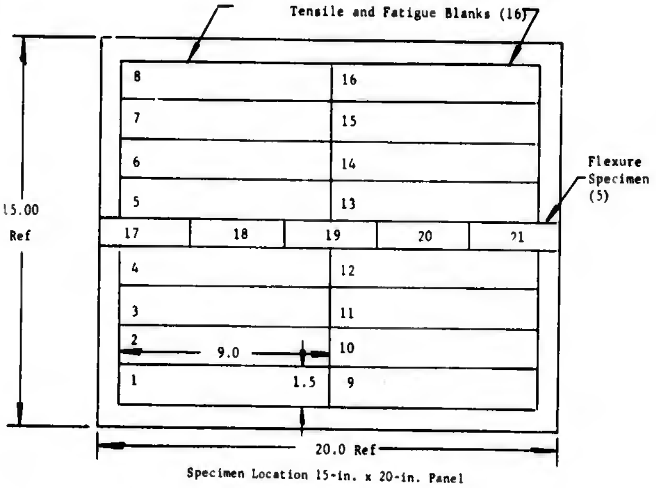
Although a wealth of information has been derived from the many tests conducted under this program, the surface has only been scratched with respect to the application of reinforced plastic materials for cryogenic structural applications.

Succeeding programs might cover the areas of impact and shear strength values, as well as further investigation of specialized resins at cryogenic temperatures.

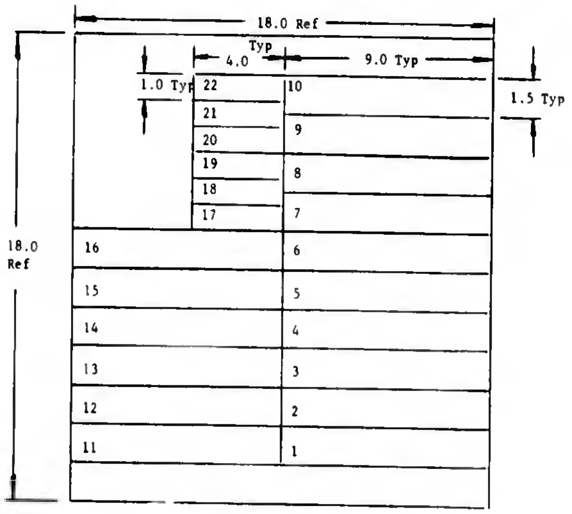
The basic characteristics of plastic laminate materials (high strength-to-weight ratio, low sensitivity to thermal shock, and low thermal conductivity, among others) make these materials among the most interesting for future use in cryogenic environments.

Another area which warrants further consideration is in the standardization of test methods for cryogenic applications. As pointed out earlier in this report, there are no recognized standard methods for testing laminate materials at temperatures as low as -423°F. Some of the standard test methods for normal temperatures, such as flexural tests, lend themselves to cryogenic testing quite well, as long as suitably modified test equipment is used. Other tests, such as bearing and compressive strength, cannot be performed at low temperatures without some modifications to the standard test methods.

If the field of plastic materials for low-temperature applications is to be fully explored, some standardization of test methods and procedures is mandatory.



Specimen Location 15-in. x 20-in. Panel



Specimen Location 18-in. x 18-in. Panel

Figure 1. Cutting Diagram and Specimen Location for 1/8-in. Panels

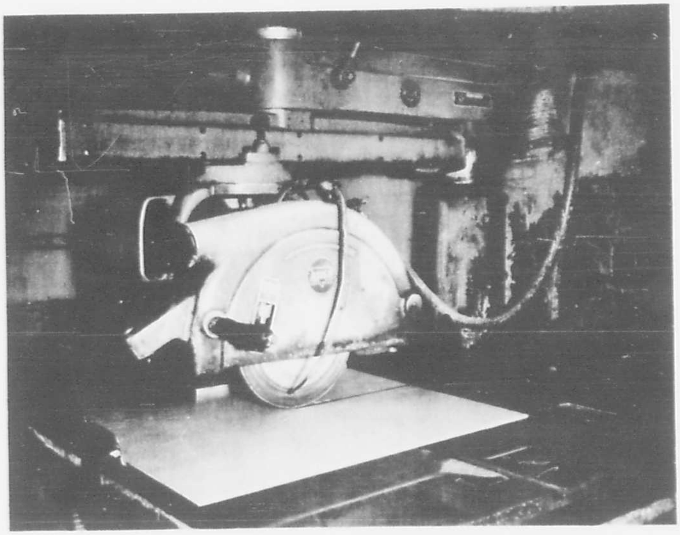


Figure 2. 1/8-in. Panel Being Cut Into Blanks on Diamond Saw

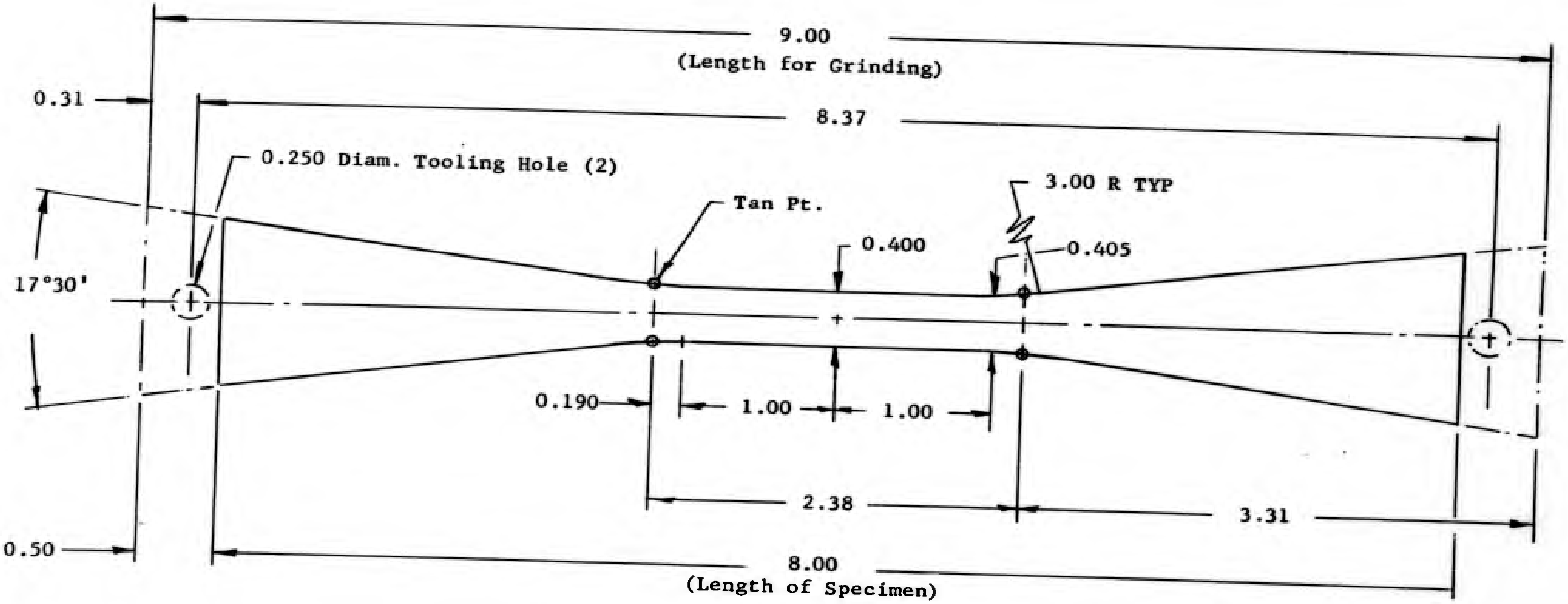


Figure 3. Tensile Test Specimen Dimensions

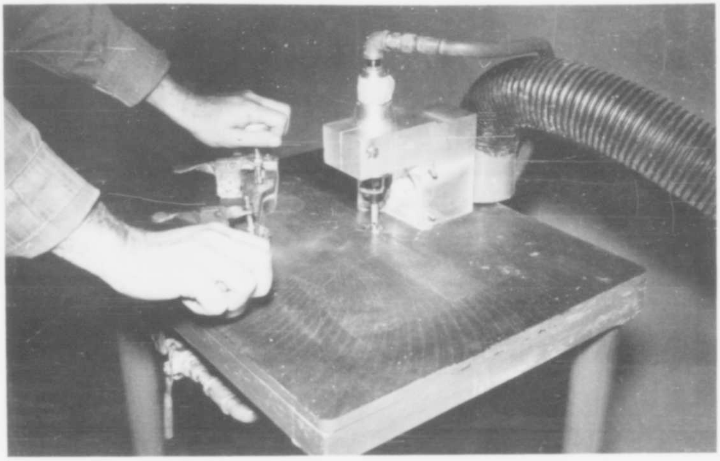


Figure 4. Air-Driven Router and Fixture for Tensile Specimens

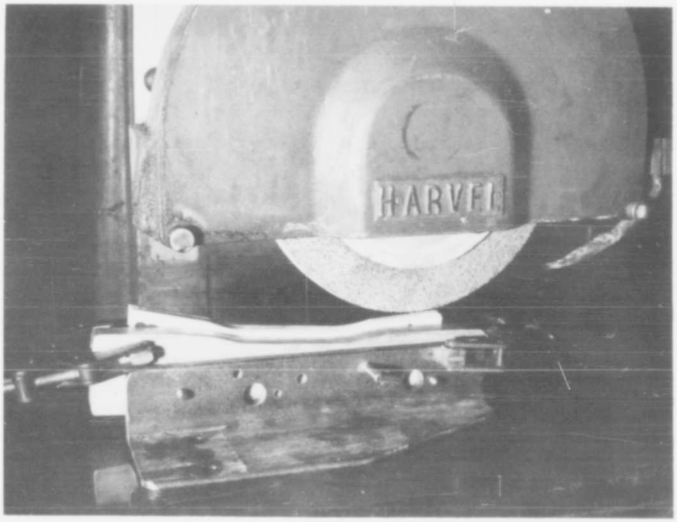


Figure 5. Tensile Specimens Ready for Grinding Paper on Gripping Area

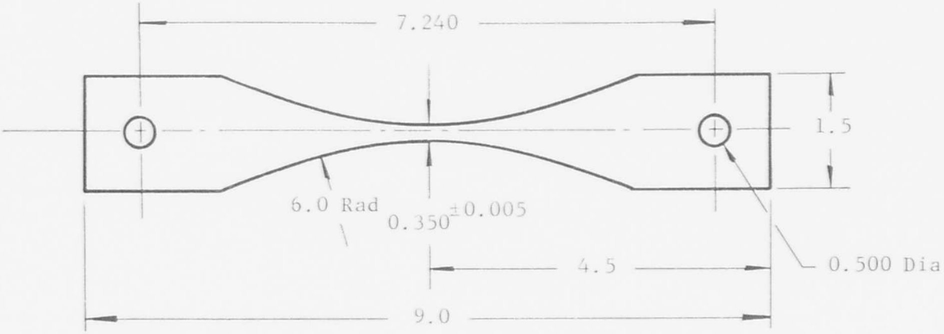


Figure 6. Tensile Fatigue Specimen Dimensions

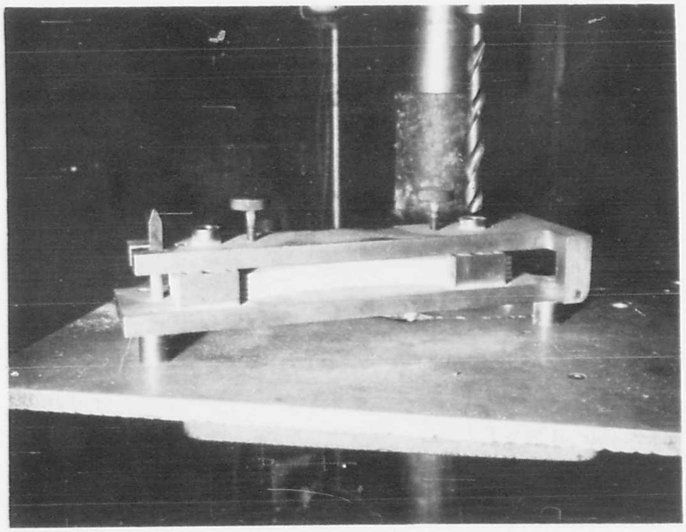


Figure 7. Reaming Holes in Fatigue Specimens

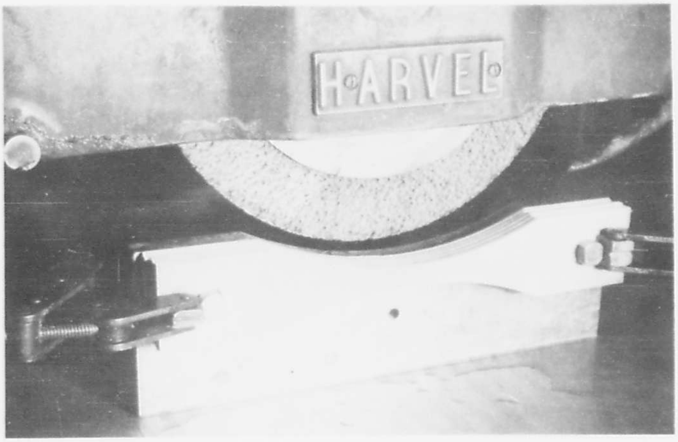


Figure 8. Fatigue Specimens Being Ground to Shape on Precision Surface Grinder

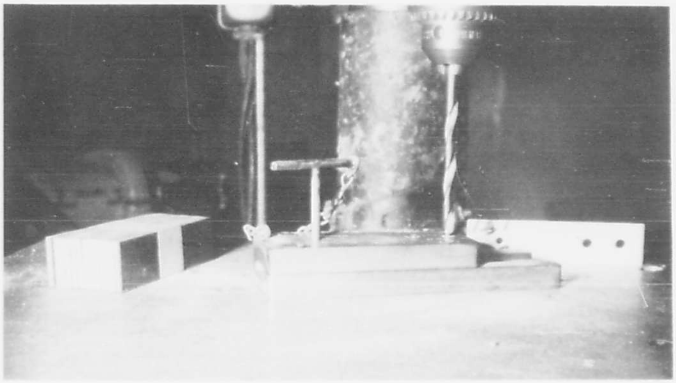


Figure 9. Reaming Holes in Bearing Strength Test Specimen

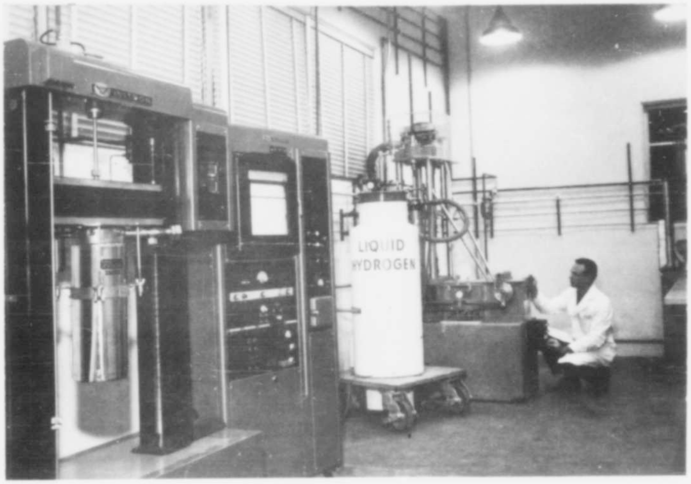


Figure 10. General Arrangement of Narmco's Cryogenic Laboratory

Figure 11. Instron Test Machine and Cryostat Ready for a Liquid Hydrogen Test

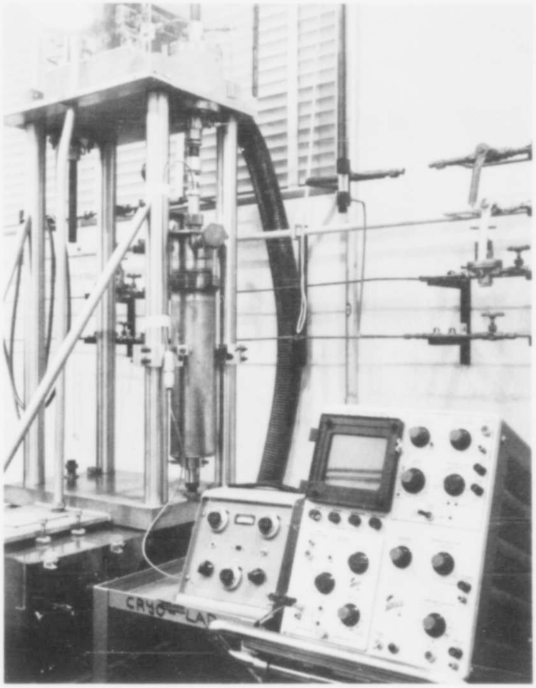


Figure 12. The Fatigue Machine with its Cryostat Installed. The Load Cell and Readout Equipment are in Place. The Load Cell is Visible Immediately Above the Cryostat, and Forms a Part of the Upper Load Rod

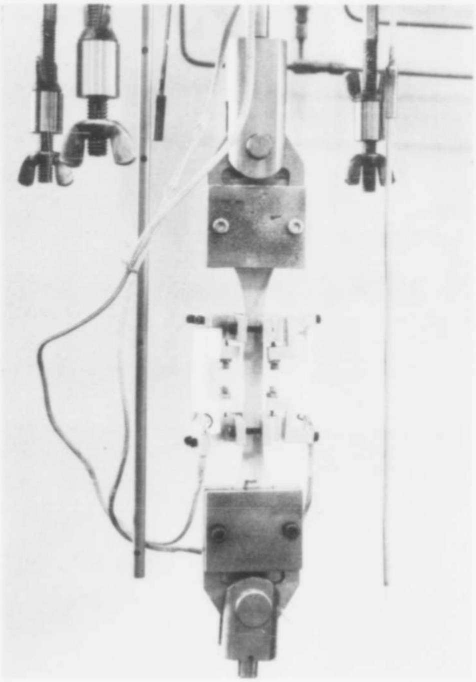


Figure 13. A Typical Tensile Test Specimen Installed in the Test Machine with the Extensometer in Place

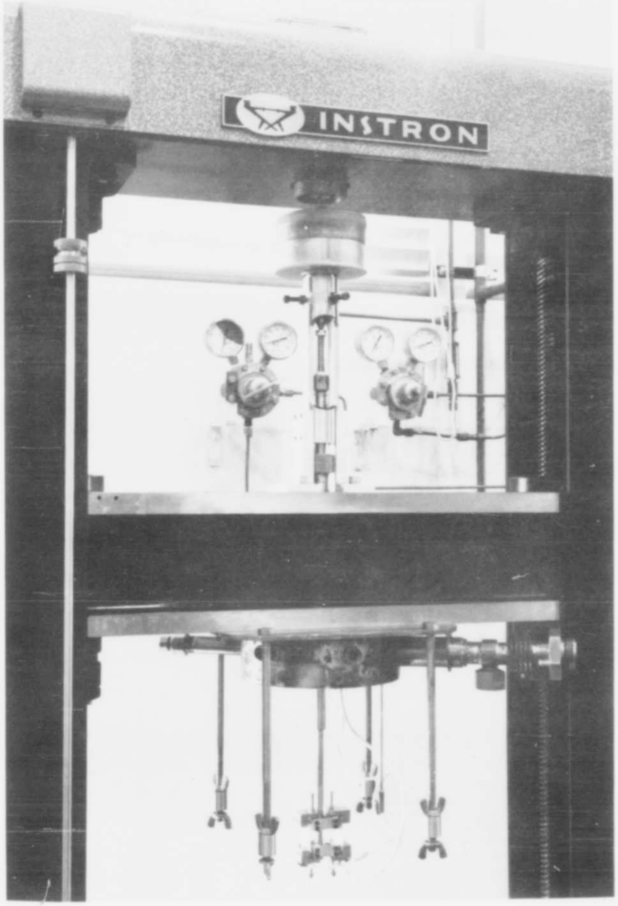


Figure 14. The Precision Calibration Device with a Strain Gage Extensometer Installed. For Low-Temperature Calibration, the Cryostat is Installed and the Extensometer Submerged in the Appropriate Cryogen

