Mechanical Properties of Epoxy Resins and Glass/Epoxy Composites at Cryogenic Temperatures*

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SYNOPSIS

The mechanical properties of four modified-epoxy resin systems were investigated. Tensile properties (including strength, modulus, and elongation), notch toughness, impact strength, the coefficient of linear thermal contraction, and thermal-shock resistance were determined for cast-resin specimens at +75, -320, and -423°F. Interlaminar shear strengths (Naval Ordnance Laboratory horizontal shear and short-span shear) were obtained for glass-filament-wound-composite specimens at the three temperatures.

The two systems with the best cryogenic properties were a hybrid-epoxy/polyurethane (Resin 4A) and a highly modified bisphenol-A epoxy (Resin 2). In choosing the optimum system, the same tests (except for notch toughness, impact strength, and short-span shear) were performed on filament-wound-composite specimens at the three temperatures. In these tests, the performance of Resin 4A was better than, or at least comparable to, that of Resin 2.

INTRODUCTION

The available data on the behavior of polymeric materials and filament-wound composites at cryogenic temperatures indicate that, in comparison with room-temperature properties, there is an increase in strength and modulus in

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tension, compression, and flexure, and a decrease in elongation (Refs. 1 through 7). A 50% or greater increase in strength is not uncommon for glass-reinforced plastics at cryogenic temperatures. (The measured strength of E-glass monofilaments increases about 60% at cryogenic temperatures.)

This paper presents data obtained in a program, sponsored by the National Aeronautics and Space Administration, undertaken to develop an improved cryogenic resin for S-901 glass-filament-wound structures - one that would provide improved shear transfer and flexural strength at -423°F as compared with presently available resin/S-901 glass systems.*

Tables 1 and 2 characterize the four resin systems investigated in this work. The resins, essentially epoxies modified in varying degree to provide superior low-temperature properties, were chosen from 41 candidate cryogenic systems after screening tests for tensile strength, elongation, and notch toughness at +75 and -423°F, as well as viscosity, pot life, and glass-transition temperature.

RESULTS AND DISCUSSION

A. CAST-RESIN PROPERTIES

1. Tensile Strength and Associated Properties

Tables 3 through 5 present data for tensile properties at three test temperatures (+75, -320, and -423°F). Toughness, an indication of the work required to fracture a test specimen, was determined by plotting a stress-strain curve for each system at each temperature and calculating the area under the curve (see Figures 1, 2, and 3). The pronounced influence of temperature on the stress-strain characteristics of Resin 4A is shown in Figure 4.

The effects of cryogenic temperatures on various tensile properties are shown in Figures 5 through 8. Figure 5 shows that the tensile stress at fracture is considerably higher at cryogenic temperatures than at room temperature. Resins 3 and 6 exhibited slightly higher strengths at -423 than at -320°F, but Resins 2 and 4A declined in strength, the latter significantly. Even so, Resin 4A remained some 50% stronger than the next strongest system. Although

*A complete account is given in Cryogenic Resins for Glass-Filament-Wound Composites, NASA CR-72114 (Aerojet-General Report 3343), January 1967.

Resins 2, 3, and 6 yielded with increased loading at room temperature, the ultimate and fractural tensile strengths were the same for all systems at cryogenic temperatures (Figures 1, 2, and 3).

To determine if the extensometer used to measure strain was creating flaws in specimens, the instrument was omitted in the testing of two of the five specimens for each resin system. No significant differences in tensile strengths were noted in the two procedures.

Figure 6 shows the "leveling effect" on strain at fracture (or elongation at fracture) experienced at cryogenic temperatures. Resin 4A, with strains of 2.95 and 1.88% at -320 and -423°F, respectively, led the other systems in this property. Although Resin 4A had the lowest tensile modulus at room temperature, its modulus values at cryogenic temperatures were significantly higher than those for the other systems (Figure 7). The superior toughness of Resins 4A and 2 is shown in Figure 8.

2. Notch Toughness

Table 6 and Figure 9 present the test results. Resin 4A exhibited the highest values at both cryogenic temperatures. All resins except Resin 6 had maximum notch toughness at -320°F. The alternate cure schedule used with Resin 3 (8 hours at 225°F) resulted in lower values at each temperature.

Examination of test-specimen fracture surfaces provided insight into modes of failure. As might be expected, the rougher the fracture surface (indicative of the increased energy required for crack propagation), the greater the notch-toughness strength. In particular, this correlation was noted for Resin 4A at -423°F and for Resin 6 at +75°F; in both instances, the fracture surfaces were rough and had strain or tear lines generally oriented in the direction of crack propagation. The fracture surfaces of other specimens (i.e., Resins 2, 3, and 6 at -423°F) exhibited a smooth, almost glasslike texture typical of brittle materials with low energy-absorption properties. In general, the fracture surfaces of test specimens failing at a notch-toughness strength above 1400 psi $\sqrt{\text{in.}}$ were distinctly rougher.

3. Impact Strength

Impact strength, another index of toughness, is a measure of the amount of energy required for rapid test-specimen fracture. Although the unnotched Charpy impact-strength tests that were performed do not yield design data

directly applicable to filament-winding conditions, they permit comparison of differences in energy absorption before fracture. Test results for 0.50 by 0.50 by 5.0-in. cast-resin specimens at +75, -320, and -423°F are presented in Table 7. Although differences between resin systems are indicated, there is considerable data scatter, which is probably due to the absence of specimen notches. (When a V-notch identical to that used in homogeneous-metal Charpy-impact specimens was incorporated into samples of Resin 6, the data exhibited less scatter at 75°F. The notched and unnotched values were 0.13 and 10.65 ft-lb/in., respectively.) The use of unnotched specimens was continued, however, because the results adequately differentiated between candidate-resin resistances to fracture by shock.

At 75°F, Resin 6 had the greatest impact strength of the resins that fractured. Because of its great resilience, Resin 4A flexed sufficiently to be tossed from the fixture without fracturing.

In the -320°F tests, during the preliminary immersion in liquid nitrogen (LN_2), all specimens except those of Resin 4A underwent thermal-shock fracture, which probably contributed to the lower impact values for Resins 2, 3, and 6 in comparison with Resin 4A.

Because the specimens were gradually cooled to the test temperature in the liquid-hydrogen (LH_2) tests, no thermal fracture occurred and the average impact-strength values were higher than those at -320°F. The actual test temperature was slightly higher than -423°F, because of the time required for specimen transfer from the conditioning bath to the impact machine. On the whole, there was less scatter in the cryogenic data than at 75°F.

4. Coefficient of Linear Thermal Contraction

Test results are presented in Figure 10 and Table 8. The average coefficient of linear thermal-contraction calculated for each resin system between -400 and +60°F ranged from 32.11 to 34.93 x 10^{-6} in./in./°F. Resins 2 and 4A had the lowest coefficients.

5. Thermal-Shock Resistance

In the initial experiments, the specimens (three of each resin system) were first placed in the cryostat and cooled (in effect, relatively slowly) to -423°F by filling the cryostat with LH_2. They were removed after 15 min, were immersed in water at room temperature for 15 min, and were dried with

compressed air. None showed signs of thermal cracking. The test procedure was then modified to provide more rapid cooling, because (a) the thermal shock resulting from the relatively slow cooling was apparently not severe enough to permit differentiation between the resins, and (b) the impact-strength specimens had been observed to crack during rapid immersion in LN_2.

Table 9 summarizes the thermal-shock results. During the first rapid cooling cycle, all Resin 3 specimens cracked and no further testing was performed on these specimens. One Resin 3 specimen cracked during the third cycle, one cracked during the fourth cycle, and the third did not crack. Two Resin 6 specimens cracked during the third cycle, but the third did not crack after five cycles. None of the Resin 4A specimens exhibited thermal cracking when exposed to either the slow or the rapid cooling cycles.

B. RESIN/GLASS-COMPOSITE PROPERTIES

1. Interlaminar Shear Strength

a. Horizontal Shear

Data from tests employing the Naval Ordnance Laboratory (NOL) horizontal-shear method are presented in Table 10. Average values ranged from 2,020 psi at +75°F for Resin 4A to 16,110 psi at -423°F for Resin 2. Increases in shear strength ranged (1) from 100 to 438% in going from +75 to -320°F, and (2) from 142 to 505% in going from +75 to -423°F. Resins 2, 3, and 4A exhibited 11 to 12% increases in shear strength at -423°F as compared with their strengths at -320°F, but Resin 6 declined about 7%. As was expected, Resin 4A exhibited the greatest increase in shear strength on cooling to cryogenic temperatures, although its strength was exceeded by each of the other resins. These results, indicating the effect of temperature on interlaminar shear stress, are illustrated in Figure 11.

Table 10 also shows resin and void contents obtained for composite segments from the same NOL ring used in fabricating the horizontal-shear specimens. The order of increasing void content in the specimens was $3<2<6<4A$. The high void content of Resin 4A was probably caused by residual methyl ethyl ketone (MEK) solvent, which was added to improve the resin-mix workability and for possible extension of pot life. When NOL rings were fabricated with a Resin 4A mix containing less MEK, there was a large increase in shear strength at all temperatures and a decrease in voids (Table 11 and Figure 12).

Complete omission of solvent was accompanied by the highest average shear-strength values at cryogenic temperatures obtained in the program. The indirect relationship between shear stress and voids at ambient temperature has been noted by many investigators. As is evident in Figure 13, a similar correlation appears to exist (at least at first approximation) at ambient temperature and possibly at -423°F for resins of significantly diverse chemical types.

Table 12 compares shear-stress results for Resin 2 at different resin and void levels. In this case, reduced voids did not result in higher average shear stresses, the values being essentially unchanged or even slightly lower than those obtained at higher void contents. The data suggest that the resin content, which was considerably different in the two specimens, may affect the horizontal shear strength, particularly in resin-poor composites.

b. Short-Span Shear

Test data are given in Table 13. Although general performance trends are clear, the results were influenced considerably by the fabrication technique, which unfortunately did not afford sufficiently rigid control over void content and resin content - two factors affecting shear-strength properties.

As in the horizontal-shear tests, Resin 4A exhibited the lowest shear stress at 75°F. Stress increases were obtained for all specimens at -320°F, with Resin 6 yielding the highest value (10,890 psi). Except for Resin 3, the shear stresses of all resins were lower at -423 than at -320°F. Resin 6 exhibited the highest shear strengths among the four systems at all test temperatures, possibly owing to some extent to its lower void content.

The low shear strengths of the Resin 4A specimens at 75°F may be attributed to great flexibility at this temperature, which hindered the efficient transfer of shear loads. The rigidity of Resin 4A specimens increased at the lower temperatures, as did the shear strength. It should be noted, however, that the unduly high void contents of Resin 2, 3, and 4A specimens undoubtedly lowered the shear strengths obtained at all test temperatures. In addition, the resin contents of Resin 2 and 4A specimens were probably too high for optimum shear strengths. The effect of temperature on short-span shear is depicted in Figure 14.

2. Tensile Strength and Associated Properties

Tables 14, 15, and 16 present test results for the various filament orientations - unidirectional 1:0, bidirectional 1:1, and bidirectional 1:2, respectively. The average filament-strength values are plotted in Figure 15 as a function of temperature. At cryogenic temperatures, Resin 4A performed better than Resin 2 in each filament orientation. In all cases, the strengths were lowest at +75°F, were highest at -320°F, and decreased somewhat at -423°F (from the value at -320°F). The sole exceptions to the general consistency are the unidirectional values for Resin 4A, particularly at -320°F; they are considered spuriously high, although no satisfactory explanation has been found. It is of interest to note in Table 14 that the high values for Resin 4A at cryogenic temperatures were achieved despite an excessive resin content (43.6 wt%) and excessive voids (16.7 vol%) resulting from an unsuccessful attempt to improve composite fabrication by modifying the procedures used.

For both resins at cryogenic temperatures, fiber strengths for the bidirectional 1:2 orientation were higher than for the 1:1 orientation; in turn, the latter were higher than for the unidirectional specimens, except for the anomalous behavior of the unidirectional Resin 4A specimens. Higher strengths exhibited by specimens having more cross fibers suggest that the interspersion of cross plies benefited the uniaxially loaded specimens, possibly because (a) they provided a more intimate bond between layers of load-carrying filaments, which in turn contributed to a more uniform load distribution, or (b) they permitted load transfer around initial fiber fractures.

Tensile-modulus and elongation-at-fracture data (Tables 14, 15, and 16) reveal that the intra-resin and inter-resin results obtained for the bidirectional orientations were also fairly consistent. On the whole, the unidirectional data were less consistent than the bidirectional with respect to these properties, perhaps because of the limited data available for Resin 2 and the excessively high resin and void contents of Resin 4A specimens.

The increase in composite tensile modulus was less pronounced at cryogenic temperatures than for the cast resins themselves (see Figure 7 and Tables 3 through 5); in fact, slight decreases were noted for Resin 2 composites at -320°F for both bidirectional orientations. Similarly in contrast to the trend noted for cast resins (Figure 6), elongation-at-fracture values were higher at cryogenic

temperatures than at 75°F for both bidirectional orientations. In general, individual-resin elongation values were highest for the 1:2 orientation and lowest for the unidirectional. There was little difference in elongations for bidirectional orientations at a specific cryogenic temperature.

3. Thermal-Shock Resistance

The test specimens, segments of NOL rings, were subjected to temperature cycling between -423°F and ambient, and were then tested in interlaminar shear at -423°F. As shown in Table 17, the Resin 2 composite was highly thermal-shock-resistant, giving little or no evidence of cycling-induced damage. The Resin 4A composite, on the other hand, exhibited a decline in shear stress from 17,940 psi to 15,390 psi, or 12.5%. At that level, the shear stresses of the two systems were approximately the same.

4. Coefficient of Linear Thermal Contraction

Linear-thermal-contraction tests were performed on rectangular composites having filaments either parallel or normal to the specimen length. Test results are given in Tables 18 and 19. Average linear-contraction values are plotted in Figure 16.

Significant variations between specimens were obtained with both Resin 2 and 4A composites, in contrast to the highly consistent thermal-contraction data obtained with the analogous cast-resin specimens. Comparison of resin content with contraction behavior reveals a direct correlation for both the longitudinal and transverse Resin 2 specimens, as well as for the transverse Resin 4A specimens. The 4A longitudinal-contraction data, however, exhibit no single consistent pattern throughout.

With regard to differences between resins, Tables 18 and 19 show that the total average thermal contractions of the Resin 4A composite specimens were about twice those of Resin 2. Because the resins exhibited highly similar thermal-contraction characteristics (Figure 10), it is reasonable to attribute the greater contraction of the 4A specimens to higher resin contents, rather than to an innate difference between the two resins. In addition, the data fail to suggest a simple relationship between a normal range of voids and thermal-contraction behavior.

The relationship between total thermal contraction and resin content is plotted for each orientation and resin in Figure 17. Least-square lines are

drawn through the data points. At least for these particular resin systems, the plots indicate that (a) filament orientation is an overriding factor in the thermal-contraction behavior of composites, (b) in parallel filament orientation, the glass filament determines the composite thermal-contraction characteristics, and (c) in normal filament orientation, the resin matrix and the resin content are dominant factors.

C. CONCLUSIONS

On the basis of tests performed on cast-resin specimens, Resins 4A and 2 had the best mechanical properties of the four resins tested. In tests on composite specimens of these two systems, the performance of Resin 4A was better than, or at least as good as, that of Resin 2. For filament-wound composites, however, Resin 2 is preferred because of its excellent processability; the short pot life and high viscosity of Resin 4A make it impractical for this purpose.

EXPERIMENTAL

Mechanical properties were determined as described below. In general, the methods adhered as closely as possible to those described in the latest American Society for Testing Materials (ASTM) Standards or in Federal Test Method (FTM) Standard No. 406. Cryogenic tests were performed using liquid nitrogen ($-320^{\circ}F$) and liquid hydrogen ($-423^{\circ}F$). The cryostat/tensile-machine assembly is shown in Figures 18 and 19.

A. CAST-RESIN PROPERTIES

1. Tensile Strength and Associated Properties

Stress-strain measurements were used to determine the tensile strength at fracture, elongation at fracture, modulus of elasticity, and toughness of the cured resin.

Each resin system was mixed according to its individual requirements (Table 1). The mixture was degassed (approximately 23 in. Hg) for 30 min, and was then bottom-drained into a 0.125-in.-thick flat-panel molding fixture pre-heated to the applicable initial cure temperature. The mold and contents were heated according to the individual cure schedules, and were then cooled. The panels were removed and routed to the configuration shown in Figure 20, and No. 600 sandpaper was used to produce a uniform surface, thereby reducing data scatter.

The test specimen was placed in the clamp fixture as shown in Figure 21. It was stressed in tension until failure occurred, using a 0.05-in./min crosshead travel rate. A 2-in. extensometer was used in conjunction with a tensile machine to provide a load-vs-strain diagram. To determine the elongation at fracture, two reference marks were inked on the specimen and were accurately measured to the nearest 0.05 in. After specimen rupture, the broken portions were fitted together and the distance between the reference marks was measured.

2. Notch Toughness

Notched tensile specimens were stressed until failure occurred. Notch toughness is a measure of the ability of a material to sustain high stresses without failure in the presence of local discontinuities such as cracks, notches, and imperfections. As modified for use in resins, the notch-toughness property demonstrates the capacity of the resin system to sustain loads despite imperfections in the matrix.

Each system was mixed according to its individual requirements (Table 1). The mixture was degassed (approximately 23 in. Hg) for 30 min, and was then bottom-drained into a 0.188-in.-thick flat-panel molding fixture preheated to the initial cure temperatures of the various resins. After curing, the mold was cooled. The panels were removed and routed to the configuration shown in Figure 22. The center notch was machined in the casting by drilling an 0.18-in.-dia hole in the center of the specimen, inserting the center-notch tool through the hole, and cutting the notch (see Figure 22).

The test specimen was placed in a clamp fixture, similar to the one shown in Figure 21, and was stressed in tension until failure occurred, using a 0.05-in./min crosshead travel.

3. Impact Strength

The relative susceptibility of a cast-resin material to shock fracture was determined at the three test temperatures. It is indicated by the energy expended by a standard pendulum-type (Charpy) impact machine in breaking the specimen in one blow.

Cast-resin panels were prepared as described for the tensile and notch-toughness panels. Specimens were cut and sanded to the dimensions shown in Figure 23.

For testing at -320°F, the specimens were cooled rapidly in LN_2 until temperature equilibrium was attained. They were then placed in aluminum-foil dishes filled with LN_2, and the test was performed with the specimens so contained. In this way, the desired -320°F temperature was obtained, with only a negligible strength contribution from the aluminum-foil dish.

For testing at -423°F, the specimens were cooled rapidly in LH_2, were stabilized at that temperature, and within a 6-sec period were transferred to the machine and tested. All specimens were supported against the steel blocks of the impact machine so that the blow was struck at their center.

4. Coefficient of Linear Thermal Contraction

The linear thermal contraction of cured resin was determined between -400 and +60°F.

Specimens were prepared from the same batch of resin mix used for tensile and notch-toughness specimens. The cure was as described in Table 1. After removal from the mold, the castings were cut and sanded into rectangular specimens. The finished specimens (three for each resin system) were 0.35 in. wide by 0.35 in. thick by 2.00 in. long. A 1/16-in. hole was drilled halfway through the center of each (normal to the length) for attachment of the thermocouple wire. The copper-constantan thermocouple was potted in the specimen with the aid of Shell 911F adhesive.

The test-specimen length between the two quartz points was measured at room temperature to the nearest 0.001 in. The specimen was placed in a fused-quartz dilatometer tube, the quartz rod was brought into contact with it, and the dial gage was positioned. The tube containing the specimen was placed in the cryostat. The system was purged with helium gas and was then cooled with LH_2 to cryogenic temperatures; the specimen was permitted to stabilize for 15 min before the temperature and gage reading were recorded. The system was gradually warmed to room temperature by slow pressurization with helium gas to replace the LH_2. Dimensional changes were recorded at 10° intervals between -400 and +60°F. Three test runs were made for each system. Before the specimens were tested, a 2-in.-long quartz sample was run to provide an instrument correction. A Leeds & Northrup Type K-3 universal potentiometer was used to monitor the temperature.

5. Thermal-Shock Resistance

The resistance of cast-resin specimens to thermal shock was determined by alternate immersions in water and in LH_2. The specimens were cut from unused impact specimens and were sanded to rectangular blocks 0.50 in. wide by 0.5 in. thick by 1.50 in. long.

