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WHITE OAK LABORATORY

THE EFFECT OF MOISTURE ON CARBON FIBER REINFORCED EPOXY COMPOSITES – II MECHANICAL PROPERTY CHANGES

BY
Joseph M. Augl

3 FEBRUARY 1977

NAVAL SURFACE WEAPONS CENTER
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The changes in the matrix dominated flexural and short beam shear strengths of six carbon fiber composites were determined as a function of temperature and absorbed moisture. Moisture absorption led to a strength reduction between 10 and 40 percent, depending on temperature, but this reduction in strength leveled out after equilibrium was reached with the surrounding environment.

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It was concluded that this loss in strength is due to a reduction in resin modulus over a wide temperature range caused by the plasticizing effect of moisture on the matrix. This loss in resin modulus was reversible, and the original dry modulus-temperature-curve was regained after the absorbed moisture was removed; simultaneously the original composite dry strength was recovered.

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THE EFFECT OF MOISTURE ON CARBON FIBER REINFORCED EPOXY COMPOSITES II MECHANICAL PROPERTY CHANGES.

It has been recognized that moisture can change certain mechanical properties of fiber reinforced composites. Since advanced composites are becoming increasingly important for future Naval hardware application, it is necessary to gain a better understanding of the mechanisms of the property changes of these materials in their environment, and ultimately, to predict their behavior and useful life time under service environment.

Part of such an investigation of property changes of advanced organic matrix composites in a humid environment is described in this report. This is the second part of an investigation which deals with the experimental results of the flexural and shear strength changes on composites, before and after humidity exposure, as a function of temperature.

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INTRODUCTION

This report is the second part of an investigation of the effect that moisture has on the properties of carbon fiber reinforced epoxy composites. The first part, which has been published as a technical report [1] described the diffusion of moisture in these composites. This report summarizes the mechanical property changes we have observed when resin and composites samples were exposed to humidity under controlled laboratory conditions. The loss in resin dominated mechanical properties is of a reversible nature, i.e., the original dry strengths can be regained after removal of moisture (at least within the limitation of the sensitivity of the mechanical testing). It seems that these moisture effects can be explained, at least qualitatively, as a result of plasticization of the resin matrix by moisture. No attempt is made here to analyze the observed results in terms of laminated plate theory. This will be the subject of a forthcoming technical report. How these results can be related to real outdoor environments will also be discussed in a separate report.

EXPERIMENTAL

Six carbon fiber epoxy composites were investigated for their changes in strength after controlled exposure to moisture. The fabrication of the prepregs and composite panels are described in Appendix A. The humidity exposure and mechanical testing of flexural and short beam shear strengths as well as the torsional braid analysis of the resins are described in Appendix B.

RESULTS AND DISCUSSION

In part I of this report we have discussed some background information on the effect of moisture on carbon fiber composites. Suffice it here to say that in view of the importance of carbon fiber composites for future Naval aircraft and other Naval hardware, where high specific stiffness and strength is required, it is important to gain a better understanding of the deterionation processes of these materials, to know their limitations and to be able to predict

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[1] J. M. Augl and A. E. Berger, "The Effect of Moisture on Carbon Fiber Reinforced Epoxy Composites, I Diffusion," NSWC/WOL/TR 76-7

their long-term behavior in service environments. Due to the complex nature of organic matrix composites the predictions of properties and failure become rather difficult, especially, since failure can be caused by various mechanisms. To predict the long-term behavior will therefore require a strong interaction of experimental and analytical procedures. Part II of this investigation will summarize some of our experimental observations leaving the discussion of the analytical methods for long-term predictions to a forthcoming report. Also, we are considering here only the pure moisture effects and not the effects of superimposed thermal or mechanical cycling. Such investigations are in progress and the results shall be reported at a later date. The six composites investigated here were the same unidirectional CF composites described in part I of this report.

A. Effect of Moisture on the Resin Modulus

Torsional braid analysis (TBA) was used to determine the change of the relative modulus of various epoxy resins that had been exposed to different relative humidities. The temperature scan was carried out at a rate of 30°C per minute from room temperature through T_g. The high heating rate was necessary to prevent excessive moisture desorption, even so, some desorption cannot be prevented. Figure 1 shows the thermomechanical behavior of the dry Narmco 5208 resin, where T_p = ultimate projected service temperature in an aircraft; T_1 = onset of transition region between glassy and rubbery region of the resin; T_2 = intersection between the flat and the step modulus-temperature curve, sometimes used as a definition for the glass transition temperature; T_g = glass transition temperature. here defined as the temperature of the maximal mechanical damping of the resin. Figures 2 to 4 show the effect on the resin stiffness after the samples had been equilibrated at various relative humidities. (Figure 5 shows the effect of moisture on Hercules 3501 resin which is another candidate resin for aircraft application. Its behavior is similar to Narmco 5208.)

From these measurements it is difficult to determine with reasonable accuracy of what the change in T_g is. However, what is even more important, is that one can see that the resin moduli are reduced over a wide temperature range; for 5208 even down to room temperature. Without discussing the quantitative details here, such a change in resin modulus will also change the resin dominated composite moduli (E_{22} and G_{12}) and the resin dominated strengths (S_{T22}, S_{C22}, S_{S12}, and S_{C11}). (E_{22}, G_{12}, S_{T22}, S_{C22}, S_{S12} and S_{C11} indicate the composite transverse modulus, the longitudinal shear modulus, the transverse tensile strength, the transverse compressive strength, the longitudinal shear strength and the longitudinal compressive strength respectively.)

In all cases of the resins investigated it was found that the dry modulus curve was regained after the moisture was removed. This indicates a reversible plasticization of the resin with moisture, at least within the sensitivity of the instrument and within the temperature range that had been scanned. This made it possible to use

a single resin specimen for the exposure experiments, thus eliminating the effects of slightly different braid geometries.

B. Effect of Moisture on the Flexural Strength of Unidirectional Carbon Fiber Composite.

The composites were prepared to a fiber volume fraction of 70% ± 1. The samples were tested dry and after three weeks of humidity exposure (at 75°C and 80% RH). The results are given in Table 1. Figure 6 shows the change in flexural strength of the six composites before moisture exposure. The T300 composites show higher strength values than the HMS composites but seem to fall off more rapidly with increasing temperature. An explanation may be that the T300 fiber had a proprietary epoxy sizing while the HMS fiber had no sizing at all. (Also, it appears that there may be a larger difference in the real strength of the HMS and T300 fibers than given in Table 2.) Figures 7 through 9 are graphs of the percent strength retention (before and after humidity exposure) as a function of temperature. Figures 10 through 15 are three dimensional projections of the flexural strength profile of these composites for the benefit of an easy and quick overview of the combined moisture and temperature effects. (The crossbars indicate the standard deviation of five test samples.)

C. Effects of Moisture on the Interlaminar Short Beam Shear Strength of CF Composites.

The interlaminar shear strength changes, determined on the same set of composites, were similar to the flexural strength changes. Again, the T300 fiber composites show a somewhat stronger temperature effect than the HMS composites (see Figure 16). Figures 17 through 19 show the percent strength retention of the dry and exposed samples. As in case of the flexural strength, the shear strength decreases with increasing temperature and increasing moisture content. Figures 20 through 25 show three dimensional projections of the interlaminar shear strengths of the composites as a function of temperature and moisture content.

D. Shape of the Interlaminar Shear Strength Degradation.

It was important to know whether or not the strength degradation would continue after the composite had been saturated with moisture and also to determine the shape of the degradation curve. Six sets of shear specimens were exposed to an accelerated moisture absorption at 75°C and 80 percent RH. The results of their strength degradation are given in Table 3 and Figure 26. The samples were exposed longer than necessary to reach 95 percent of their equilibrium concentration. (Estimates of how long it takes to reach a particular fraction of the equilibrium concentration can be easily obtained when the diffusion coefficient of moisture in the composite is known, see Part I of this report. [1]

Table 1

Flexural and Shear Strengths of Carbon Fiber Epoxy Composites as a Function of Temperature and Absorbed Moisture