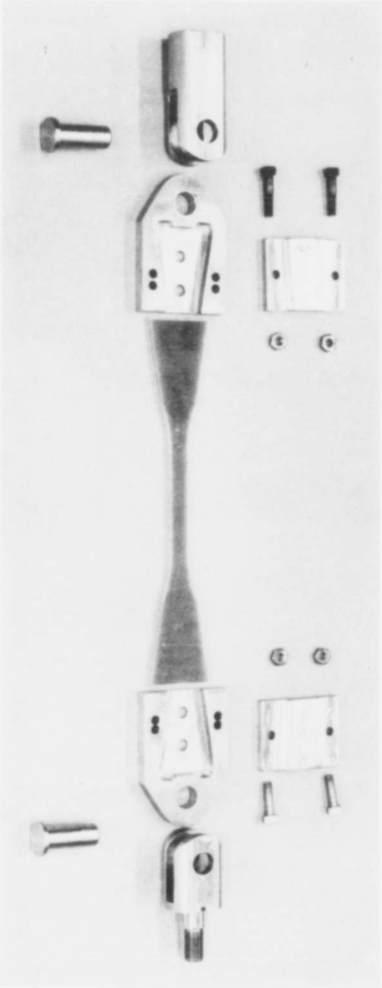


Figure 15. A Typical Tensile Specimen and the Titanium Tensile Grips

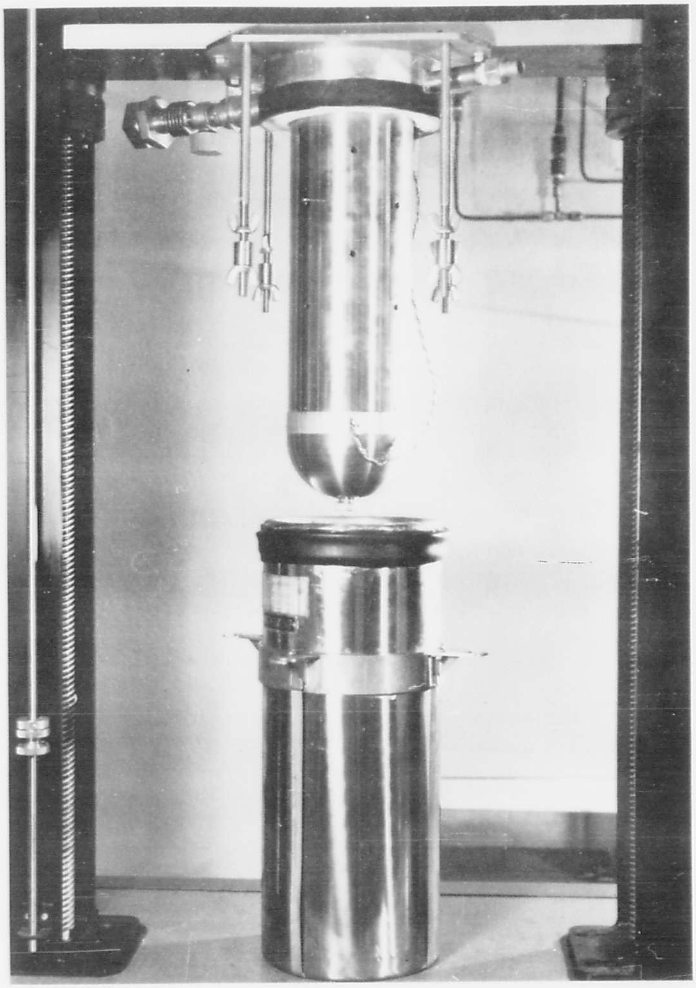


Figure 16. Partially Disassembled Cryostat Showing Load Cylinder and Cup in Place

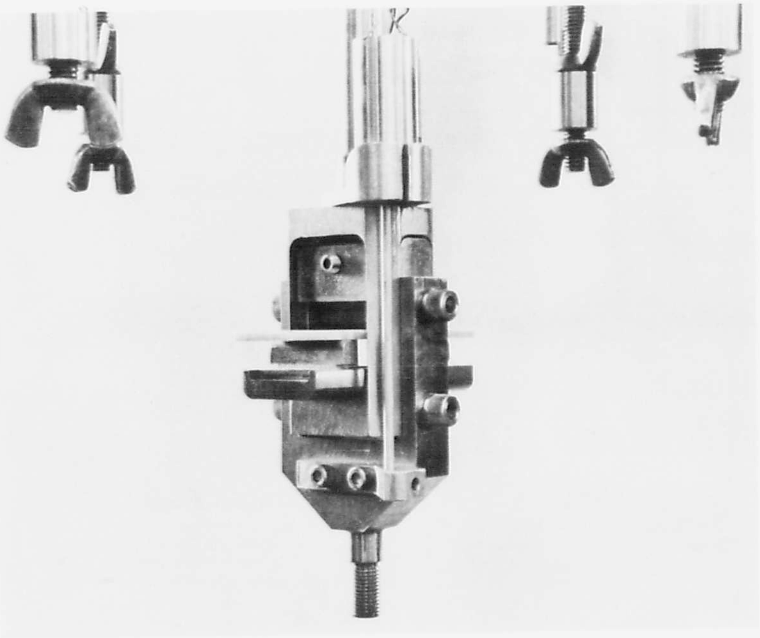


Figure 17. A Flexure Test Specimen Installed in the Test Fixture. The LVDT Type Extensometer is Mounted on the Upper Portion of the Fixture

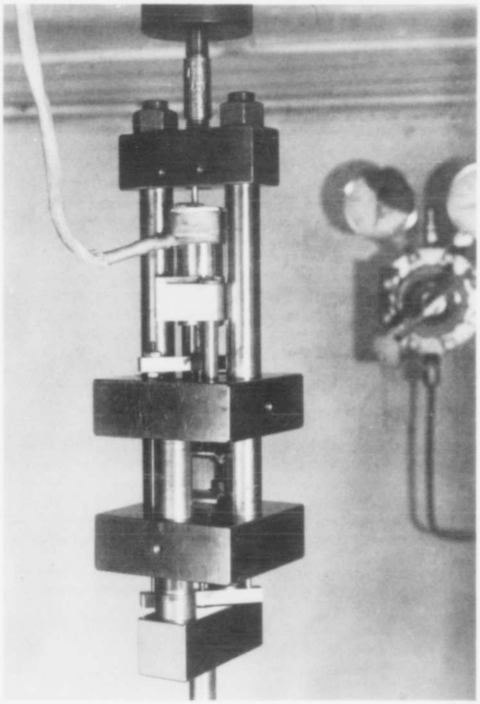


Figure 18. Compression Test Fixture with Test Specimen Installed. The Specimen is Visible Between the Two Large Square Plates. The Extensometer is an Integral, Axially Mounted LVDT Type, and can be seen in the Upper Part of the Fixture

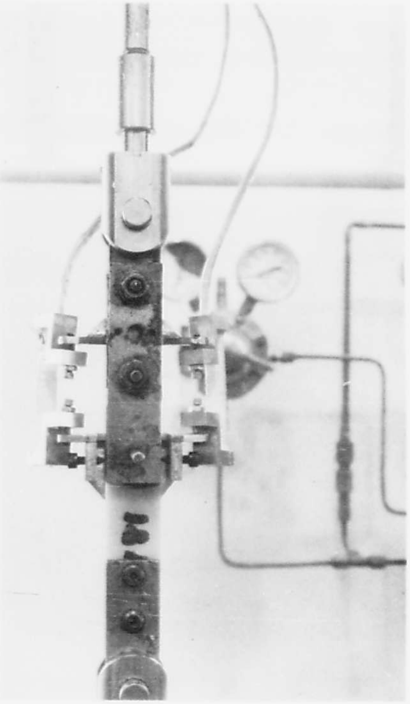


Figure 19. Typical Bearing Strength Specimen with Extensometer in Place Ready for Testing

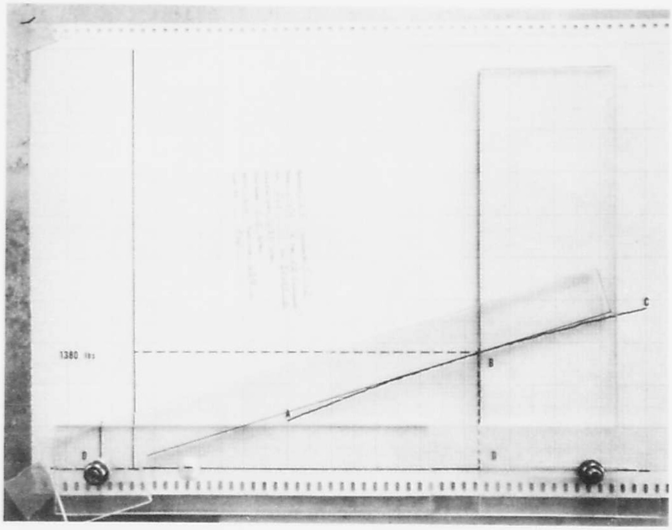


Figure 20. Plastic Template for Determining
Bearing Strength from Chart

Figure 21. Cryostat Being Installed in Fatigue Machine

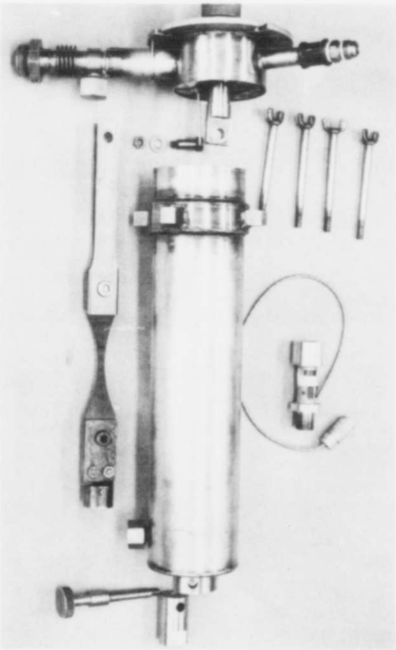


Figure 22. Disassembled Fatigue Cryostat and Test Specimen

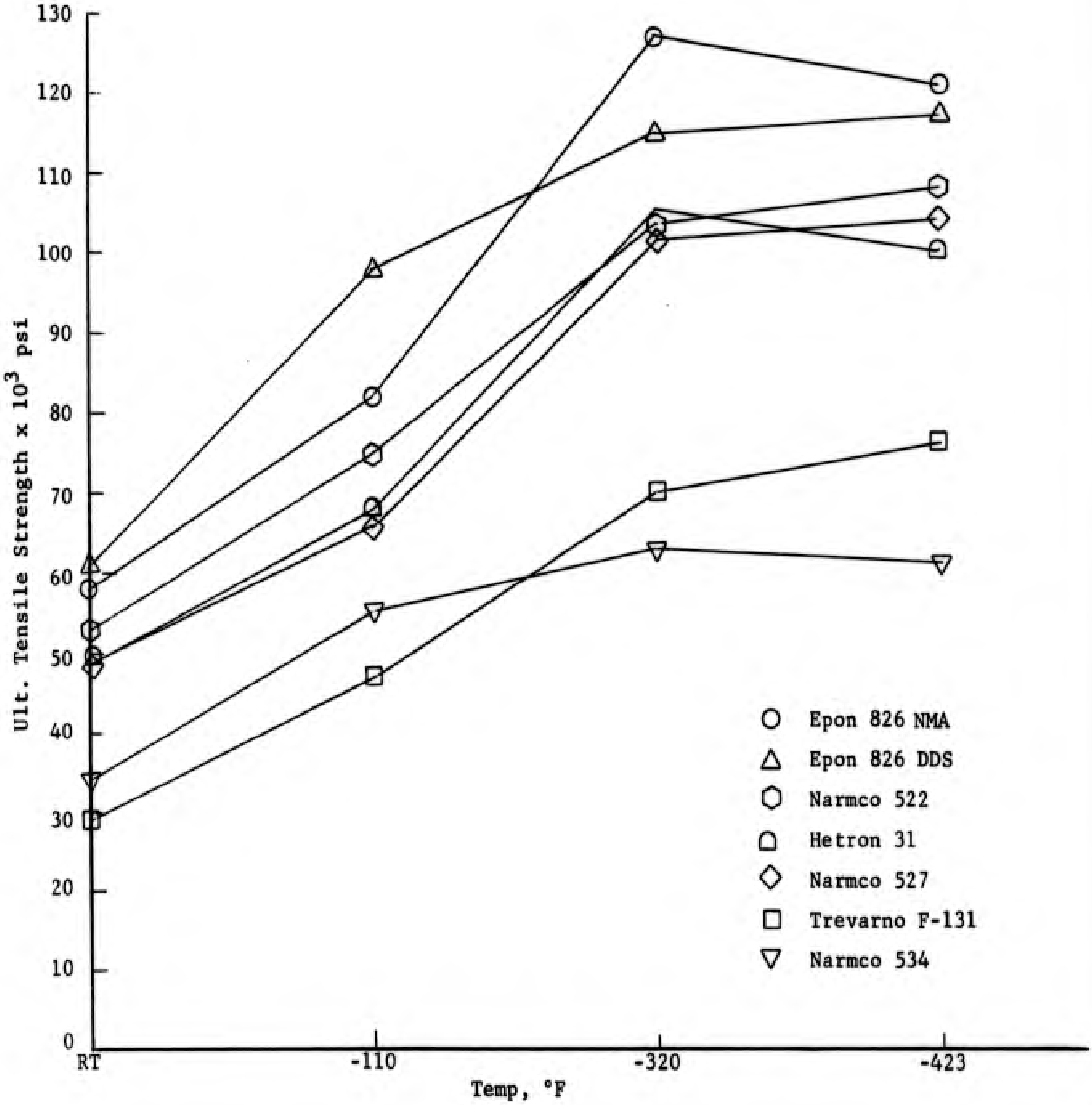


Figure 23. Average Tensile Strength of Phase I Materials

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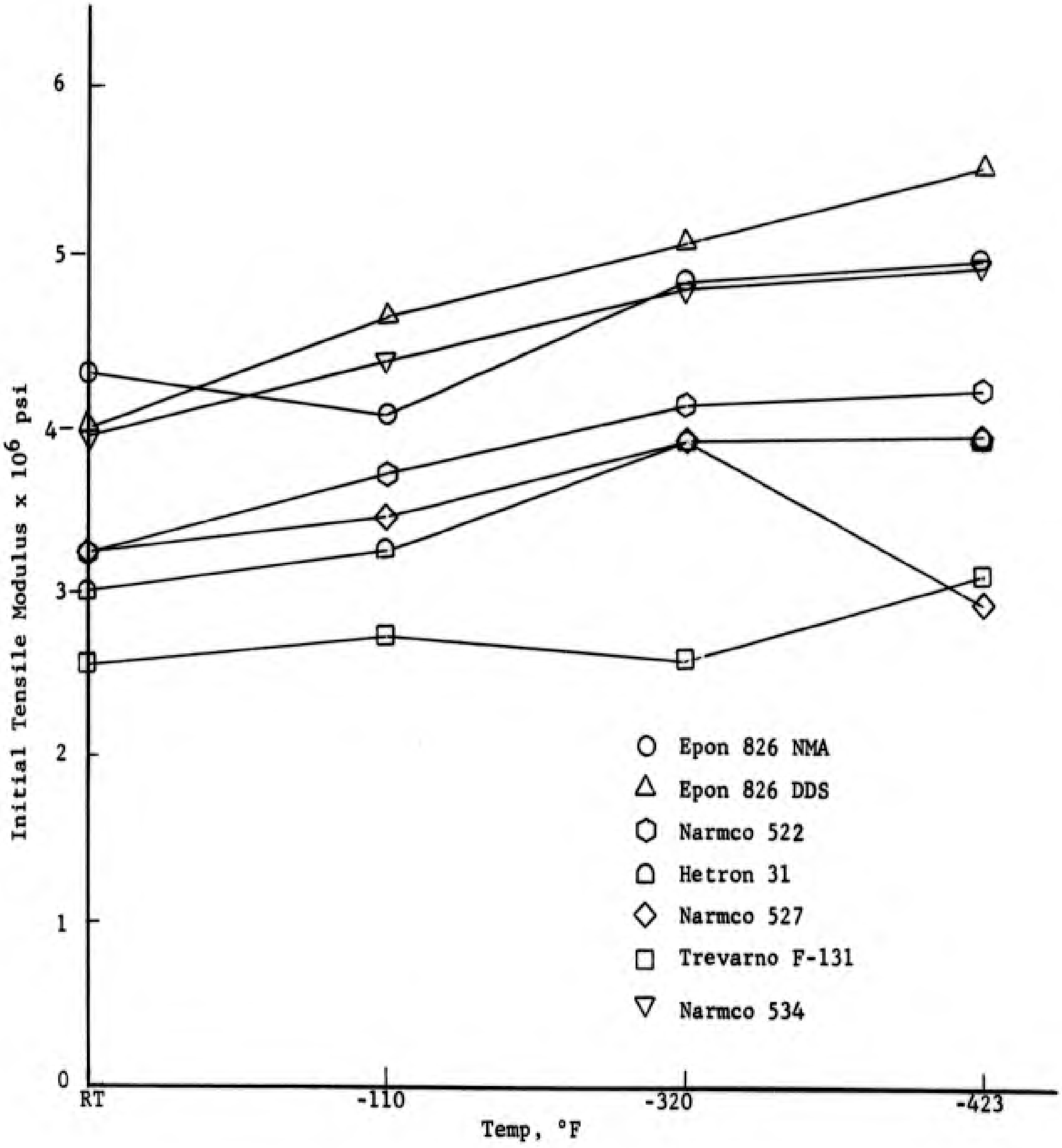