After immersion in an LH_2-filled Dewar flask and equilibration for 15 min, the specimen was removed from the flask and placed in a 2000-ml beaker of water at room temperature. After 15 min, it was removed from the water, dried with compressed air, and inspected for signs of thermal cracking. The test procedures were repeated for a total of five cycles. Three specimens were tested for each resin system.

B. RESIN/GLASS-COMPOSITE PROPERTIES

1. Interlaminar Shear Strength

The interlaminar shear strengths of the glass/resin-composite specimens were determined by two test methods at +75, -320, and -423°F. Both methods employed centrally loaded, simply supported, laminate beams with geometric configurations that induced failure in interlaminar shear.

a. NOL Horizontal Shear

The interlaminar shear strengths of unidirectional composite specimens were determined by the horizontal-shear method at the three test temperatures.

Resins 2, 3, and 6 were prepared according to the procedure given in Table 1. Using the NOL-ring winding machine, the roving was passed through a dip tank containing the resin and was then wound directly on the mandrel (70 turns) under a tension of 15 ±2 lb. The mandrel was heated (150 to 175°F).

A diluted mix of Resin 4A was prepared as follows: Pulverized MOCA (55.2 g) was dissolved in 100 ml of analytical-grade MEK (15 min). Adiprene L-100 (100 g), Epon 826 (70 g), and Epon 871 (30 g) were successively added, with stirring. The solution was placed in the dip tank, and winding was performed as with the other systems.

After cures according to individual requirements, the rings were taken from the winding fixture and were routed to a thickness of approximately 0.125 in. Excess resin was removed with No. 280 sandpaper, and the

required arc segments (15) were cut from each ring; the width and thickness were measured to the nearest 0.001 in. The finished specimen was 0.250 in. wide, 0.635 in. long, and 0.125 in. thick. These dimensions conformed to those recently approved in the tentative test method under ASTM Standard D2344-65 T.

The specimen was aligned between the loading nose and supports, and was center-loaded on the convex side (Figure 24). The crosshead travel rate was 0.05 in./min and the specimen was stressed until failure occurred.

b. Short-Span Shear

The interlaminar shear strengths of unidirectional composite specimens were determined by the short-span-shear method at +75, -320, and -423°F.

Resins 2, 3, and 6 were prepared according to the procedures given in Table 1. With the drum-winding fixture, roving was passed through a dip tank containing the resin system and was then wound directly on the mandrel (under a roving tension of 7 ±2 lb) as unidirectional, side-by-side filaments. Three windings were prepared, each 24 by 56 in. in size.

The viscosity of Resin 4A made it unsuitable for "wet winding." Immediately after mixing (Table 1), the resin system was diluted with MEK (0.16 ml per gram of resin mix). This solution was brushed onto a cellophane release wrap that enveloped the mandrel. Roving was wound over the wet cellophane. Resin-poor areas were touched up with a brush after winding. Three windings were prepared, each 24 by 56 in. in size.*

The subsequent procedure was the same for all four systems. The windings were cut into six equal lengths perpendicular to the roving wind. These lengths were laid up so as to make an 18-ply, unidirectional, flat-panel laminate. The laminate was sandwiched between four plies of Dacron bleeder cloth and a layer of 181-glass cloth to facilitate release. The sandwich was placed on a steel plate that had been wrapped with Teflon-coated glass cloth. This assembly was then vacuum-bagged and cured in a vacuum according to the resin-system requirements. After curing, the laminate was removed and cut into short-span-shear specimens 2.00 in. long by 1.00 in. wide by 0.125 in. thick. The specimen width and thickness were measured to the nearest 0.001 in. Any necessary polishing was done only in the lengthwise direction.

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*It is now believed that the MEK-diluted Resin 4A mix would have provided comparable or even better results if it had been applied to the roving from a dip tank, although this was not actually attempted.

The specimen was mounted in the test fixture in the calibrated testing machine, and was carefully aligned between the loading nose and supports (Figure 25). The crosshead travel rate was 0.05 in./min and the specimen was stressed to failure.

2. Tensile Strength and Associated Properties

The tensile properties of composite material prepared with alternate filament orientations were determined as described below.

a. Specimens

Various steps in specimen preparation are considered individually in succeeding paragraphs.

Mixing of Resin 2 - Empol 1040 (80 g) and DSA (463.6 g) were mixed at ambient temperature and Epon 828 (400 g), preheated to 150°F, was added. The mixture was cooled to room temperature and 4.0 g BDMA was added, with stirring.

Mixing of Resin 4A - Adiprene L-100 (200 g) and Epon 871 (60 g) were heated separately to 150°F and were then mixed; Epon 826 (140 g) was heated to 150°F and stirred into the mixture. Molten MOCA (110.4 g) was added, and the resulting solution was stirred until homogeneous. Because of the short pot life of this system, four successively mixed batches of resin were required for each layer of winding.

Winding - A release film (Thalco 225) was applied to the mandrel of the winding machine. The roving was led through a resin bath and was wound directly over the film at a winding tension of 7 lb. With Resin 2, winding was performed at room temperature; with Resin 4A, the mix (temperature ≥150°F) was poured into a resin pot preheated to 150 to 170°F, and winding was begun promptly. Two heat lamps were trained on the revolving mandrel to keep the winding at 175 to 200°F and thus improve the resin flow and wetting properties. Four separate layers of glass were prepared (54 by 20 in.) for each resin and filament-orientation pattern.

Filament-Orientation Patterns - Three different patterns of filament orientation were used for each resin system (Figure 26): (1) unidirectional, 1:0 longitudinal-to-transverse filament ratio, (2) bidirectional, 1:1 longitudinal-to-transverse filament ratio, and (3) bidirectional, 1:2 longitudinal-to-transverse filament ratio.

Layup and Cure - Each 54 by 20-in. layer was cut into three equal pieces, approximately 18 in. square. The resulting 12-ply laminates were sandwiched between two layers of burlap (alternating orientation) and three layers of Dacron bleeder cloth on a Teflon-coated steel plate. (For the unidirectional laminate, cross plies of 181-glass cloth were placed between every third layer to reinforce the grip sections of the test specimens and prevent failure in these areas.) The laminate was fitted with a thermocouple in contact with the panel. It was placed in a vacuum bag and was cured on the following day in accordance with the appropriate schedule (Resin 2: 2 hours at 150°F, 4 hours at 300°F; Resin 4A: 5 hours at 285°F). For the Resin 4A panels, the vacuum bag also contained calcium sulfate drying agent.

Post-cure Handling - After curing, the panels were cut and routed to a tapered-end specimen configuration (Figure 20). No. 600 sandpaper was used to provide a uniform specimen surface and reduce data scatter.

b. Procedure

The test specimen was placed in the clamp fixture (Figure 21). It was stressed in tension until failure occurred, using a 0.05-in./min crosshead travel rate. An extensometer with a 2.0-in. gage length was used to record a continuous load-vs-deflection curve for each specimen. Deflection monitoring had to be discontinued before failure because the total specimen elongation exceeded the travel limits of the extensometer. The maximum elongation (at failure) could not be determined by measuring the post-failure distance between inked reference marks, because the composite specimens fractured in such a way that the marks were not legible. Elongation-at-failure values were therefore extrapolated from the graphs by extending the load-deflection curve to the load at failure.

Premature failure caused by the shearing of bolts or specimen slippage in the clamped grips (the latter particularly with unidirectional orientation) necessitated rerouting of the gage width of some specimens from approximately 0.400 in. to 0.250 in. and, in some cases, to 0.125 in. This approach assured tensile failure in the gage area, and the filament-strength values were not significantly different from those obtained with the wider specimens.

Filament strengths were not determined from the composite stresses (which are a function of the load at failure, the original cross-sectional area, and the resin content), but were calculated on the basis of the actual number of

fibers wound and oriented in the direction of loading. The methods used for, and examples of, filament-tensile-strength calculations are given in the Appendix.

3. Thermal-Shock Resistance

Composite specimens were tested in interlaminar shear after being cycled between room temperature and -423°F. They were NOL-ring segments used for NOL horizontal-shear tests, and were cut to the same finished dimensions (Figure 24).

After immersion for 15 min in a Dewar flask filled with LH_2, the specimens were placed in a 2000-ml beaker of water at room temperature. After 15 min, they were removed, dried with compressed air, and examined for signs of thermal cracking. This test procedure was repeated for a total of five cycles.

Upon completion of temperature cycling, the specimens were mounted in the interlaminar-shear fixture (Figure 24) for testing in LH_2. The specimen was aligned between the loading nose and supports, and was center-loaded on the convex side. The crosshead travel rate was 0.05 in./min, and the specimens were stressed to failure. The interlaminar-shear-strength values were compared with -423°F data for noncycled specimens to determine the extent of degradation in the resin-to-glass bond caused by thermal cycling.

4. Coefficient of Linear Thermal Contraction

The linear thermal contraction of composite specimens with unidirectionally oriented filaments was determined from measurements made parallel and transverse to the filaments.

a. Specimens

Various steps in specimen preparation are considered separately below.

Mixing of Resins - The two resins were mixed as described for the tensile-test specimens.

Winding - For Resin 2, the winding fixture was used to pass the roving through the dip tank containing the resin and wind it as unidirectional side-by-side filaments on a released 6 by 8-in. flat mandrel (roving tension, 13 lb). Each side of the mandrel had 50 plies (a number later found to be excessive).

A dip tank was not used for Resin 4A; instead, the mix was brush-coated directly on the mandrel. A layer of roving (tension, 12 lb) was

wound over the mandrel, and additional resin was brushed on. This alternate brushing/winding procedure was continued for 32 plies on each side of the mandrel. A heat gun, trained on the winding, maintained a temperature of approximately 150°F. The resin was held in a 150°F oven during winding, except when it was actually being applied.

It was not possible with either resin to complete the winding in a single day. The partially wound mandrels were removed from the machine and were enclosed in an air-tight cellophane bag that contained calcium sulfate drying agent. On the following day a fresh batch of resin was prepared and the winding was completed. (The resin on the No. 2 panel from the previous day was still workable; that on the No. 4A panel was thick, but blended smoothly when the new batch of resin was applied.)

Layup and Cure - Adjacent to each side of the mandrel-containing panels were placed four layers of Dacron bleeder cloth; each outer layer of Dacron was backed by a 1-in. layer of burlap that had been wrapped in 181-glass cloth. The resulting sandwich was placed on a Teflon-coated steel plate and was fitted with a thermocouple in contact with the panel. The assembly was vacuum-bagged and cured in accordance with the required schedule (see the layup and cure discussion in paragraph B,2,a, foregoing).

Post-cure Handling - After the cure, the panels were cut and sanded into rectangular beam specimens 2 in. long by 0.35 in. square. Six specimens were prepared for each system, three for testing in the longitudinal direction (parallel to the filament orientation) and three for testing in the transverse direction (normal to the filament orientation). With a 1/16-in. bit, a hole was drilled halfway through the center of each specimen (normal to the length) for attachment of a copper-constantan thermocouple wire. The latter was potted in the specimen with Shell 911F adhesive.

b. Procedure

The test procedure was the same as that used to determine the coefficient of linear thermal contraction of cast resin.

APPENDIX

CALCULATION OF FILAMENT AND COMPOSITE TENSILE STRESS IN COMPOSITE SPECIMENS

In analyzing the tapered-end test specimens employed in this program, it was assumed that the glass filaments are the primary structural material and therefore carry all the load. A method based on the total number of glass fibers oriented in the direction of loading was used in determining the filament stress; modification of the filament stress by the resin content yielded the composite stress.

To determine these stress levels, an equivalent filament thickness was first calculated from the specific amount of glass roving laid in the test-specimen gage area during winding. This method was used in order (1) to provide a proper basis for comparison of data with regard to filament orientation and resin content, and (2) to minimize differences in fabrication results, such as varying resin and void contents. In this manner, the composite specimens were analyzed on the basis of actual winding data and laboratory tests, thus eliminating the effects of differences in specimen thickness caused by nonuniform resin contents or unequal vacuum pressures on the test panels. The equivalent filament thickness (t_f) is given by

$$t_f = A_e N_1 N_2 N_3 N_4$$

where

A_e = area of single end = 21.0×10^{-6} sq in.

N_1 = number of ends per strand = 20

N_2 = number of strands per turn = 1

N_3 = number of turns per inch per layer = 11.5

N_4 = number of layers in test specimen = 12

Therefore,

$$t_f = (21.0 \times 10^{-6})(20)(1)(11.5)(12) = 0.058 \text{ in.}$$

Filament stress was determined by the ratio of load to area, factored by the fraction of filament layers oriented in the direction of loading. The lead of the winding machine and the width of the roving create an angular difference between the filament orientation and the direction of loading, but one so small that it may be ignored in the following equations:

$$\sigma_f = \frac{P}{t_f w} \quad \frac{N_4}{N_5}$$

where

σ_f = ultimate filament stress, psi

P = ultimate load, lb

w = test-specimen width, in.

N_5 = actual layers oriented in direction of load

and

$$\alpha_c = \frac{P}{t_c w} = \frac{P}{t_f w} \quad \frac{V_g}{100}$$

where

σ_c = ultimate composite stress, psi

t_c = total equivalent composite thickness = $\dfrac{t_f}{V_g/100}$

V_g = amount of glass filament in composite, vol% (see Figure 27)

Sample calculations follow. For a 1:2 filament orientation,

$$\sigma_f = \frac{4340}{(0.058)(0.407)} \left(\frac{12}{4}\right) = 551{,}500 \text{ psi}$$

$$\sigma_c = \frac{4340}{(0.058)(0.407)} \quad (0.65) = 119{,}500 \text{ psi}$$

For a 1:1 filament orientation,

$$\sigma_f = \frac{5200}{(0.058)(0.402)} \left(\frac{12}{6}\right) = 446{,}000 \text{ psi}$$

$$\sigma_c = \frac{5200}{(0.058)(0.402)} (0.6875) = 153{,}300 \text{ psi}$$

For a 1:0 filament orientation,

$$\sigma_f = \frac{5080}{(0.058)(0.233)} \left(\frac{12}{12}\right) = 375{,}900 \text{ psi}$$

$$\sigma_c = \frac{5080}{(0.058)(0.233)} (0.6237) = 234{,}400 \text{ psi}$$

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TABLE 1

CANDIDATE RESIN SYSTEMS FOR MECHANICAL-PROPERTY TESTING

No.	Resin Formulation (Parts by Weight)	Mixing Procedure and Cure Schedule
2	Epon 828/DSA/Empol 1040/BDMA (100/115.9/20/1)	Mix and warm epoxy and Empol 1040 to 212°F. Cool to room temperature and add DSA and BDMA. Cure: 2 hours at 150°F, 4 hours at 300°F.
3	Epon 828/DSA/BOHET/BDMA (100/134/26/1)	Mix epoxy, DSA, and BOHET; then add BDMA. Cure: 2 hours at 150°F, 4 hours at 300°F. Alternate cure: 8 hours at 225°F.
4A	Epon 826/Epon 871/Adiprene L-100/MOCA (35/15/50/27.6)	Heat the epoxies and L-100 to 212°F and degas them for 10 min, with continuous stirring, in a 29-in.-Hg vacuum. Melt MOCA (ground with mortar and pestle) at 225°F and add it to the mix. Degas the mix as before, with stirring, for 3 min. Cure: Pour into heated mold at 200°F, heat 5 hours at 285°F.
6	Epon 826/Empol 1040/Z-6077/DSA/BDMA (80/20/20/115.9/1)	Heat Epon 826 and Empol 1040 to 212°F and mix thoroughly at this temperature. Cool to ambient temperature and add, while stirring, Z-6077, DSA, and finally BDMA. Cure: 8 hours at 260°F.

TABLE 2

RESIN-SYSTEM INGREDIENTS

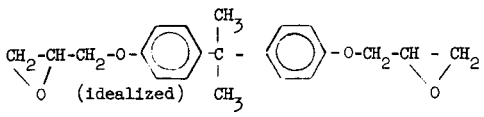
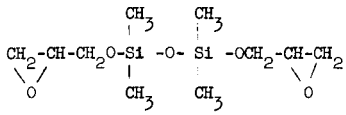
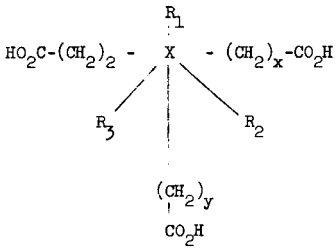
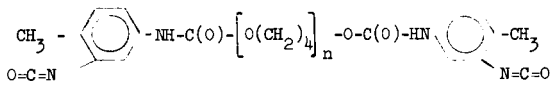
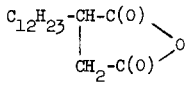
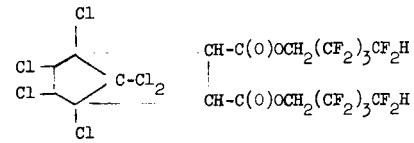
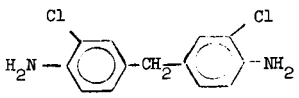
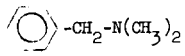
Ingredient	Structure	Description & Source
Epon 828 Epon 826		Bisphenol-A epoxy. Shell Chemical Company, Plastics and Resins Division, Downey, Calif.
Epon 871	Proprietary compound	Aliphatic epoxy. Shell Chemical, Downey.
Z-6077		1,3-Bis(3-glycidoxypropyl)-tetramethyl-disiloxane. Dow-Corning Corporation, Midland, Mich.
Empol 1040		Aliphatic tricarboxy acid (molecular weight 845). Emery Industries, Inc., Cincinnati, Ohio.
Adiprene L-100		Polyurethane prepolymer. E. I. du Pont de Nemours & Company, Wilmington, Del.
DSA		Dodecenylsuccinic anhydride. Allied Chemical Corporation, New York, N.Y.
BOHET		HET-acid bis(octafluoropentyl)ester. Aerojet-General Corporation, Azusa, Calif.
MOCA		4,4'-Methylene-bis(2-chloroaniline). Du Pont, Wilmington.
BDMA		Benzyldimethylamine. Sumner Chemical Company, New York, N.Y.

TABLE 3

TENSILE-TEST RESULTS (75°F)

Resin No.	Tensile Stress, 10^3 psi		Tensile Modulus 10^3 psi	Elongation, %		Toughness* lb-in./cu in.
	Ultimate	At Fracture		At Ultimate Stress	At Fracture	
2	6.80	6.15	253.1	3.6	5.0	233.1
	6.72	6.13	308.0	3.6	4.4	
	6.60	6.17	308.0	3.7	4.0	
	6.87	5.92	-	-	-	
	6.65	5.13	-	-	-	
	Av 6.73	5.90	289.7	3.6	4.5	
3**	6.14	6.14	353.4	2.2	2.2	157.1
	7.27	7.27	349.0	3.5	3.5	
	7.22	7.00	330.7	3.8	4.2	
	7.43	6.47	-	-	-	
	7.55	7.55	-	-	-	
	Av 7.12	6.89	344.3	3.2	3.3	
4A	1.86	1.86	-	142	142	1715.0
	2.55	2.55	16.0	149	149	
	2.49	2.49	15.8	149	149	
	2.67	2.67	9.1	152	152	
	2.47***	2.47***	11.2***	120***	120***	
	Av 2.41	2.41	13.0	142.4	142.4	
6	5.57	5.57	278.5	3.0	3.0	109.6
	5.75	5.75	282.4	3.1	3.1	
	5.57	5.57	294.2	3.0	3.0	
	5.70	4.37	-	-	-	
	5.66	4.26	-	-	-	
	Av 5.65	5.10	285.0	3.0	3.0	

*Calculated from area under stress-strain curve.

**Specimens prepared using alternate cure (8 hours at 225°F).

***Valve included in averages, although specimen did not fail but slipped out of grip at level shown.

TABLE 4

TENSILE-TEST RESULTS (-320°F)

Resin No.	Tensile Stress, 10^3 psi: Ultimate	Tensile Stress, 10^3 psi: At Fracture	Tensile Modulus 10^3 psi	Elongation, %: At Ultimate Stress	Elongation, %: At Fracture	Toughness* lb-in./cu in.
2	15.26	15.26	758	2.0	2.0	145.0
	**	**	**	**	**	
	14.44	14.44	745.8	2.0	2.0	
	13.75	13.75	656.8	2.0	2.0	
	15.76	15.76	-	-	-	
	Av 14.80	14.80	720.2	2.0	2.0	
3***	10.65	10.65	683.9	1.5	1.5	95.1
	****	****	****	****	****	
	11.79	11.79	694.9	1.7	1.7	
	****	****	727.4	****	****	
	12.33	12.33	764.5	1.6	1.6	
	Av 11.59	11.59	728.9	1.6	1.6	
4A	29.25	29.25	1004.9	3.1	3.1	446.0
	25.90	25.90	1066.8	2.8	2.8	
	27.61	27.61	1033.8	-	-	
	27.13	27.13	-	-	-	
	30.13	30.13	-	-	-	
	Av 28.00	28.00	1035.1	2.95	2.95	
6	12.17	12.17	746.7	1.8	1.8	109.6
	12.89	12.89	-	1.4	1.4	
	11.16	11.16	753.3	1.5	1.5	
	15.28	15.28	766.6	1.9	1.9	
	-	-	771.9	-	-	
	Av 12.87	12.87	759.6	1.65	1.65	

* Calculated from area under stress-strain curve.