Panel No.	Fiber	Resin	Exposure Conditions RH	Exposure Conditions Temp °C	Exposure Conditions Days	Average Wgt Increase Flex Spec (%)	Fiber Volume %	Test Temp (°C)	Flex. Str. MPasc. (Psi x 10^{-3})	Flex. Str. MPasc. (Psi x 10^{-3}) Normalized 70% Fib. Vol.	Coeff. Var. (%)	S.B. Shear Strength MPasc (Psi x 10^{-3})	Coeff. Var. (%)	Av. Wt. Increase of Shear Spec. (%)
6	T300	5208	-	-	-	-	70.34	25	1,793 (260 F)	1,786 (259)	5.3	63.4 (9.20)	1.8	-
18	T300	332	-	-	-	-	71.3	25	1,655 (240 F)	1,627 (236)	4.8	73.7 (10.7)	5.2	-
28	T300	1031	-	-	-	-	69.2	25	1,524 (221 F)	1,544 (224)	3.1	71.7 (10.4)	2.8	-
24	HMS	5208	-	-	-	-	68.7	25	875 (127 F)	889 (129)	4.8	66.8 (9.69)	11.2	-
21	HMS	332	-	-	-	-	68.9	25	1,056 (153 F)	1,069 (155)	3.5	51.4 (7.45)	3.8	-
26	HMS	1031	-	-	-	-	67.1	25	1,089 (158 F)	1,138 (165)	4.4	58.3 (8.46)	2.4	-
6	T300	5208	-	-	-	-	70.34	100	1,269 (184 C)	1,262 (183)	3.9	49.6 (7.20)	8.8	-
18	T300	332	-	-	-	-	71.3	100	1,082 (157 C)	1,062 (156)	3.7	60.1 (8.72)	5.3	-
28	T300	1031	-	-	-	-	69.2	100	1,059 (155 C)	1,082 (157)	2.8	58.4 (8.47)	1.9	-
24	HMS	5208	-	-	-	-	68.7	100	799 (116 F)	813 (118)	4.8	66.9 (9.70)	5.2	-
21	HMS	332	-	-	-	-	68.9	100	806 (117 F)	812 (118)	7.1	48.1 (6.98)	2.1	-
26	HMS	1031	-	-	-	-	67.1	100	820 (119 C)	858 (124.5)	1.4	53.8 (7.80)	3.9	-
6	T300	5208	-	-	-	-	70.34	150	1,151 (167 V)	1,145 (166)	3.4	47.3 (6.86)	10.5	-
18	T300	332	-	-	-	-	71.3	150	931 (135 C)	917 (133)	1.4	52.1 (7.55)	9.7	-
28	T300	1031	-	-	-	-	69.2	150	944 (137 C)	955 (138.5)	3.1	51.6 (7.49)	9.1	-
24	HMS	5208	-	-	-	-	68.7	150	751 (109 F)	772 (112)	2.9	64.5 (9.39)	3.0	-
21	HMS	332	-	-	-	-	68.9	150	724 (105 C)	737 (107)	2.6	45.1 (6.54)	7.9	-
26	HMS	1031	-	-	-	-	67.1	150	696 (101 C)	720 (104.5)	1.9	39.4 (5.71)	2.9	-
6	T300	5208	-	-	-	-	70.34	177	889 (129.5 S)	889 (129)	5.9	37.8 (5.48)	4.3	-
18	T300	332	-	-	-	-	71.3	177	724 (105 C)	713 (103.5)	1.6	35.9 (5.21)	6.5	-
28	T300	1031	-	-	-	-	69.2	177	779 (113 C)	786 (114)	1.5	45.4 (6.59)	3.7	-
24	HMS	5208	-	-	-	-	68.7	177	588 (85.4 F)	600 (87)	8.6	49.2 (7.14)	2.7	-
21	HMS	332	-	-	-	-	68.9	177	594 (86.2 C)	603 (87.5)	1.2	35.3 (5.12)	3.5	-
26	HMS	1031	-	-	-	-	67.1	177	469 (68.1 C)	489 (71)	6.8	40.1 (5.81)	4.0	-
6	T300	5208	80	75	21	0.64	70.34	25	1,579 (229 C)	1,572 (228)	1.3	59.7 (8.65)	8.8	0.69
18	T300	332	80	75	21	0.46	71.3	25	1,351 (196 F)	1,324 (192)	1.6	60.4 (8.76)	9.4	0.47
28	T300	1031	80	75	21	0.54	69.2	25	1,303 (189 C)	1,317 (191)	3.5	50.7 (7.35)	13.1	0.56
24	HMS	5208	80	75	21	0.66	68.7	25	827 (120 F)	841 (122)	1.9	62.5 (9.06)	1.8	0.54
21	HMS	332	80	75	21	0.52	68.9	25	931 (135 F)	945 (137)	1.5	44.2 (6.41)	2.4	0.50
26	HMS	1031	80	75	21	0.55	67.1	25	869 (126 F)	910 (132)	3.3	51.4 (7.46)	7.9	0.56
6	T300	5208	80	75	21	0.64	70.34	100	1,193 (173 S)	1,186 (172)	2.1	37.8 (5.43)	8.9	0.69
18	T300	332	80	75	21	0.46	71.3	100	1,055 (153 C)	1,041 (151)	3.1	42.3 (6.14)	3.5	0.47
28	T300	1031	80	75	21	0.54	69.2	100	972 (141 S)	986 (143)	3.5	42.0 (6.09)	8.4	0.56
24	HMS	5208	80	75	21	0.66	68.7	100	765 (111 F)	783 (113.5)	2.1	49.6 (7.20)	4.9	0.64
21	HMS	332	80	75	21	0.52	68.9	100	834 (121 C)	848 (123)	2.0	40.7 (5.91)	1.6	0.50
26	HMS	1031	80	75	21	0.55	67.1	100	827 (120 C)	862 (125)	2.8	40.2 (5.83)	[illegible]	[illegible]
6	T300	5208	80	75	21	0.64	70.34	150	965 (140 S)	932 (135)	4.1	34.7 (5.04)	4.0	0.69
18	T300	332	80	75	21	0.46	71.3	150	744 (108 C)	731 (106)	1.5	28.6 (4.15)	3.7	0.47
28	T300	1031	80	75	21	0.54	69.2	150	855 (124 S)	868 (126)	1.4	33.7 (4.89)	4.5	0.56
24	HMS	5208	80	75	21	0.66	68.7	150	710 (103 S)	724 (105)	9.3	40.0 (5.80)	4.4	0.64
21	HMS	332	80	75	21	0.52	68.9	150	642 (93.1 C)	652 (94.5)	1.4	36.2 (5.25)	2.3	0.50
26	HMS	1031	80	75	21	0.55	67.1	150	506 (73.4 S)	527 (76.5)	2.9	29.4 (4.26)	3.5	0.56
6	T300	5208	80	75	21	0.64	70.34	177	806 (117 S)	800 (116)	2.7	40.4 (5.86)	3.5	0.69
18	T300	332	80	75	21	0.46	71.3	177	542 (78.6 C)	531 (77)	3.9	31.6 (4.58)	7.8	0.47
28	T300	1031	80	75	21	0.54	69.2	177	735 (107 C)	710 (103)	1.4	28.3 (4.11)	[illegible]	0.56
24	HMS	5208	80	75	21	0.66	68.7	177	623 (90.4 C)	634 (92)	1.2	40.5 (5.87)	5.5	0.64
21	HMS	332	80	75	21	0.52	68.9	177	453 (65.7 C)	458 (66.5)	1.1	35.6 (5.17)	9.5	[illegible]
26	HMS	1031	80	75	21	0.55	67.1	177	433 (62.8 C)	452 (65.5)	3.4	26.9 (3.90)	5.7	0.56

The flexural strength values shown in column 10 are average values of five specimens each. The samples were tested at a crosshead speed of 0.1 cm per minute; the samples were equilibrated to temperature for three minutes before testing. Failure mode of flexural strength tests: T (tensile failure), C (Compressive Failure), S (Shear Failure), V (Variable Failure within groups of five samples), F (Total fracture, mode could not be ascertained).

The shear samples were exposed for 24 days at 75°C and 80% RH. The shear strengths of column 13 are average values of 5 specimens.

Table 2: Carbon Fiber Properties[a]

Properties	Hercules HMS	Thornel 300
Strand Modulus, psi x 10^6 (G Pasc.)	50 - 55 (345-379)	33 - 34.5 (227-238)
Strand Break Strength, psi x 10^3 (G Pasc.	340 (2.34)	361 (2.49)
Density, g/cc (lot average)	1.85 - 1.90	1.74 - 1.78
Filament Diameter, microns	7.2 - 7.5	8
Number of Filaments	10,000	3,000

a) The data were obtained from the manufacturer at the time of purchase.

Table 3

Change in Short Beam Shear Strength of Narmco 5208/HMS as a Function of Exposure Time (after Exposure at 75°C and 80% RH; Test Temperature: 125°C)

Exposure Time, days	Avg. Moisture Uptake, Percent	Coeff. Var. (%)	S. B. Shear Strength MPasc. (KSI)	Coeff. Var. (%)
0	-	-	73.16 (10.61)	13.68
1	.347	9.57	73.74 (10.70)	8.82
4	.561	8.9	74.19 (10.76)	8.30
10	.667	5.0	69.70 (10.11)	11.31
28	.805	19.11	52.72 (7.65)	17.84
66	.854	5.15	46.62 (6.76)	8.15
115	.906	9.20	48.45 (7.03)	18.0

a) An average of six specimens were tested for each exposure condition.

From Figure 26 it can be seen that the interlaminar shear strength does not fall as rapidly as one might expect from the rather rapid increase of moisture content. However, this can be easily understood if one considers the internal moisture distribution in the composite at the time of testing (see Part I on moisture distribution) and the stress distribution in a short beam shear sample. While the higher shear stresses are generated in the center plan of the composite, this area has initially the lowest moisture content. Therefore there is a lag in strength degradation. On the other hand, the shape of the flexural strength degradation should be somewhat different since the maximal stresses are generated at the surface layers where the humidity saturation level is reached rather quickly. Thus the initial shape of the degradation curve is dominated by the failure mode and the distance of the initial failure (leading to catastrophic fracture) from the exposed surface.

The strength degradation caused by resin plasticization through moisture stops however after moisture equilibrium is reached. There is no essential difficulty to predict the time to reach moisture equilibrium in a composite, or any fraction of it, if the environmental temperature and moisture fluctuations are known (from weather data for instance). How to do this by simple averaging procedures, or more precisely by use of a high speed computer, will be discussed at a later date.

E. Reversibility of Flexural and S.B. Shear Strengths Losses.

A set of each composite was redried after the 3 weeks exposure to 80% RH at 75°C by heating it for 96 hours at 120°C in vacuo. In all cases were the flexural and shear strengths comparable with the original strengths within the experimental errors. (The test temperature was 125°C.) This is an other indication that the loss in strength after humidity exposure is due to resin plasticization and is reversible at least wihin the sensitivity of the mechanical testing.

CONCLUSIONS

1. The epoxy matrices investigated in this work all absorb moisture which leads to a substantial reduction in the resin modulus even at temperatures well below glass transition temperature. Thus moisture acts as a resin plasticizer. This effect is reversible after the moisture has been removed again, at least within the sensitivity of the test method.

2. In all cases it was observed that the matrix dominated strength properties of the composites (flexural and shear strengths) were reduced after the samples had been exposed to moisture. Again, within the sensitivity of the test methods, this strength degradation is reversible, i.e., after removal of the moisture the original strength values were regained.

3. The loss in strength levels out after the composite has reached equilibrium with the humidity of the surrounding environment. The shape of the strength loss curve with time depends probably on the failure mechanism and on the distance of the initiation site of catastrophic failure from the exposed surface.

RECOMMENDATIONS

1. It is recommended that further experimental investigations of moisture effects be carried out on crossplied laminates since these composites may be more sensitive to the formation of internal stresses, due to moisture swelling, and to temperature changes due to mismatch between the thermal expansion coefficients of resin and fiber.

2. We further recommend that the laminated plate and shell theory be expanded to include moisture and temperature effects. This could be accomplished by incorporating the variability of the matrix modulus (as a function of moisture concentration and temperature) into the stiffness tensor of the laminate. It is expected that the continuously changing matrix modulus through the thickness of the composite can be approximated successfully by a finite difference approach, so that no major change will be required in the formalism of composite analysis.

Appendix A

MATERIALS

A. Resins

The resins used for the prepregging operations were:

1. Narmco 5208 (a commercial resin manufactured by the Whittaker Corporation, Narmco Division). It is a one component system (resin plus curing agent).

2. DER 332/DADS. The resin system consists of 100 parts of DER 332 (a diglycidyl ether of bisphenol A) and 36 parts of DADS (4,4' diaminodiphenyl sulfone).