Figure 24. Average Initial Tensile Modulus of Phase I Materials

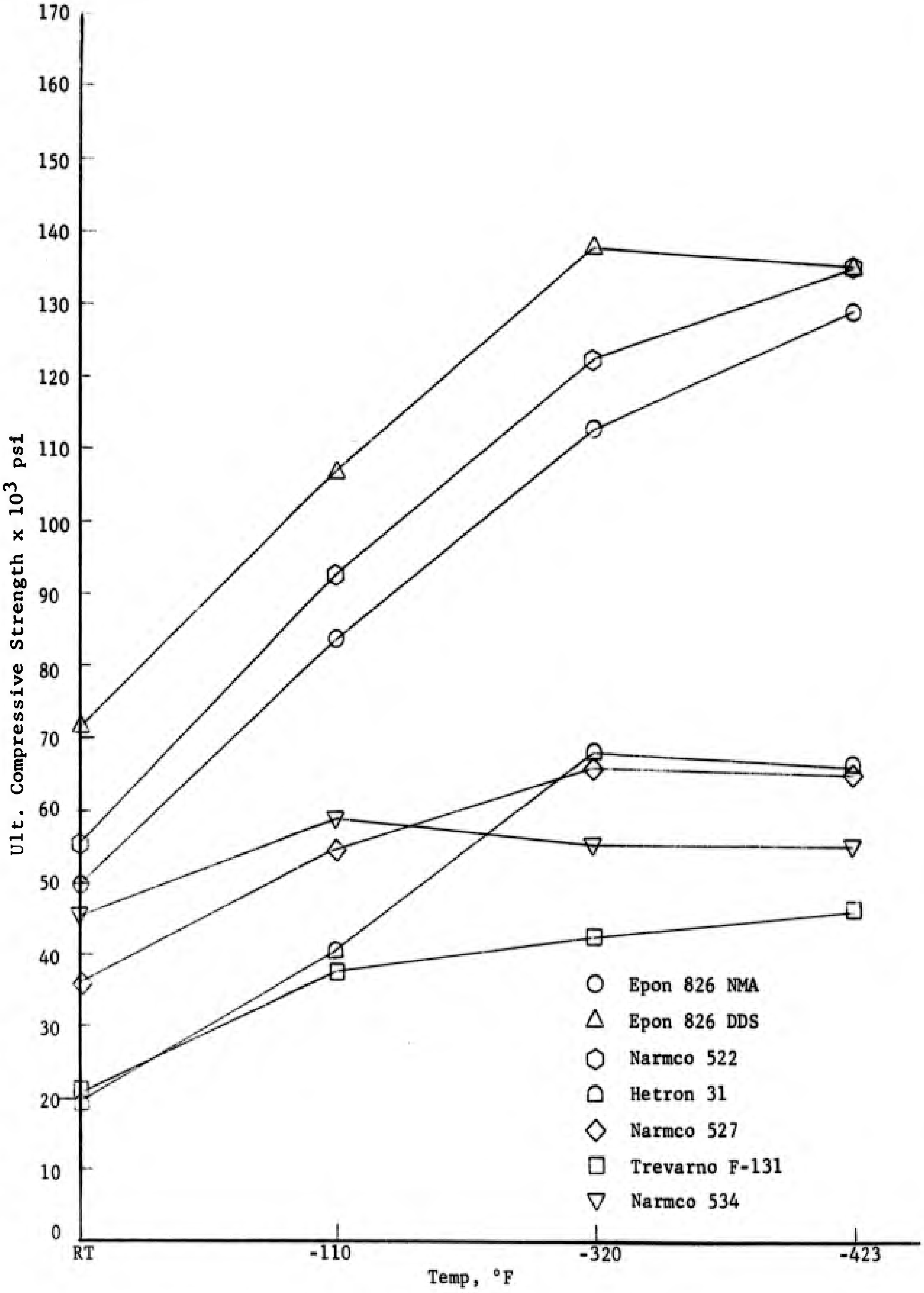


Figure 25. Average Ultimate Compressive Strength for Phase I Materials

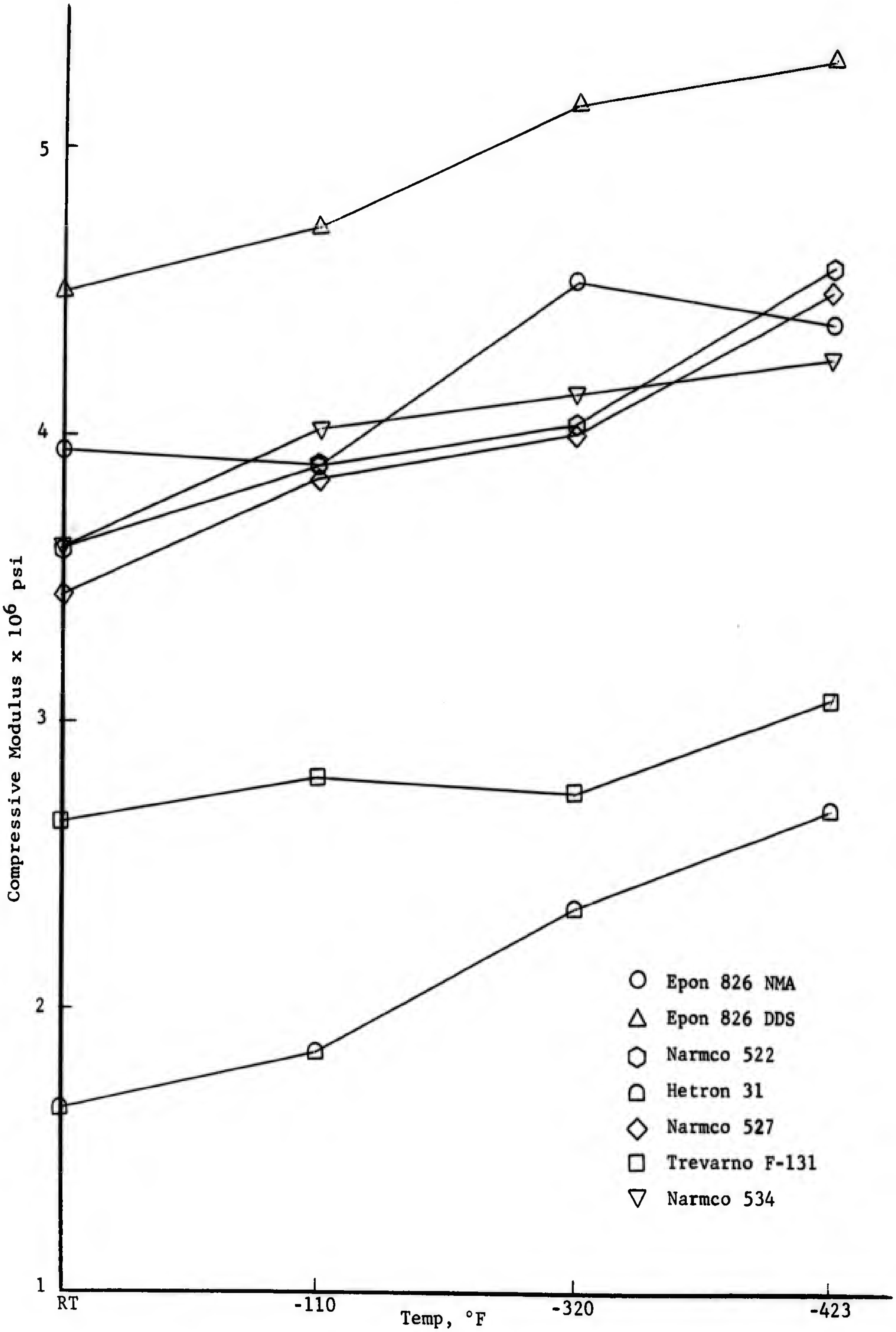


Figure 26. Average Compressive Modulus Values for Phase I Materials

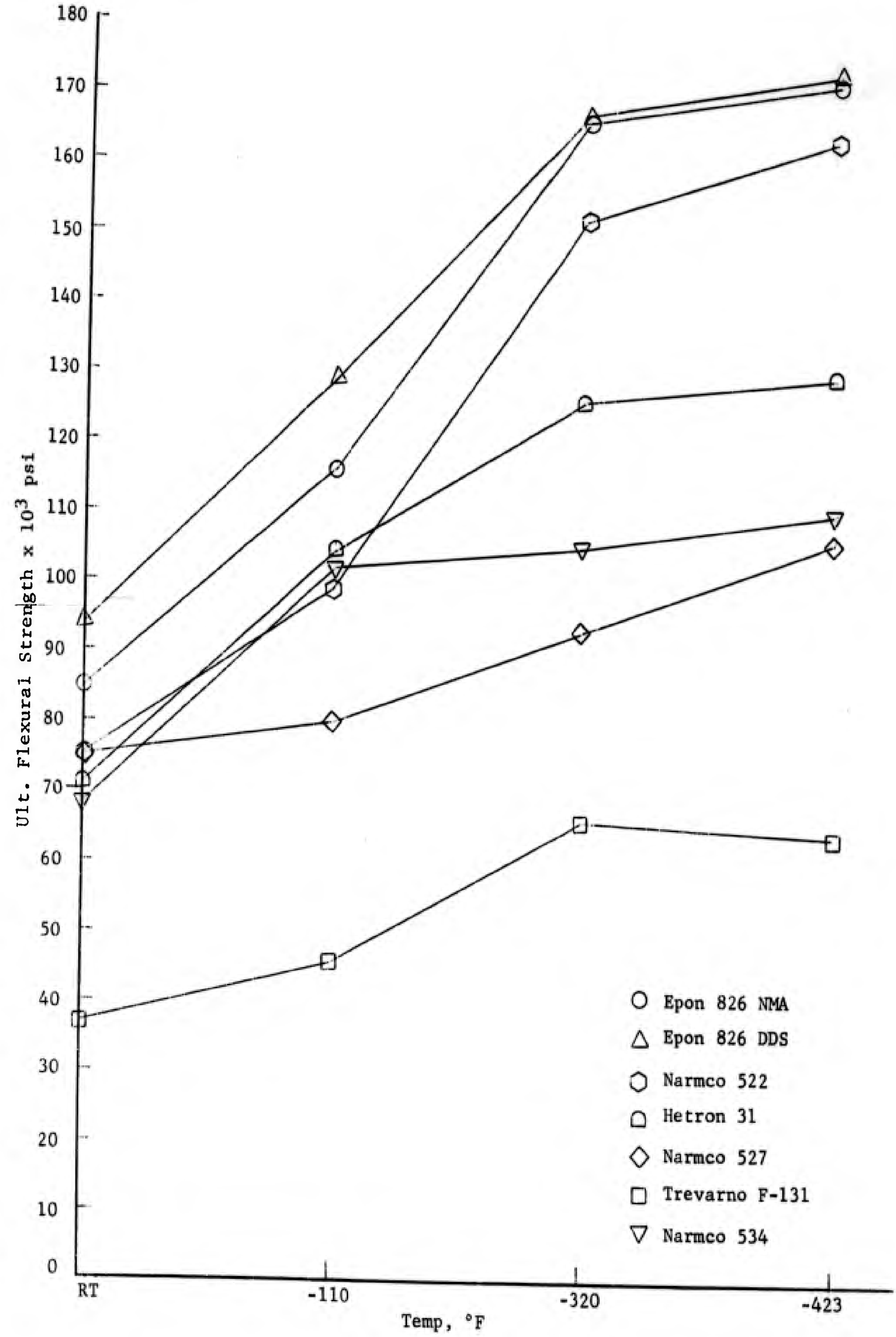


Figure 27. Ultimate Flexural Strength for Phase I Materials

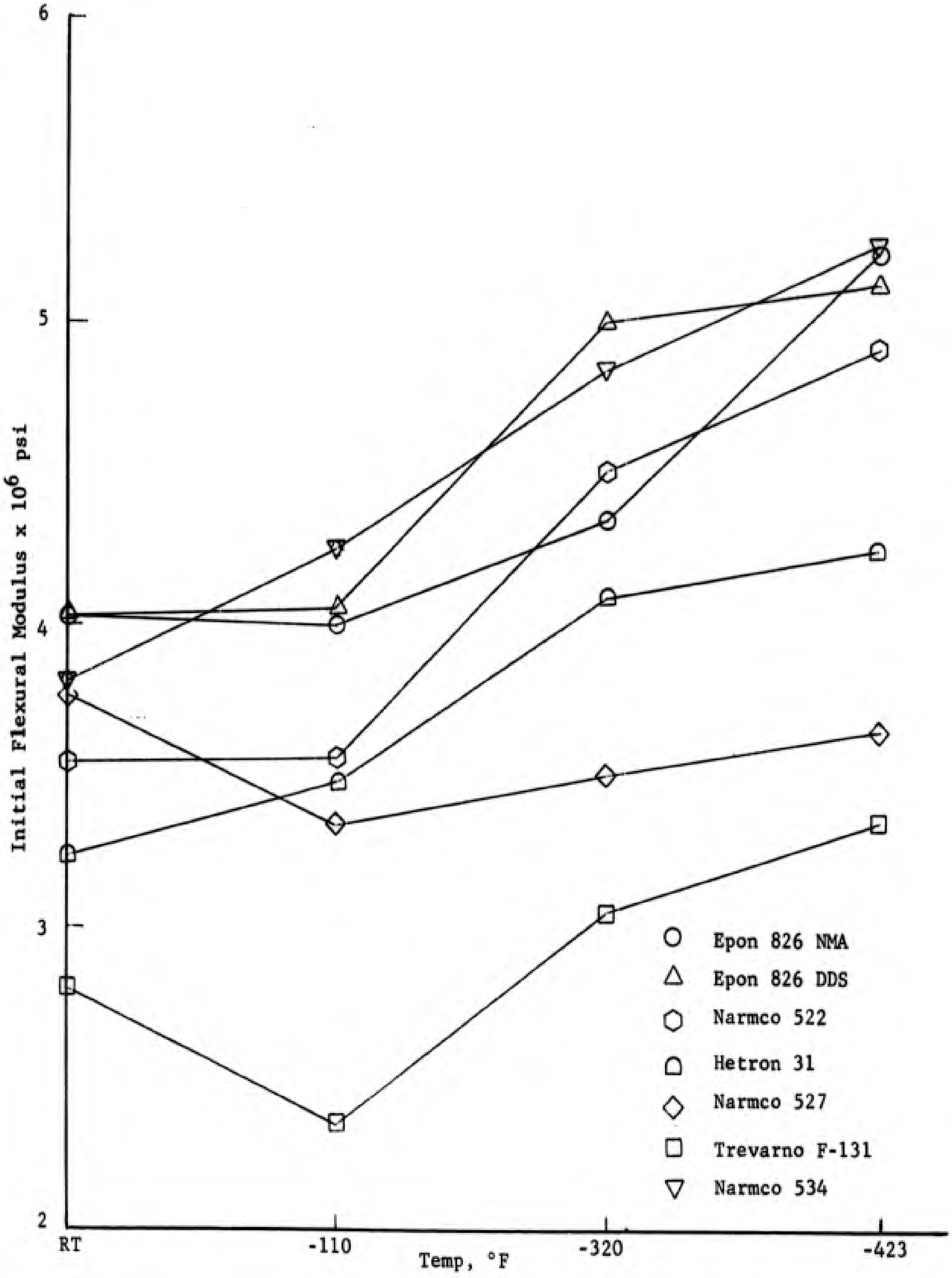


Figure 28. Initial Flexural Modulus for Phase I Materials

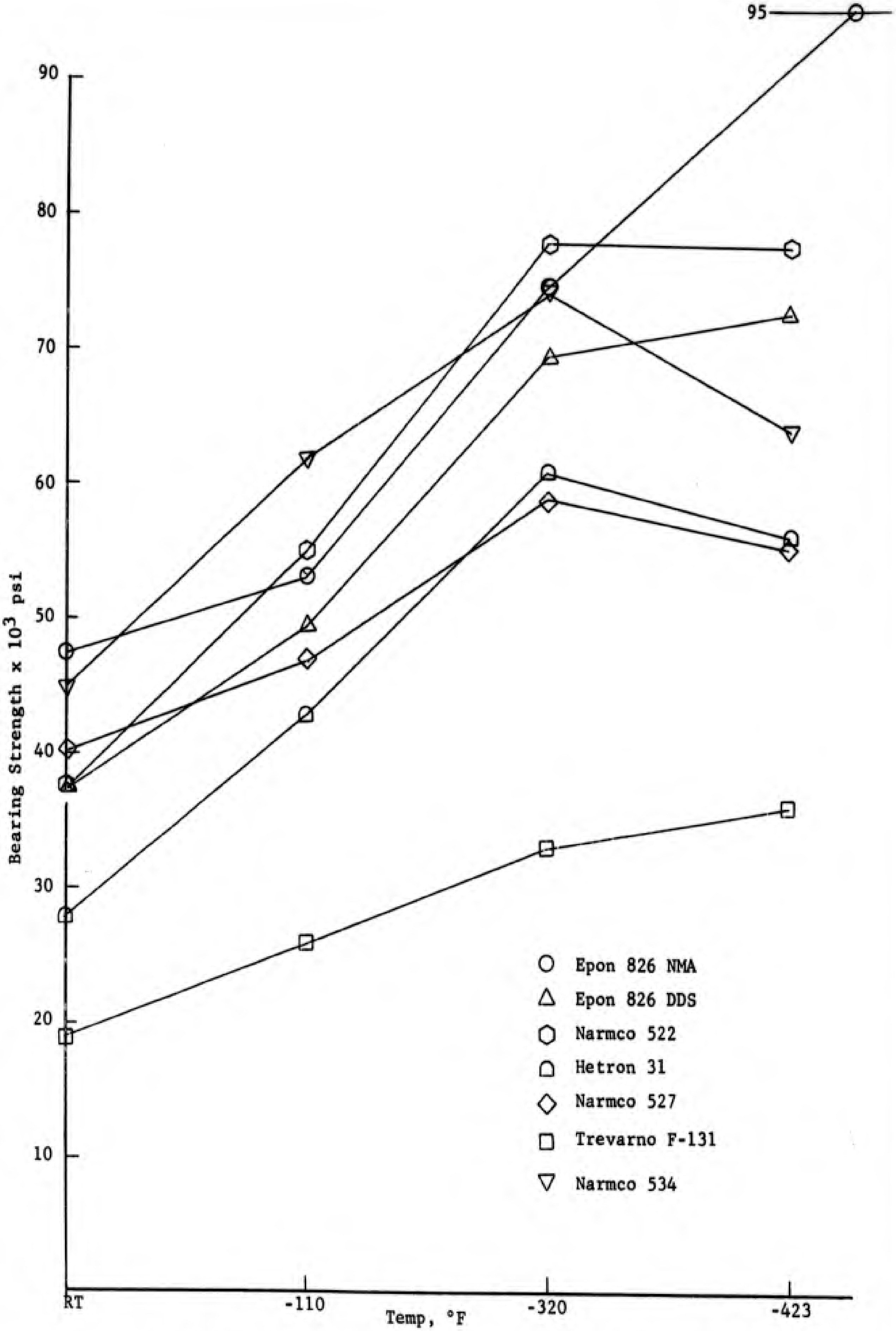


Figure 29. Average Bearing Strength for Phase I Materials

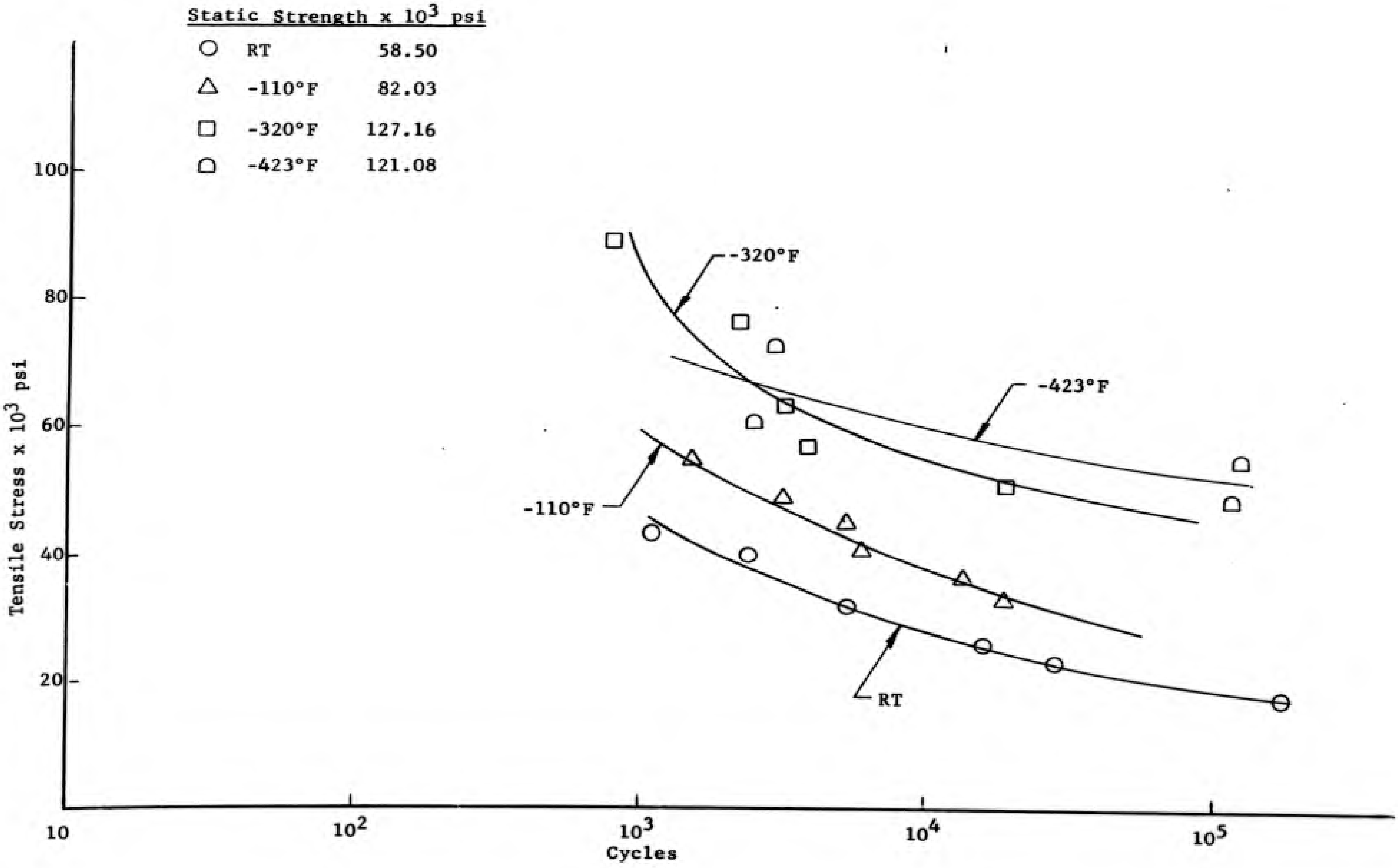


Figure 30. S-N Diagram of Epon 826/NMA-181

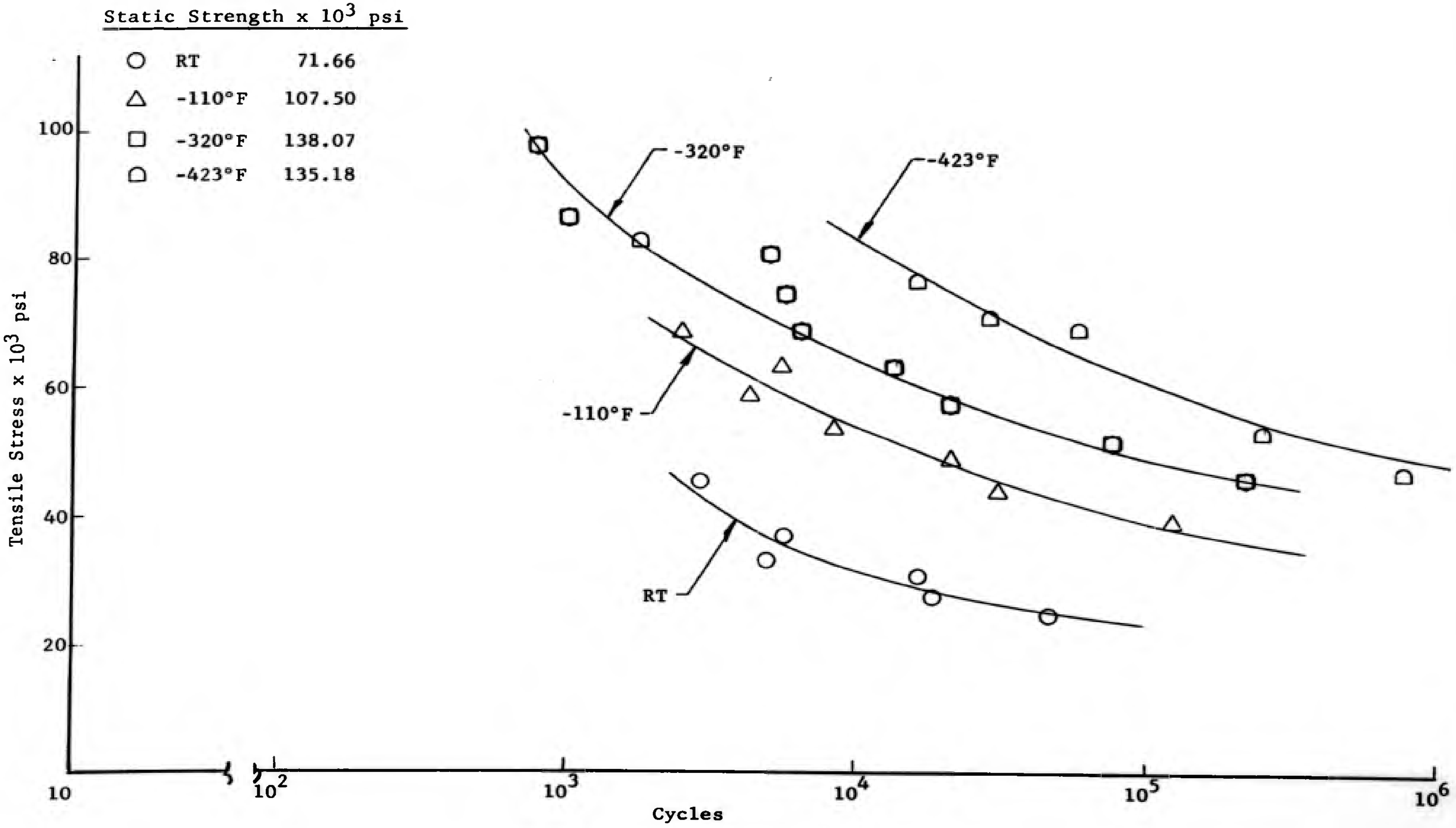


Figure 31. S-N Diagram of Epon 826/DDS-181

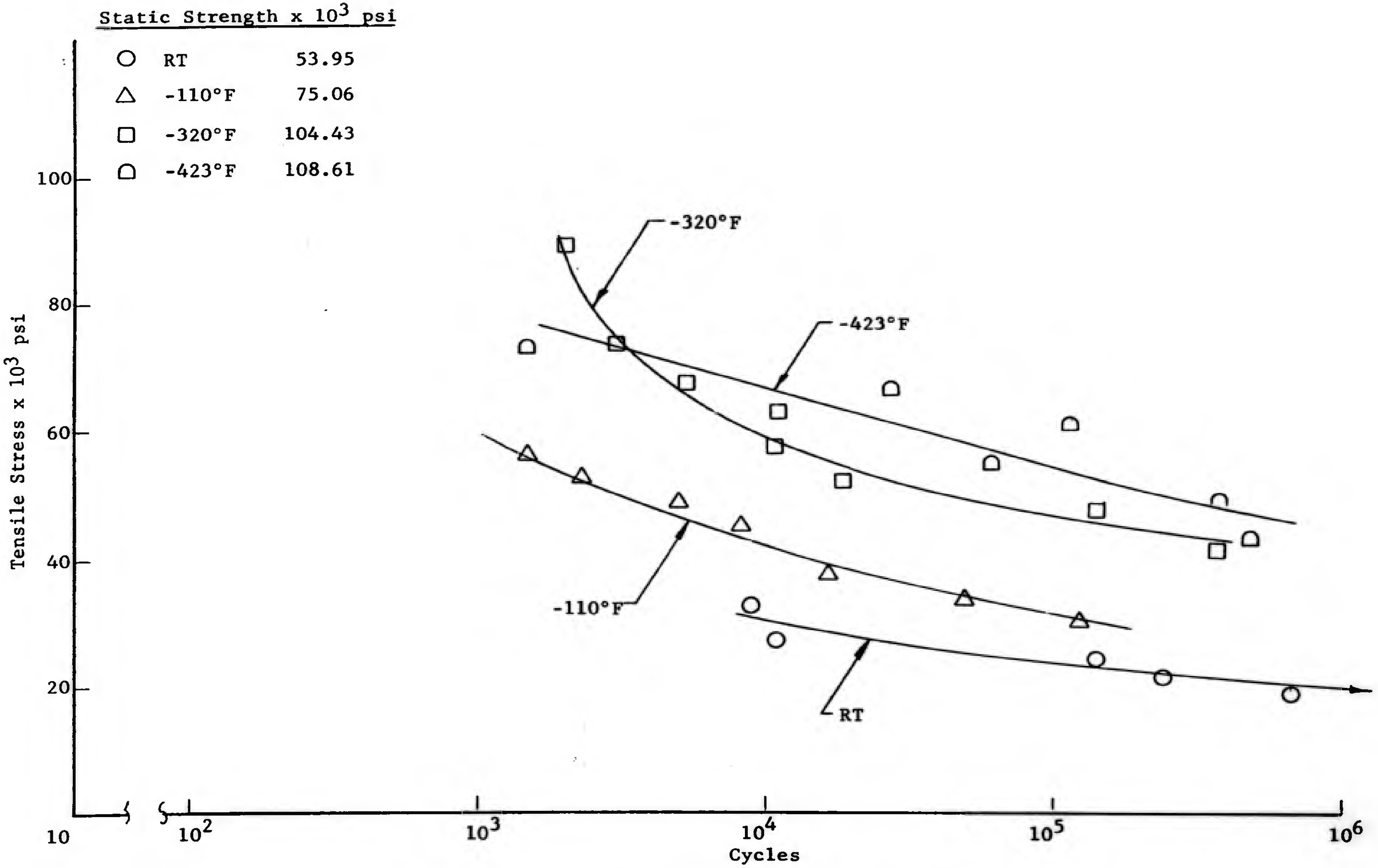


Figure 32. S-N Diagram of Narmco 522-181

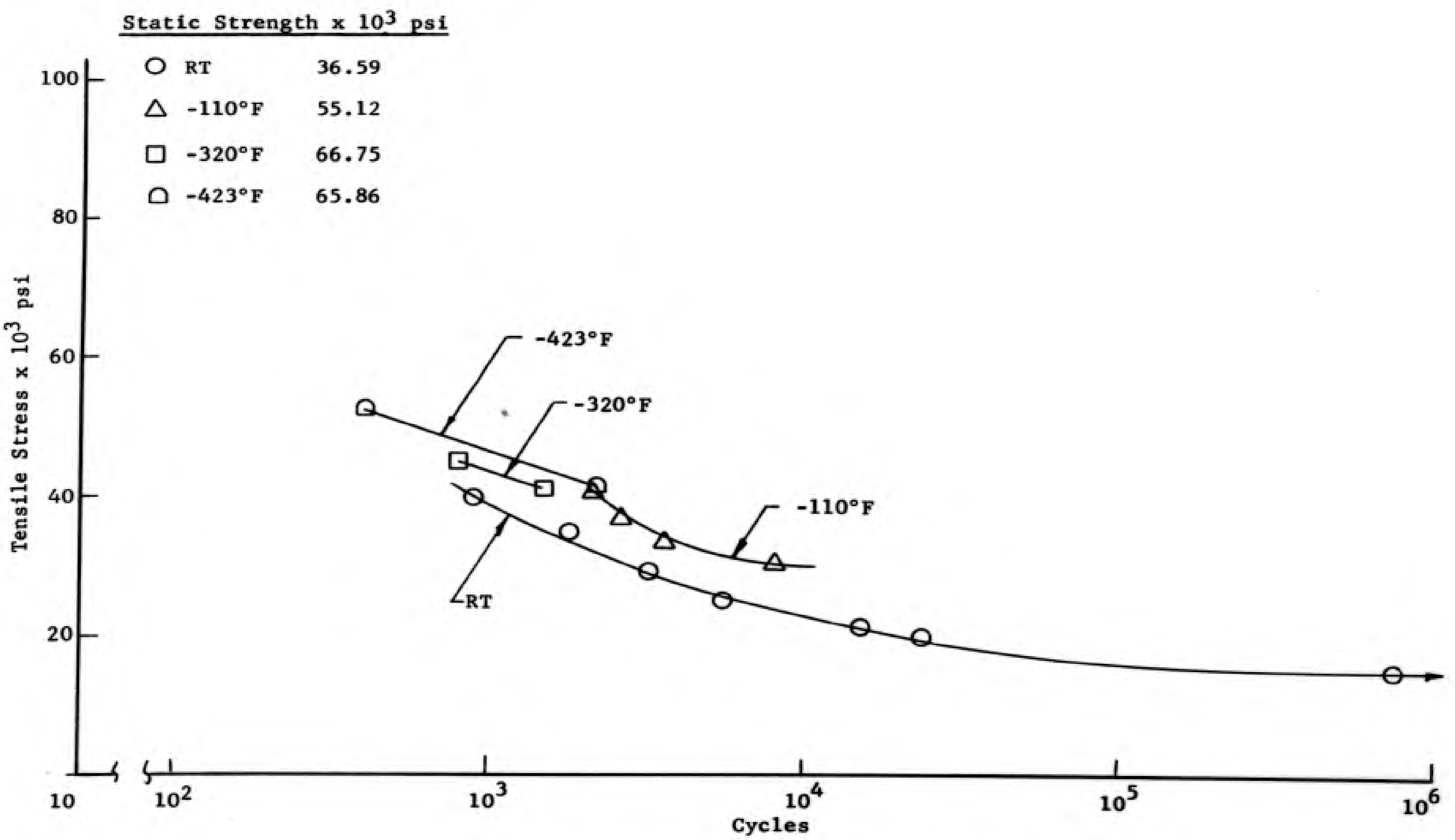


Figure 33. S-N Diagram of Narmco 527-181

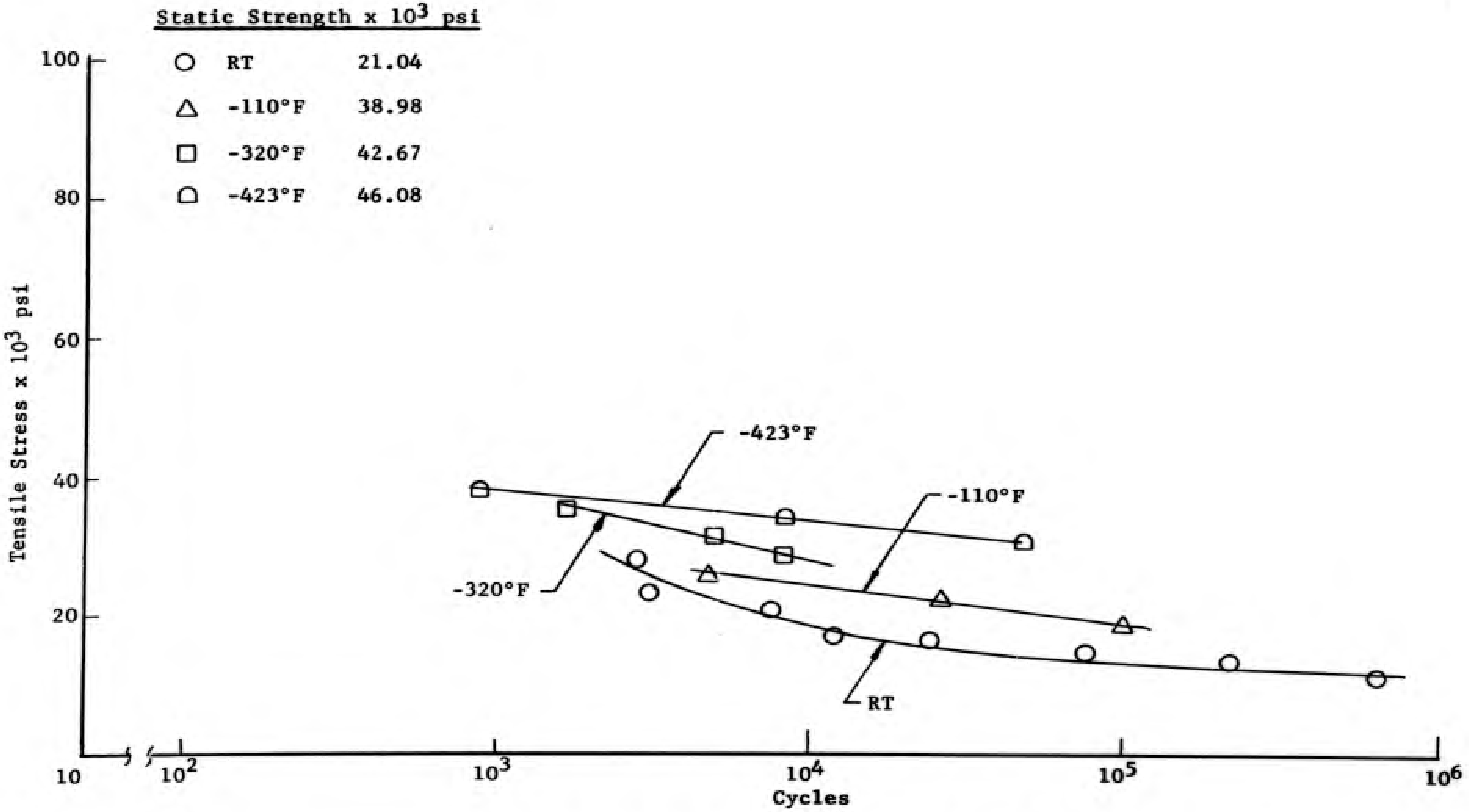


Figure 34. S-N Diagram of Trevarno F-131-181

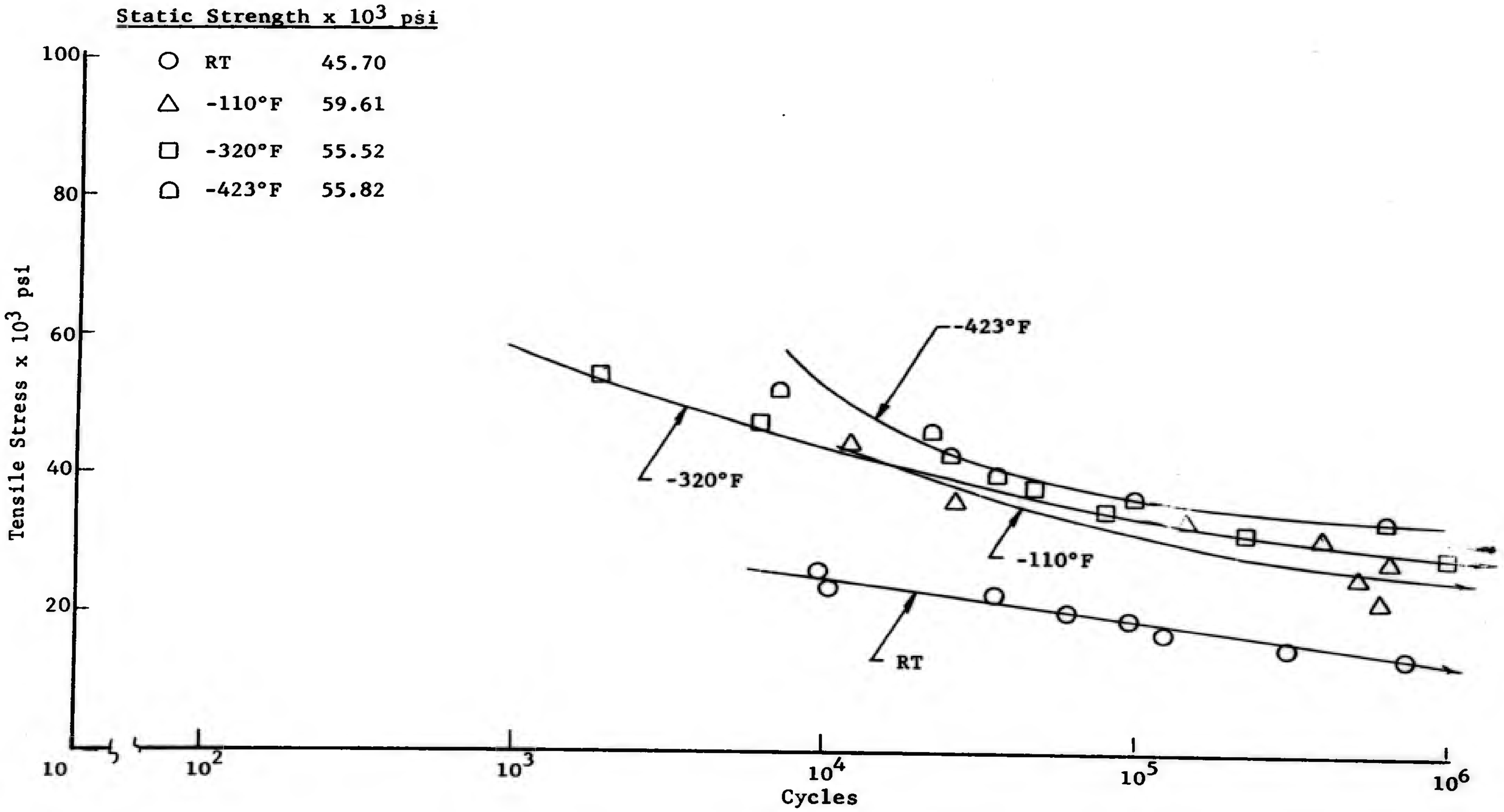


Figure 35. S-N Diagram of Narmco 534-181

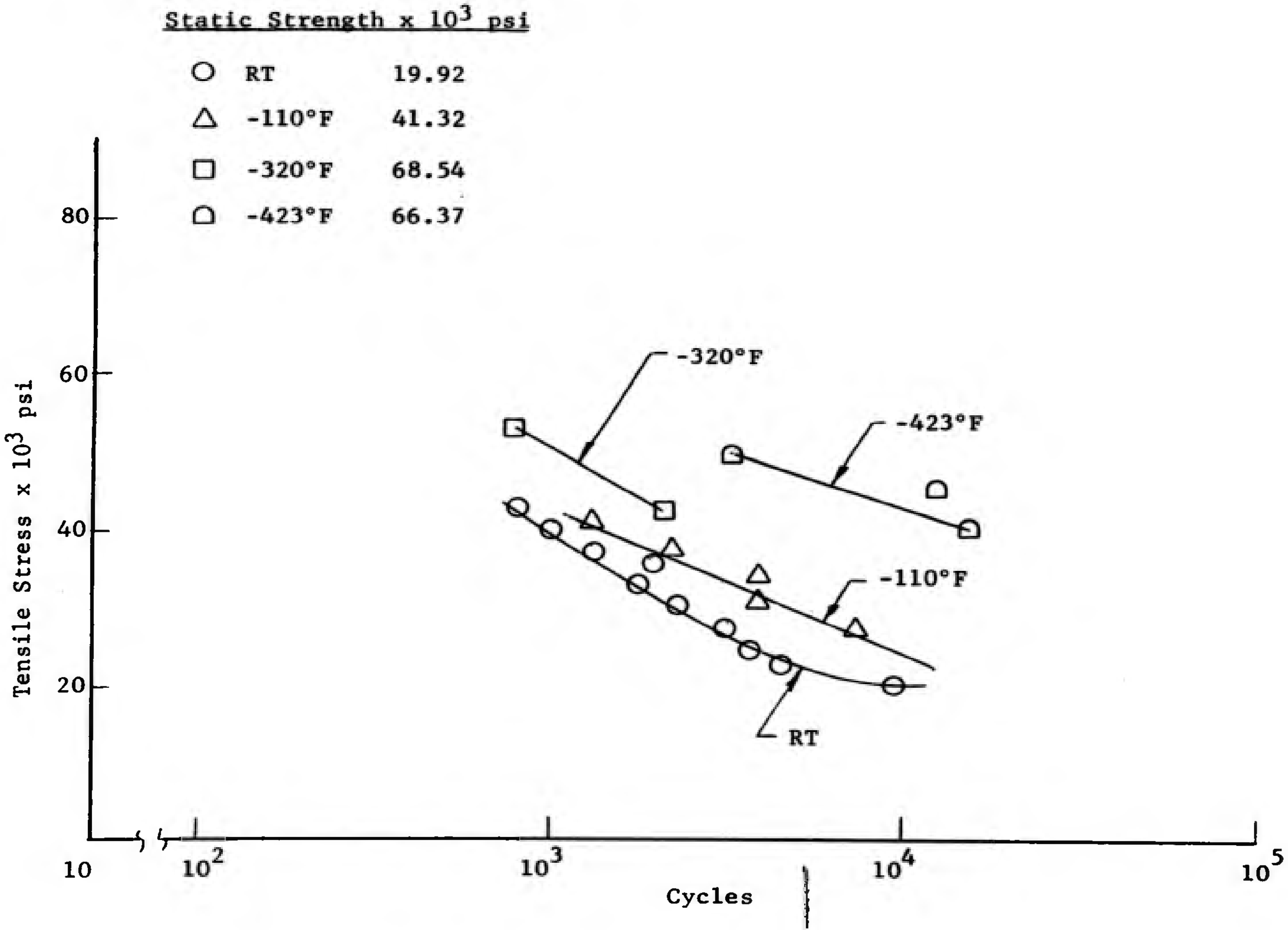


Figure 36. S-N Diagram of Hetron H-31-181

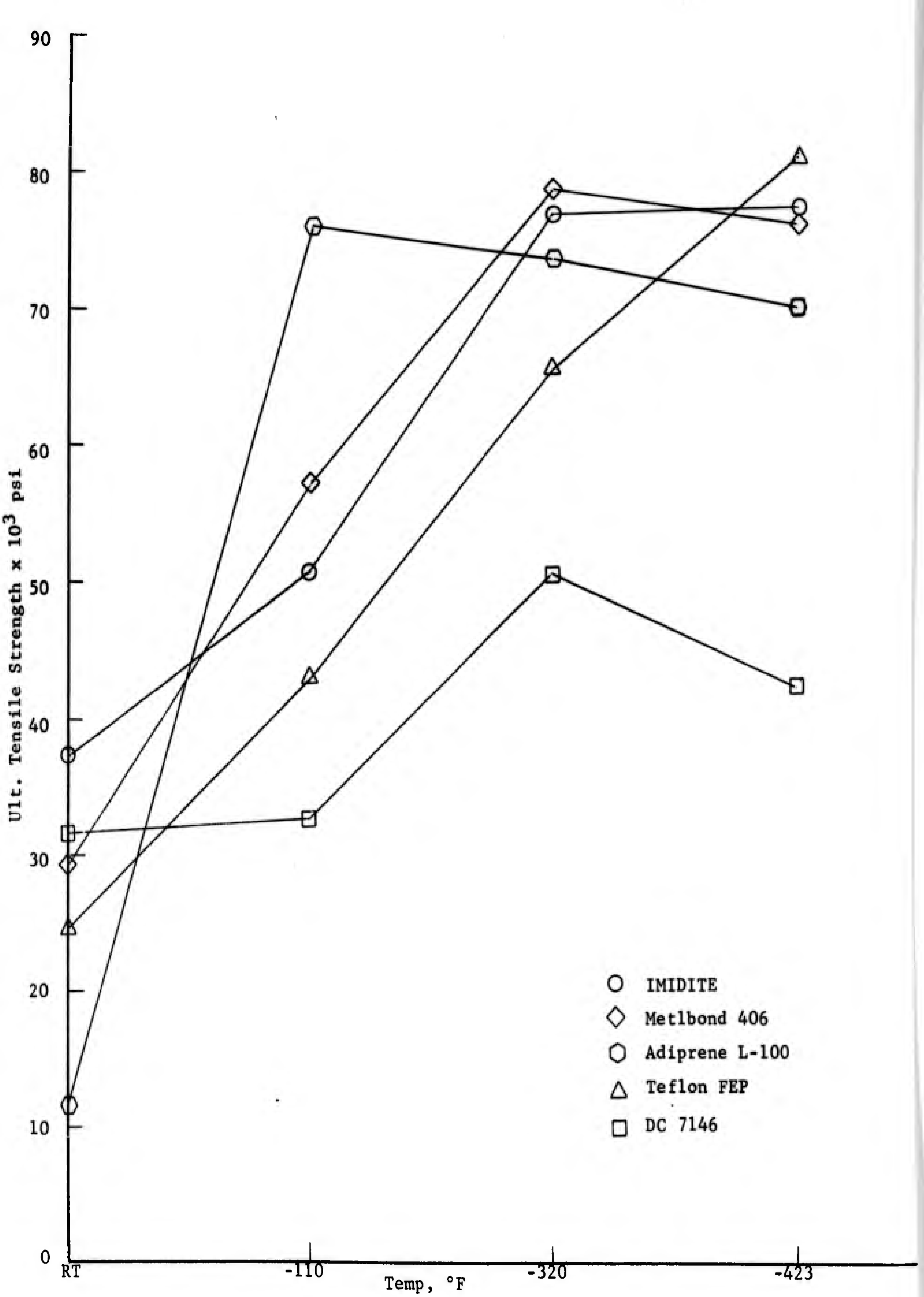


Figure 37. Ultimate Tensile Strength for Phase II Materials

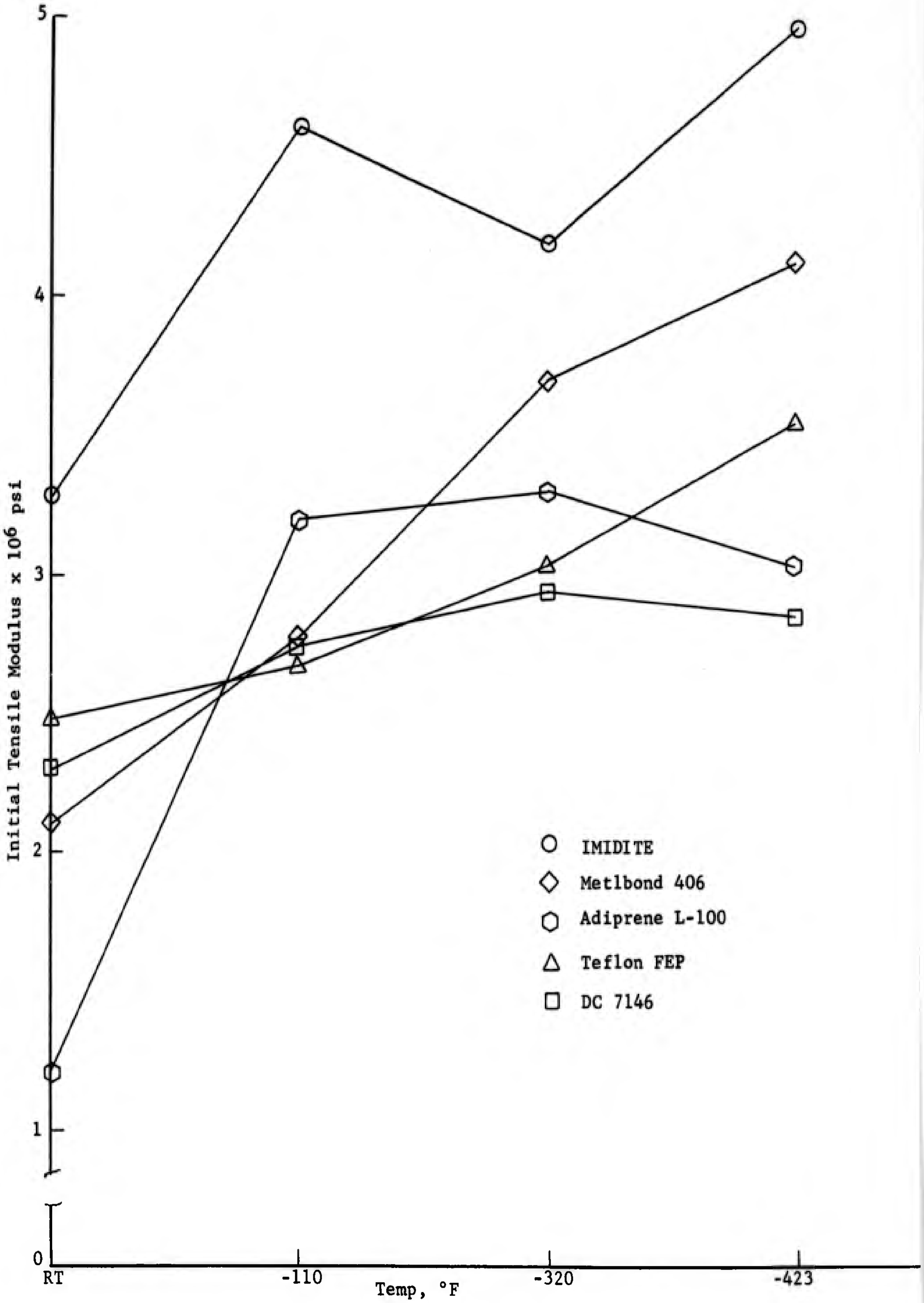


Figure 38. Initial Tensile Modulus for Phase II Materials

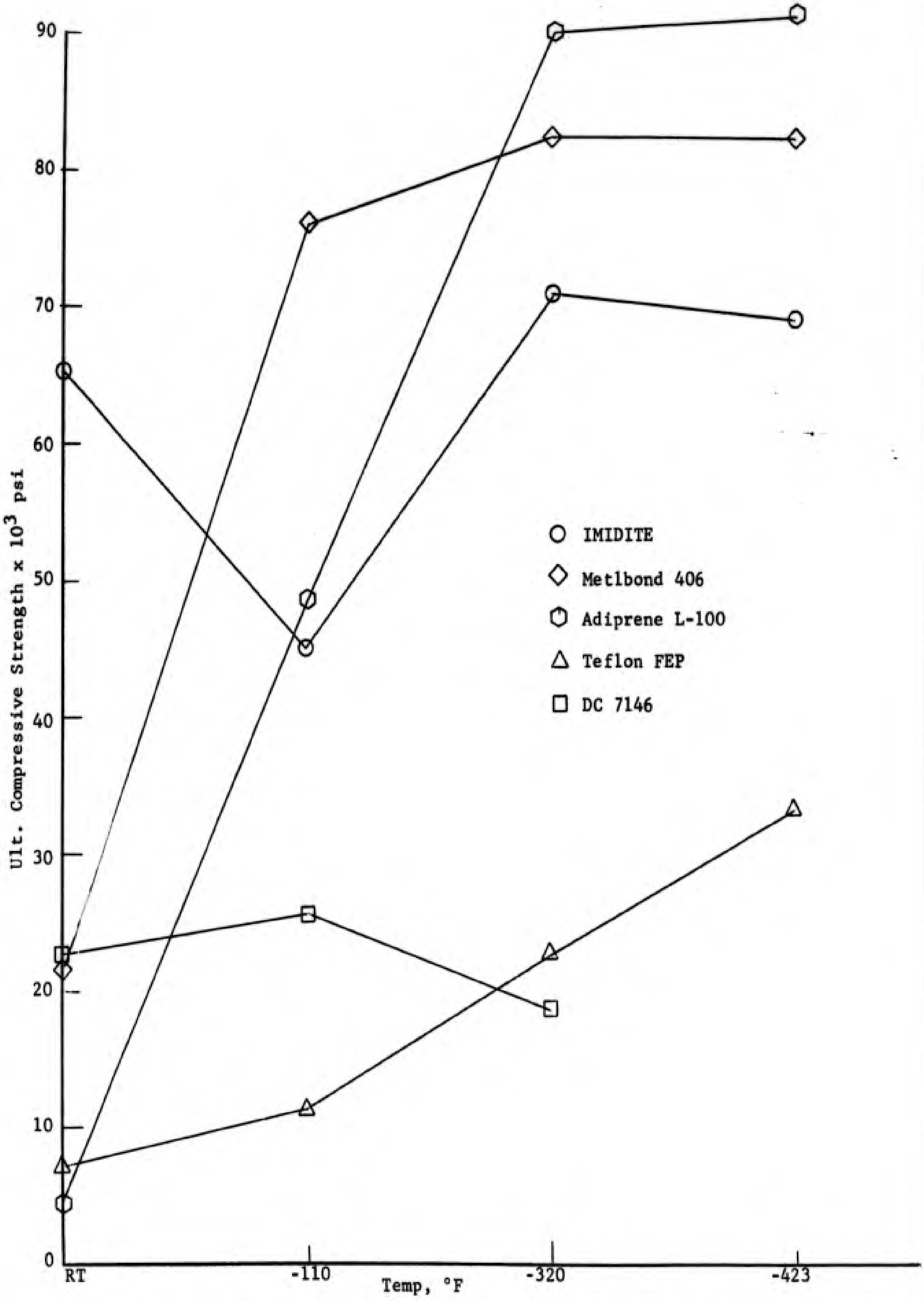


Figure 39. Ultimate Compressive Strength for Phase II Materials

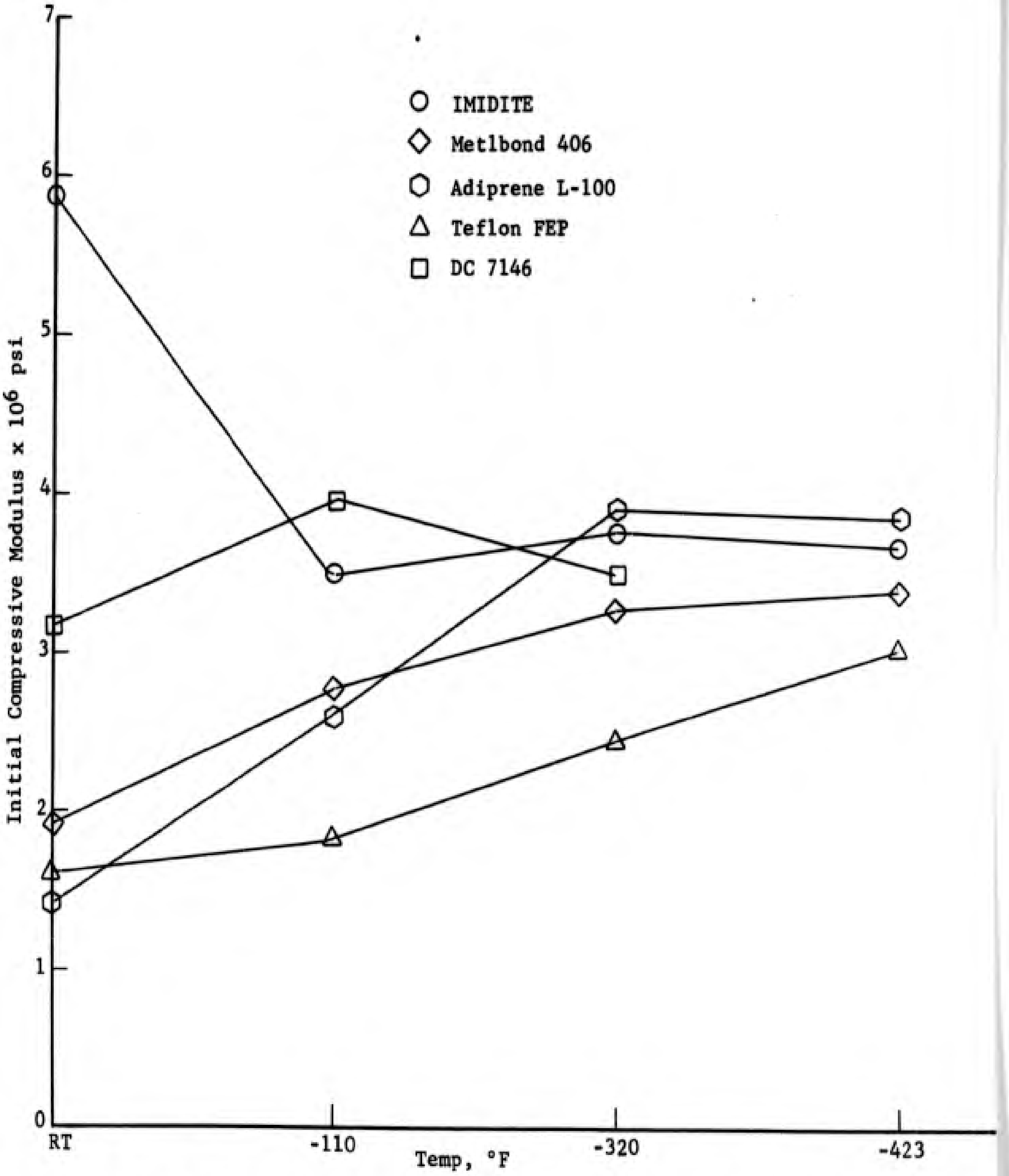


Figure 40. Initial Compressive Modulus for Phase II Materials

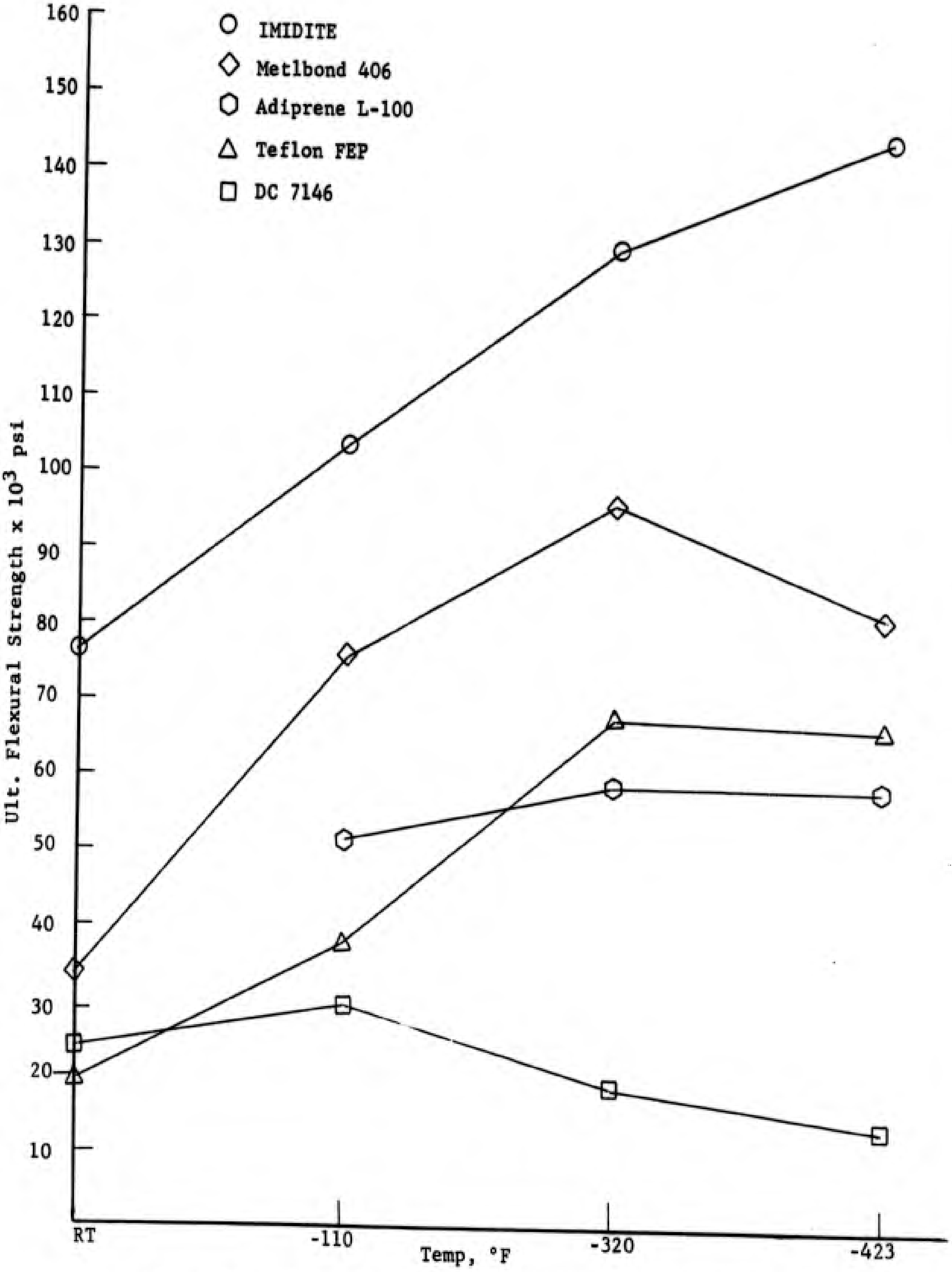


Figure 41. Ultimate Flexural Strength for Phase II Materials

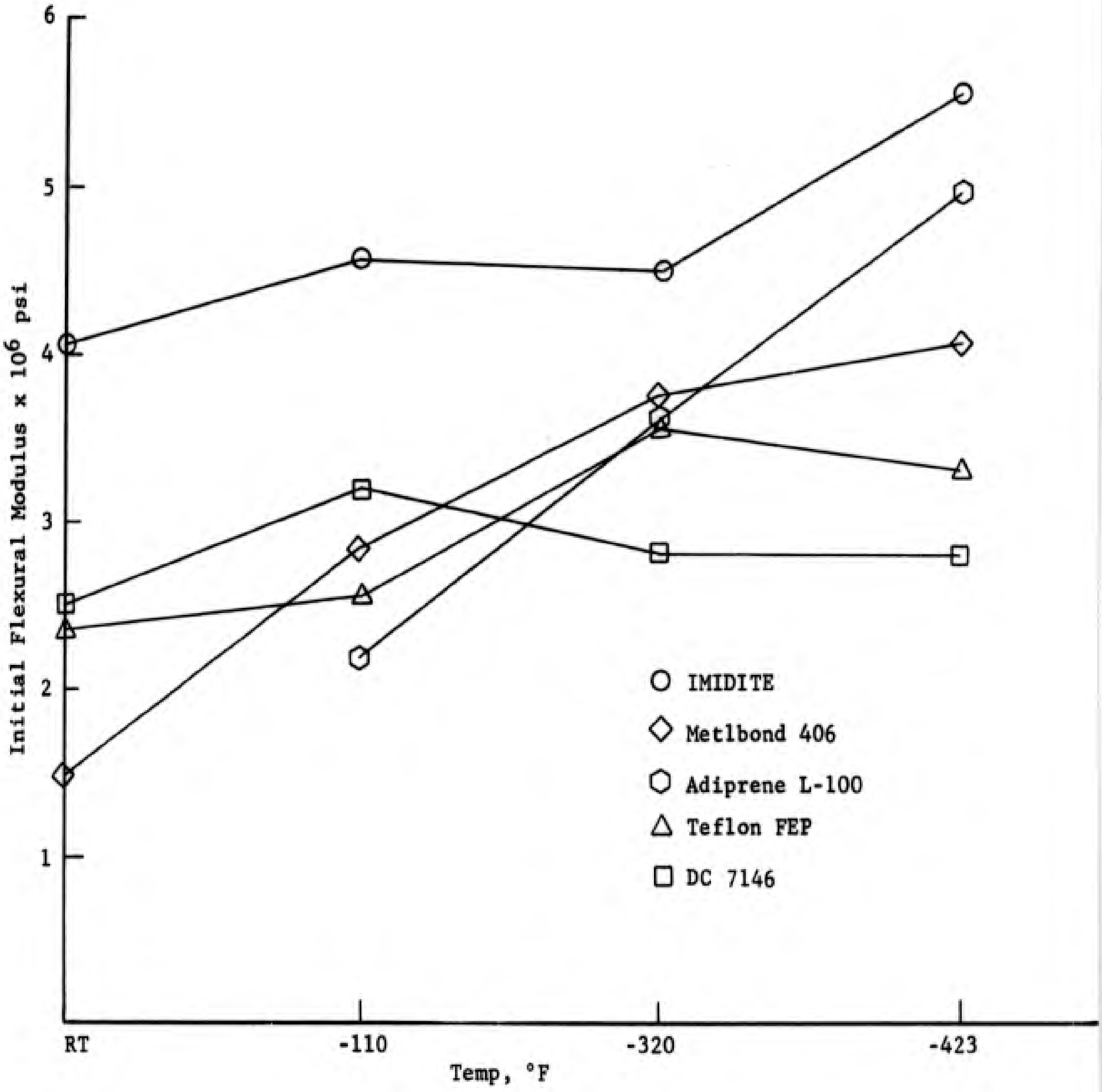