** Specimen fractured during setup in grips.

*** Alternate cure (8 hours at 225°F).

**** Specimen fractured during testing in LN_2.

TABLE 5

TENSILE-TEST RESULTS (-423°F)

Resin No.	Tensile Stress, 10^3 psi		Tensile Modulus 10^3 psi	Elongation, %		Toughness* lb-in./cu in.
	Ultimate	At Fracture		At Ultimate Stress	At Fracture	
2	14.53	14.53	978.8	1.6	1.6	137.0
	11.12	11.12	990.7	1.6	1.6	
	17.13	17.13	963.2	1.9	1.9	
	13.73	13.73	-	-	-	
	15.80	15.80	-	-	-	
	Av 14.46	14.46	977.5	1.7	1.7	
3**	12.86	12.86	972.6	1.3	1.3	86.3
	11.65	11.65	-	-	-	
	13.08	13.08	951.7	1.4	1.4	
	10.39	10.39	-	-	-	
	13.14	13.14	-	-	-	
	Av 12.23	12.23	962.1	1.35	1.35	
4A	23.13	23.13	1835.5	2.0	2.0	226.0
	21.57	21.57	998.0	2.0	2.0	
	22.18	22.18	1145.4	2.0	2.0	
	17.15	17.15	1246.0	1.5	1.5	
	27.90	27.90	-	-	-	
	22.18	22.18	-	-	-	
	Av 22.35	22.35	1306.2	1.9	1.9	
6	13.13	13.13	827.2	1.9	1.9	111.3
	11.14	11.14	934.2	1.5	1.5	
	12.13	12.13	942.6	1.3	1.3	
	15.75	15.75	-	-	-	
	14.10	14.10	-	-	-	
	Av 13.25	13.25	901.3	1.6	1.6	

*Calculated from area under stress-strain curve.

**Alternate cure (8 hours at 225°F).

TABLE 6

NOTCH TOUGHNESS, TEST RESULTS

Resin No.	Notch Toughness, psi $\sqrt{\text{in.}}$				
	At +75°F		At -320°F		At -423°F
2	1484.5		1639.6		1272.3
	1566.1		1742.1		1347.7
	1236.7		1541.2		1426.6
	1361.2		1585.4		1367.5
	1561.2		1678.1		1313.0
	Av 1441.9		1637.3		1345.4
3	603.7	346.7	*	1399.7	745.3
	1263.8	372.8	1141.9	1316.8	819.2
	1147.1	403.1	833.5	1139.3*	746.8
	915.4	500.3	688.3	-	739.9
	2178.7	443.5	*	-	762.4
	Av 1221.7**	413.3***	887.9***	1285.3**	762.7***
4A	346.5		3954.2		3115.0
	377.0		3681.1		3093.4
	365.1		3744.1		2751.1
	399.1		3772.2		2893.7
	385.7		3417.5		2600.8
	Av 374.7		3713.8		2890.8
6	2890.9		2035.7		1334.8
	2811.9		953.1*		1385.8
	2930.7		1586.8		1241.5
	2951.3		944.2*		1341.5
	2928.6		1379.9		1337.6
	Av 2920.7		1667.5		1328.3

*Specimen fractured when immersed in LN_2.

**Cured 2 hours at 150°F and 4 hours at 300°F.

***Cured 8 hours at 225°F.

TABLE 7

IMPACT STRENGTH, TEST RESULTS (CHARPY UNNOTCHED)

Resin No.	Type of Value	Impact Strength, ft-lb/in.* At +75°F	At -320°F	At -423°F
2	High	4.97	3.13**	7.99
	Low	1.27	1.65	3.68
	Av	3.23	2.52	6.54
3	High	4.36	2.67**	8.08
	Low	3.16	1.84	3.13
	Av	3.48	2.27	5.48
4A	High	41.9***	19.59	14.80
	Low	34.6	6.52	10.36
	Av	37.7	12.16	12.38
6	High	18.72	9.89**	6.34
	Low	5.75	1.23	2.96
	Av	10.65	3.80	4.42

*Minimum of five specimens tested for each resin system at each test temperature. -320°F data: Specimens rapidly cooled, stabilized at -320°F, and tested in aluminum-foil dish filled with LN_2. -423°F data: Specimens gradually cooled, stabilized at -423°F, and tested with 6-sec time lag (maximum).

**Thermal-shock cracking of specimens noted during immersion in LN_2.

***Specimens did not fracture but were tossed from fixture.

TABLE 8

CAST-RESIN LINEAR THERMAL CONTRACTION, TEST RESULTS

Temperature °F	$\Delta L/L$, 10^{-3} in./in.*			
	Resin 2	Resin 3	Resin 4A	Resin 6
+60	0	0	0	0
0	2.507	2.801	3.637	3.031
-50	4.664	4.999	6.161	5.423
-100	6.853	7.145	8.237	7.741
-150	8.701	9.157	10.014	9.873
-200	10.416	10.992	11.538	11.712
-250	11.868	12.845	12.713	13.349
-300	13.156	14.172	13.635	14.592
-350	13.966	15.088	14.083	15.465
-400	14.769	15.904	14.814	16.069
-407	14.944	-	-	-
-410	-	15.948	14.839	-
-417	-	-	-	16.238

*$\Delta L/L$ = Change in length due to cooling/test-specimen length at room temperature. Values are averages of three runs. Calculated average coefficients of linear thermal contraction between +60 and -400°F:

Resin 2 - 32.11 x 10^{-6} in./in./°F
Resin 3 - 34.46 x 10^{-6} in./in./°F
Resin 4A- 32.20 x 10^{-6} in./in./°F
Resin 6 - 34.93 x 10^{-6} in./in./°F.

TABLE 9

THERMAL-SHOCK RESISTANCE, TEST RESULTS*

Resin No.	Specimen No.	Slow Cooling	Rapid Cooling				
			Cycle 1	Cycle 2	Cycle 3	Cycle 4	Cycle 5
2	1	N	N	N	C	**	-
	2	N	N	N	N	C	-
	3	N	N	N	N	N	N
3	1	N	C	-	-	-	-
	2	N	C	-	-	-	-
	3	N	C	-	-	-	-
4A	1	N	N	N	N	N	N
	2	N	N	N	N	N	N
	3	N	N	N	N	N	N
6	1	N	N	N	C	**	-
	2	N	N	N	C	**	-
	3	N	N	N	N	N	N

*Specimens cooled slowly or rapidly to -423°F and then placed in water at room temperature. N = no discernible change. C = thermal cracking.

**No further testing attempted after specimen cracked.

TABLE 10

INTERLAMINAR SHEAR, TEST RESULTS (NOL HORIZONTAL-SHEAR METHOD)*

Resin No.	Resin wt%	Voids vol%	Composite Specific Gravity	Shear Stress, 10^3 psi		
				+75°F	-320°F	-423°F
2	17.86	4.70	1.903	6.41	14.31	15.93
				6.79	16.41	16.87
				6.53	12.91	15.48
				6.91	14.61	15.28
				6.62	14.31	16.97
				Av 6.65	14.11	16.11
3**	17.60	3.41	1.954	6.64	15.00	15.56
				6.40	13.01	15.28
				6.45	16.21	18.47
				6.95	12.51	15.18
				6.45	12.91	15.00
				Av 6.58	13.93	15.90
4A	13.11	9.56	1.931	1.93	10.00	13.34
				2.08	9.81	13.01
				2.08	12.61	13.02
				2.04	12.11	10.50
				1.98	9.71	11.25
				Av 2.02	10.87	12.22
6	17.47	5.63	1.891	5.64	15.11	13.50
				5.72	16.31	15.00
				5.67	15.51	14.28
				5.30	15.71	14.71
				5.63	11.70***	15.54
				Av 5.59	15.66	14.61

*Values shown for resin content, void content, and specific gravity are averages of three tests for each resin system. Specimens were segments from the same NOL ring used in fabricating horizontal-shear specimens.

**Specimens prepared using alternate cure cycle (8 hours at 225°F).

***Data rejected because specimen slipped in fixture during test.

TABLE 11

RESIN 4A INTERLAMINAR SHEAR, TEST RESULTS
(NOL HORIZONTAL-SHEAR METHOD)

Specimen	Ratio of MEK (ml) to L-100 (g)	Resin wt%	Voids vol%	Composite Specific Gravity	Shear Stress, 10^3 psi		
					+75°F	-320°F	-423°F
A	1/1	13.11	9.56	1.931	1.93	10.00	13.34
					2.08	9.81	13.01
					2.08	12.61	13.02
					2.04	12.11	10.50
					1.98	9.71	11.25
					Av 2.02	10.87	12.22
B	1/4	10.64	5.66	2.069	3.06	12.55	15.77
					3.12	12.44	12.15
					3.14	15.93	12.03
					3.09	17.41	16.29
					3.04	18.09	14.74
					Av 3.09	15.28	14.20
C	0/1	13.31	3.92	2.053	5.17	17.11	15.67
					5.09	15.13	17.41
					5.14	12.85	19.82
					5.01	16.82	17.14
					5.27	20.06	19.65
					Av 5.14	16.40	17.94

TABLE 12

RESIN 2 INTERLAMINAR SHEAR, TEST RESULTS
(NOL HORIZONTAL-SHEAR METHOD)

Specimen	Resin, wt%	Voids vol%	Composite Specific Gravity		Shear Stress, 10^3 psi +75°F	-320°F	-423°F
A	17.86	4.70	1.903		6.41	14.31	15.93
					6.79	16.41	16.87
					6.53	12.91	15.48
					6.91	14.61	15.28
					6.62	14.31	16.97
				Av	6.65	14.11	16.11
B	11.44	2.54	2.086		5.92	13.71	14.50
					5.85	13.76	16.75
					6.36	15.85	15.48
					6.23	15.04	15.58
					6.50	12.45	14.64
				Av	6.17	14.16	15.39

TABLE 13

INTERLAMINAR SHEAR, TEST RESULTS (SHORT-SPAN-SHEAR METHOD)

Resin No.	Resin wt%*	Voids vol%*	Composite Specific Gravity	Shear Stress, 10^3 psi		
				+75°F	-320°F	-423°F
2	27.54	9.42	1.644	4.25	9.32	9.29
				4.40	8.45	8.57
				4.58	9.10	9.37
				4.16	9.48	8.46
				4.24	10.31	8.49
				Av 4.32	9.33	8.84
3	22.85	11.91	1.701	4.03	7.31	7.77
				4.17	7.94	8.54
				4.11	8.46	8.18
				4.05	7.97	8.29
				3.82	7.51	8.30
				Av 4.04	7.84	8.42
4A	30.54	15.44	1.550	0.53	8.46	7.25
				0.52	8.31	7.16
				0.54	8.39	8.54
				0.57	7.67	7.61
				0.53	7.86	7.79
				Av 0.54	8.14	7.76
6	23.49	6.31	1.766	4.57	11.80	9.49
				4.53	10.37	10.16
				4.26	11.41	10.47
				4.21	10.94	9.64
				4.18	9.93	9.76
				Av 4.35	10.89	9.90

*Averages of three tests for each resin system.

TABLE 14

COMPOSITE TENSILE STRENGTH, TEST RESULTS (UNIDIRECTIONAL 1:0 ORIENTATION)

Resin No.	Temp °F	Resin wt%	Voids vol%	Ult Load lb	Specimen Width in.	Ultimate Tensile Strength, 10^3 psi Filament	Ultimate Tensile Strength, 10^3 psi Composite	Tensile Modulus 10^3 psi	Maximum Elongation, %*
2	+75	21.0	7.03	**	0.197	-		-	-
				2260	0.110	354.2		8.7	2.3
				2625	0.121	374.0		8.2	2.6
				2380	0.111	369.6		8.6	2.3
				**	0.122	-		-	-
						Av 365.9	228.2	8.5	2.4
2	-320	21.0	7.03	**	0.241	-		-	-
				**	0.245	-		-	-
				2970	0.123	416.3		8.9	2.7
				3325	0.125	458.6		8.1	1.8
				3195	0.116	474.8		10.3	2.8
						Av 449.9	280.6	9.1	2.4
2	-423	21.0	7.03	5080	0.233	375.9		***	***
				5320	0.251	365.4		14.5	1.4
				2820	0.122	398.5		10.3	2.2
				5040	0.243	357.6		***	***
				**	0.248	-		-	-
						Av 374.3	233.5	12.4	1.8
4A	+75	43.6	16.7	**	0.249	-		-	-
				1990	0.127	270.1		3.8	2.0
				2025	0.125	279.3		3.8	2.1
				1975	0.130	261.9		3.5	2.1
				1820	0.125	251.0		3.8	1.9
						Av 265.6	101.5	3.8	2.0
4A	-320	43.6	16.7	4500	0.130	596.8		5.4	3.1
				****	0.248	-		-	-
				4550	0.123	637.7		9.7	2.0
				4370	0.124	607.6		10.7	1.5
				4200	0.128	565.7		8.2	1.1
						Av 601.9	229.9	8.5	1.9
4A	-423	43.6	16.7	7950	0.252	543.9		3.7	4.0
				3510	0.123	492.0		6.2	2.9
				6850	0.253	466.8		6.2	3.7
				7090	0.243	503.0		6.2	2.4
				6820	0.253	464.7		7.1	2.7
						Av 494.0	188.7	5.9	3.1

*Extrapolated values.

**Specimen slipped in jaws of fixture during test.

***Inconsistent load-elongation curve.

****Bolt failure in test fixture.

TABLE 15

COMPOSITE TENSILE STRENGTH, TEST RESULTS (BIDIRECTIONAL 1:1 ORIENTATION)

Resin No.	Temp °F	Resin wt%	Voids vol%	Ult Load lb	Specimen Width in.	Ultimate Tensile Strength, 10^3 psi Filament	Ultimate Tensile Strength, 10^3 psi Composite	Tensile Modulus 10^3 psi	Maximum Elongation, %*
2	+75	16.7	1.09	2400	0.249	332.2		4.1	2.6
				2375	0.242	338.4		4.3	2.6
				2610	0.248	362.8		4.1	2.9
				2525	0.244	356.8		4.2	2.8
				2570	0.238	372.2		4.1	3.0
						Av 352.4	121.1	4.2	2.8
2	-320	16.7	1.09	3950	0.241	565.1		4.4	4.2
				3675	0.242	523.6		3.9	4.4
				4065	0.252	556.2		4.1	4.4
				3175	0.244	448.6		3.8	3.7
				3750	0.240	538.6		3.6	4.6
						Av 526.4	181.0	4.0	4.3
2	-423	16.7	1.09	5200	0.402	446.0		4.0	4.5
				5490	0.402	470.8		4.3	3.7
				5620	0.413	469.2		5.2	2.7
				5680	0.409	478.8		4.1	3.7
				5550	0.409	467.9		5.1	3.0
						Av 466.6	160.4	4.5	3.5
4A	+75	17.3	5.10	1920	0.250	264.8		3.7	2.1
				1900	0.255	256.8		3.7	2.1
				1875	0.252	256.4		3.8	2.0
				1700	0.245	239.2		3.6	2.0
				1830	0.251	251.4		3.7	2.1
						Av 253.7	88.2	3.7	2.1
4A	-320	17.3	5.10	4280	0.250	590.2		4.2	4.4
				4505	0.250	621.2		4.9	3.8
				4125	0.249	571.2		4.0	4.4
				3770	0.248	524.0		4.1	3.9
				4500	0.251	618.2		4.0	4.6
						Av 584.9	203.2	4.2	4.2
4A	-423	17.3	5.10	6140	0.404	524.0		**	**
				6400	0.404	546.2		4.4	3.2
				5600	0.402	480.2		4.2	3.5
				3680	0.251	505.4		4.1	4.1
				***	-	-		-	-
						Av 513.9	178.6	4.2	3.6

*Extrapolated values.

**Inconsistent load-elongation curve.

***Specimen broke during assembly in test fixture.

TABLE 16

COMPOSITE TENSILE STRENGTH, TEST RESULTS (BIDIRECTIONAL 1:2 ORIENTATION)

Resin No.	Temp °F	Resin wt%	Voids vol%	Ult Load lb	Specimen Width in.	Ultimate Tensile Strength, 10^3 psi Filament	Ultimate Tensile Strength, 10^3 psi Composite	Tensile Modulus 10^3 psi	Maximum Elongation, %*
2	+75	19.2	1.70	2000	0.244	423.9		2.8	3.3
				1980	0.247	417.6		2.6	3.4
				1945	0.246	408.9		2.7	3.2
				1950	0.256	393.9		2.5	3.2
				1830	0.246	384.6		2.7	3.1
						Av 405.7	87.9	2.7	3.2
2	-320	19.2	1.70	2125	0.244	450.3		-	-
				3375	0.252	692.7		2.6	5.4
				3100	0.264	607.2		2.4	5.3
				3125	0.245	658.5		2.8	4.7
				3025	0.271	577.2		2.7	4.6
						Av 597.2	129.4	2.6	4.7
2	-423	19.2	1.70	4340	0.407	551.5		**	**
				4180	0.401	539.1		2.9	3.3
				4520	0.403	580.1		3.2	3.8
				3920	0.408	496.9		3.8	2.9
				4750	0.402	611.1		2.6	4.9
						Av 555.7	120.4	3.1	3.8
4A	+75	19.2	5.27	700	0.250	144.8		2.2	1.2
				940	0.254	191.4		2.4	1.7
				1325	0.250	274.1		2.5	2.2
				705	0.253	144.1		2.3	1.3
				1240	0.252	254.5		2.4	2.2
						Av 201.8	44.9	2.4	1.7
4A	-320	19.2	5.27	1405	0.247	***		-	-
				2950	0.249	612.6		2.6	5.0
				2970	0.242	635.7		2.8	4.8
				3305	0.298	689.1		2.7	5.1
				2940	0.248	612.9		2.5	4.9
						Av 637.5	141.7	2.6	4.9
4A	-423	19.2	5.27	1860	0.400	****		-	-
				3300	0.412	****		-	-
				4750	0.414	608.1		2.8	4.4
				2800	0.396	****		-	-
				4580	0.407	582.0		2.4	4.4
						Av 595.0	132.3	2.6	4.4

* Extrapolated values.

** Inconsistent load-elongation curve.

*** Statistical outlier.

**** Specimen delaminated and broke in shoulder section.

TABLE 17

COMPOSITE THERMAL-SHOCK RESISTANCE, TEST RESULTS
(NOL HORIZONTAL-SHEAR METHOD)

Resin No.	Resin wt%	Voids vol%	Shear Stress at -423°F, 10^3 psi	
			Cycled Specimens	Noncycled* Specimens
2	11.44	2.54	15.00	-
			16.99	-
			13.83	-
			12.89	-
			17.22	-
			Av 15.19	15.39
4A	13.31	3.92	14.87	-
			14.83	-
			15.13	-
			16.61	-
			17.41	-
			Av 15.70	17.94

*Noncycled specimens (see Tables 11 and 12) cut from same NOL ring as cycled specimens.