3. Epon 1031/NMA. This resin consists of 100 parts of Epon 1031 (1,1,2,2-tetra (p-glycidyloxyphenyl)ethane), 77 parts of Nadic methyl anhydride, and 1 part of BDMA (benzyldimethylamine).

B. Fibers

1. Thornel 300. This material was obtained from Union Carbide Corporation and consists of a continuous strand of 3,000 filaments which have an epoxy sizing for better handleability. (For properties reported by the manufacturer see Table 1.)

2. HMS fibers. This material is a tow with 10,000 continuous filaments manufactured by the Hercules Corporation. The fibers had no sizing. (For properties reported by the manufacturer see Table 2.)

C. Prepregs

The prepregs were made by filament winding the dry carbon fiber yarn onto an aluminum cylinder (60 cm in diameter). Before the winding operation two strips of double sided adhesive tapes were placed onto the cylinder surface (parallel to the direction of the cylinder axis). A 60 cm wide band of fibers was now wound onto the cylinder. Then two more adhesive tapes were placed over the first strips so that the band of fibers was held in place. The winding density of the T300 fibers was 15.48 strands per cm width and the density of the HMS fibers was 3.72 strands per cm. A cut was made between the adhesive strips so that the band of carbon fibers could be transferred to a flat table (covered with a mylar film) by carefully lifting it up from the winding cylinder. The

fiber band was now stretched on the table and fixed with the adhesive tape. A sixty percent solution of the resin in acetone was carefully poured over the fiber band. The amount of resin used was adjusted so that the ratio of resin to fiber was such that a prepreg with 40 weight percent of resin was obtained. The impregnated band was then covered with a 1 mil Teflon film and the resin solution was carefully worked into the fiber band by means of a rubber roller. The Teflon film was then removed to allow the acetone to evaporate (beware of fire hazard! The prepreg was cut into 241 x 23 cm sheets. The mylar backing was removed just before stacking to form the laminate by rubbing the Mylar film with dry-ice. This made the tacky resin brittle enough to allow to pull off the Mylar backing.

D. Laminate Fabrication

The unidirectional laminate plates were fabricated by a combined vacumn pressure technique. The 41 x 23 cm prepreg sheets were stacked and placed onto a steel plate between layers of porous materials to allow resin bleeding. The layup was as follows: (1) Al-foil, (2) bleeder paper, (3) porous teflon, (4) thin glass scrim cloth, (5) laminate, (6) thin glass crim cloth, (7) porous teflon, (8) bleeder paper, (9) perforated metal caul plate, (10) two layers of thick 181 glass cloth, and (11) Silicon or Mylar cover film that was sealed with a zink chromate sealing compound. On one side of the steel plate had a channel drilled through which vacuum could be applied (see Figure 27).

The whole assemblage was placed between the preheated plates of a Preco press to be cured under pressure and applied vacuum.

The curing conditions for the composites are given in Table 4. After cure the panels were allowed to cool over night under pressure. They were post cured between metal plates in an oven at 204°C for 4 hours. No significant difference in strength was observed between panels post cured in air or under nitrogen. The average panel thicknesses were 0.203 cm.

Table 4

Sequence of Curing Conditions

Composite	1. Initial Temp. °C	2. Initial Pressure	3. Heat Rise	4. Dwell	5. Pressurize Applied	6. Final Heat rise (°C Per Min)	7. Cure Temp. °C	8. Post Cure (°C)
Narmco 5208 T300 and HMS	90	applied vacuum (5 mm)	2-3° per Min.	60 Min. at 135°C	100 psi	2-3° per Min.	180, 2 hrs.	204, 4 hrs.
DER 332/ T300 and HMS	110	4 mm Vac.	2-3° per Min.	76 Min. at 135°	100 psi	2-3° per Min.	180, 2 hrs.	204, 4 hrs.
EPON 1031/ T300 and HMS	250	4 mm Vac.	2-3° per Min.	36 Min. at 132°	100 psi	2-3° per Min.	180, 2 hrs.	204, 4 hrs.

Appendix B

Mechanical Testing and Humidity Exposure

Flexural and short beam shear specimens were machined (size: 8.9 x 1.27 cm^2 and 1.45 x .635 cm^2 respectively) and tested under 3 point loading before and after humidity exposure. The samples were considered dry after heating for 96 hours at 120°C in a vacuum desiccator (no further weight loss was observed after this time). The humidity exposure conditions were: 80 percent relative humidity for 21 days at 75°C. (A beaker containing the samples was placed into a wide-mouth screw cap container (23.5 cm high, 11.5 cm diameter). A potassium chloride-water mixture (2 cm high, with undissolved solute) served to maintain the relative humidity constant at 80 percent. The container was closed and kept submerged in a water bath at a temperature of 75°C $\pm$ 1°). The results are given in Table 1.

To determine the shape of the shear strength degradation curve. one set of samples was exposed up to 115 days. These results are given in Table 3.

The mechanical testing at elevated temperature was carried out in an environmental chamber with forced air heating. By placing a thermocouple in the center of one specimen it was established that it required three minutes to equilibrate the sample to the chamber temperature. To minimize moisture desorption during testing three minutes were allowed for all specimens for temperature equilibration before the load was applied.

Torsional Braid Analysis (TBA)

The Torsional Braid Analysis of the resins was carried out with a Chemical Instrument Corporation Torsional Braid Analyzer Mod 100-1B. The method of TBA has been described in two review articles [2, 3] by J. K. Gillham and will not be discussed here. The braid samples were impregnated with the same resins as used for the panel fabrication and cured under the same condition as the composite panels. (The cure and post cure were carried out inside the TBA instrument under nitrogen atmosphere at atmospheric pressure.)

After the resin had been cured the "dry" rigidity curve was determined.

---

[2] J. K. Gillham, Rev. Macrom, Sci. 1, 83 (1972)
[3] J. K. Gillham, AIRCHE Journal, 20, 1066 (1974)

The same sample was then used to determine the change of the rigidity after exposure to moisture. The exposure time of the thin braid to various levels of moisture was ten days in each case which was sufficient to let the braid come to equilibrium.

In order to prevent excessive desorption of moisture during the temperature scan the highest heating rate (30°C per minute) was used for the determination of the rigidity curve. Even at these high heating rates some moisture will desorb from the resin, therefore, these curves have to be considered as upper limits in the reduction of resin rigidity. Since it was found that the original, dry rigidity curve, was regained with each resin after the moisture had been removed, only one sample each was used to determine the change in the modulus curves as a result of exposure to various levels of relative humidity. The results are shown in Figure 1 to 5.