Figure 42. Flexural Modulus for Phase II Materials

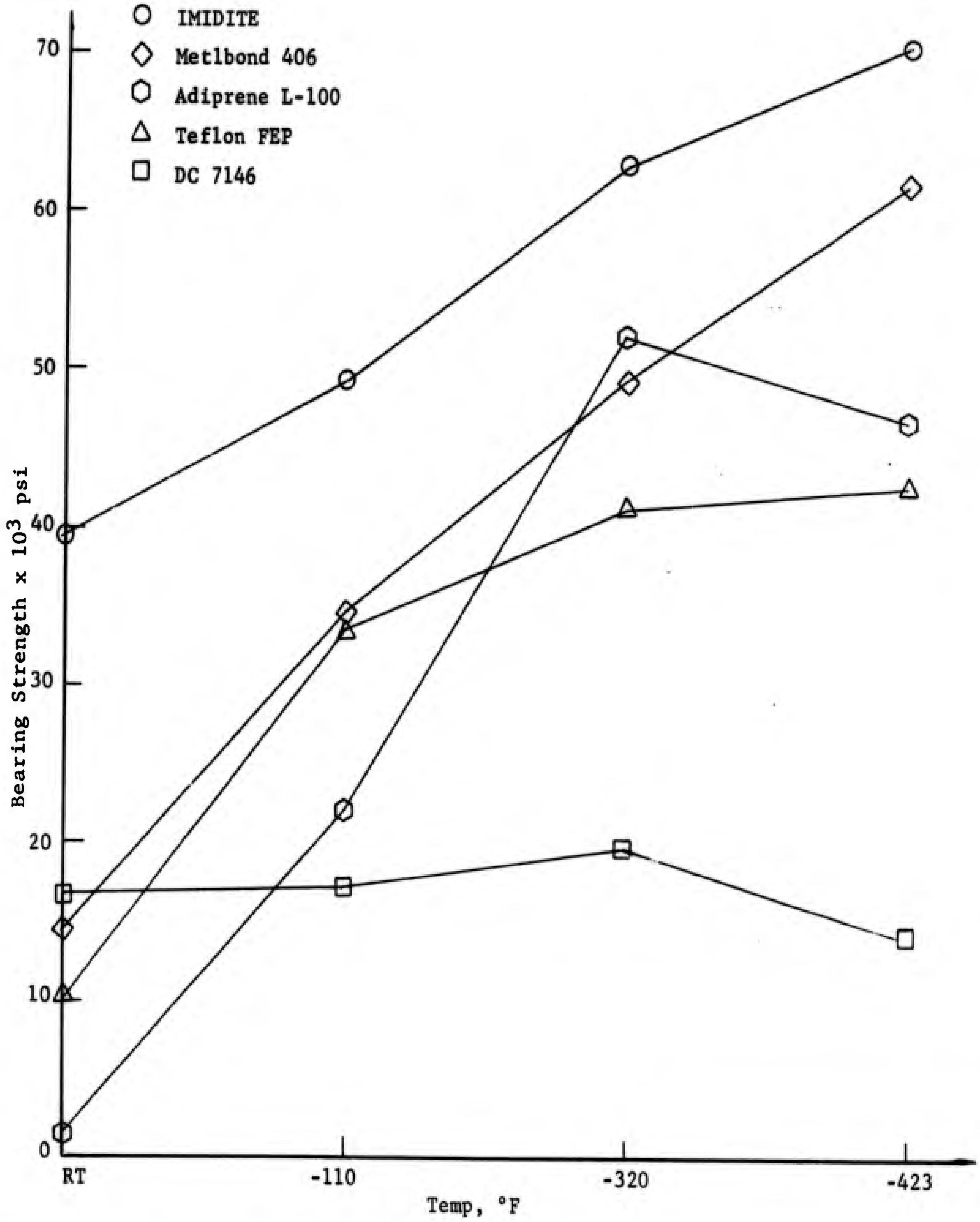


Figure 43. Bearing Strength for Phase II Materials

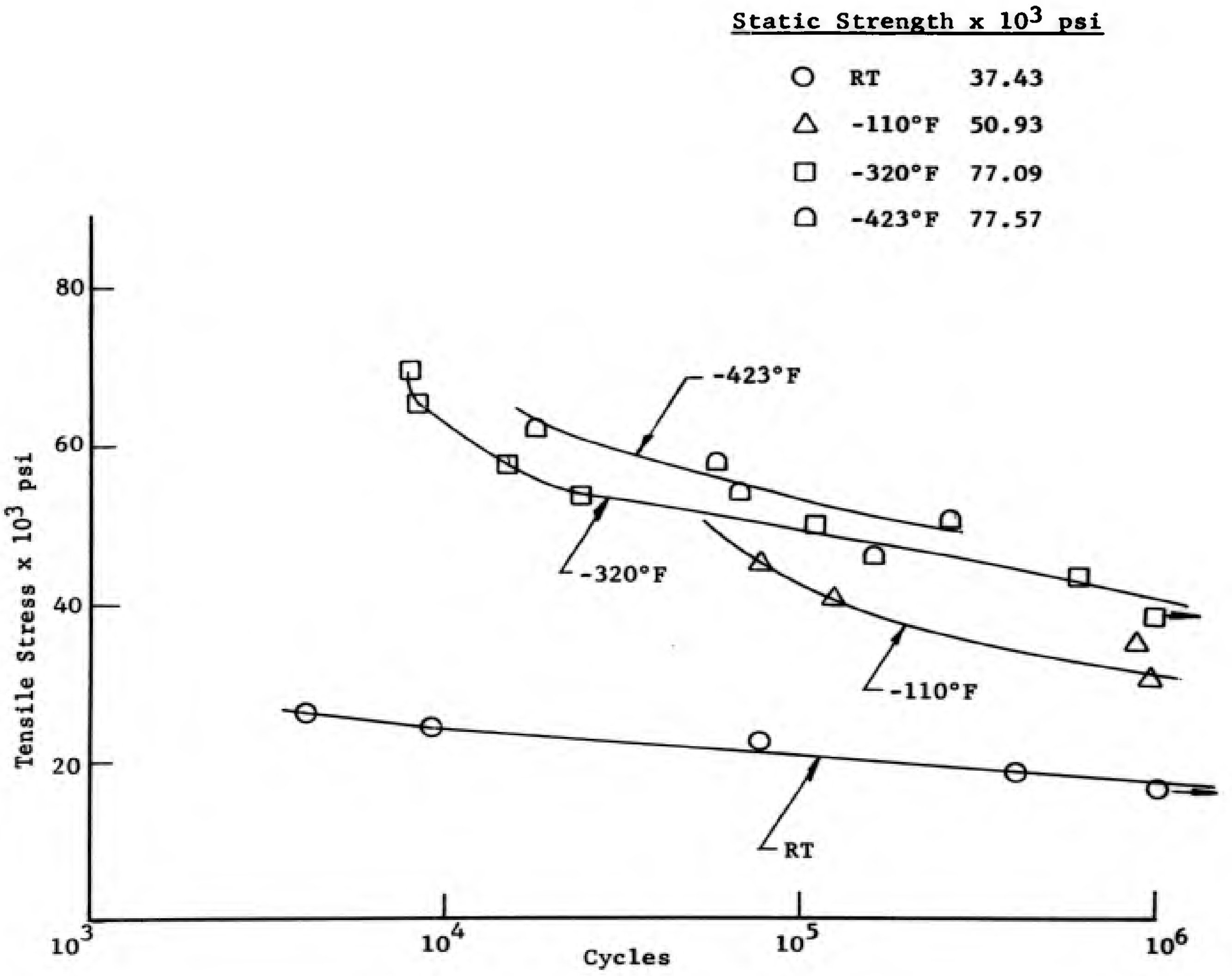


Figure 44. S-N Diagram of IMIDITE

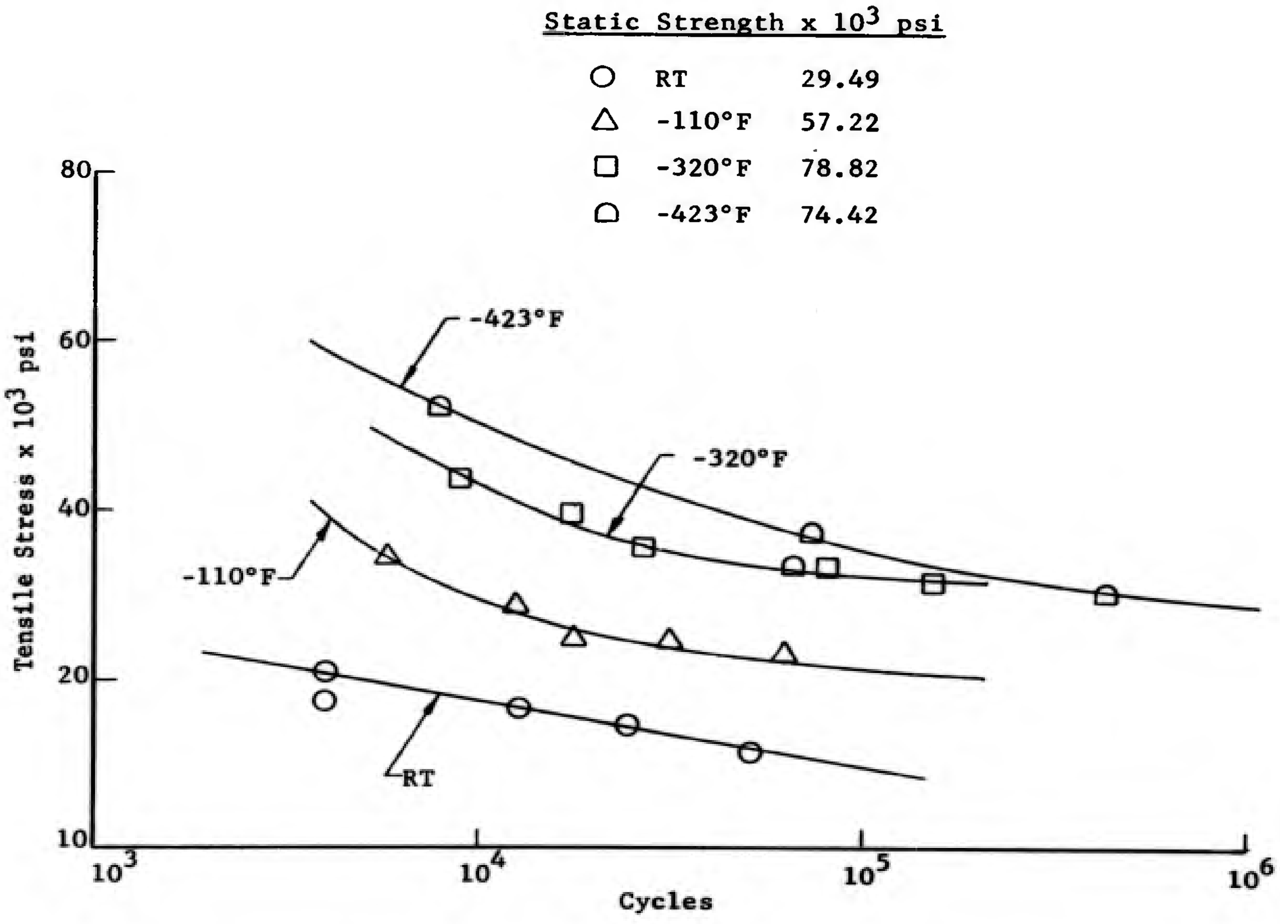


Figure 45. S-N Diagram of Metlbond 406

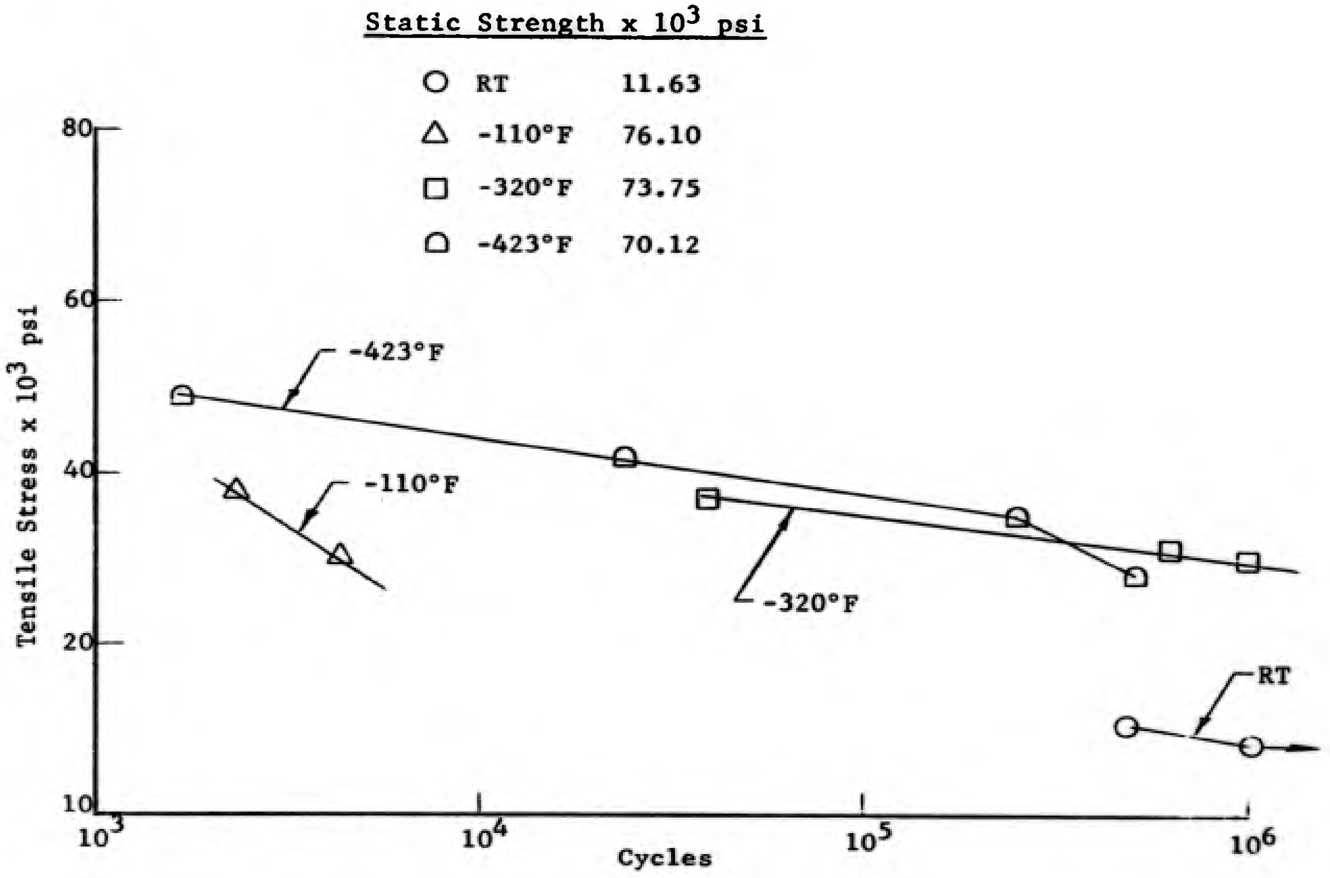


Figure 46. S-N Diagram Adiprene L-100

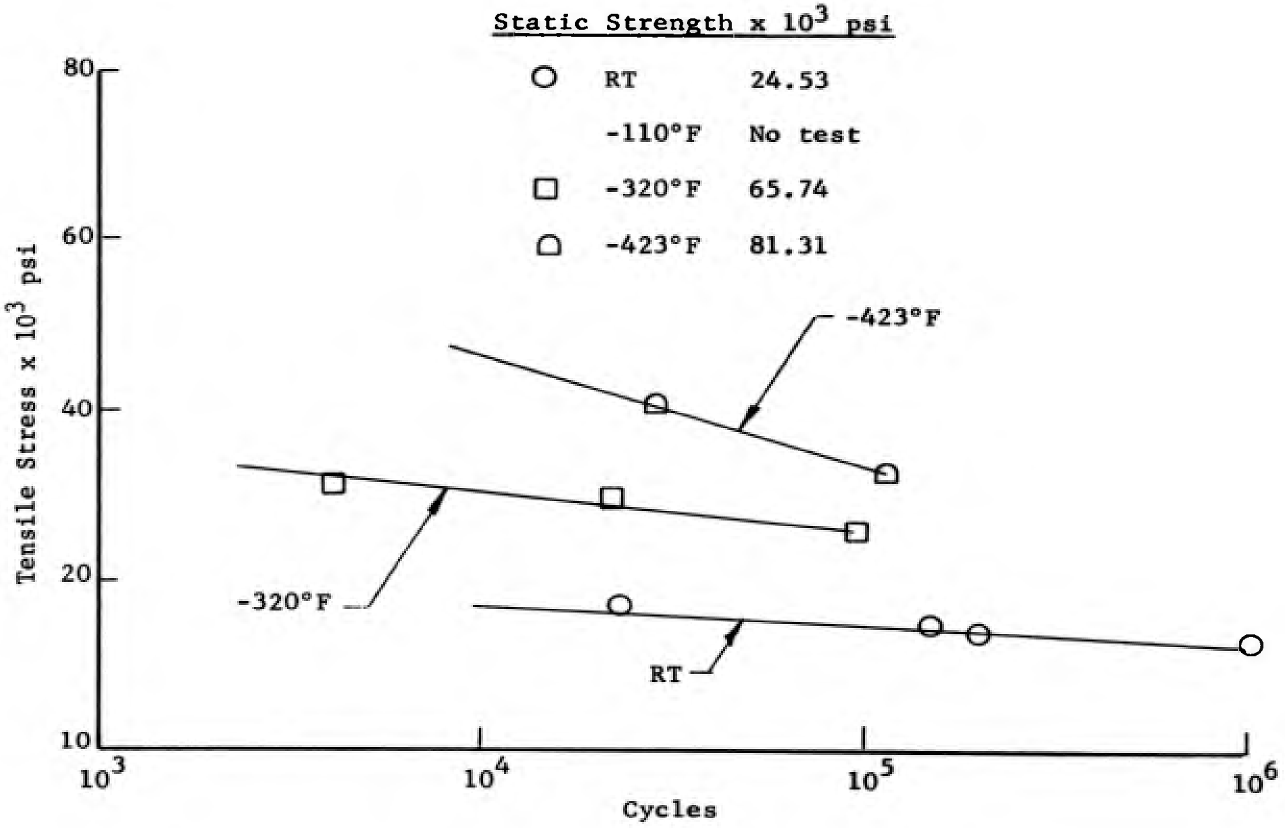


Figure 47. S-N Diagram of Teflon FEP

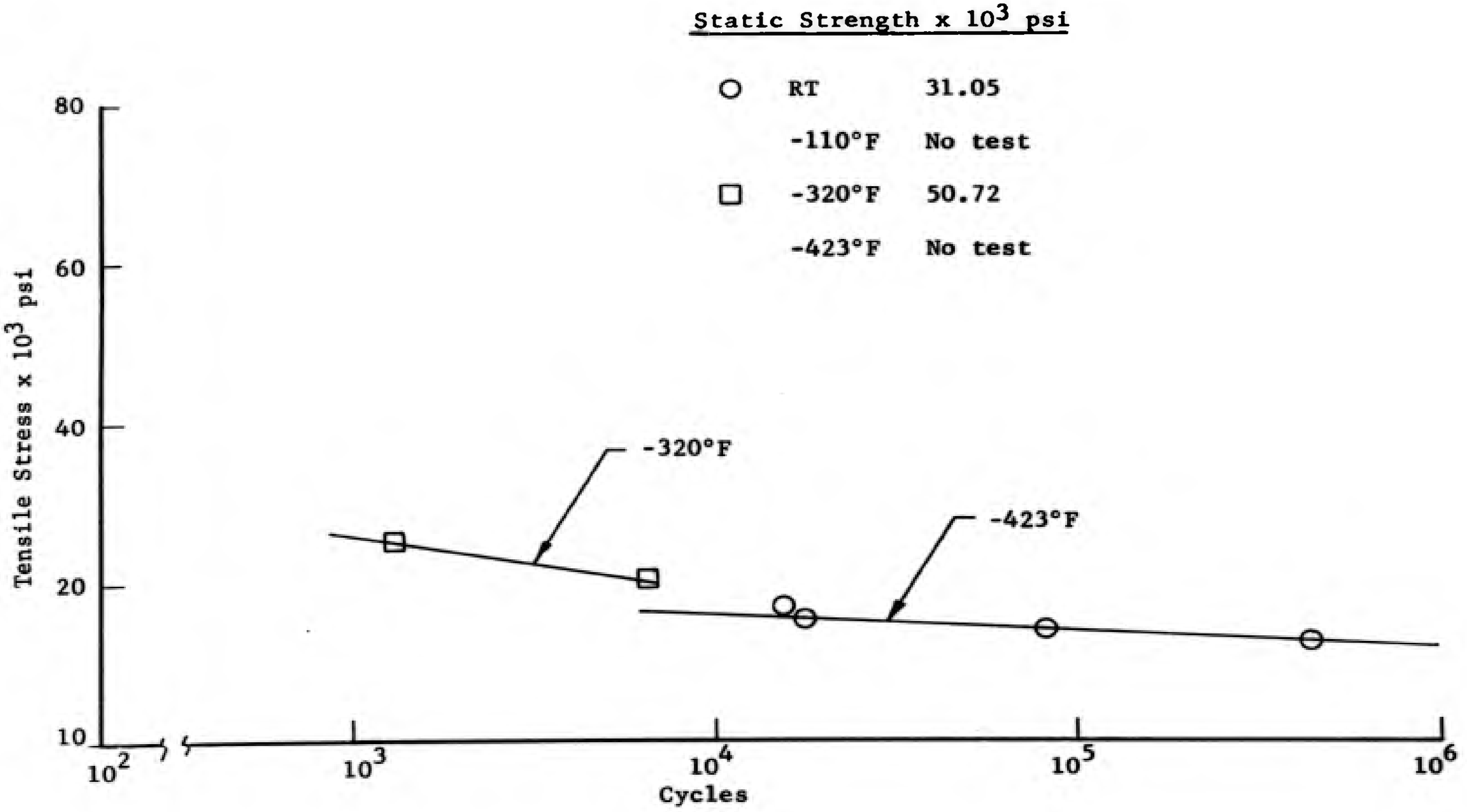


Figure 48. S-N Diagram of DC-7146

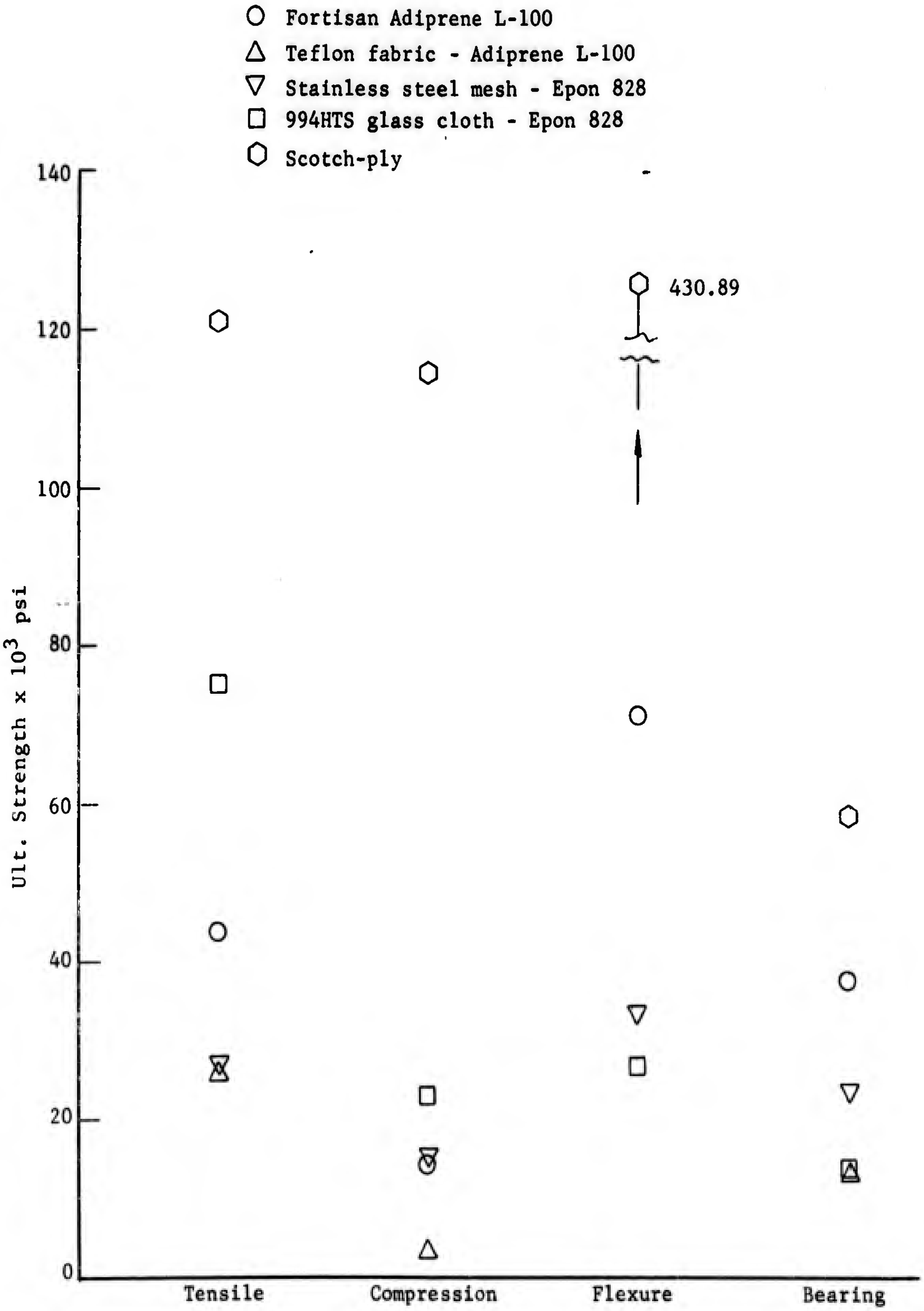


Figure 49. Average Strength of Phase III Materials at -320°F

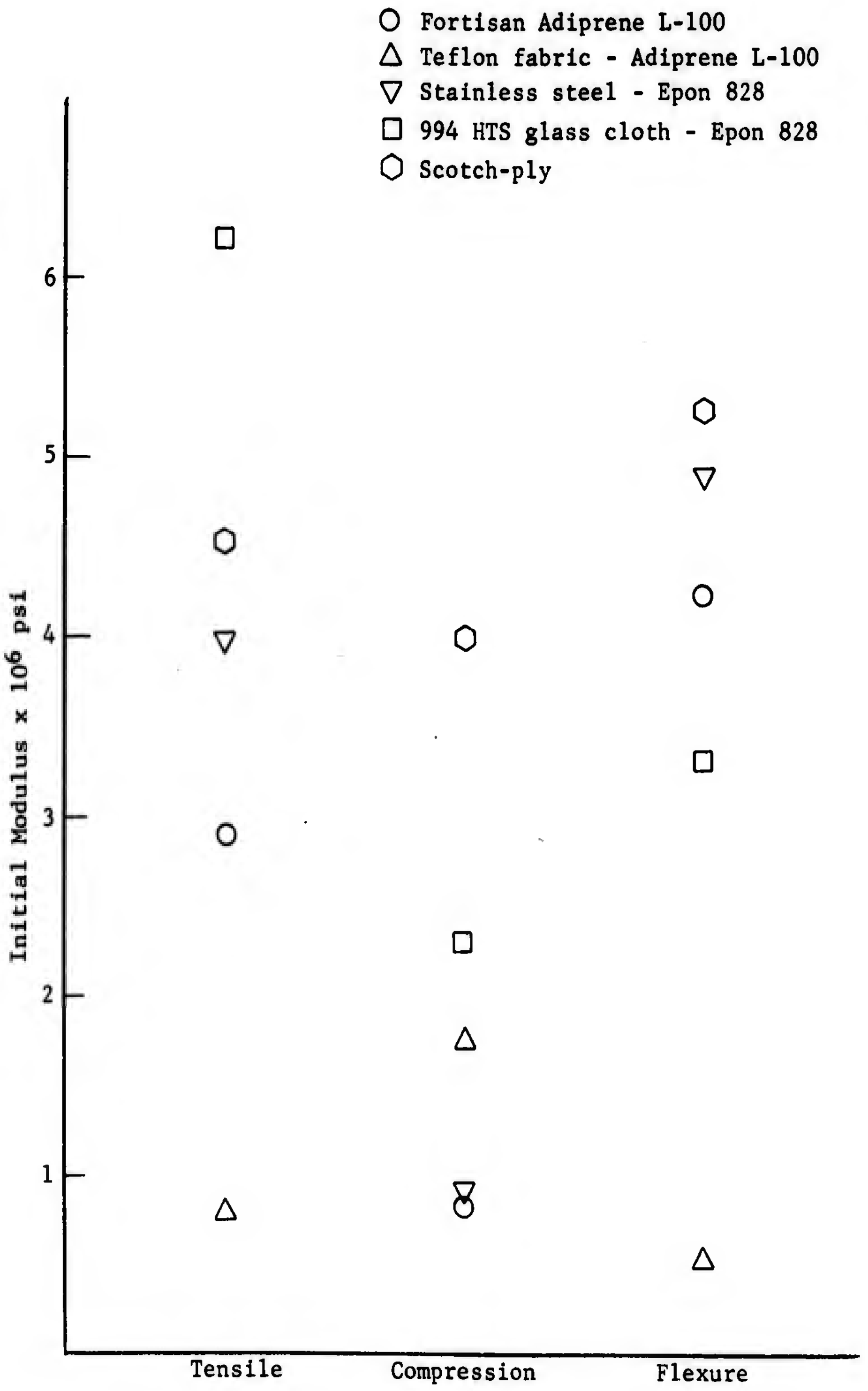
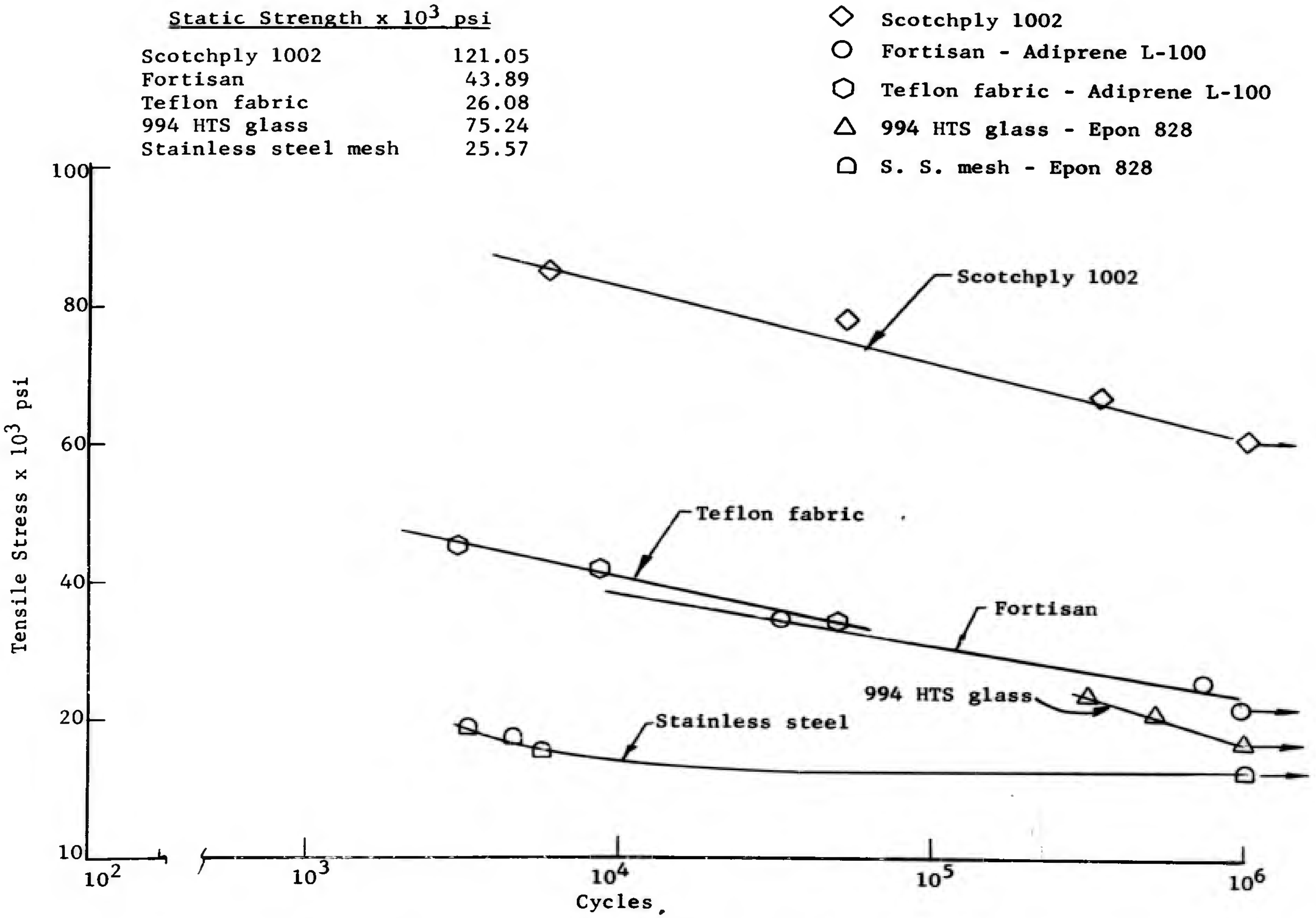


Figure 50. Average Modulus of Phase III Materials at -320°F



Static Strength x 10^3 psi	
Scotchply 1002	121.05
Fortisan	43.89
Teflon fabric	26.08
994 HTS glass	75.24
Stainless steel mesh	25.57

Figure 51. S-N Diagrams for Phase III Materials at -320°F

TABLE 1

LIST OF MATERIALS

Phase I - Existing Resin Systems

Epoxy Resin Systems	MIL-R-9300A Type I
Epon 826/NMA-181	Shell Chemical Company A Division of Shell Oil Company New York 20, New York
Epon 826/DDS-181	Shell Chemical Company A Division of Shell Oil Company New York 20, New York
Narmco 522-181	Narmco Materials Division Telecomputing Corporation Costa Mesa, California
Polyester Resin Systems	**MIL-R-7575**
Narmco 527-181	Narmco Materials Division Telecomputing Corporation Costa Mesa, California
Hetron 31-181	Durez Plastics Division Hooker Chemical Corporation North Tonowanda, New York
Silicone Resin System	**MIL-R-25506A**
Trevarno F-131-181	Coast Manufacturing Supply Company Livermore, California
Phenyl - Silane	
Narmco 534-181	Narmco Materials Division Telecomputing Corporation Costa Mesa, California