TABLE 18

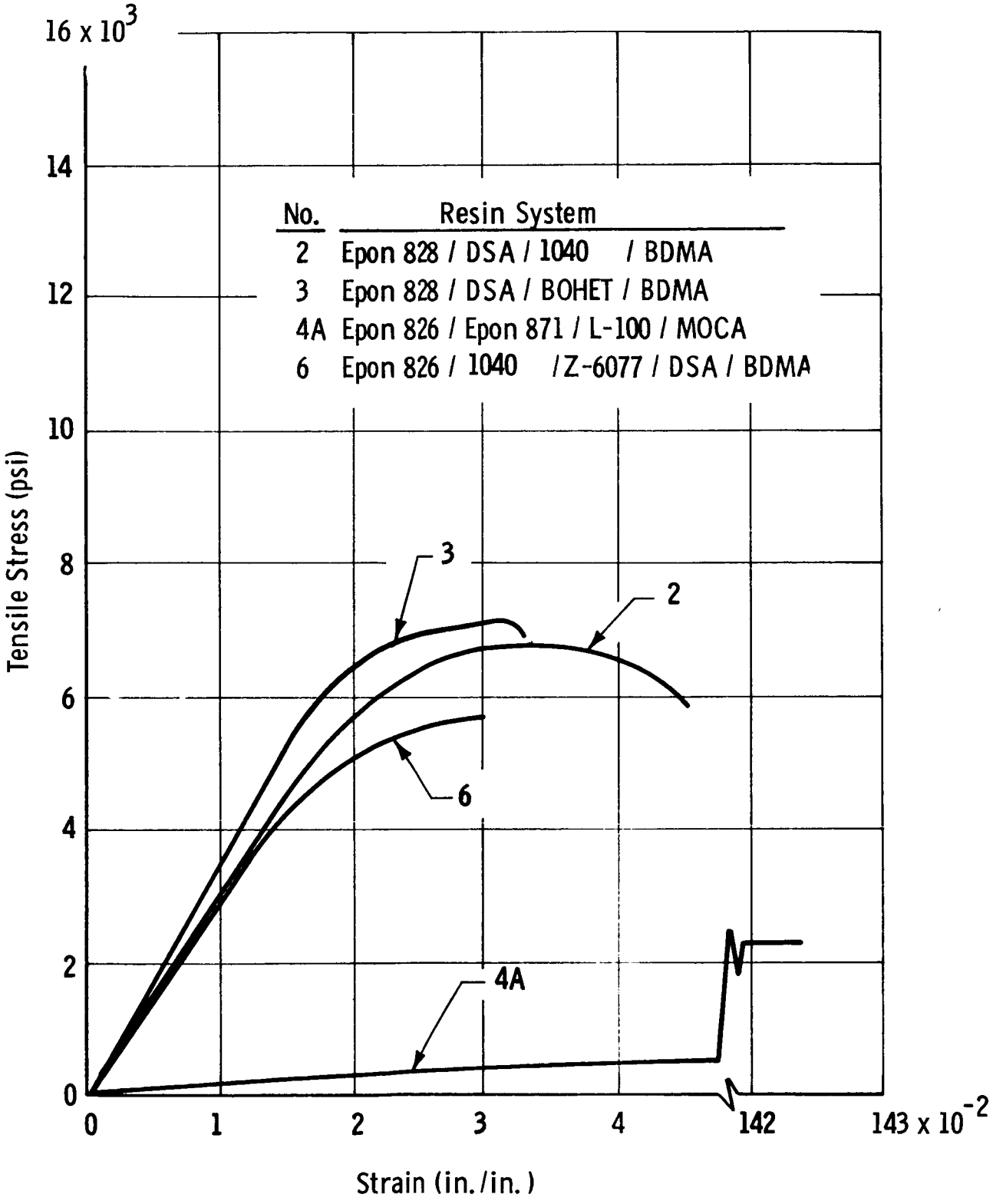
COMPOSITE LINEAR THERMAL CONTRACTION, TEST RESULTS (RESIN 2)

Temp °F	ΔL/L, 10^{-3} in./in.							
	Parallel (Longitudinal)				Normal (Transverse)			
	Specimen 1	Specimen 2	Specimen 3	Average	Specimen 1	Specimen 2	Specimen 3	Average
+70	0	0	0	0	0	0	0	0
+50	0.086	0.087	0.113	0.095	0.162	0.162	0.319	0.212
0	0.111	0.136	0.138	0.128	0.636	0.636	0.010	0.761
-50	0.173	0.198	0.227	0.199	1.073	1.114	1.696	1.305
-100	0.210	0.284	0.265	0.254	1.585	1.634	2.407	1.875
-150	0.284	0.383	0.340	0.336	2.059	2.133	2.931	2.374
-200	0.371	0.420	0.353	0.381	2.421	2.569	3.443	2.811
-250	0.445	0.445	0.378	0.423	2.770	2.969	3.917	3.219
-300	0.457	0.457	0.391	0.435	3.057	3.305	4.279	3.547
-350	0.470	0.470	0.403	0.448	3.170	3.490	4.466	3.709
-400	0.519	0.495	0.429	0.481	3.419	3.817	4.790	4.009
Resin, wt%	19.1	18.5	13.8	17.1	12.9	14.7	15.4	14.4
Voids, vol%	9.0	8.85	2.73	6.9	2.84	4.3	4.26	3.8

TABLE 19

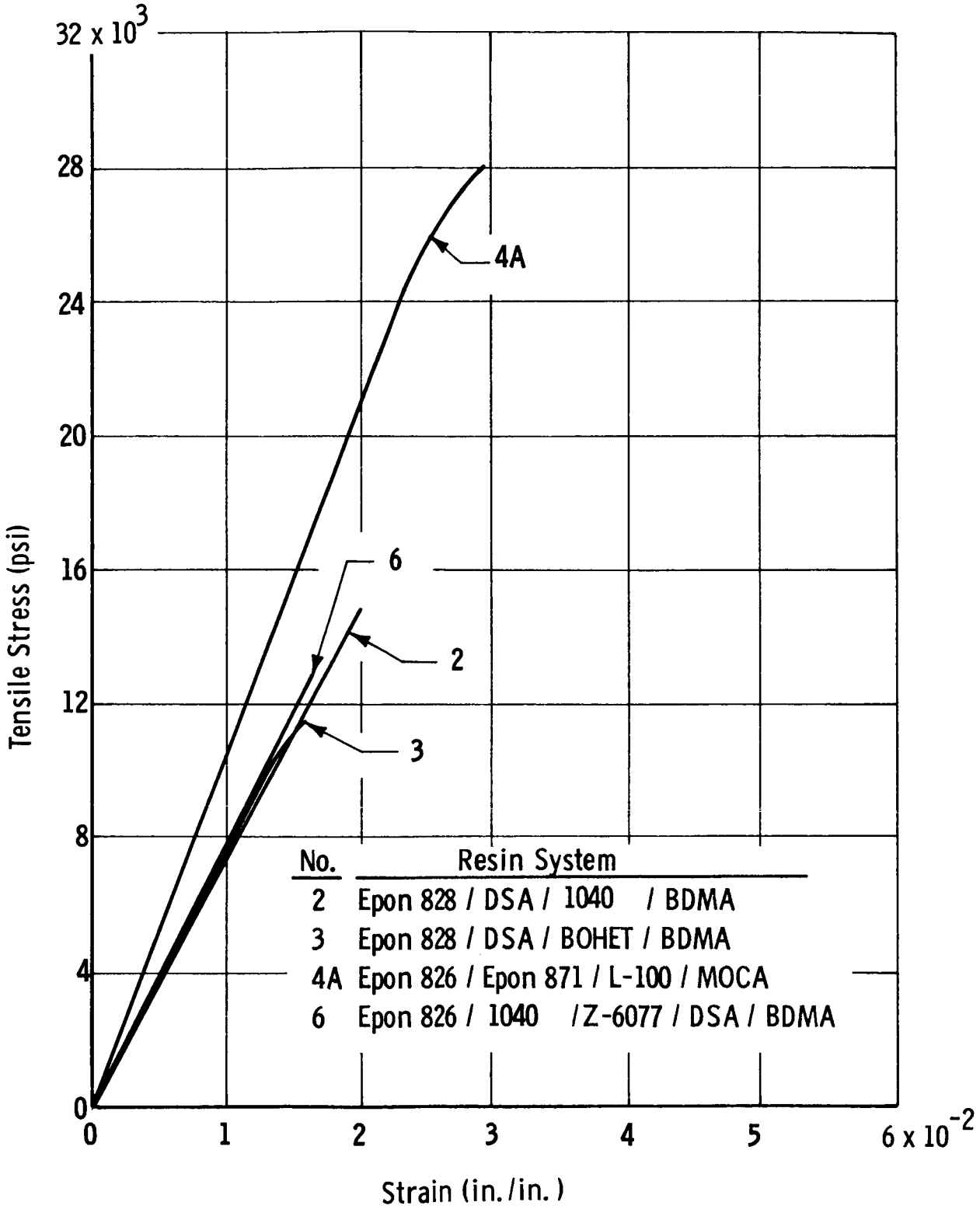
COMPOSITE LINEAR THERMAL CONTRACTION, TEST RESULTS (RESIN 4A)

Temp °F	ΔL/L, 10^{-3} in./in.							
	Parallel (Longitudinal)				Normal (Transverse)			
	Specimen 1	Specimen 2	Specimen 3	Average	Specimen 1	Specimen 2	Specimen 3	Average
+70	0	0	0	0	0	0	0	0
+50	0.113	0.112	0.112	0.112	0.534	0.742	0.783	0.686
0	0.213	0.212	0.212	0.212	1.975	2.429	2.549	2.318
-50	0.352	0.324	0.324	0.333	3.007	3.700	3.805	3.504
-100	0.490	0.462	0.462	0.471	3.963	4.745	4.863	4.524
-150	0.641	0.587	0.586	0.605	4.833	5.652	5.883	5.456
-200	0.780	0.674	0.724	0.726	5.541	6.445	6.741	6.242
-250	0.881	0.749	0.824	0.818	6.113	7.099	7.412	6.875
-300	0.969	0.812	0.936	0.905	6.523	7.590	7.873	7.329
-350	1.032	0.849	0.974	0.951	6.734	7.754	8.134	7.541
-400	1.132	0.924	1.048	1.035	7.032	7.854	8.507	7.798
Resin wt%	33.9	39.7	39.6	37.7	26.7	31.7	32.8	30.4
Voids, vol%	8.10	7.66	6.80	7.5	6.30	8.53	7.16	7.3



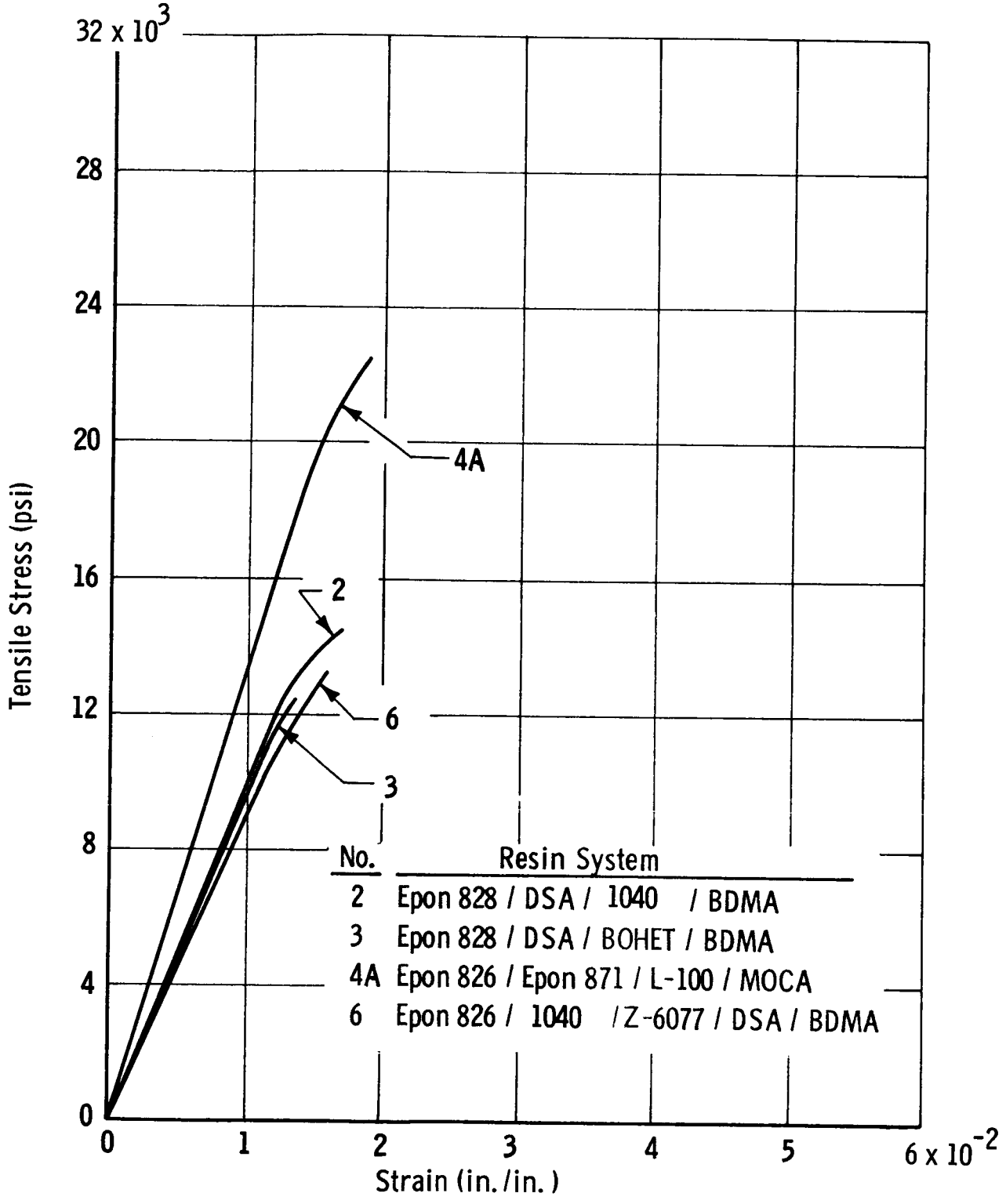
Resin Stress-Strain Curves (75°F)

Figure 1



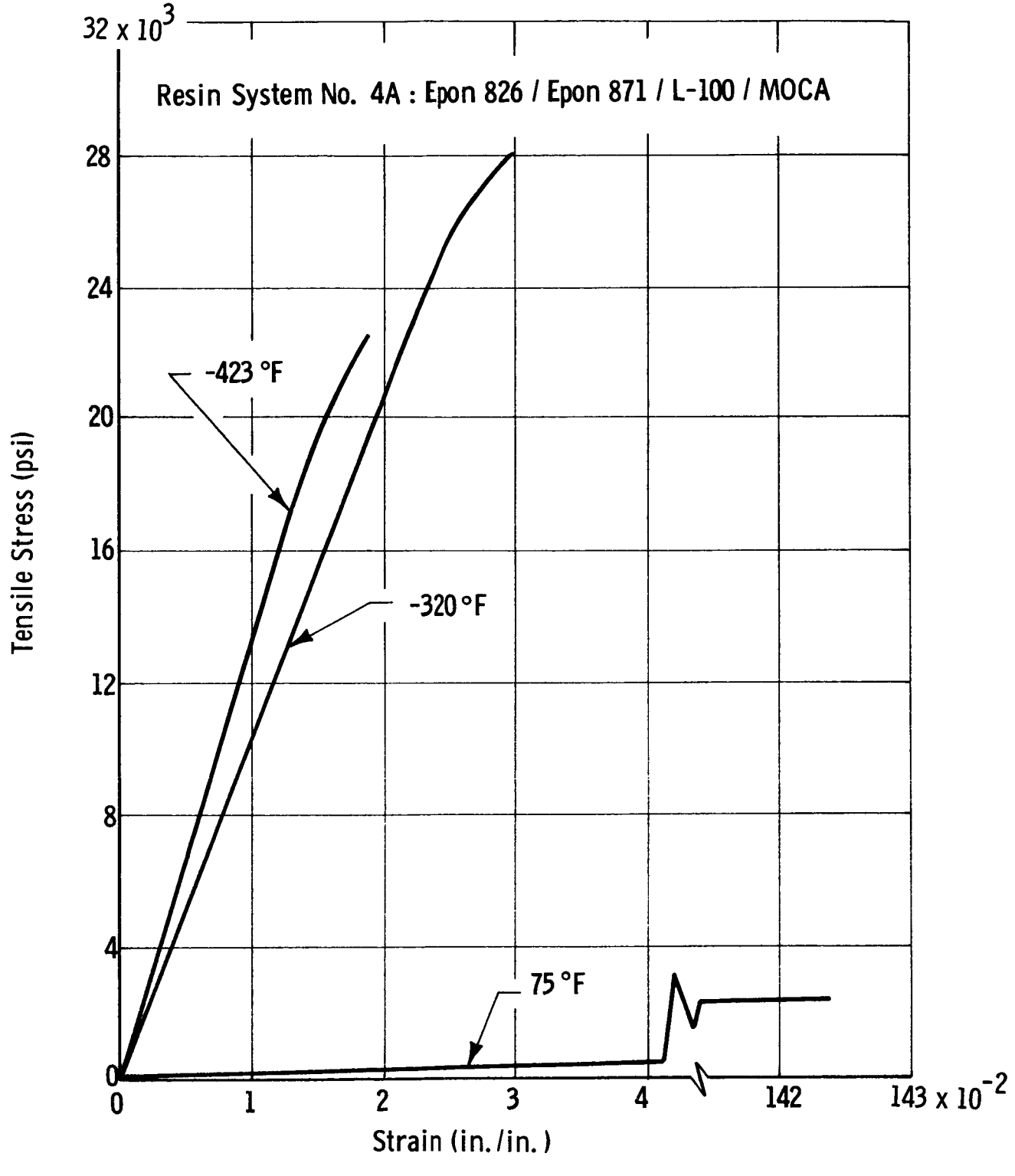
Resin Stress-Strain Curves (-320°F)

Figure 2



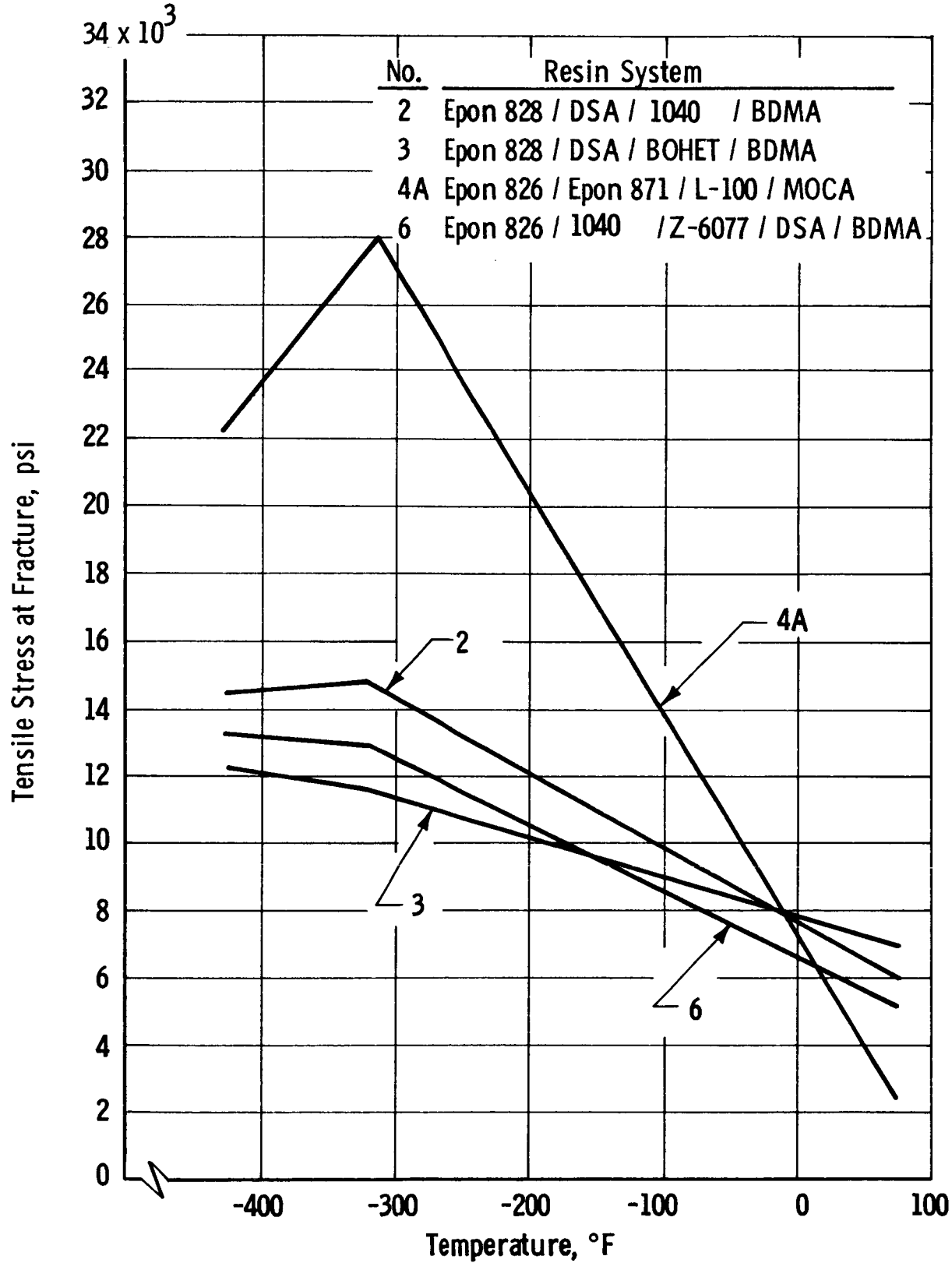
Resin Stress-Strain Curves (-423°F)