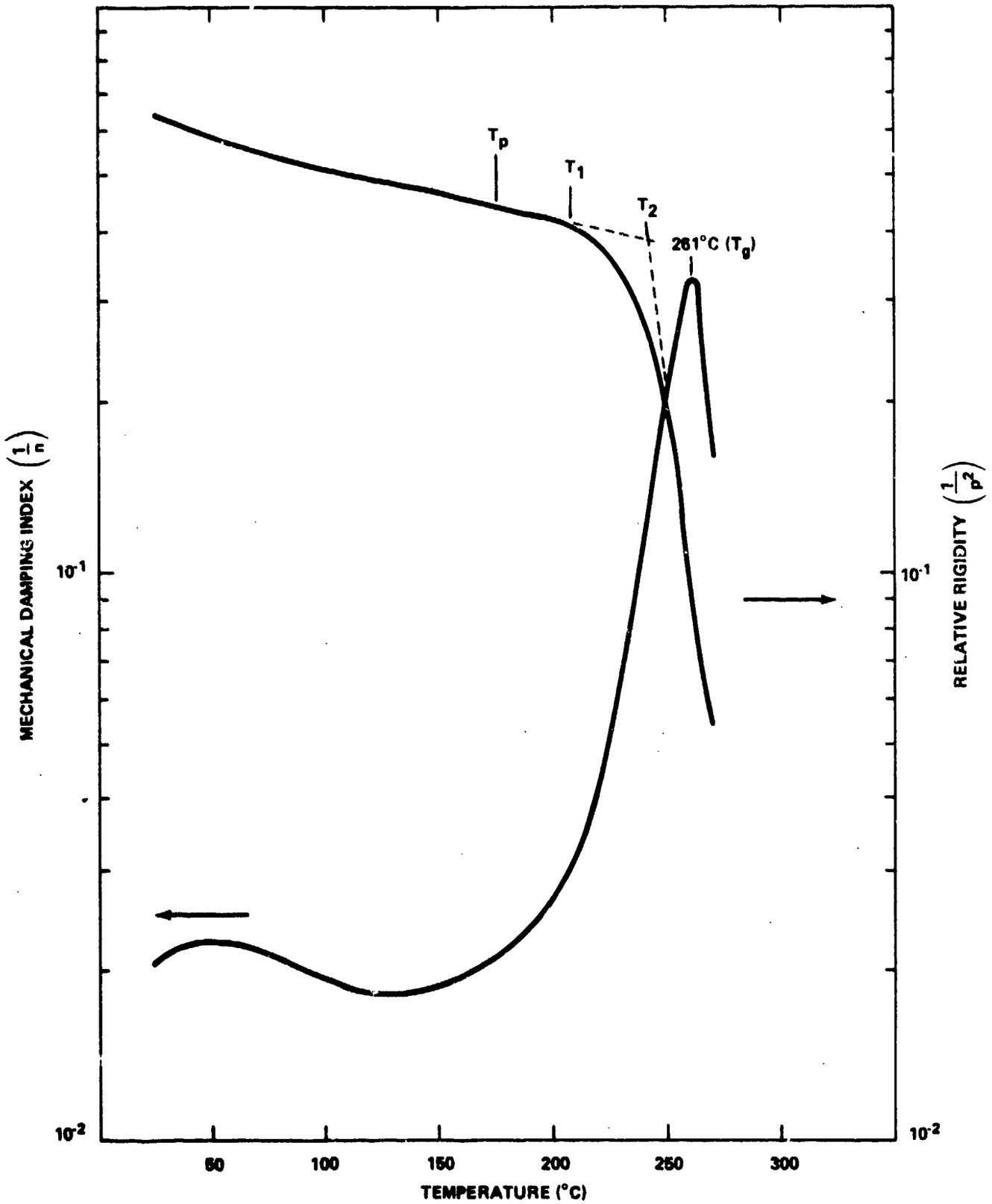

FIG. 1 THERMOMECHANICAL BEHAVIOR OF THE NARMCO 5208 RESIN

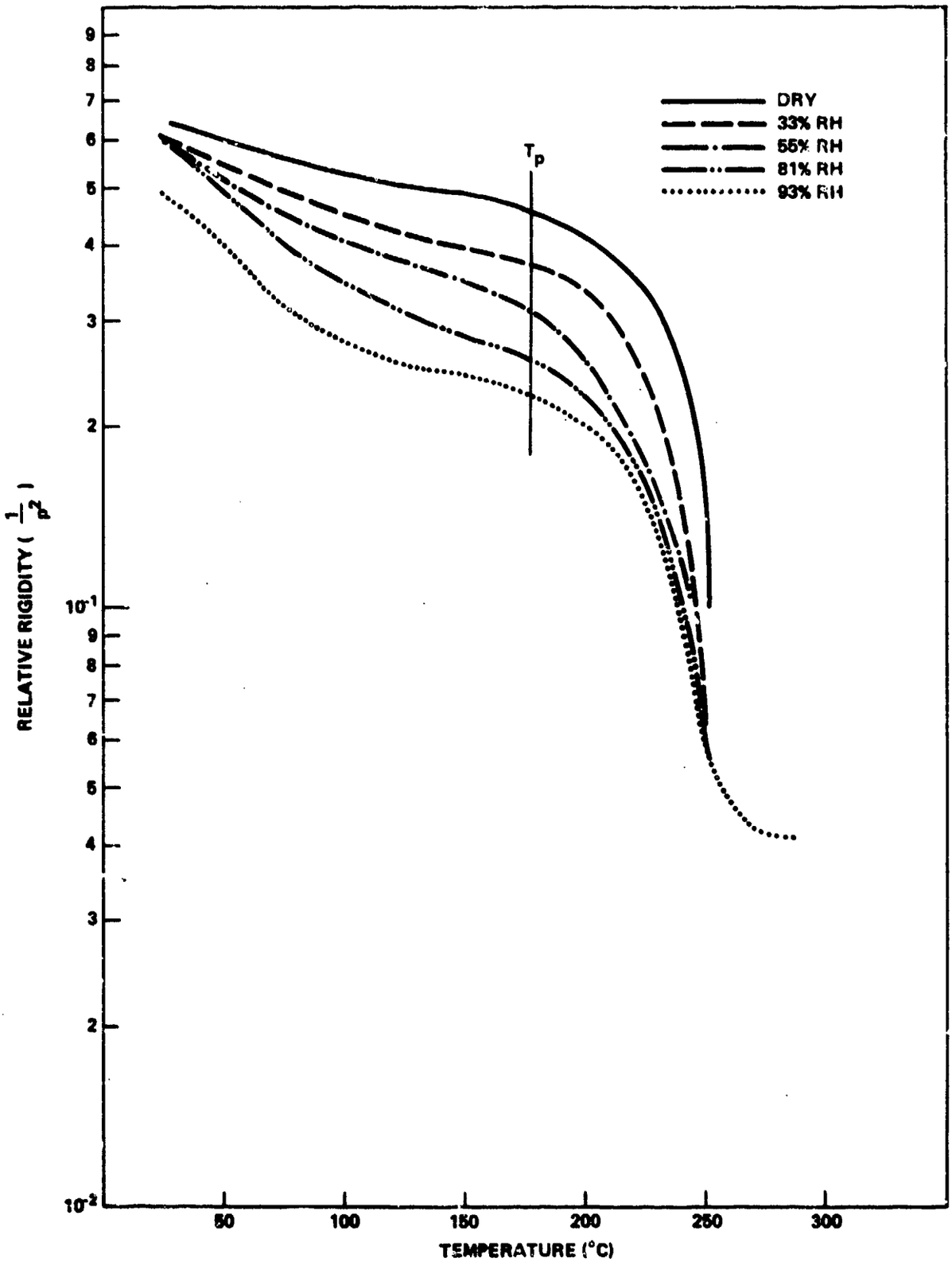

FIG. 2 EFFECT OF VARIOUS RELATIVE HUMIDITIES ON RESIN MODULUS (DETERMINED BY TBA AFTER 10 DAYS EXPOSURE. RESIN: NARMCO 5208).

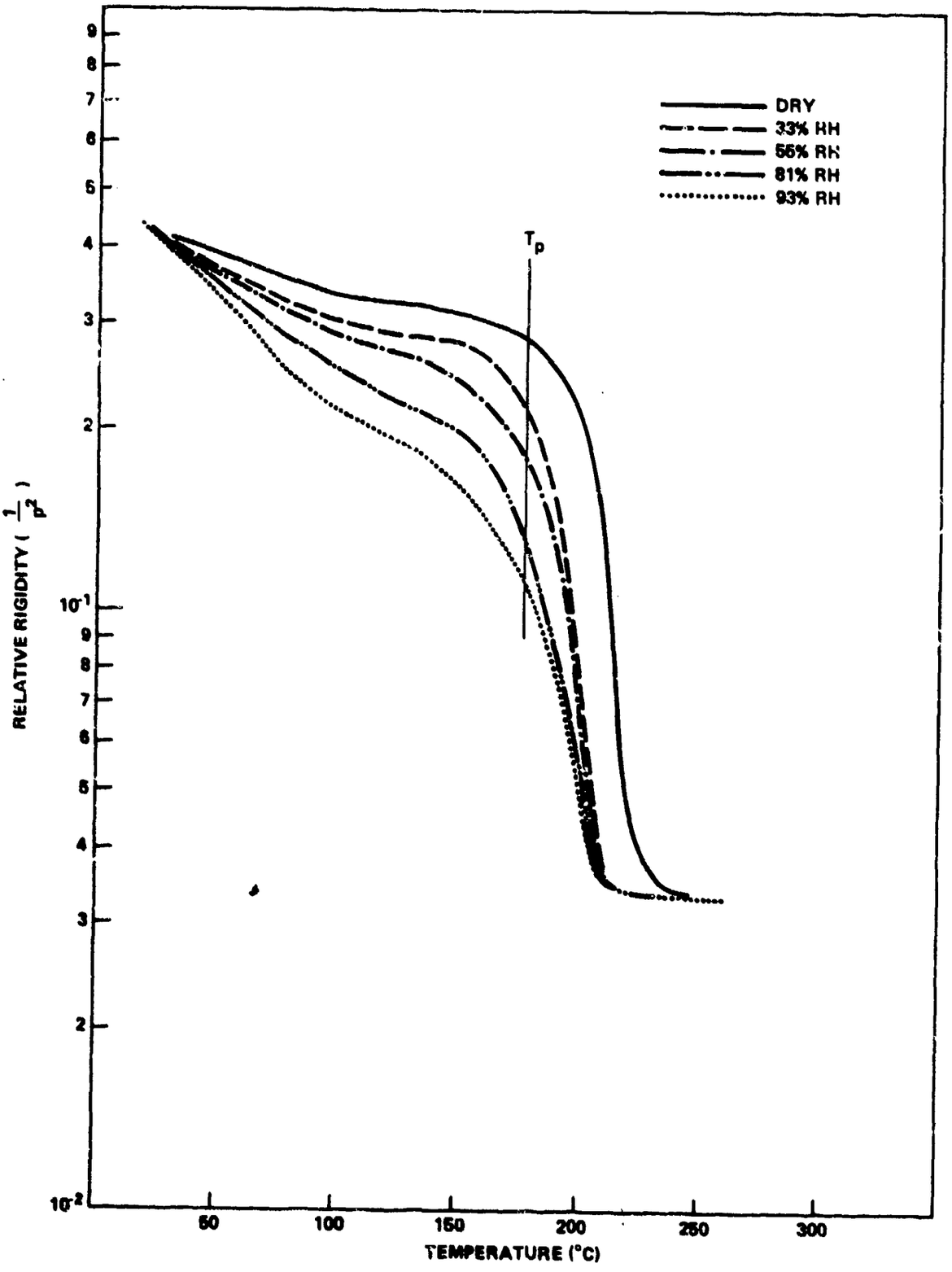

FIG. 3 EFFECT OF VARIOUS RELATIVE HUMIDITIES ON RESIN MODULUS (DETERMINED BY TBA AFTER 10 DAYS EXPOSURE. RESIN: DER 332/DADS).