TABLE 1 (Continued)

Phase II - Experimental Resin Systems

Nylon-Epoxy System	
Metlbond 406-181	Narmco Materials Division Telecomputing Corporation Costa Mesa, California
IMIDITE-181	Narmco Materials Division Telecomputing Corporation Costa Mesa, California
Flexible Polyurethane	
Adiprene L-100-181	DuPont de Nemours Elastomers Department 2930 East 44th Street Los Angeles, California
Teflon System	
FEP-181	DuPont de Nemours Elastomers Department 2930 East 44th Street Los Angeles, California
Silicone System	
Dow-Corning 7146-181	Dow-Corning Corporation Midland, Michigan

TABLE 1 (Continued)

Phase III - Experimental Reinforcing Materials

Cellulose Fabric Fortisan Fabric Adiprene L-100 Resin	Celanese Fibers Company Charlotte, North Carolina
Fluorocarbon Fabric Teflon Fabric Type T21 Adiprene L-100 Resin	W. S. Shambaun & Company 11617 West Jefferson Boulevard Culver City, California
Unidirectional Glass Scotchply - 1002	Minnesota Mining & Manufacturing Company Reinforced Plastics Division 900 Bush Avenue St. Paul, Minnesota
Special Filament Glass 994-Glass HTS EPON 828 Epoxy Resin	Hess Goldsmith & Company, Incorporated 4820 East District Boulevard Los Angeles, California
Space Age Metals 304 Stainless Steel Wire Mesh Cloth - Epon 828 Epoxy Resin	W. S. Tyler Company 3540 Wilshire Boulevard Los Angeles 5, California

TABLE 2

LAMINATING AND POSTCURE CYCLES

Material	Cure	Postcure
Phase I — Existing Resin Systems		
Epoxy		
Epon 826/NMA	1 hr @ 200 psi @ 350°F press temp	2 hr @ 392°F
Epon 826/DDS	4-min contact, 1 hr @ 200 psi @ 345°F press temp	2 hr @ 392°F
Narmco 522 (prepreg)	2½-min contact, 1 hr @ 20 psi @ 350°F press temp	None
Polyester		
Narmco 527-181 (prepreg)	20 min @ 15 psi @ 285°F press temp	None
Hetron 31	N.A.	N.A.
Silicone		