Figure 3



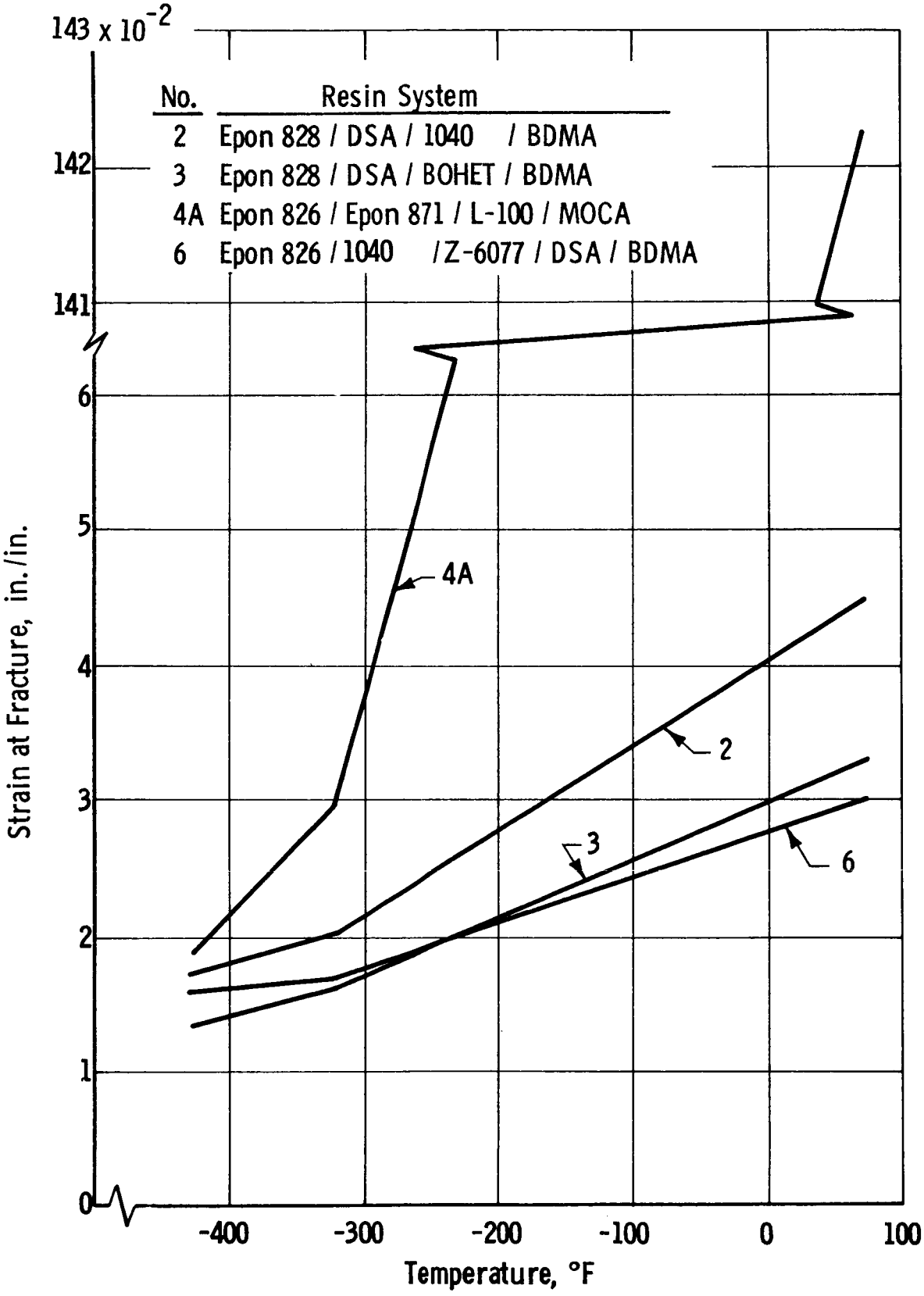
Resin 4A Stress-Strain Curves

Figure 4



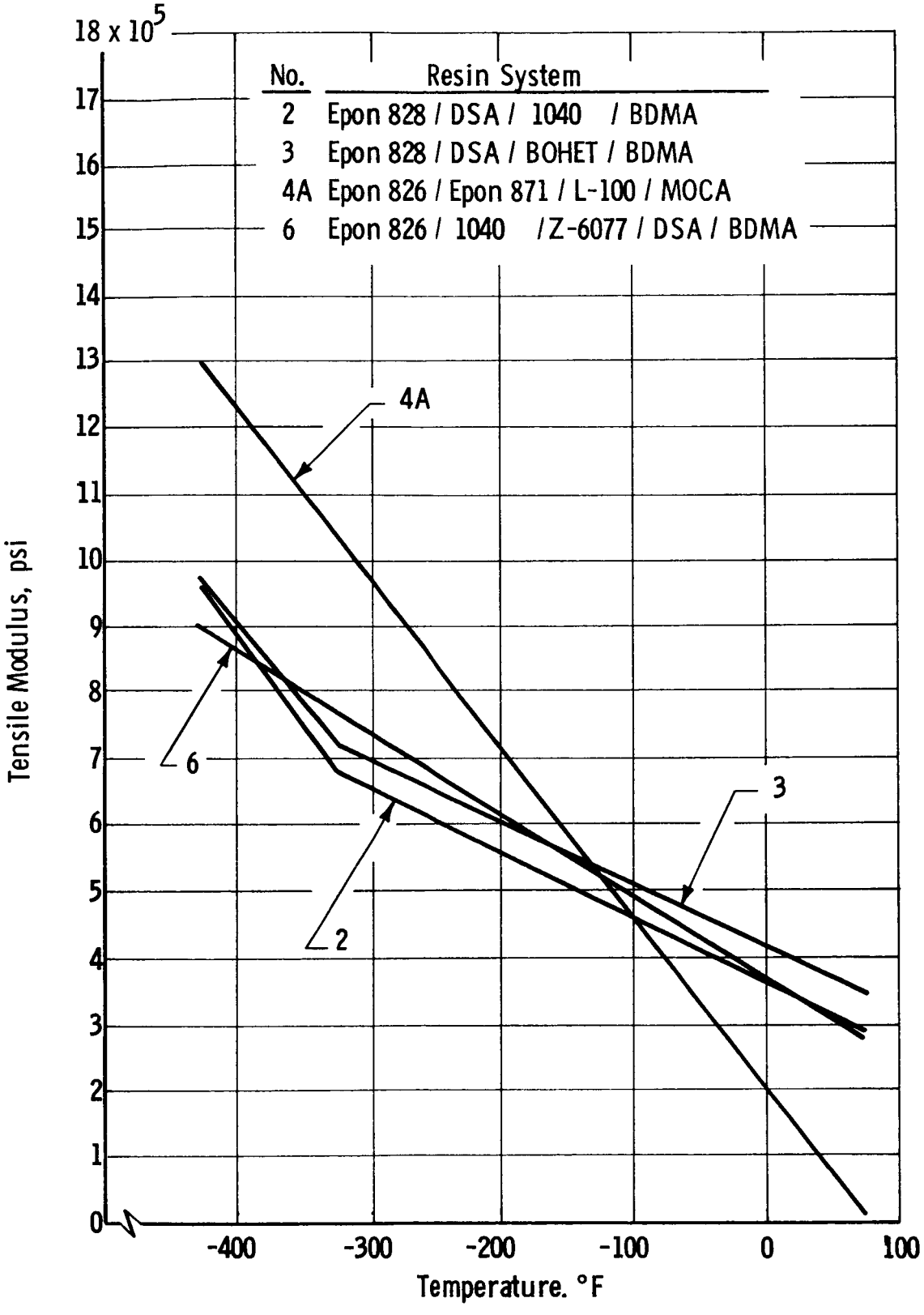
Effect of Temperature on Resin Tensile Stress at Fracture

Figure 5



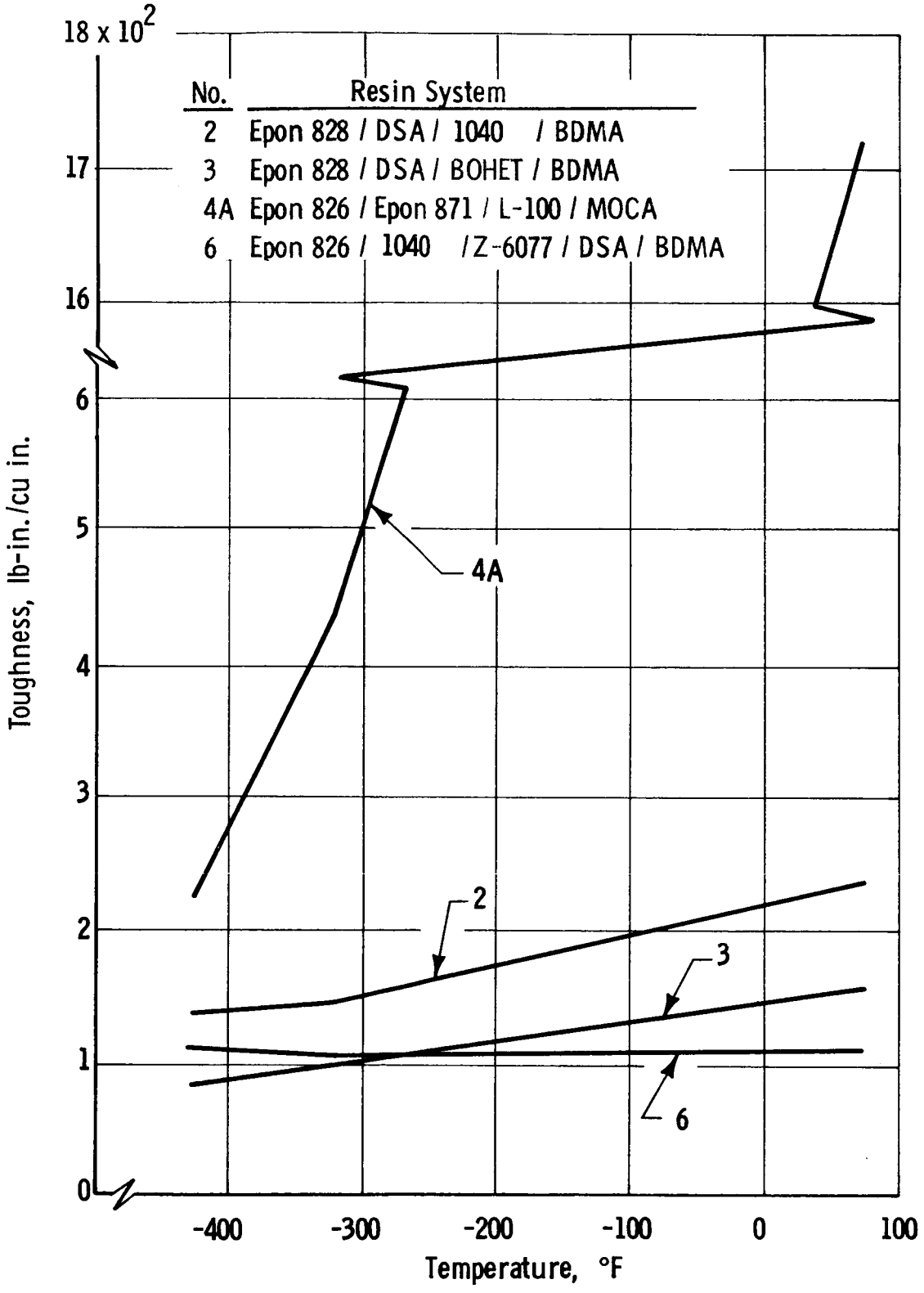
Effect of Temperature on Resin Strain at Fracture

Figure 6



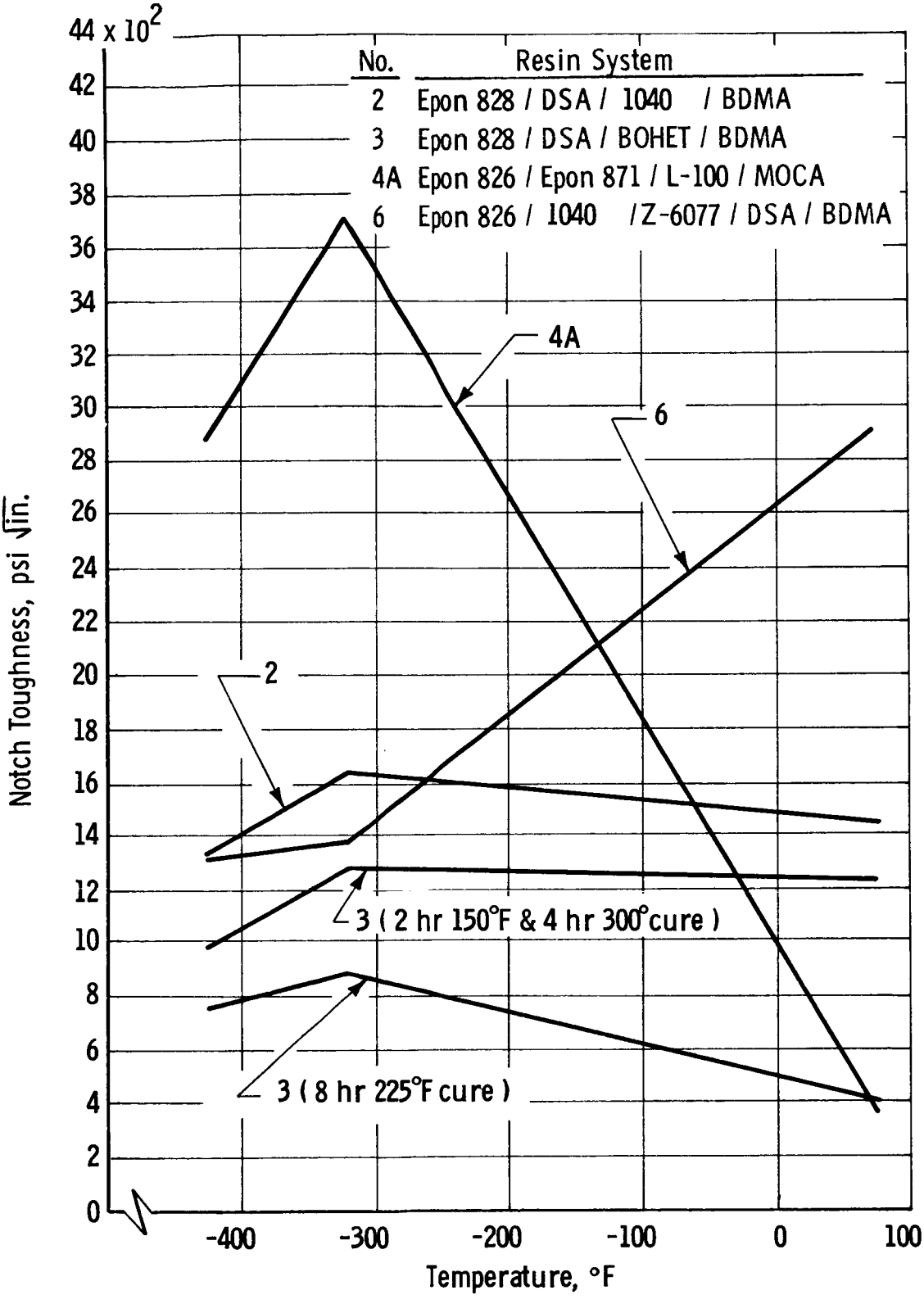
Effect of Temperature on Resin Tensile Modulus

Figure 7



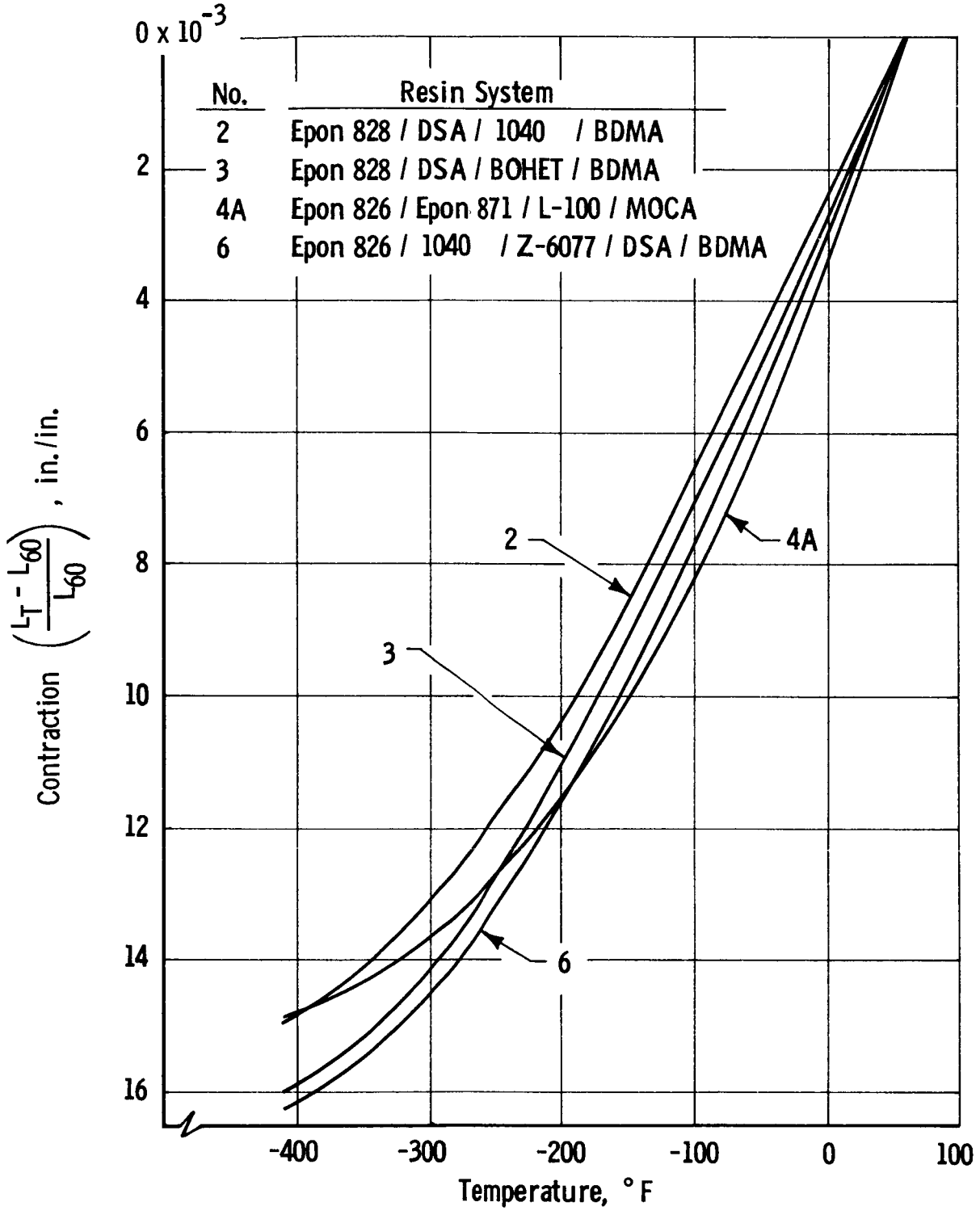
Effect of Temperature on Resin Toughness (Area Under Stress-Strain Curve)