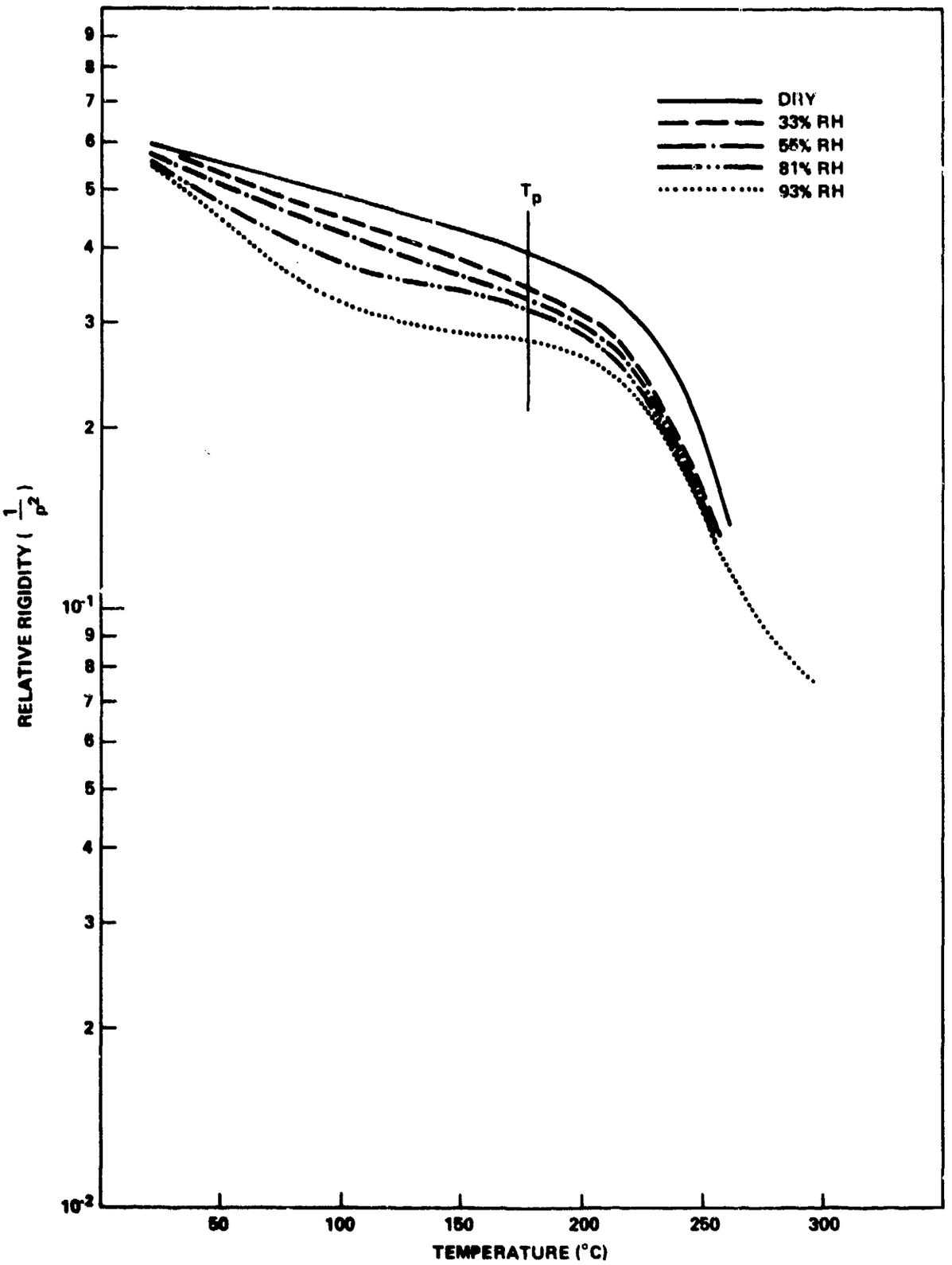

FIG. 4 EFFECT OF VARIOUS RELATIVE HUMIDITIES ON RESIN MODULUS (DETERMINED BY TBA AFTER 10 DAYS EXPOSURE. RESIN: EPON 1031/NMA).

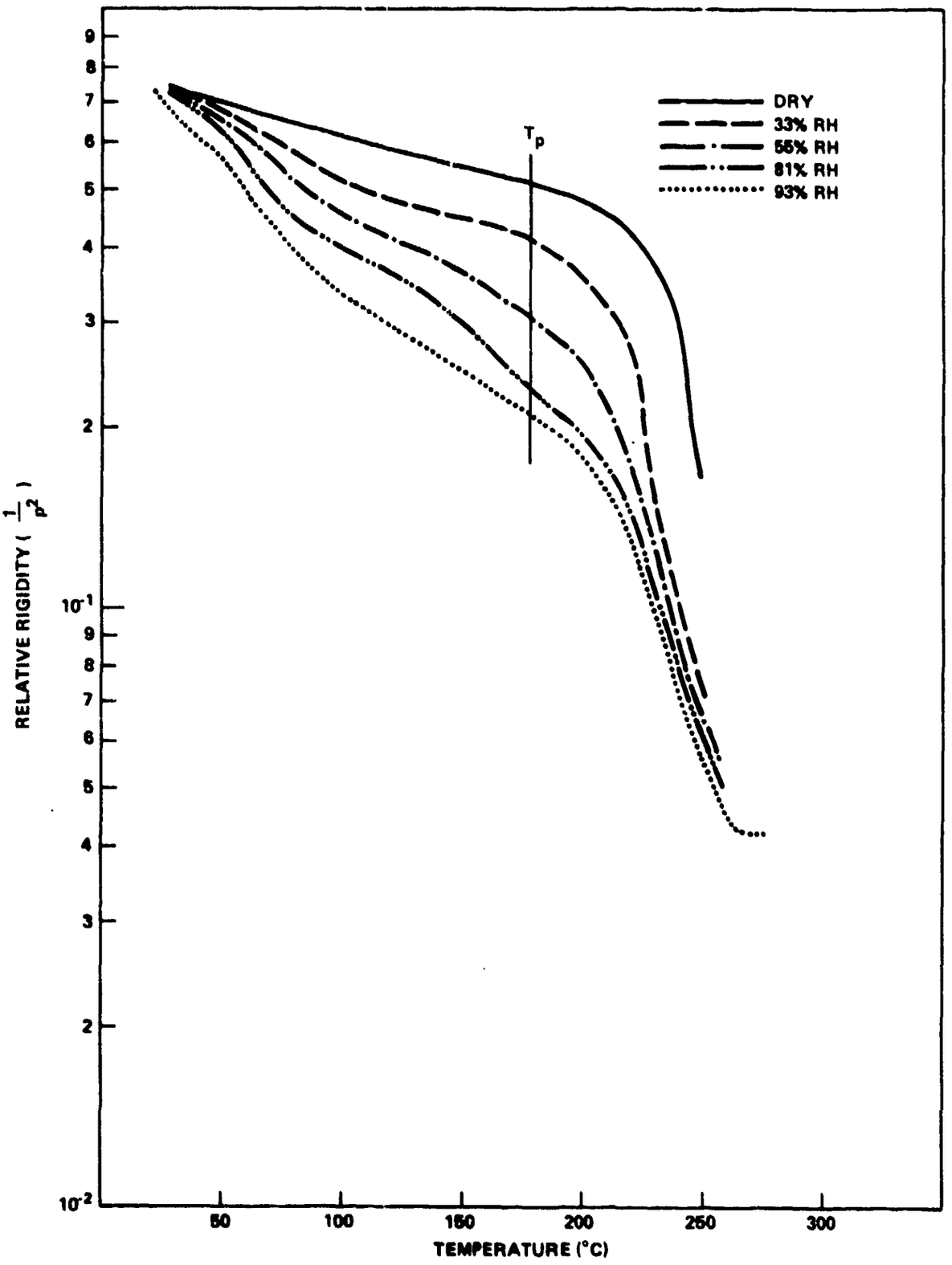

FIG. 5 EFFECT OF VARIOUS RELATIVE HUMIDITIES ON RESIN MODULUS (DETERMINED BY TBA AFTER 10 DAYS EXPOSURE. RESIN: HERCULES 3501)

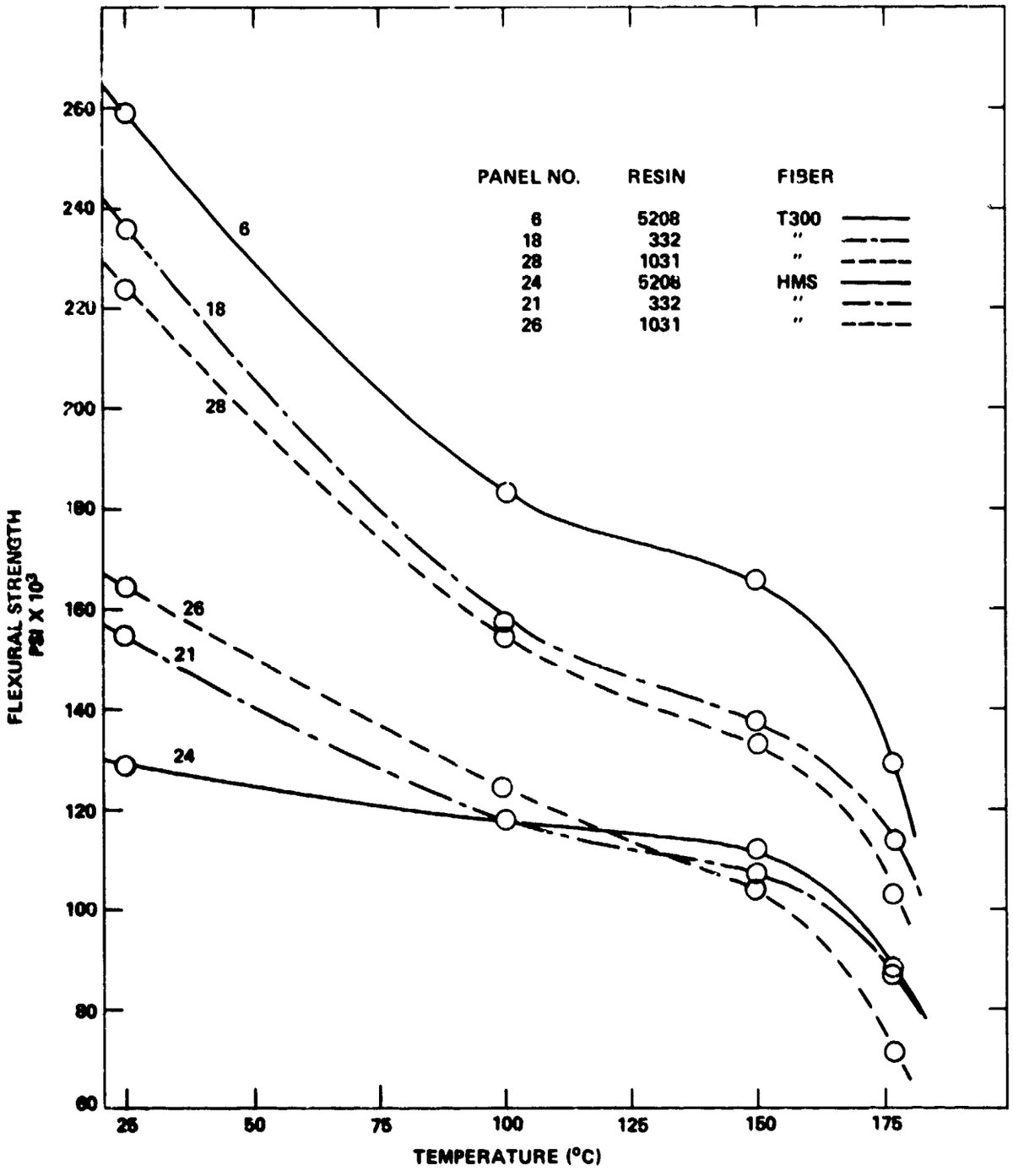

FIG. 6 FLEXURAL STRENGTH OF CARBON FIBER COMPOSITES AS A FUNCTION OF TEMPERATURE (BEFORE EXPOSURE)