Trevarno F-131-181 (prepreg)	30 min @ vacuum bag or higher pressure @ 350°F	16 hr @ 200°F, then 2 hr each @ 260°F, 300°F, 350°F, 400°F, 440°F; then 12 hr @ 482°F
Phenyl-silane		
Narmco 534-181 (prepreg)	3½-min contact, 1 hr @ 200 psi @ 250°F press temp	24 hr each @ 250°F, 300°F, 350°F, then 8 hr @ 400°F, & 48 hr @ 500°F
Phase II — Experimental Resin Systems		
Nylon-Epoxy		
Metlbond 406-181	1 hr @ 5-100 psi @ 350°F press temp	None
IMIDITE-181 (prepreg)	3 hr @ 200 psi @ 700°F	Inert N_2 atmosphere @ following time/temp: 24 hr @ 600°F, 650°F, 700°F, 750°F; then 8 hr @ 800°F
Flexible-Polyurethane		
Adiprene L-100-181	1 hr @ 30 psi @ 250°F	None
Teflon		
FEP-181	15 min @ 100 psi @ 700°F press temp	None
Silicone		
Dow-Corning 7146-181	6 hr @ 15 psi @ 300°F press temp	None
Phase III — Experimental Reinforcing Material		
Cellulose Fabric		
Fortisan Fabric Adiprene L-100 Resin	1 hr @ 30 psi @ 250°F press temp	None
Fluorocarbon Fabric		
Teflon Type Fabric Adiprene L-100 Resin	1 hr @ 30 psi @ 250°F press temp	None
Unidirectional Glass		
Scotchply 1002	N.A.	N.A.
Special Filament Glass		
994 HTS Glass Cloth Epon 828 Epoxy Resin	2-min contact, ½ hr @ 200 psi @ 300°F press temp	1½ hr @ 392°F
Space Age Metals		
304 Stainless Steel Wire Mesh Cloth Epon 828 Epoxy Resin	2-min contact, ½ hr @ 200 psi @ 300°F	1½ hr @ 392°F

TABLE 3

SPECIFIC GRAVITY AND RESIN CONTENT

Materials & Panel		Specific Gravity	Resin Content, %
Epon 826/NMA-181			
A	A-1	1.996	14.9
	A-2	1.925	21.3
	A-3	1.891	23.2
	A-4	1.857	26.3
	A-5	2.016	14.4
	A-6	1.986	23.5
Epon 826/DDS-181			
C	C-1	1.96	27.9
	C-2	2.03	23.9
	C-3	1.98	26.9
	C-4	1.99	26.5
	C-5	1.95	28.6
	C-6	1.98	26.7
Narmco 522-181			
	22-2	1.85	28.8
	22-3	1.88	32.9
	22-4	1.85	35.7
	22-5	1.86	33.9
Narmco 527-181			
	27-1	1.95	39.9
	27-2	1.97	37.8
	27-3	1.98	37.5
	27-4	1.97	35.8