Figure 8



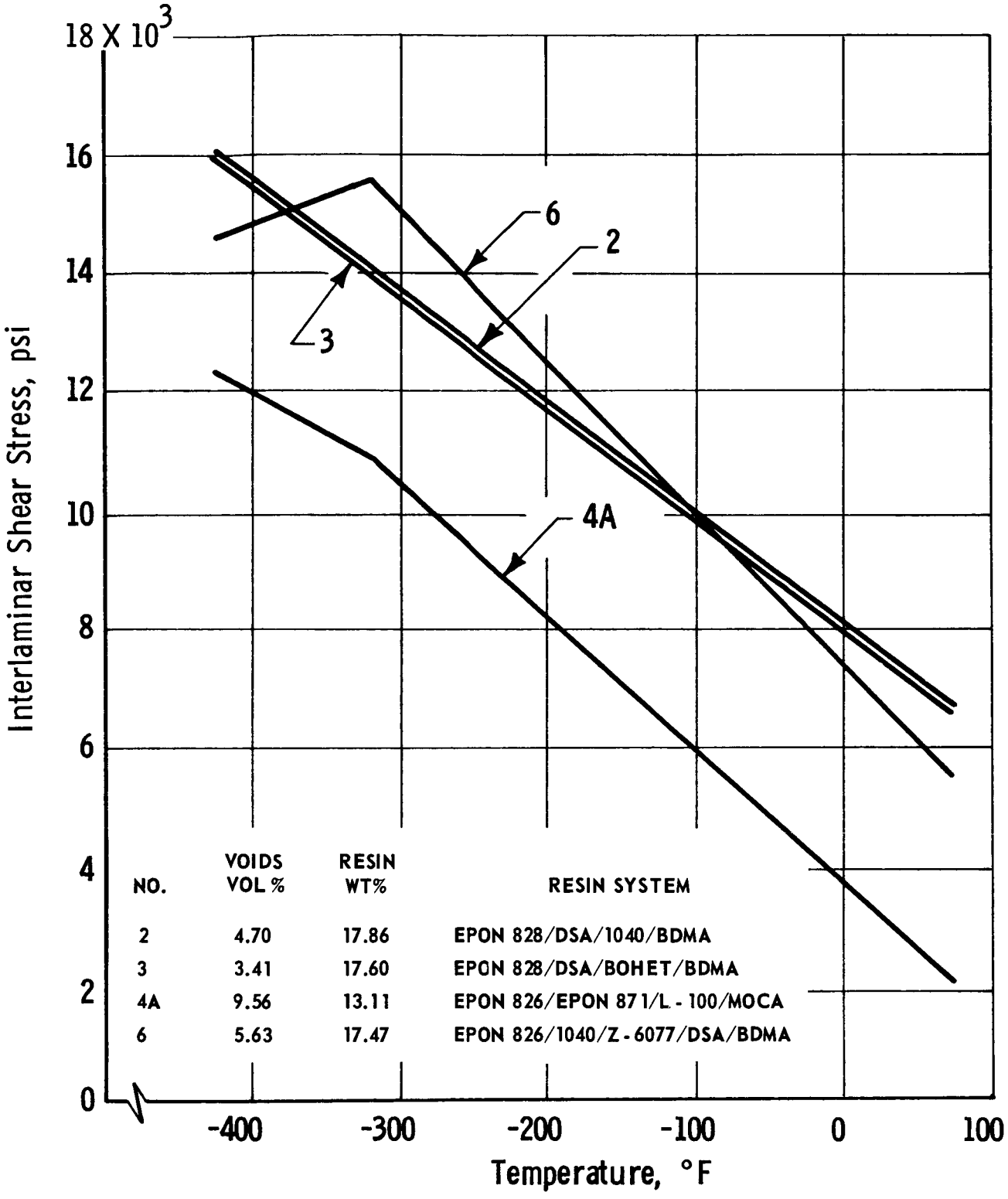
Effect of Temperature on Resin Notch Toughness

Figure 9



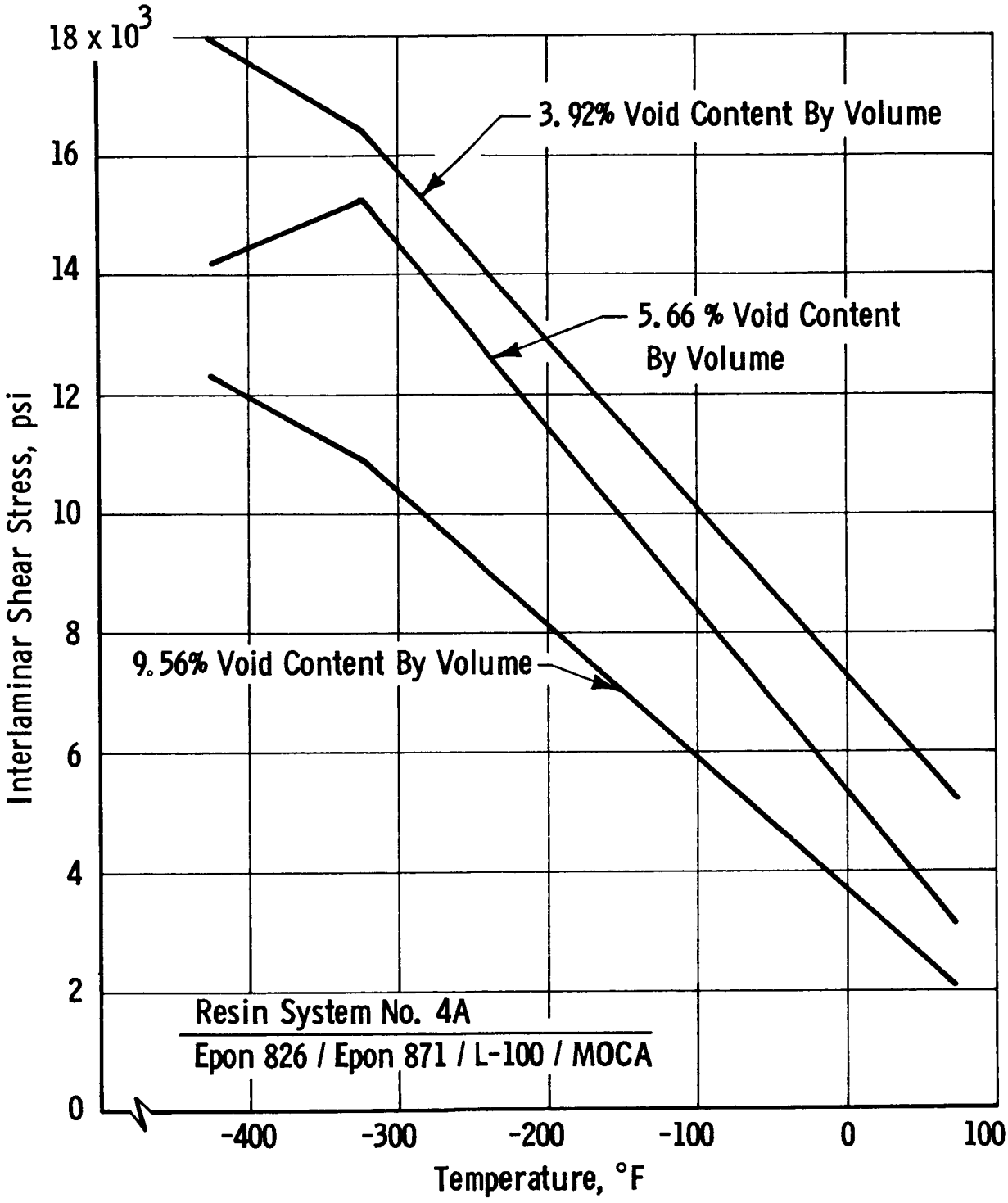
Linear Thermal Contraction of Resins

Figure 10



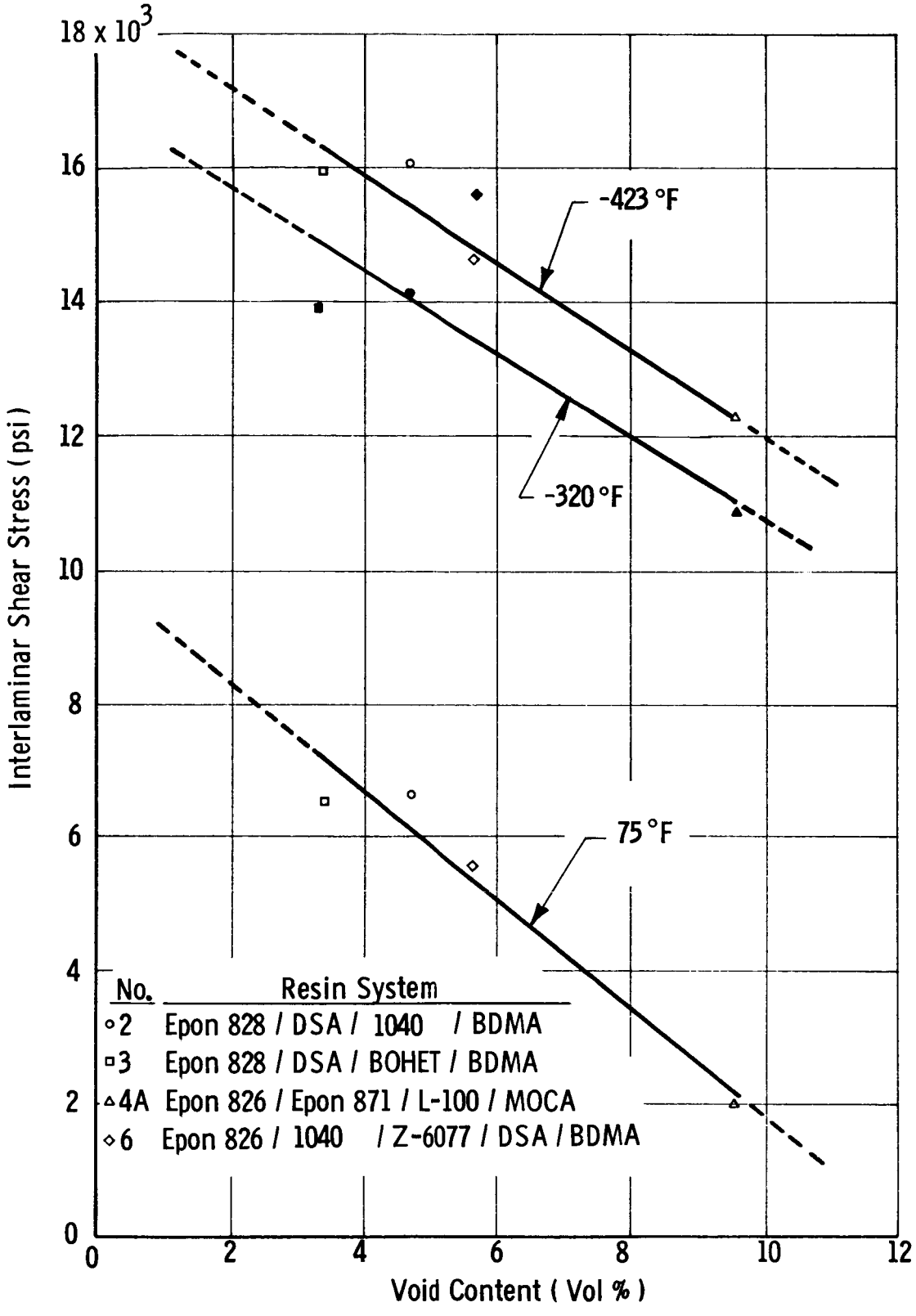
Effect of Temperature on Interlaminar Shear Stress
Glass/Resin Composites (Horizontal-Shear Method)

Figure 11



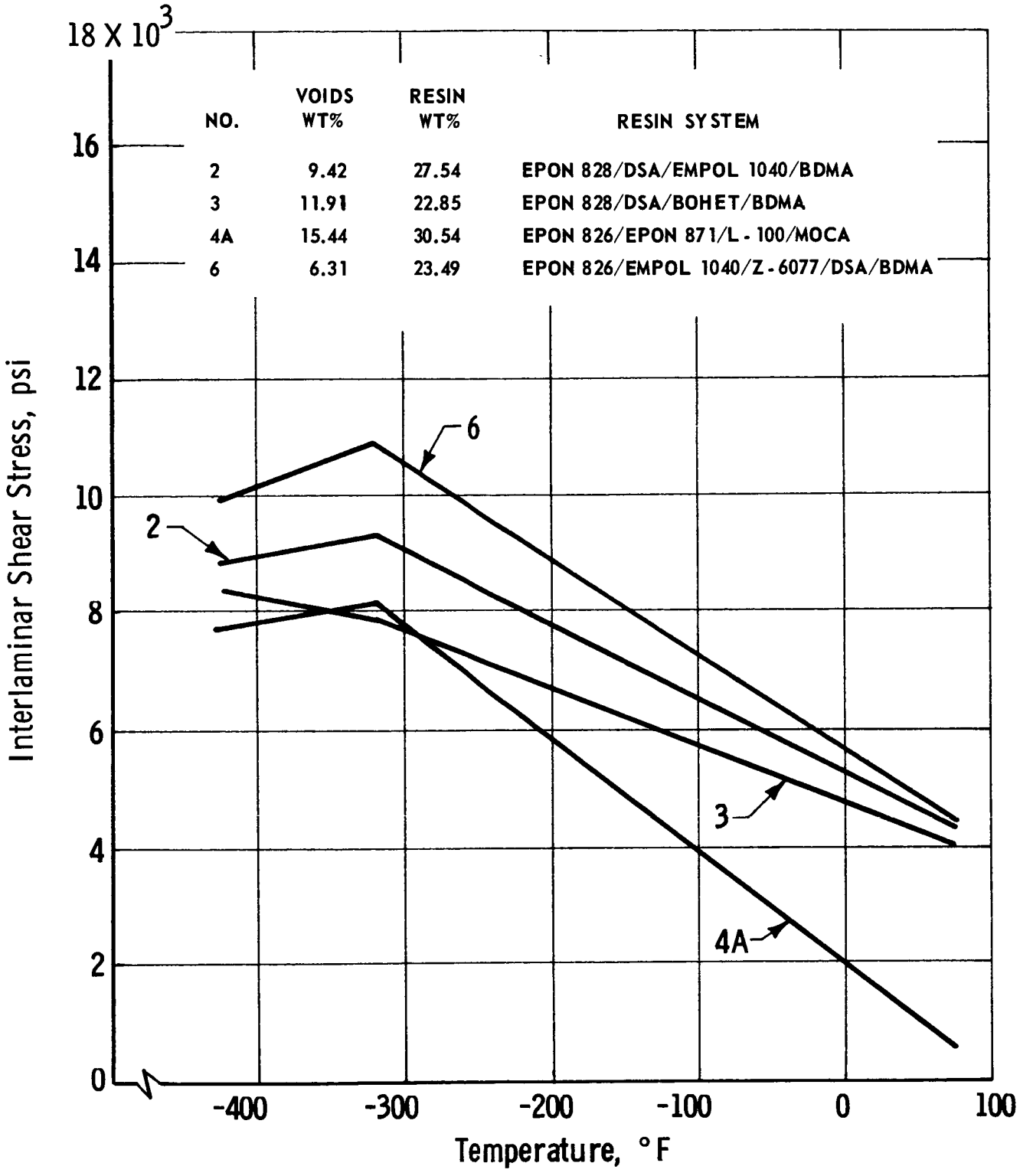
Effect of Temperature and Voids on Interlaminar Shear Stress
Resin 4A Composites (Horizontal-Shear Method)

Figure 12



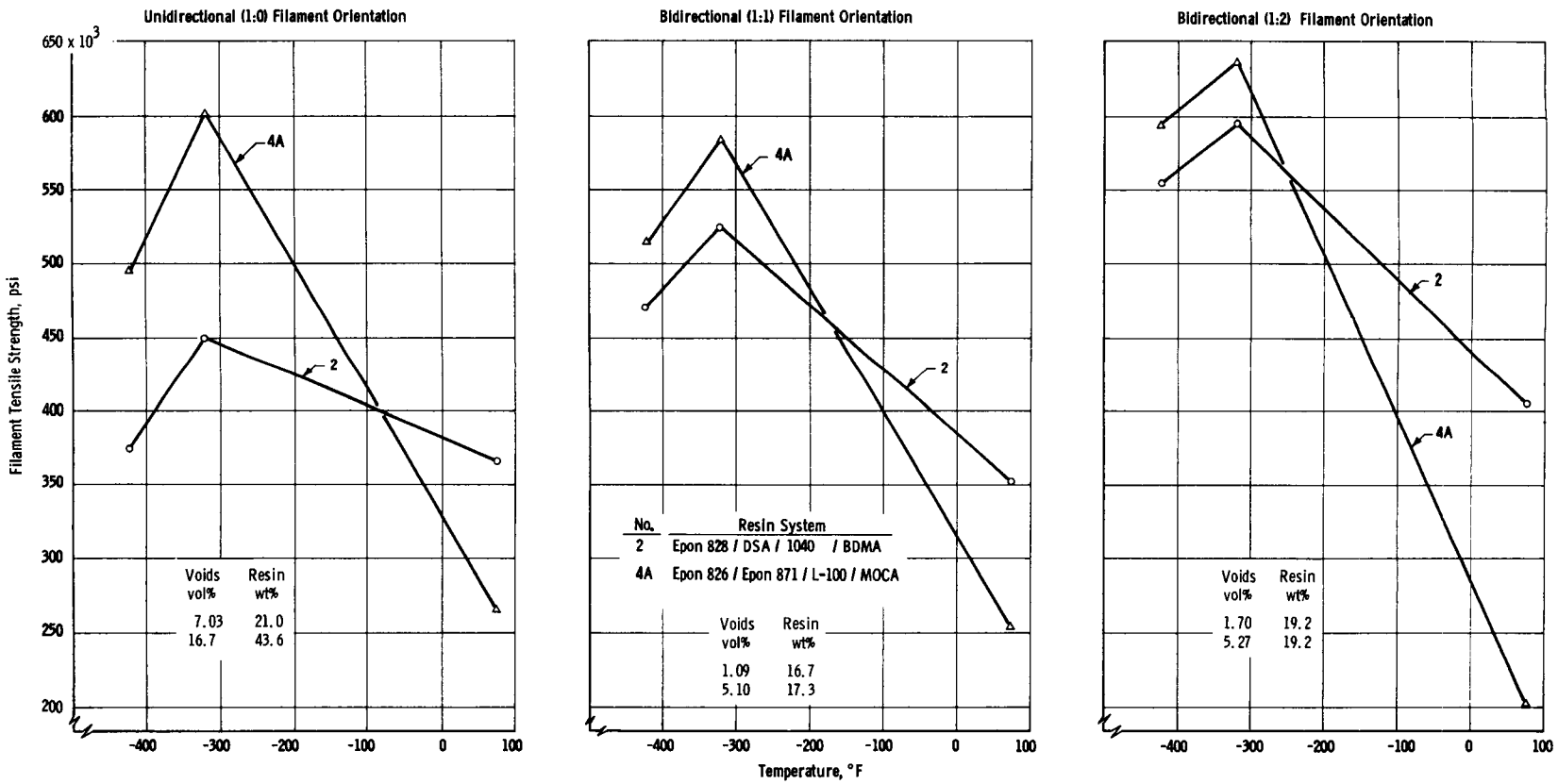
Relationship Between Interlaminar Shear Stress and Composite Voids

Figure 13



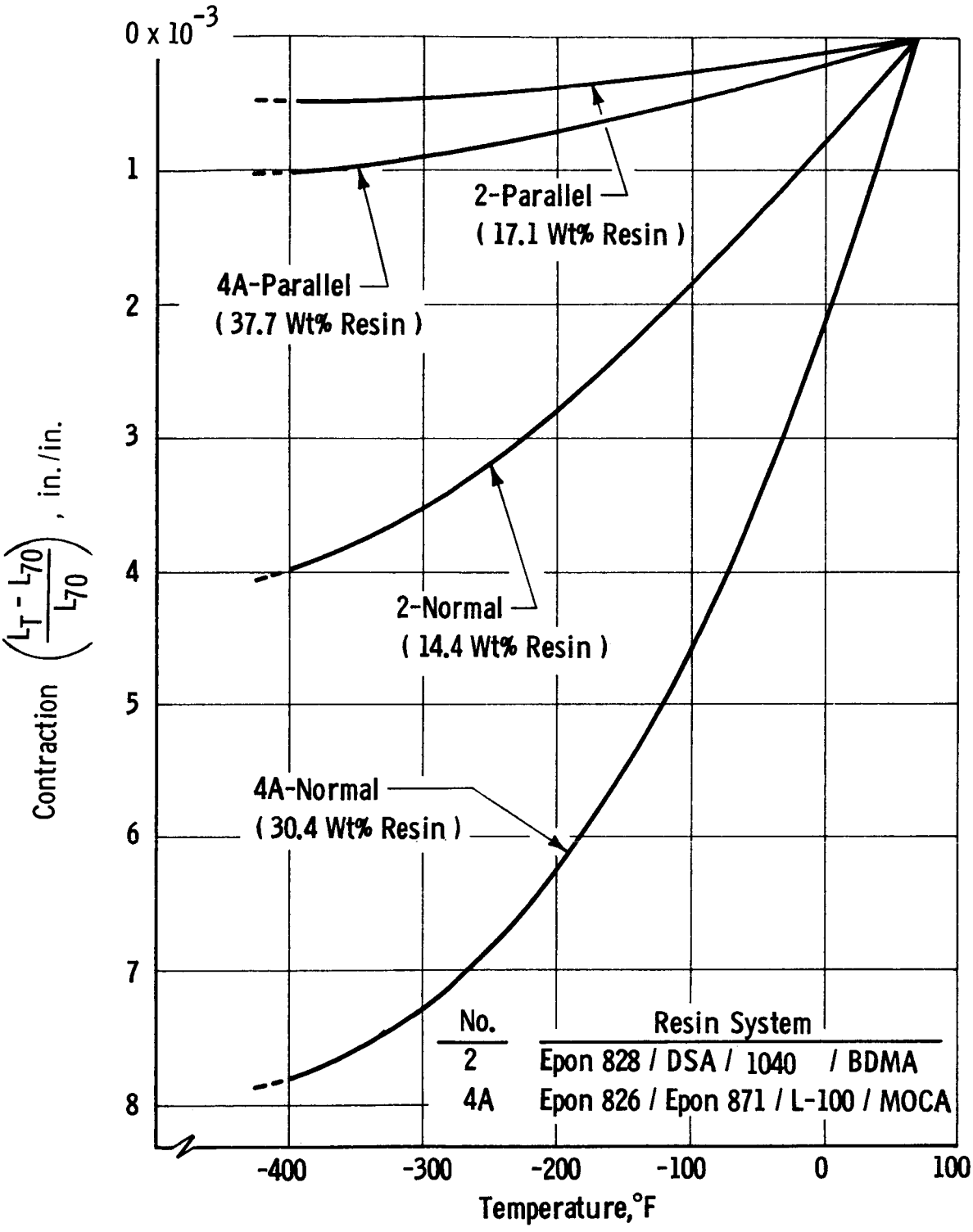
Effect of Temperature on Interlaminar Shear Stress of Composites
(Short-Span-Shear Method)