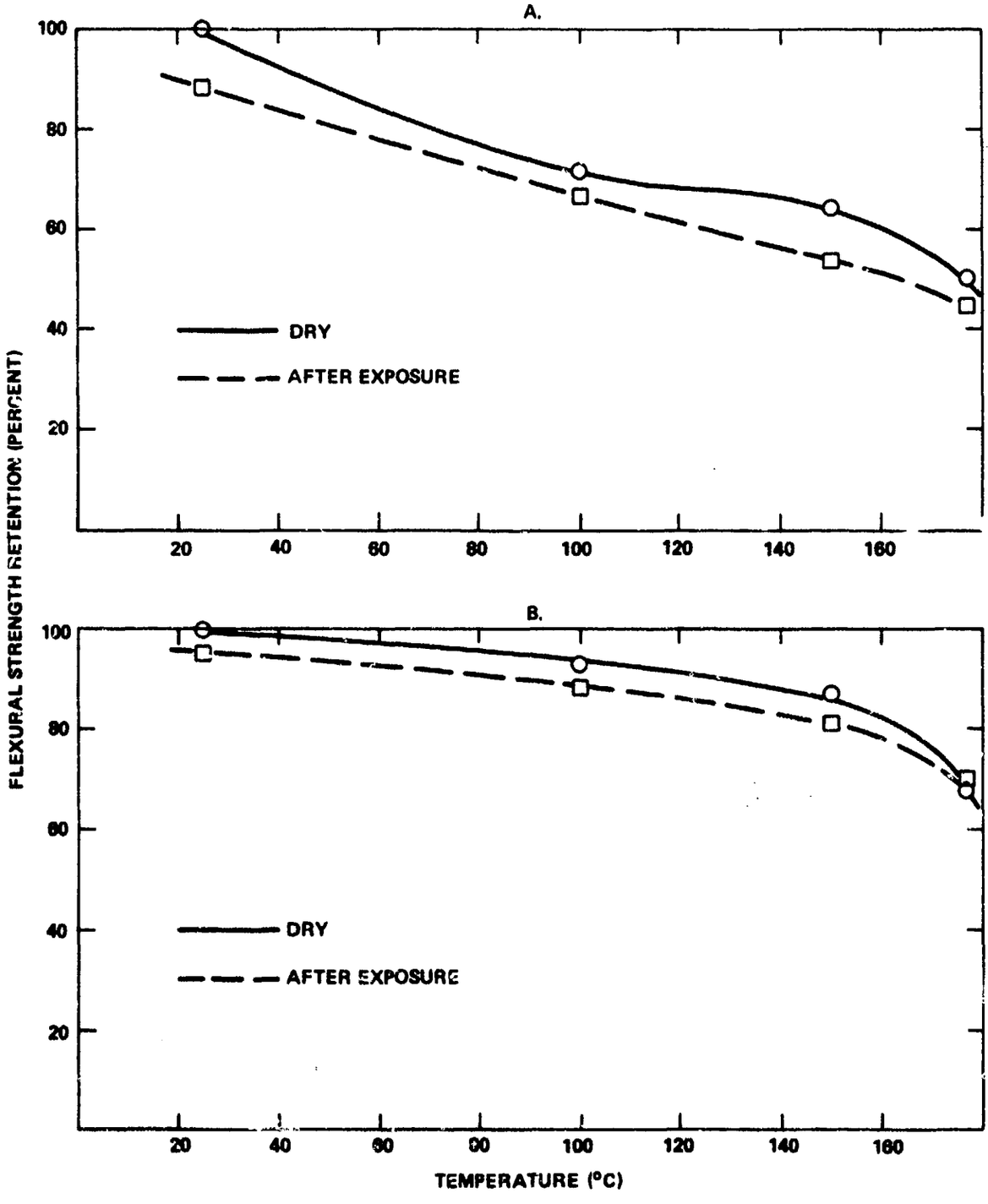

FIG. 7 PERCENT FLEXURAL STRENGTH RETENTION OF (A) NARMCO 5208/T300 EPOXY COMPOSITE, (B) NARMCO 5208/HMS EPOXY COMPOSITE BEFORE AND AFTER HUMIDITY EXPOSURE

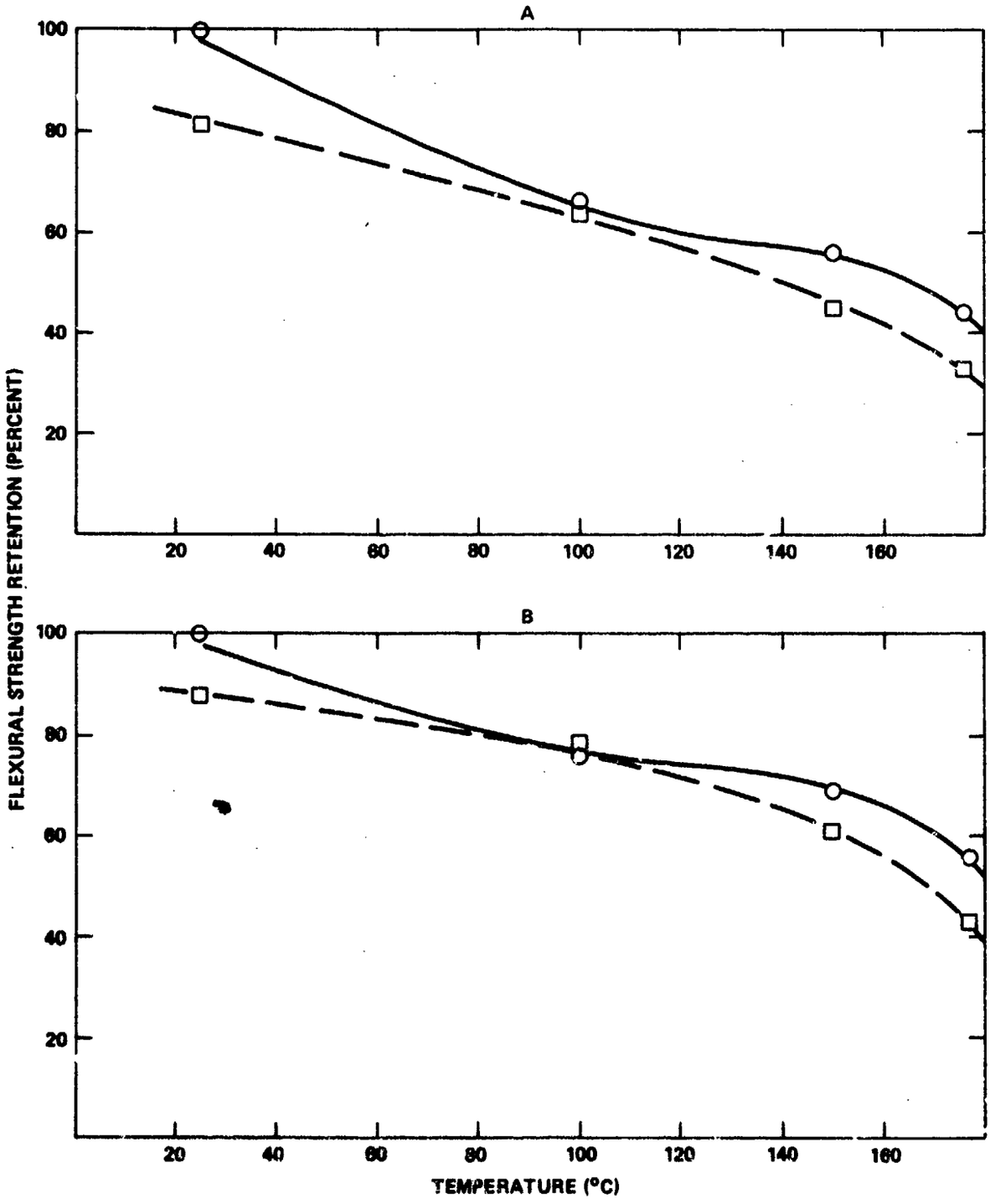

FIG. 3 PERCENT FLEXURAL STRENGTH RETENTION OF (A) DER 332/T300 EPOXY COMPOSITE, (B) DER 332/HMS EPOXY COMPOSITE, BEFORE AND AFTER HUMIDITY EXPOSURE

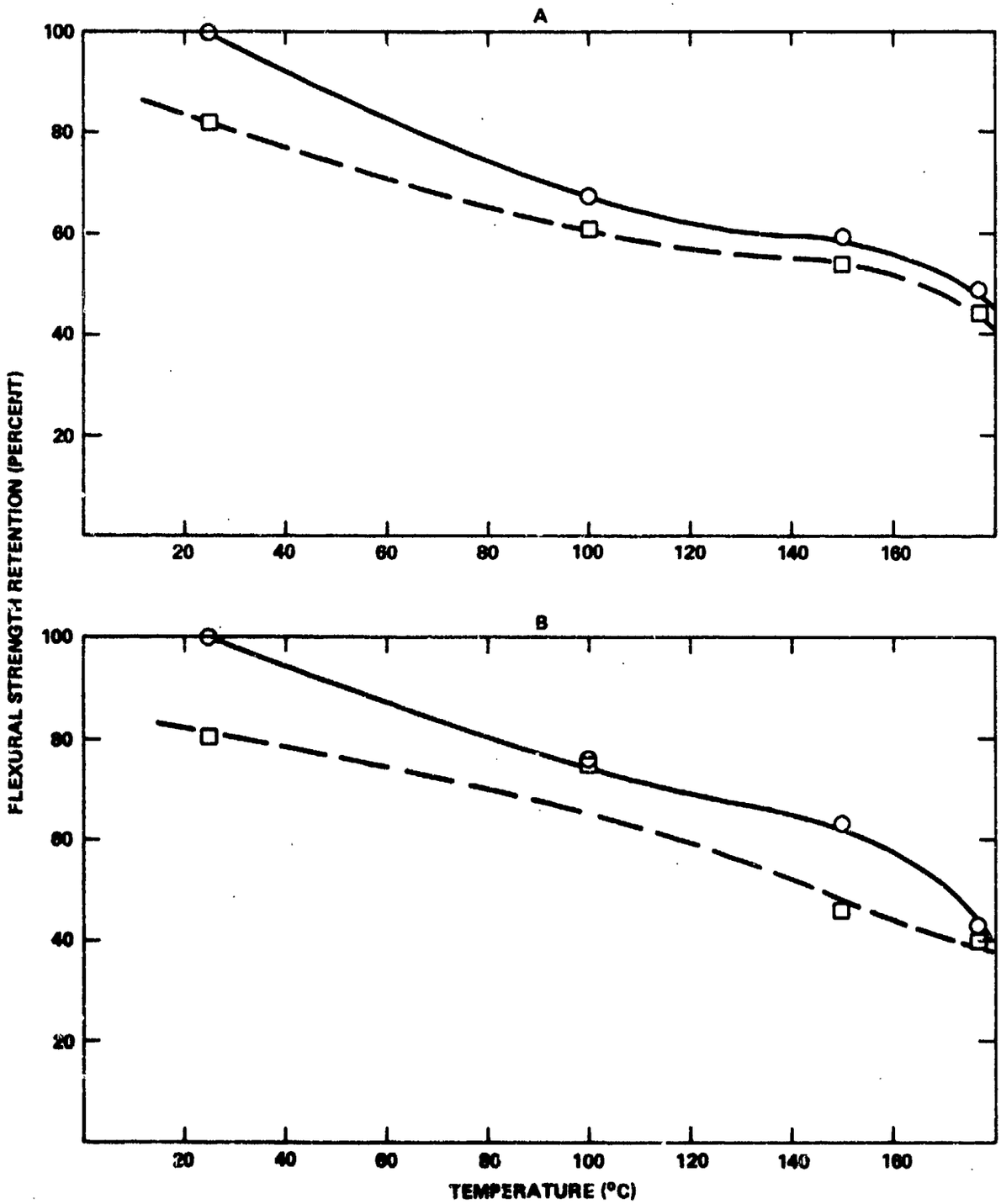

FIG. 9 PERCENT FLEXURAL STRENGTH RETENTION OF (A) EPON 1031/T300 EPOXY COMPOSITE; (B) EPON 1031/HMS EPOXY COMPOSITE BEFORE AND AFTER HUMIDITY EXPOSURE