	27-5	2.02	35.4
Hetron 31-181			
	H-31-2	1.89	40.7
	H-31-3	1.89	40.6
	H-31-4	1.90	40.1
	H-31-5	1.90	39.9
	H-31-6	1.89	40.8

NOTE: The procedures given in Federal Test Method Standard No. 406 Methods 5011 and 7061-1 were used for these determinations.

TABLE 3 (Continued)

Materials & Panel	Specific Gravity	Resin Content, %
Trevarno F-131-181		
F-131-2	1.84	25.0*
F-131-3	1.82	25.0*
F-131-4	1.84	25.0*
F-131-5	1.83	25.0*
Narmco 534-181		
34-1	1.94	25.3
34-2	1.94	25.6
34-3	1.98	25.6
34-4	1.96	24.6
34-5	1.90	27.3
Metlbond 406-181		
A-1	1.67	58.4
B-1	1.66	61.2
C-1	1.65	57.1
IMIDITE-181		
A	1.86	22.8
B	1.90	21.5
C	1.85	22.2
Adiprene L-100-181		
1	1.585	31.58
FEP-181		
1	2.34	41.4
2	2.32	41.5
3	2.33	40.7
4	2.30	41.9
6	2.31	41.8
Dow-Corning 7146-181		
A	1.78	25.0**
B	1.67	25.0**
C	1.75	25.0**

* Percent resin as estimated by the manufacturer. Resin content by method 7061-1 of LP 406b not applicable to silicone materials.

** Percent resin as estimated by Narmco Laboratory, based on previous experience with similar materials. Resin content by method 7061-1 of LP 406b not applicable to silicone materials.

NOTE: The procedures given in Federal Test Method Standard No. 406 Methods 5011 and 7061-1 were used for these determinations.

TABLE 3 (Continued)

Materials & Panel	Specific Gravity	Resin Content %
<u>Adiprene L-100-F</u>		
B	1.29	42.1***
C	1.28	38.4***
<u>Adiprene L-100-T</u>		
1	1.66	28.3***
<u>Scotchply</u>		
1	1.85	34.4
<u>Epon 828-994 HTS</u>		
1	1.57	17.7
<u>Epon 828-304 SS</u>		
1	3.045	18.4

***Percent resin estimated from known fabric weight in specimen. Resin content by method 7061-1 of FTM not applicalbe to organic fabrics.

NOTE: The procedures given in Federal Test Method Standard No. 406 Methods 5011 and 7061-1 were used for these determinations.