Figure 14



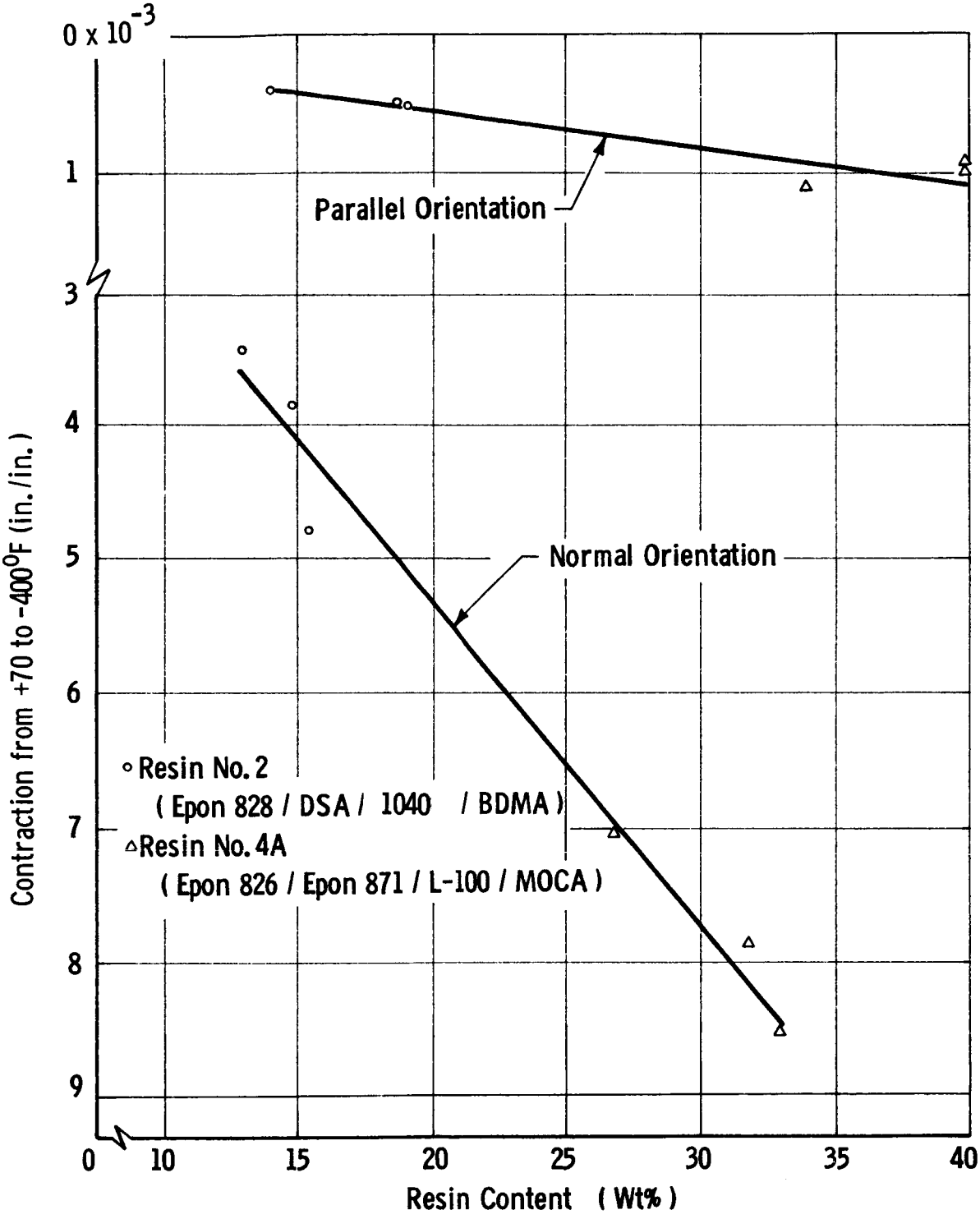
Effect of Temperature on Tensile Strength of Composite Filaments

Figure 15



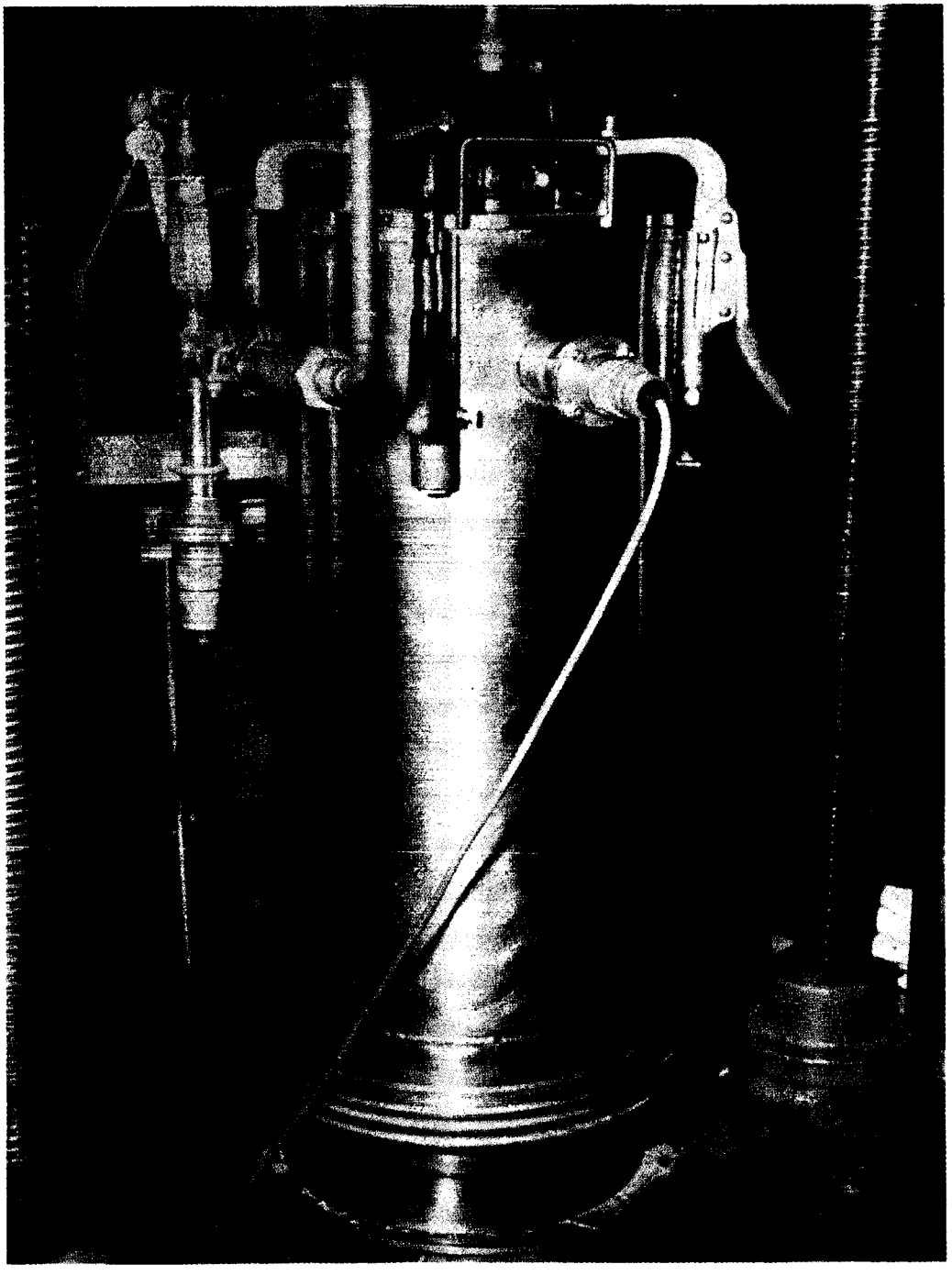
Linear Thermal Contraction of Composites

Figure 16



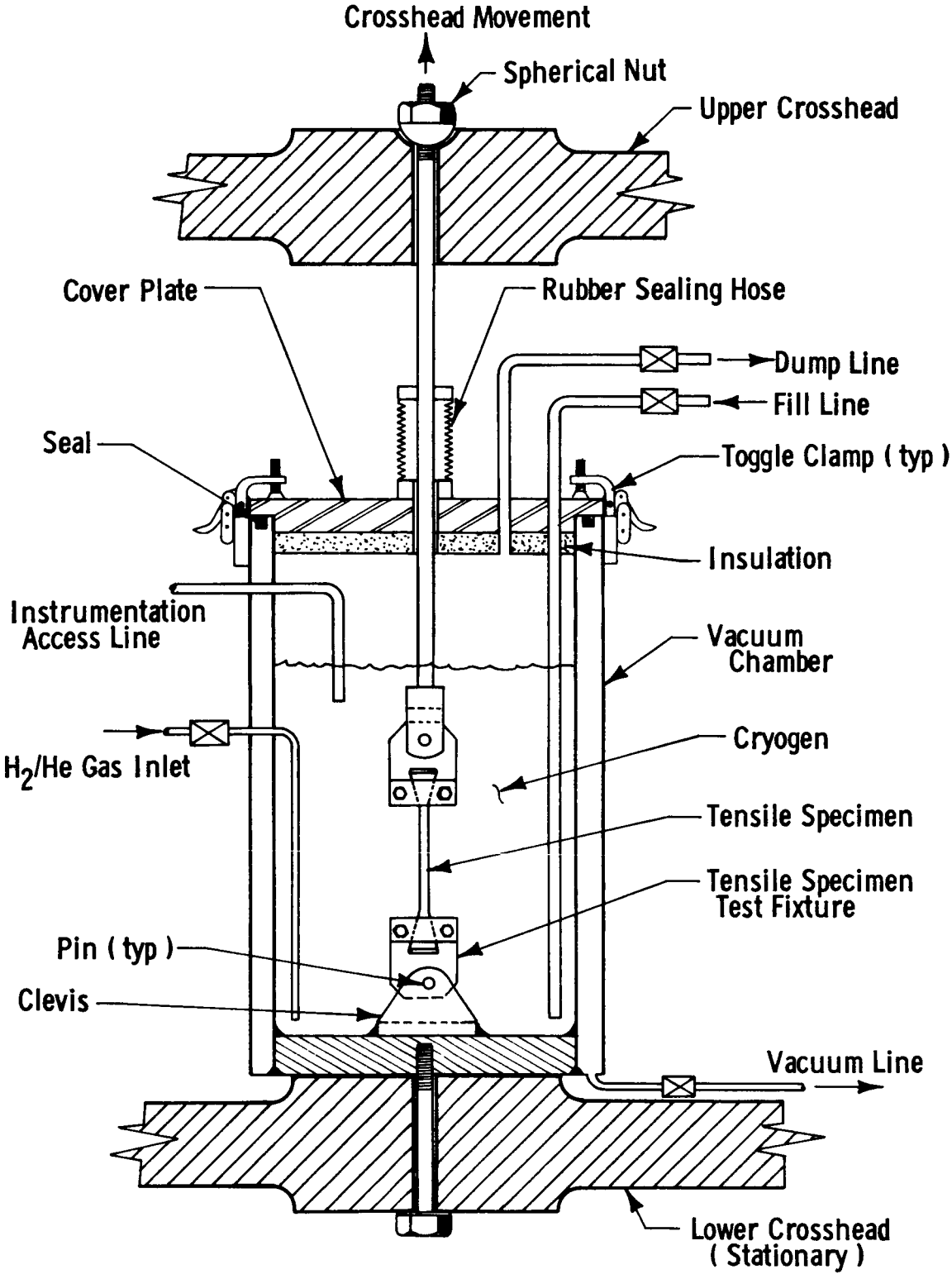
Effect of Resin Content and Filament Orientation on
Linear Thermal Contraction of Composites

Figure 17



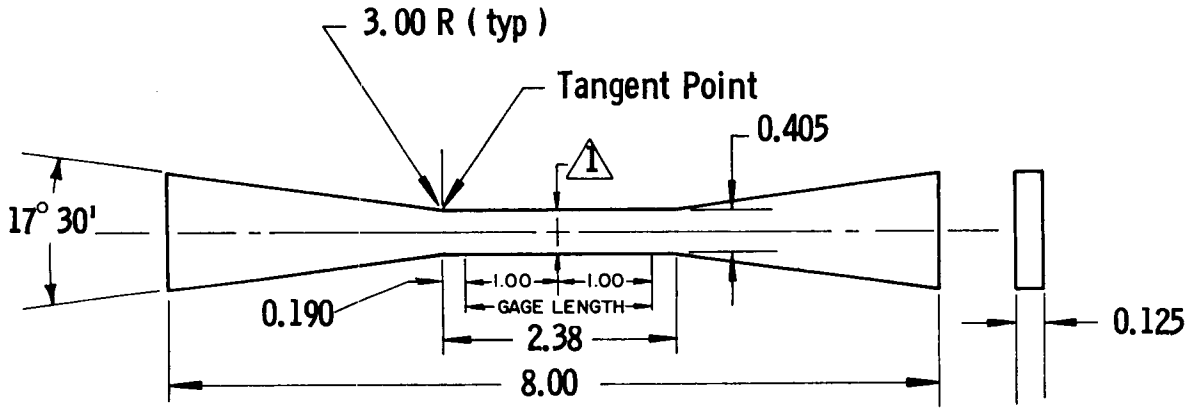
Cryostat/Tensile-Test Machine

Figure 18



Cryostat/Tensile-Test Machine (Sketch)

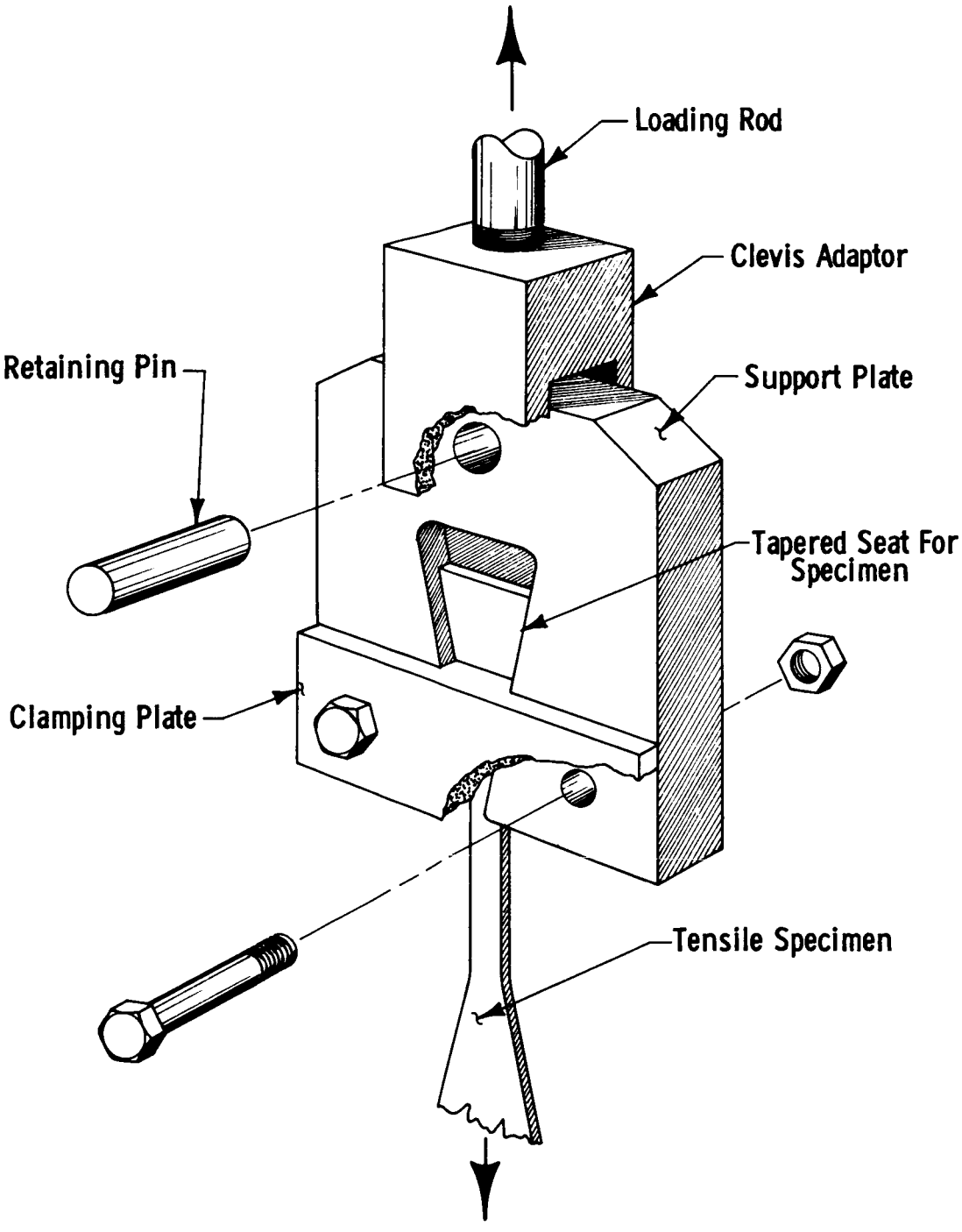
Figure 19



Note: 1 Width at center shall be $^{+\ 0.000}_{-\ 0.004}$ compared with minimum dimension of reduced section