TABLE 4

AVERAGE TENSILE STRENGTH AND MODULUS AT VARIOUS TEMPERATURES FOR PHASE I MATERIALS

Material	Test Temp															
	RT				-110°F				-320°F				-423°F			
	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus
Epoxy																
Epon 826/NMA-181	18.9	58.50	4.31	3.49	26.63	82.03	4.05	2.89	28.60	127.17	4.87	3.75	N.A.	121.08	4.97	N.A.
$\sigma_{Est.}$	0.15	5.46	0.28	0.26	2.47	5.92	0.36	0.21	2.82	12.16	0.50	0.31	N.A.	7.70	0.34	N.A.
Epon 826/DDS-181	23.20	61.03	3.97	2.94	25.51	98.06	4.64	3.21	32.57	115.17	5.06	3.08	29.29	117.95	5.52	2.99
$\sigma_{Est.}$	4.83	4.03	0.23	0.19	1.51	3.97	0.11	0.06	0.82	8.73	0.14	0.72	2.15	7.23	0.20	0.19
Narmco 522-181	N.A.	53.95	3.22	2.14	16.26	75.06	3.69	1.93	N.A.	104.43	4.10	3.54	N.A.	108.62	4.20	N.A.
$\sigma_{Est.}$	N.A.	3.94	0.22	0.51	0.69	4.21	0.22	0.37	N.A.	9.29	0.37	0.64	N.A.	7.77	0.50	N.A.
Polyester																
Hetron 31-181	20.15	49.96	2.97	2.28	13.65	68.12	3.24	1.74	21.37	105.30	3.89	2.35	13.25	100.06	3.91	2.49
$\sigma_{Est.}$	7.34	2.45	0.30	0.18	2.04	1.16	0.15	0.15	0.72	2.88	0.21	0.22	1.53	1.12	0.22	0.11
Narmco 527-181	14.22	49.47	3.23	2.45	8.96	66.93	3.43	2.33	4.02	102.07	3.89	2.71	N.A.	104.09	2.92	N.A.
$\sigma_{Est.}$	3.10	1.99	0.34	0.19	3.40	4.93	0.42	0.25	9.24	2.63	0.15	0.47	N.A.	2.97	0.18	N.A.
Silicone																
Trevarno F-131-181	17.61	29.42	2.54	2.22	22.17	47.42	2.73	2.31	N.A.	70.39	2.59	2.38	N.A.	76.17	3.07	N.A.
$\sigma_{Est.}$	5.43	1.96	0.22	0.30	4.54	2.94	0.14	0.31	N.A.	6.90	0.16	0.20	N.A.	15.20	0.22	N.A.
Phenyl Silane																
Narmco 534-181	16.48	34.44	3.93	2.46	17.69	55.33	4.37	2.58	16.21	63.42	4.80	2.86	14.49	61.23	4.94	3.15
$\sigma_{Est.}$	4.26	2.17	0.70	0.42	1.82	1.64	0.29	0.21	2.41	1.36	0.21	0.19	1.03	2.94	0.42	0.40

TABLE 5

AVERAGE COMPRESSION STRENGTH AND MODULUS AT VARIOUS TEMPERATURES FOR PHASE I MATERIALS

Material	Test Temp							
	RT		-110°F		-320°F		-423°F	
	Ult. Strength x 10^3psi	Modulus x 10^6psi	Ult. Strength x 10^3psi	Modulus x 10^6psi	Ult. Strength x 10^3psi	Modulus x 10^6psi	Ult. Strength x 10^3psi	Modulus x 10^6psi
Epoxy								
Epon 826/NMA-181	50.85	3.94	84.36	3.91	113.85	4.59	129.05	4.38
$\sigma_{Est.}$	5.26	0.06	6.13	0.65	7.94	0.11	10.53	0.19
Epon 826/DDS-181	71.66	4.50	107.40	4.73	138.07	5.17	135.18	5.32
$\sigma_{Est.}$	5.01	0.43	6.67	0.12	12.26	0.37	12.17	0.38
Narmco 522-181	55.75	3.67	93.22	3.89	122.85	4.08	135.48	4.64
$\sigma_{Est.}$	3.27	0.11	4.52	0.14	2.12	0.02	2.14	0.14
Polyester								
Hetron 31-181	19.92	1.64	41.32	1.86	68.54	2.36	66.37	2.70
$\sigma_{Est.}$	0.59	0.18	2.13	0.06	3.33	0.26	7.53	0.09
Narmco 527-181	36.59	3.44	55.12	3.86	66.75	4.02	65.82	4.51
$\sigma_{Est.}$	2.09	0.17	2.87	0.15	4.56	0.22	9.93	0.43
Silicone								
Trevarno F131-181	21.04	2.68	38.98	2.80	42.67	2.76	46.08	3.21
$\sigma_{Est.}$	1.87	0.04	0.98	0.13	3.48	0.61	9.34	0.05
Phenyl Silane								
Narmco 534-181	45.70	3.61	59.61	4.03	55.52	4.16	55.82	4.27
$\sigma_{Est.}$	2.49	0.08	5.50	0.38	3.40	0.24	0.25	0.00

TABLE 6

AVERAGE FLEXURAL STRENGTH AND MODULUS AT VARIOUS TEMPERATURES FOR PHASE I MATERIALS

Material	Test Temp															
	RT				-110°F				-320°F				-423°F			
	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus
Epoxy																
Epon 826/NMA-181	36.80	85.95	4.05	3.40	35.50	116.47	4.00	3.45	41.29	166.68	4.70	4.05	N.A.	171.17	5.49	N.A.
$\sigma_{Est.}$	6.77	4.85	0.40	0.18	6.04	10.77	0.43	0.40	7.11	11.59	0.37	0.25	↓	27.10	2.55	↓
Epon 826/DDS-181	33.76	94.88	4.04	3.35	35.93	129.56	4.13	3.33	38.12	167.46	5.01	4.45	↓	172.39	5.25	↓
$\sigma_{Est.}$	2.45	2.64	0.28	0.30	1.63	1.89	0.21	0.10	2.60	6.02	0.31	0.37	↓	6.48	0.61	↓
Narmco 522-181	20.41	75.64	3.60	3.32	22.49	99.13	3.63	3.31	25.78	152.61	4.53	4.07	↓	163.69	4.83	↓
$\sigma_{Est.}$	2.49	5.08	0.23	0.28	7.63	4.68	0.37	0.27	3.29	16.65	0.25	0.23	↓	10.18	0.23	↓
Polyester																
Hetron 31-181	27.55	71.20	3.24	2.82	32.11	104.13	3.47	3.05	31.39	126.69	4.18	3.55	N.A.	129.00	4.26	N.A.
$\sigma_{Est.}$	4.67	1.27	0.09	0.09	1.15	0.89	0.05	0.06	1.77	3.71	0.19	0.15	↓	3.51	0.28	↓
Narmco 527-181	N.A.	75.21	3.78	N.A.	13.88	80.08	3.34	3.05	22.93	93.75	3.51	3.11	↓	106.28	3.67	↓
$\sigma_{Est.}$	N.A.	2.61	0.05	N.A.	3.04	6.73	0.21	0.11	5.14	6.85	0.24	0.43	↓	10.86	0.14	↓
Silicone																
Trevarno F-131-181	21.80	37.88	2.82	2.14	18.56	45.77	2.37	2.14	19.67	66.87	3.06	2.45	N.A.	64.99	3.35	N.A.
$\sigma_{Est.}$	3.17	1.71	0.10	0.09	1.21	1.76	0.16	0.14	3.13	6.15	0.21	0.23	N.A.	4.54	0.68	N.A.
Phenyl silane																
Narmco 534-181	39.56	68.96	3.80	3.10	24.81	102.10	4.25	3.66	31.52	105.36	4.87	4.00	N.A.	110.42	5.25	N.A.
$\sigma_{Est.}$	2.34	3.46	0.20	0.21	2.04	6.20	0.26	0.42	2.98	11.76	0.39	0.42	N.A.	5.92	0.97	N.A.

TABLE 7

AVERAGE BEARING STRENGTH AT VARIOUS TEMPERATURES FOR PHASE I MATERIALS

Material		Bearing Strength x 10^3 psi			
		RT	-110°F	-320°F	-423°F
<u>Epoxy</u>					
Epon 826/NMA-181		47.14	53.17	74.88	95.32
	$\sigma_{Est.}$	1.83	0.29	2.39	9.71
Epon 826/DDS-181		37.78	49.07	69.56	72.94
	$\sigma_{Est.}$	5.07	1.93	2.28	0.00
Narmco 522-181		37.27	55.23	78.39	74.64
	$\sigma_{Est.}$	3.16	2.44	1.85	7.45
<u>Polyester</u>					
Hetron 31-181		28.16	43.03	61.90	56.39
	$\sigma_{Est.}$	1.02	1.83	2.38	3.31
Narmco 527-181		40.04	47.22	59.14	55.15
	$\sigma_{Est.}$	0.88	4.29	2.31	10.18
<u>Silicone</u>					
Trevarno F-131-181		19.83	26.30	33.81	36.23
	$\sigma_{Est.}$	0.90	1.31	7.75	2.92
<u>Phenyl Silane</u>					
Narmco 534-181		45.52	62.19	74.61	64.57
	$\sigma_{Est.}$	1.38	2.06	1.75	5.20

TABLE 8

PHASE I
AVERAGE BEARING STRENGTHS FOR MATERIALS

(Previously Tested in First Portion of this Program)

Material	Bearing Strength x 10^3 psi	
	RT	-320°F
Narmco 506	--	50.96
$\sigma_{Est.}$	--	0.00
Narmco 513	--	32.63
$\sigma_{Est.}$	--	1.24
CTL 91 LD	48.61	64.25
$\sigma_{Est.}$	4.39	0.97
Paraplex P-43	46.81	46.31
$\sigma_{Est.}$	10.07	4.06
Trevarno F-130	18.66	30.66
$\sigma_{Est.}$	1.21	1.80
Laminac 4232	35.37	46.88
$\sigma_{Est.}$	13.09	3.81

TABLE 9

SUMMARY OF TENSILE FATIGUE TESTS FOR PHASE I MATERIALS

Material	Av. Ult. Tensile Strength, psi	Test Temp							
		RT		-110°F		-320°F		-423°F	
		% Ult.	Cycles	% Ult.	Cycles	% Ult.	Cycles	% Ult.	Cycles
Epon 826/NMA-181	RT: 58,500	75	1,000	65	1,500	70	800	60	2,900
	-110°F: 82,030	65	2,400	60	3,200	60	2,200	50	2,500
	-320°F: 127,160	55	5,300	55	5,200	50	3,200	45	124,500
	-423°F: 121,080	45	16,000	50	5,900	45	3,800	40	118,700
		40	28,300	45	13,500	40	18,900		
		30	174,000	40	18,600				
Epon 826/DDS-181	RT: 61,030	75	2,900	70	2,500	85	800		
	-110°F: 98,060	60	5,600	65	5,500	75	1,000	70	1,800
	-320°F: 115,169	55	4,900	60	4,300	70	5,000	65	16,300
	-423°F: 117,955	50	16,400	55	7,400	65	5,700	60	29,000
		45	18,500	50	21,300	60	6,500	55	25,000
		40	47,000	45	31,900	55	13,400	50	58,800
				40	125,000	50	21,200	45	256,000
						45	76,200	40	785,000
						40	223,400		
Narmco 522-181	RT: 53,950	60	8,600	75	1,500	85	2,000	65	1,500
	-110°F: 75,060	50	11,000	70	2,300	70	3,000	60	27,700
	-320°F: 104,430	45	139,400	65	5,000	65	5,400	55	115,900
	-423°F: 108,616	40	242,700	60	8,300	60	11,100	50	62,300
		35	665,800	55	4,400	55	10,900	45	387,800
		30	1,000,000+	50	16,400	50	18,400	40	486,000
				45	49,500	45	141,300		
				40	122,500	40	386,500		
Narmco 527-181	RT: 49,468	80	900	60	2,100	45	700	50	400
	-110°F: 66,935	70	1,800	55	2,600	40	1,500	40	2,200
	-320°F: 102,070	60	3,200	50	3,600				
	-423°F: 104,091	50	5,500	45	8,000				
		45	15,000	40	7,900				
		40	23,300						
		30	751,500						
Trevarno F-131-181	RT: 29,420	85	2,800	55	4,700	50	1,700	50	900
	-110°F: 47,424	80	3,100	45	26,500	45	5,000	45	8,400
	-320°F: 70,390	70	7,400	40	101,300	40	8,200	40	48,900
	-423°F: 76,168	60	12,000						
		55	24,100						
		50	79,800						
		45	224,100						
		40	647,500						
Narmco 534-181	RT: 34,440	75	9,600	80	12,100	85	1,900	85	7,200
	-110°F: 55,339	70	10,300	70	23,200	75	6,200	80	6,200
	-320°F: 63,420	65	35,500	65	26,900	70	13,300	75	22,100
	-423°F: 61,229	60	61,600	60	146,700	65	17,100	70	25,500
		55	95,300	55	402,200	60	47,300	65	36,500
		50	125,200	50	659,700	55	81,700	60	100,200
		45	304,000	45	511,300	50	225,500	55	664,300
		40	728,300	40	604,700	45	1,013,400	50	1,015,300
		30	1,790,900						
Hetron 31-181	RT: 49,960	85	800	60	1,300	50	800	50	3,300
	-110°F: 68,120	80	1,000	55	2,200	40	2,100	45	12,300
	-320°F: 105,300	75	1,300	50	3,800			40	15,100
	-423°F: 100,061	70	1,900	45	3,800				
		65	1,800	40	7,300				
		60	2,300						
		55	3,200						
		50	3,600						
		45	4,500						
		40	9,500						

TABLE 10

AVERAGE TENSILE STRENGTH AND MODULUS AT VARIOUS TEMPERATURES FOR PHASE II MATERIALS

Material	Test Temp															
	RT				-110°F				-320°F				-423°F			
	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus
IMIDITE-181	N.A.	37.43	3.28	N.A.	13.82	50.93	4.60	3.17	21.78	77.09	4.38	3.22	23.72	77.56	4.98	3.39
$\sigma_{Est.}$	↓	2.24	0.12	↓	0.81	2.90	0.41	0.17	1.01	7.49	0.30	0.18	3.35	5.15	0.26	0.12
Metlbond 406-181	↓	29.49	2.10	↓	N.A.	57.22	2.79	N.A.	N.A.	78.82	3.71	N.A.	N.A.	76.37	4.11	N.A.
$\sigma_{Est.}$	↓	1.47	0.21	↓	↓	2.53	0.02	↓	N.A.	2.03	0.35	N.A.	N.A.	6.99	0.08	N.A.
Adiprene L-11-181	↓	11.63	1.20	↓	↓	76.10	3.21	↓	9.68	73.75	3.30	1.94	9.60	70.17	3.02	2.27
$\sigma_{Est.}$	↓	1.94	0.91	↓	↓	6.73	0.15	↓	2.72	4.95	0.56	0.15	0.44	2.62	0.07	0.36
Teflon FEP-181	↓	24.53	2.49	↓	↓	43.02	2.68	↓	N.A.	65.74	3.03	N.A.	16.43	81.28	3.54	2.90
$\sigma_{Est.}$	↓	3.27	0.09	↓	↓	2.79	0.18	↓	↓	1.05	0.43	↓	3.47	4.11	0.17	0.70
Dow-Corning-7146-181	↓	31.05	2.30	↓	↓	32.62	2.75	↓	↓	50.72	2.93	↓	N.A.	42.42	2.84	N.A.
$\sigma_{Est.}$	↓	1.65	0.11	↓	↓	0.43	0.08	↓	↓	1.68	0.31	↓	N.A.	1.21	0.09	N.A.

TABLE 11

AVERAGE COMPRESSION STRENGTH AND MODULUS AT VARIOUS TEMPERATURES FOR PHASE II MATERIALS

Material	Test Temp							
	RT		-110°F		-320°F		-423°F	
	Ult. Strength x 10^3psi	Modulus x 10^6psi	Ult. Strength x 10^3psi	Modulus x 10^6psi	Ult. Strength x 10^3psi	Modulus x 10^6psi	Ult. Strength x 10^3psi	Modulus x 10^6psi
IMIDITE -181	65.40	5.88	45.19	3.50	71.08	3.76	69.10	3.66
$\sigma_{Est.}$	1.92	0.17	4.95	0.28	4.67	0.28	7.39	6.80
Metlbond 406-181	21.64	1.92	76.06	2.71	82.42	3.27	82.16	3.40
$\sigma_{Est.}$	0.11	0.07	2.17	0.12	8.06	0.77	4.73	0.15
Adiprene L-100-181	4.89	1.41	48.60	2.63	90.05	3.91	91.10	3.84
$\sigma_{Est.}$	0.17	0.18	0.83	0.37	5.05	0.05	0.51	0.24
Teflon FEP-181	7.25	1.51	11.43	1.83	22.64	2.45	33.32	3.04
$\sigma_{Est.}$	0.28	0.04	1.48	0.24	4.53	0.21	8.35	0.28
Dow-Corning 7146-181	22.87	3.17	35.94	3.98	18.97	3.51	No test	
$\sigma_{Est.}$	0.54	0.09	3.09	0.12	4.00	0.22	4.25	0.46

TABLE 12

AVERAGE FLEXURAL STRENGTH AND MODULUS AT VARIOUS TEMPERATURES FOR PHASE II MATERIALS

Material	Test Temp															
	RT				-110°F				-320°F				-423°F			
	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus	Initial Strength	Ult. Strength	Initial Modulus	Secondary Modulus
Metlbond 406-181	N.A.	33.84	1.48	N.A.	N.A.	76.02	2.86	N.A.	N.A.	96.02	3.77	N.A.	N.A.	81.67	4.09	N.A.
$\sigma_{Est.}$	N.A.	2.38	0.09	N.A.		1.73	0.13			8.42	0.15			5.34	0.09	
Adiprene L-100-181		No test:				51.82	2.20			58.70	3.63			48.50	4.97	
$\sigma_{Est.}$		Material too flexible				2.32	0.41			20.41	0.15			20.63	0.74	
Teflon FEP-181	N.A.	19.38	2.35	N.A.		37.40	2.57			67.31	3.56			66.29	3.31	
$\sigma_{Est.}$		1.16	0.07			2.64	0.02			9.76	0.05			1.64	0.02	
Dow-Corning-7146-181		23.81	2.50			29.40	3.22			18.85	2.82			13.39	2.80	
$\sigma_{Est.}$		4.25	0.46			1.12	0.54			1.26	0.17			0.51	0.12	
IMIDITE-181		76.86	4.05			103.67	4.59			129.80	4.51			144.01	5.57	
$\sigma_{Est.}$		6.01	0.15			10.58	0.02			11.53	0.90			9.10	0.24	

TABLE 13

AVERAGE BEARING STRENGTH FOR PHASE II MATERIALS

Material	Bearing Strength x 10^3 psi			
	RT	-110°F	-320°F	-423°F
IMIDITE-181	39.70	49.39	62.99	70.38
$\sigma_{Est.}$	1.38	5.51	0.94	11.11
Metlbond 406-181	14.55	34.86	49.45	61.69
$\sigma_{Est.}$	1.83	3.69	3.47	9.96
Adiprene L-100-181	1.65	22.07	52.08	46.85
$\sigma_{Est.}$	0.11	3.74	0.00	10.34
Teflon FEP-181	10.13	33.71	41.22	42.62
$\sigma_{Est.}$	1.74	0.00	4.72	2.51
Dow-Corning 7146-181	16.81	17.18	19.77	13.86
$\sigma_{Est.}$	2.23	1.96	3.68	1.55

TABLE 14

SUMMARY OF TENSILE FATIGUE TESTS FOR PHASE II MATERIALS

Material	Av. Ult. Tensile Strength, psi	Test Temp							
		RT		-110°F		-320°F		-423°F	
		% Ult.	Cycles	% Ult.	Cycles	% Ult.	Cycles	% Ult.	Cycles
IMIDITE-181	RT: 37,432 -110°F: 50,925 -320°F: 77,087 -423°F: 77,565	70 65 60 50	4,000 9,000 76,400 404,000	90 80 70 60	17,800 126,900 796,000 969,000	90 85 75 70 65 60 50	7,900 8,400 14,800 24,000 110,500 608,000 1,000,000	80 75 70 65 60 50	15,000 59,100 69,000 260,300 161,300 1,000,000
Metlbond 406-181	RT: 29,492 -110°F: 57,224 -320°F: 78,824 -423°F: 74,415	70 60 55 50 40	4,900 4,300 12,800 24,900 51,600	60 50 45 42.5 40	5,800 12,500 17,800 32,200 63,400	55 50 45 42.5 40	9,200 17,500 27,300 84,600 152,600	70 60 50 45 40	8,000 9,400 74,300 66,700 440,600
Adiprene L-100-181	RT: 11,630 -110°F: 76,100 -320°F: 73,750 -423°F: 70,123	90 70	500,000 1,000,000	50 40	2,300 4,300	50 45 42.5 40	38,500 613,700 1,000,000+	70 60 50 40	1,700 24,300 252,500 517,100
Teflon FEP-181	RT: 24,530 -110°F: -320°F: 65,741 -423°F: 81,312	70 60 55 50	22,600 145,700 201,400 1,000,000+			47.5 45 40	4,100 22,300 92,800	50 40	28,200 115,900
Dow-Corning 7146-181	RT: 31,051 -320°F: 50,724	55 50 45 40	15,300 17,600 81,200 430,200			50 40	1,300 6,400		

TABLE 15

AVERAGE TENSILE, COMPRESSIVE, FLEXURAL, AND BEARING STRENGTH AT -320°F FOR PHASE III MATERIALS

Material		Tensile		Compressive		Flexural		Bearing
		Ult. Strength x 10^3psi	Modulus	Ult. Strength x 10^3psi	Modulus	Ult. Strength x 10^3psi	Modulus	Ult. Strength x 10^3psi
Adiprene L-100-F		43.89	2.90	14.16	0.81	71.50	4.22	37.98
	$\sigma_{Est.}$	3.75	0.23	5.32	0.14	3.36	0.24	0.69
Adiprene L-100-T		26.08	0.81	3.56	1.76	Specimen bends	0.55	13.40
	$\sigma_{Est.}$	0.83	0.01	0.47	0.33		0.01	3.78
Epon 828-994 HTS		75.24	6.21	23.07	2.98	26.86	3.30	13.65
	$\sigma_{Est.}$	10.45	0.59	1.39	1.25	1.84	0.37	6.57
Epon 828-304 SS		25.57	3.97	14.42	0.868	33.29	4.89	23.54
	$\sigma_{Est.}$	1.21	0.48	8.98	2.12	16.14	1.58	6.92
Scotchply		121.05	4.53	114.96	4.00	430.89	5.26	58.78
	$\sigma_{Est.}$	4.91	0.27	11.82	0.19	6.51	0.15	2.32

TABLE 16

SUMMARY OF TENSILE FATIGUE TESTS AT -320°F FOR PHASE III MATERIALS

Material	Av. Ult. Tensile Strength, psi	% Ult. Strength	No. Cycles to Failure
Adiprene L-100-F	43,896	80	32,000
		60	738,700
		50	1,000,000
Adiprene L-100-T	26,080	90	313,700
		80	526,600
		65	1,000,000
Epon 828-994 HTS	75,237	60	3,000
		55	8,500
		45	49,800
Epon 828-304 SS	27,572	70	3,300
		65	4,400
		60	4,500
		55	5,600
		50	1,000,000
Scotchply	121,052	70	5,900
		65	53,000
		60	52,000
		55	347,000
		50	1,000,000

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