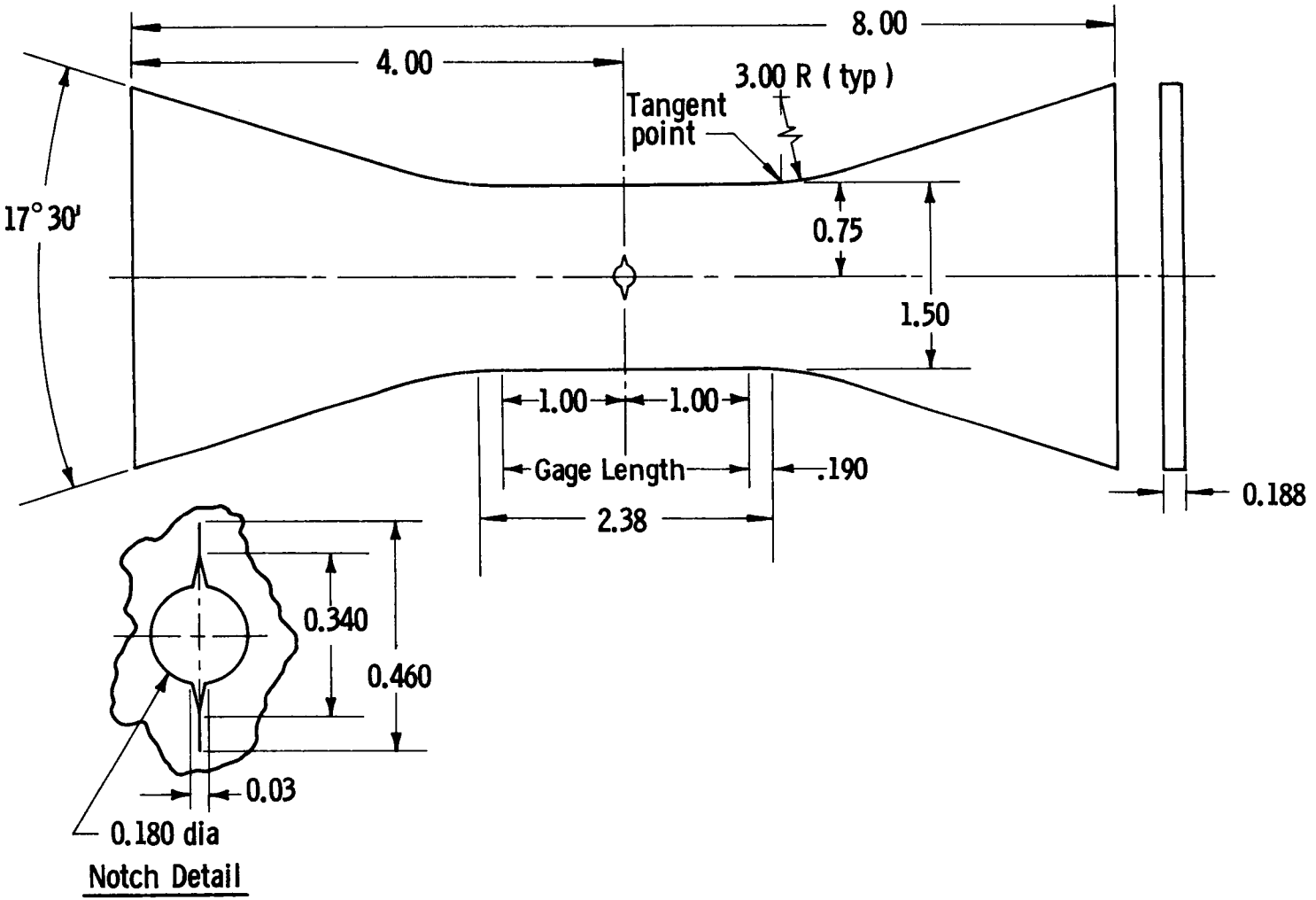
Tensile-Test Specimen

Figure 20



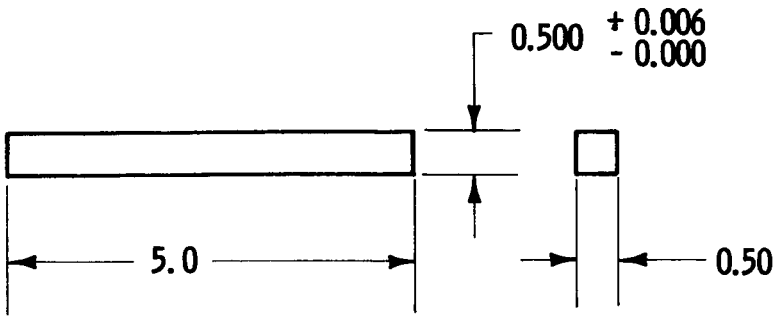
Tensile-Test Fixture

Figure 21

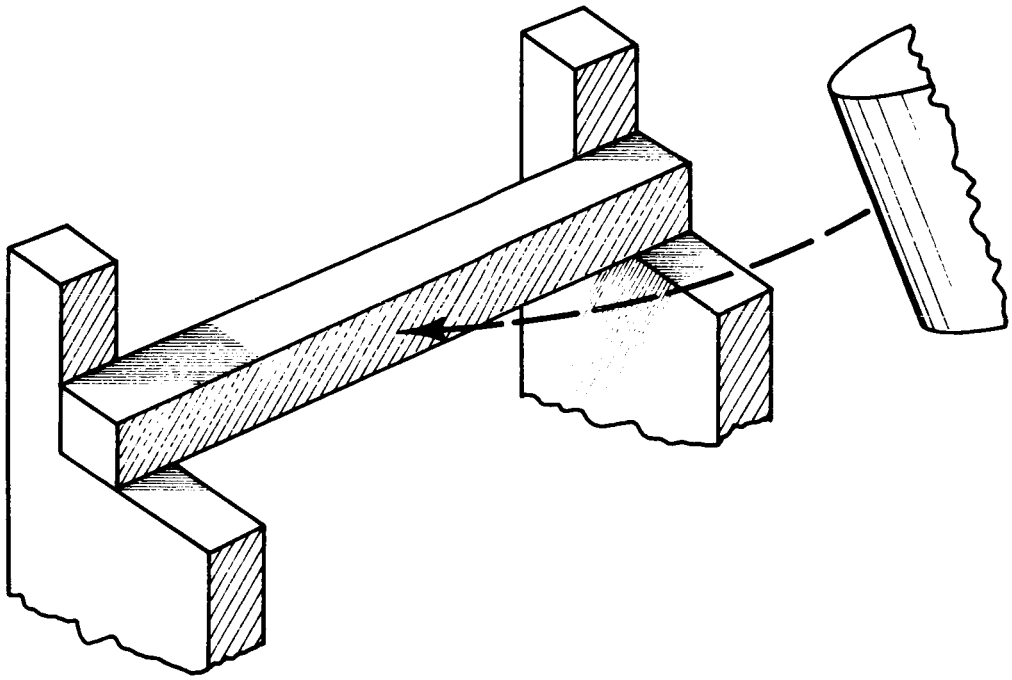


Notch-Toughness Specimen

Figure 22

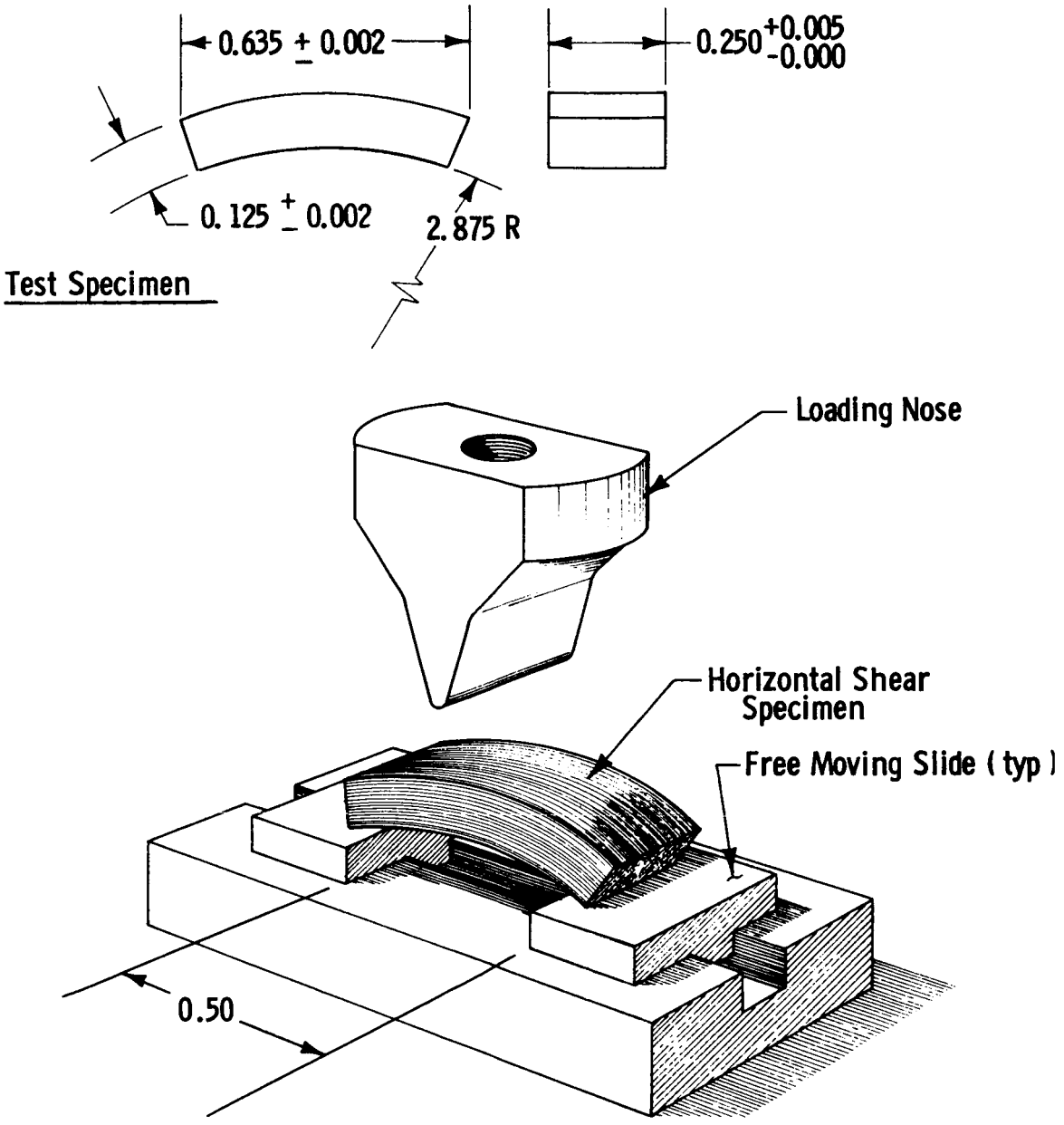


Test Specimen



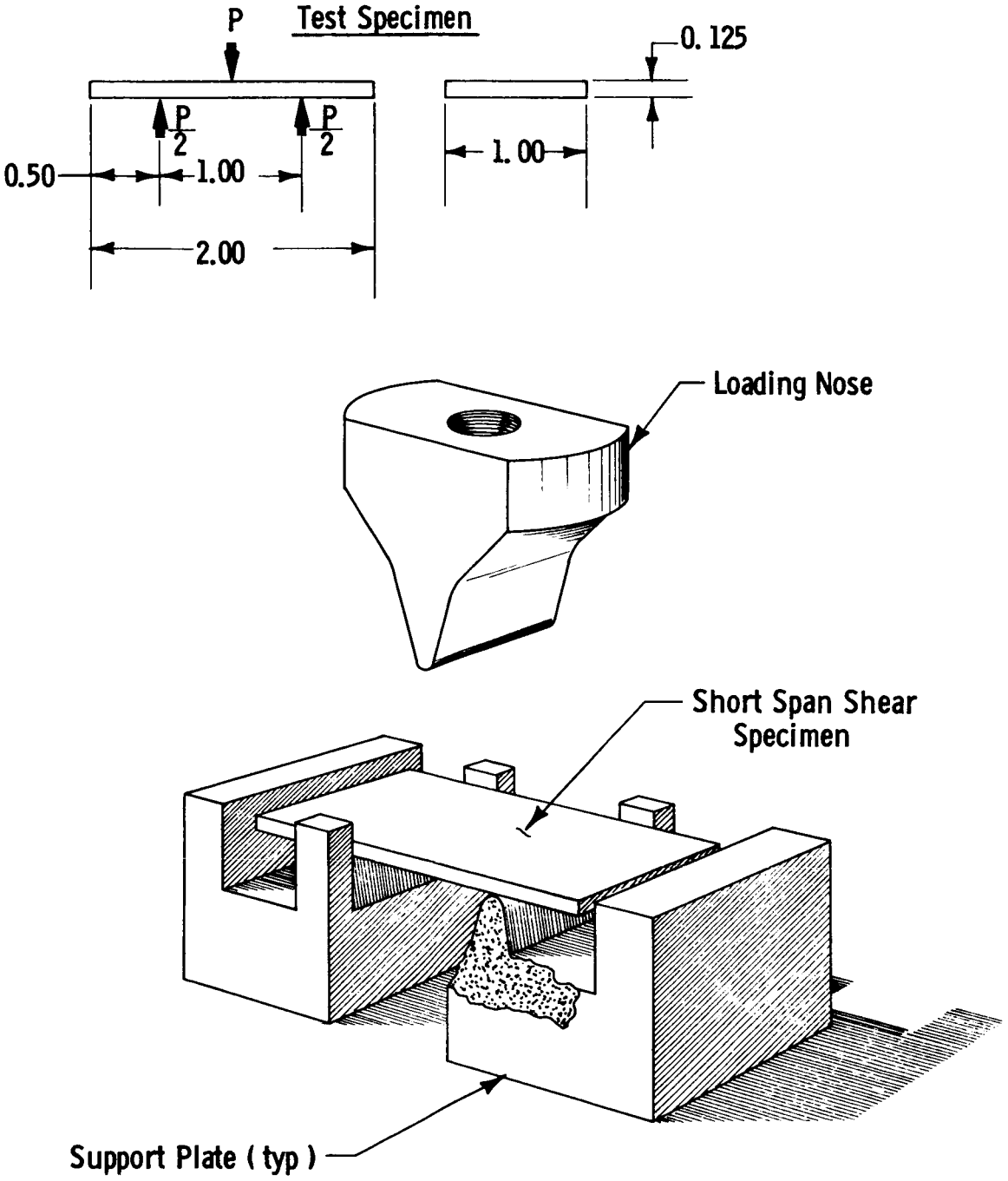
Simple-Beam (Charpy-Type) Impact Test

Figure 23



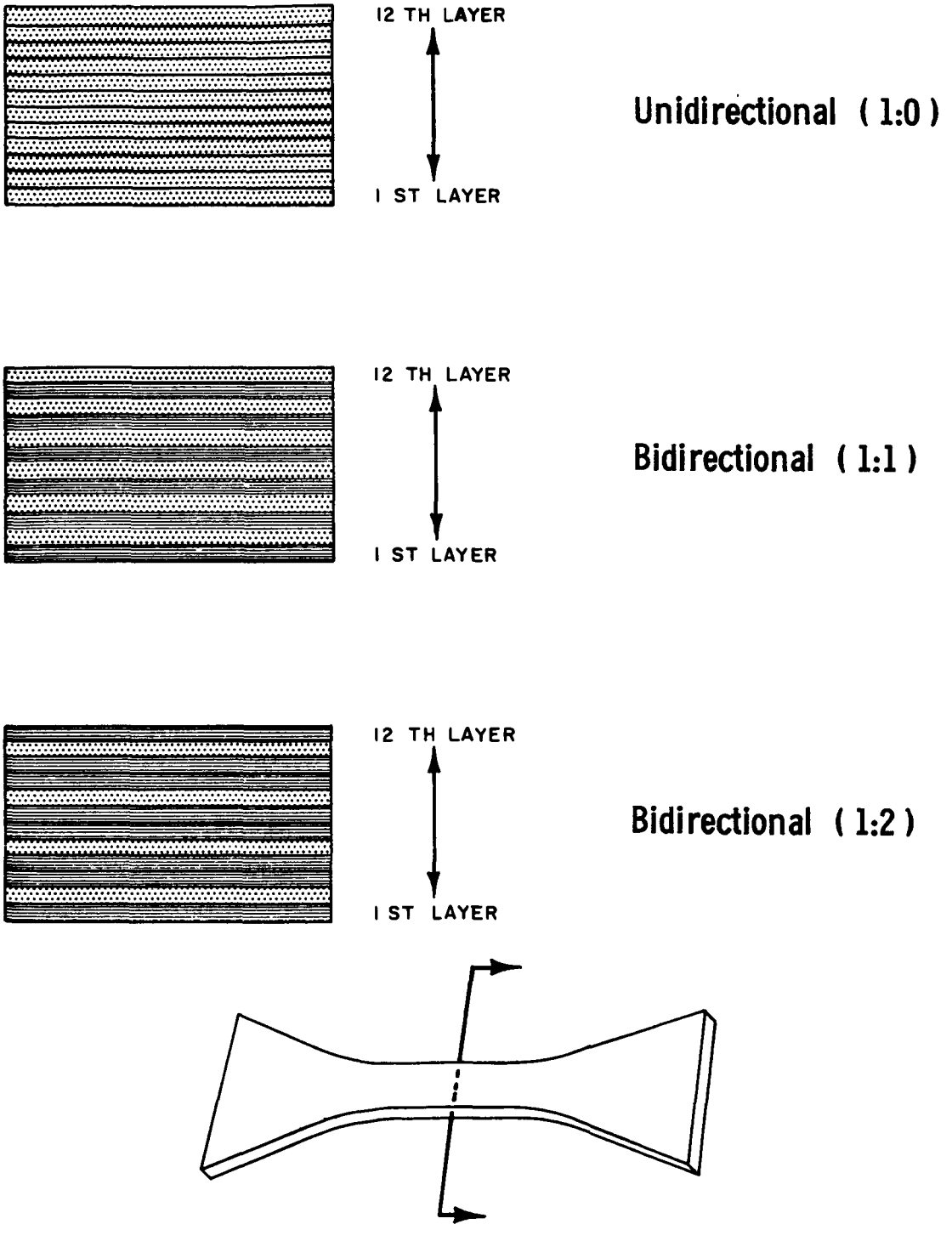
Horizontal-Shear Test Setup

Figure 24



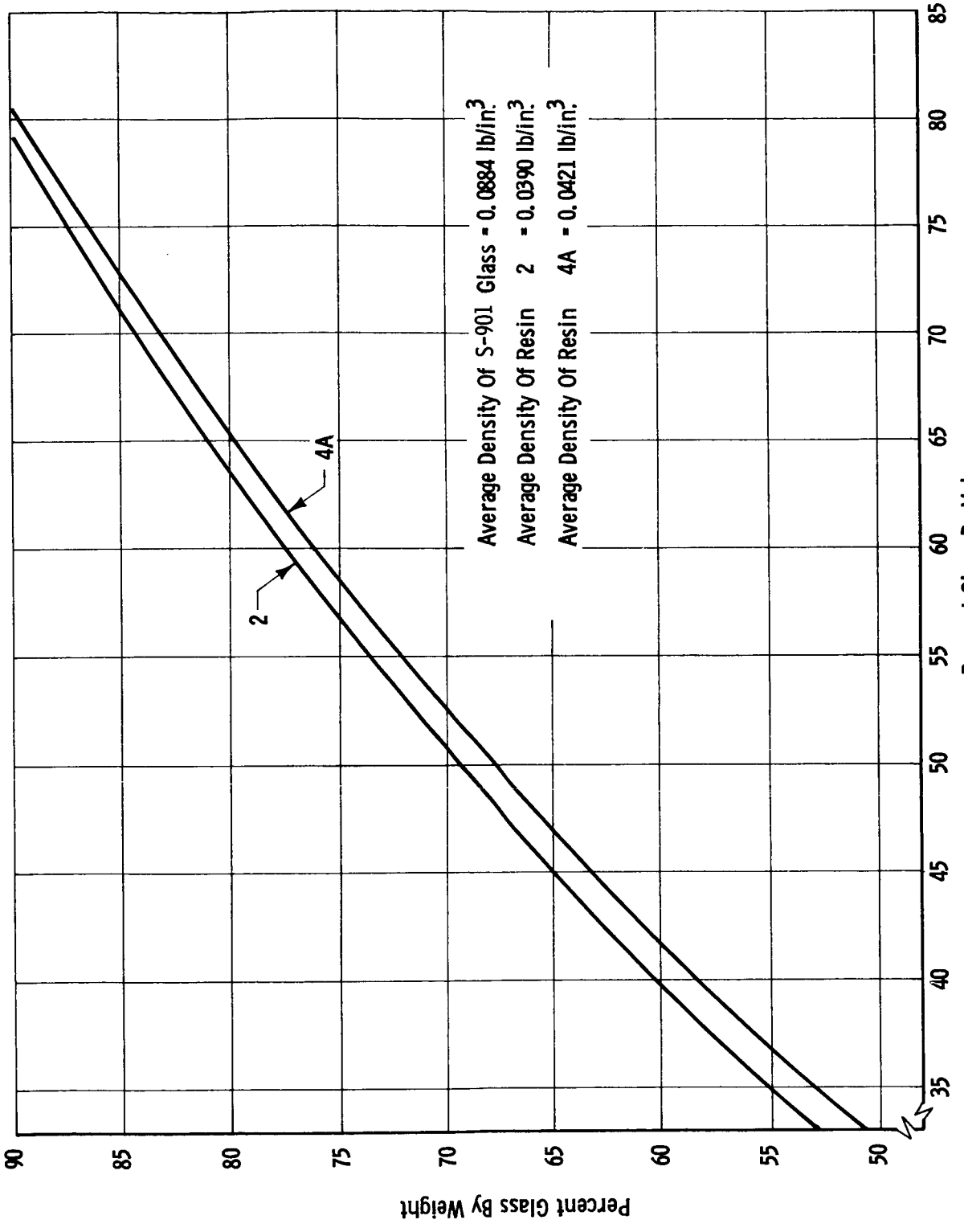
Short-Span-Shear Test Setup

Figure 25



Filament-Orientation Patterns for Tensile-Strength Specimens

Figure 26



Glass Weight/Volume Relationships in Resin Composites

Figure 27