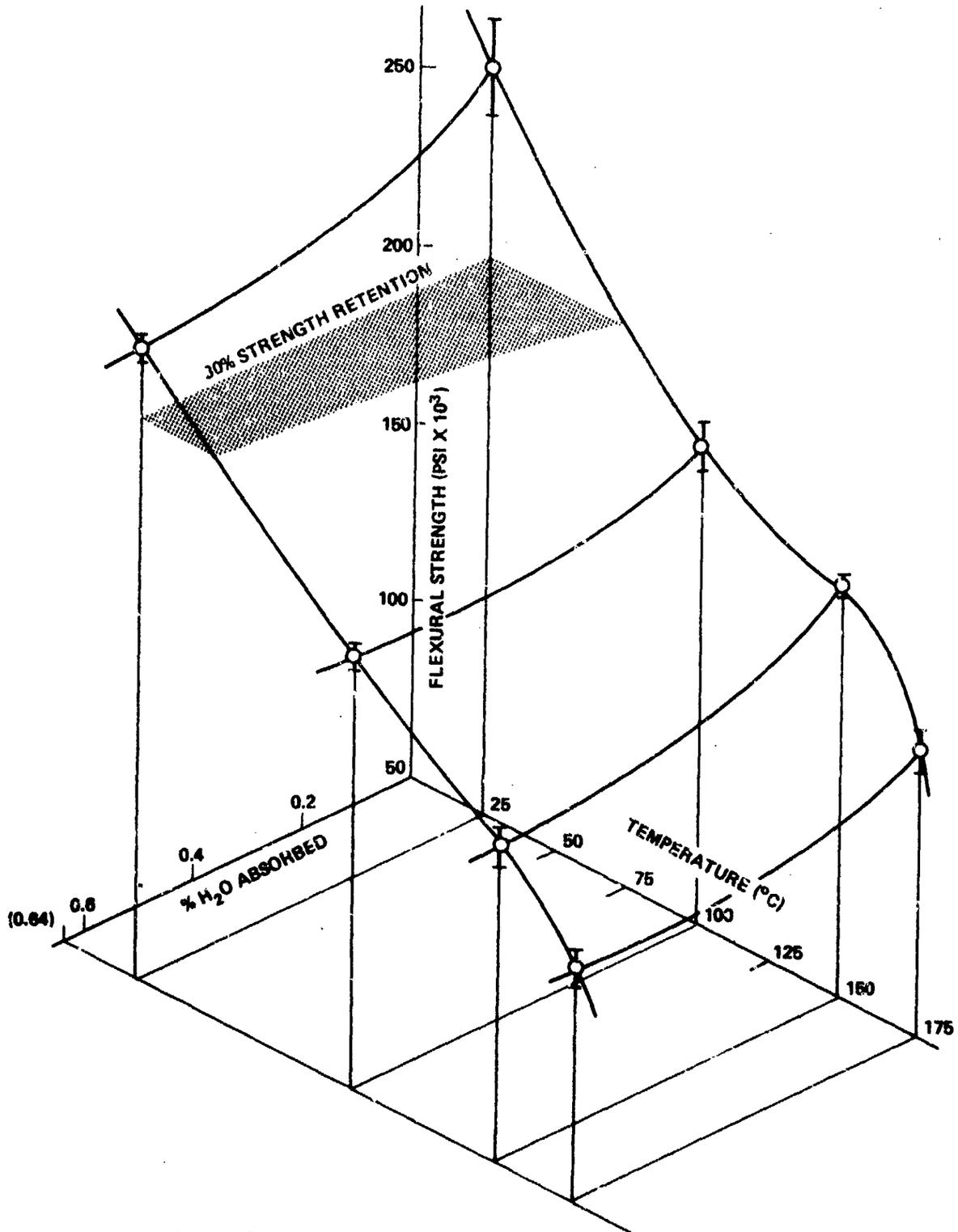

FIG. 10 FLEXURAL STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (NARMCO 5208/T300) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED

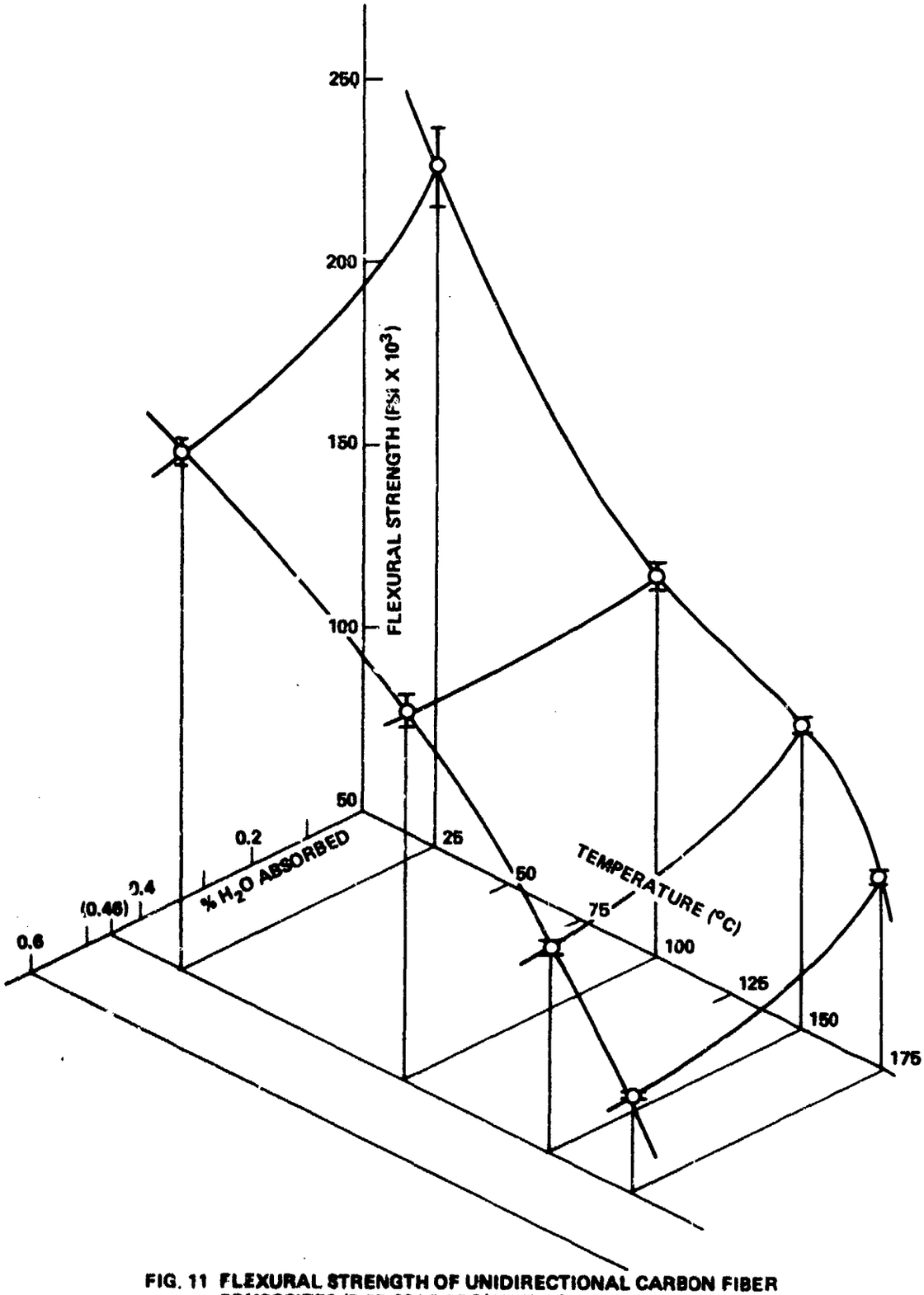

FIG. 11 FLEXURAL STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (DER 332 DADC/T300) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED

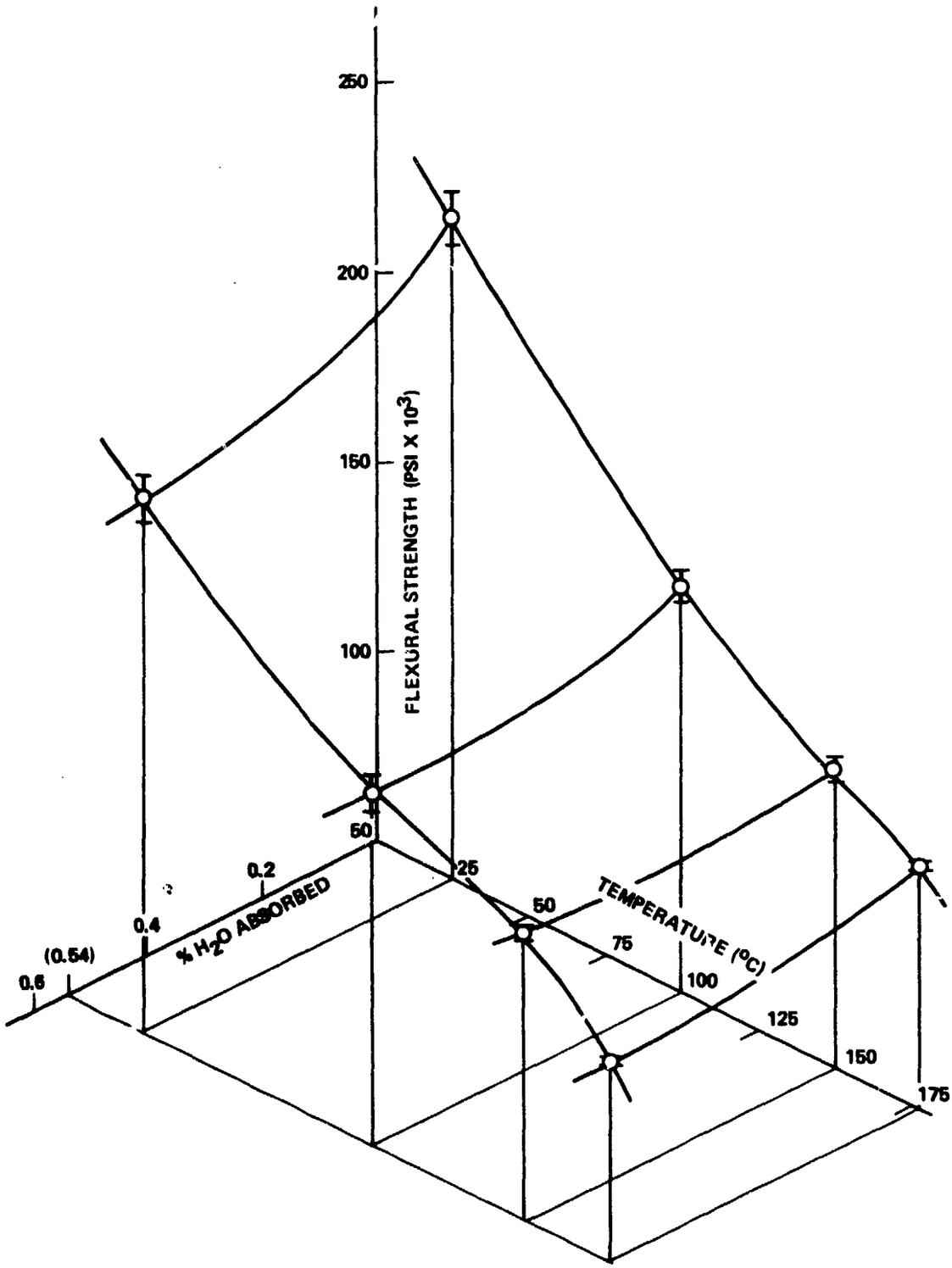

FIG. 12 FLEXURAL STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (EPON 1031 NMA/T300) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED

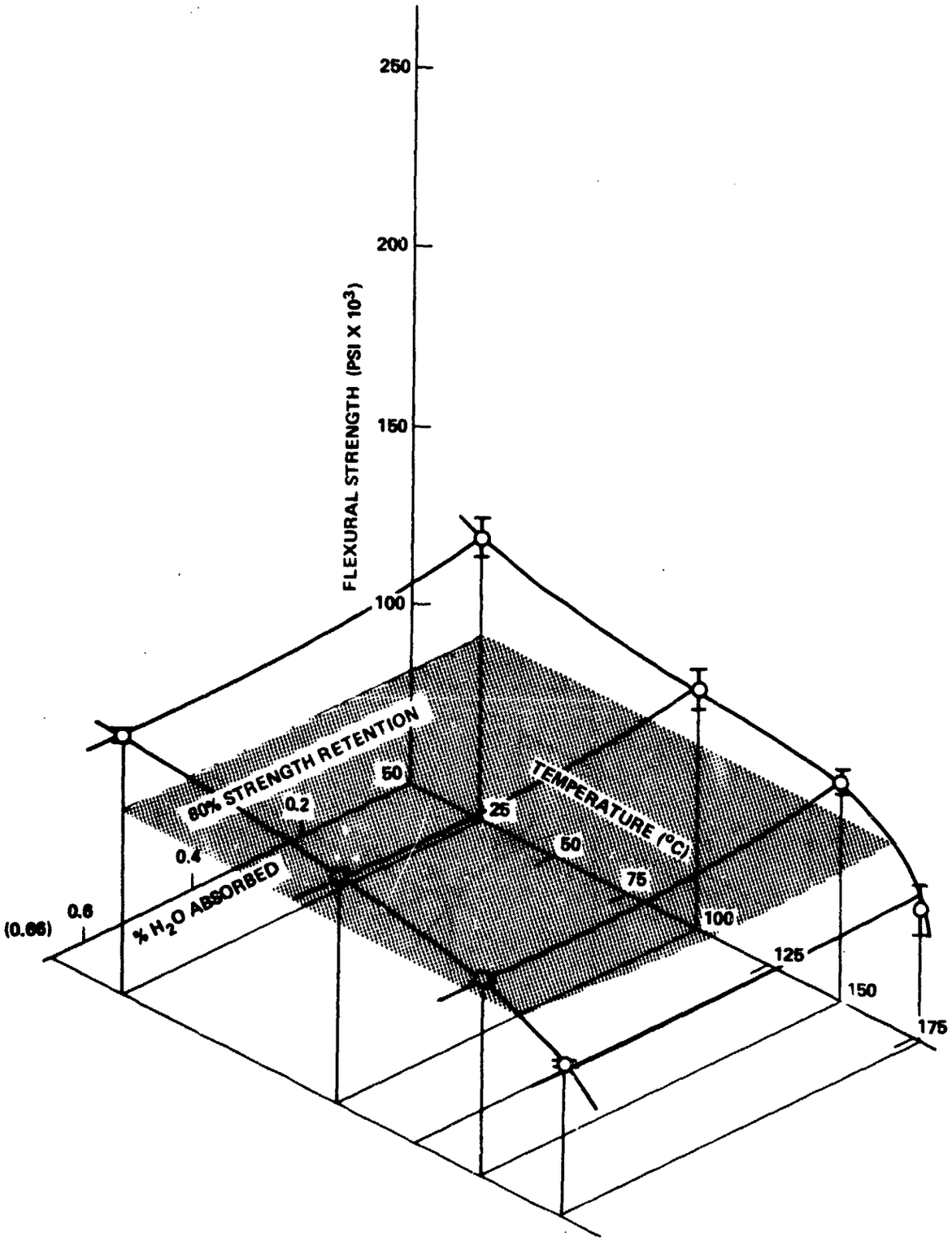

FIG. 13 FLEXURAL STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (NARMCO 5208/HMS) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED

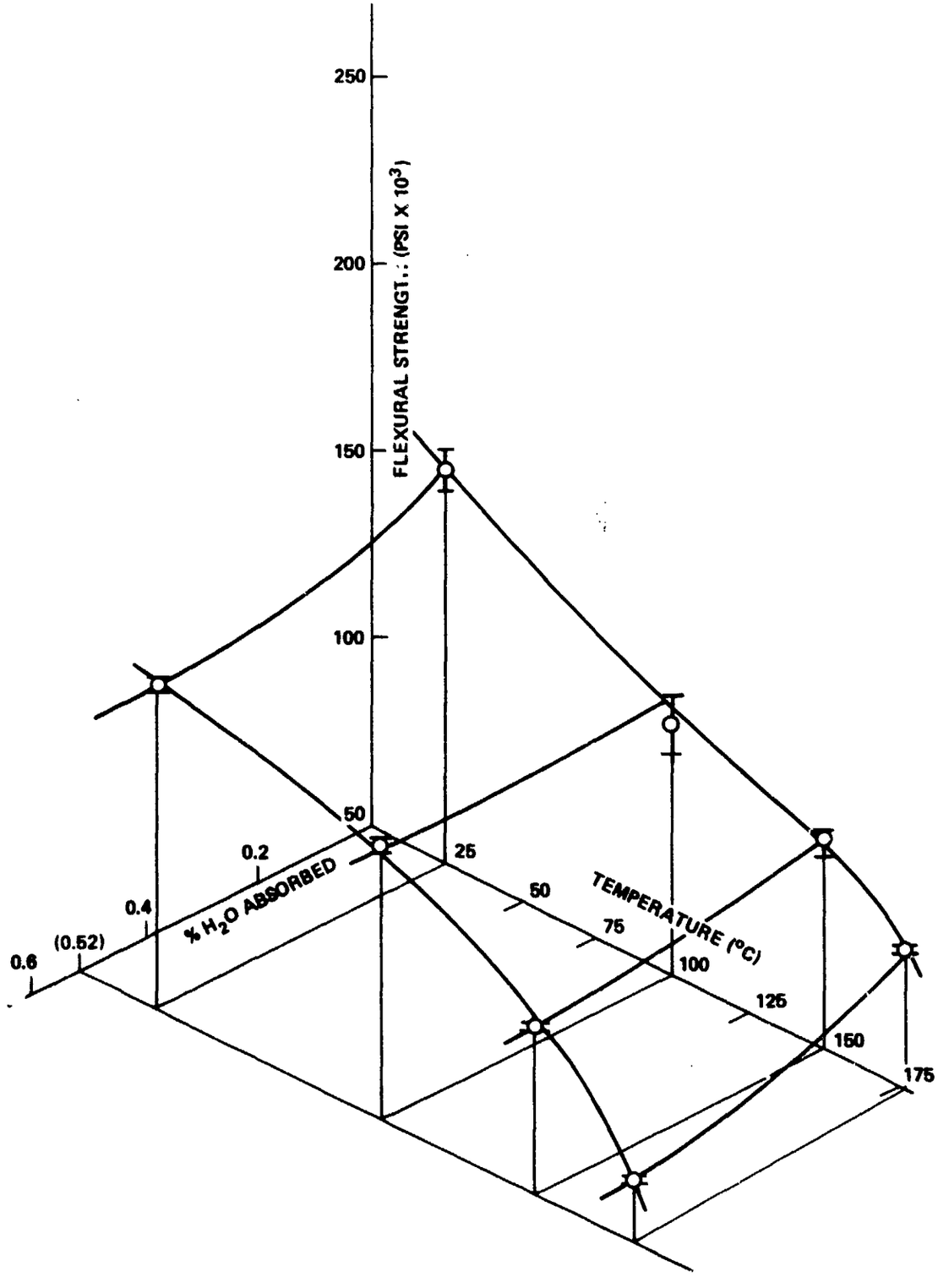

FIG. 14 FLEXURAL STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (DER 332 DADS/HMS) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED

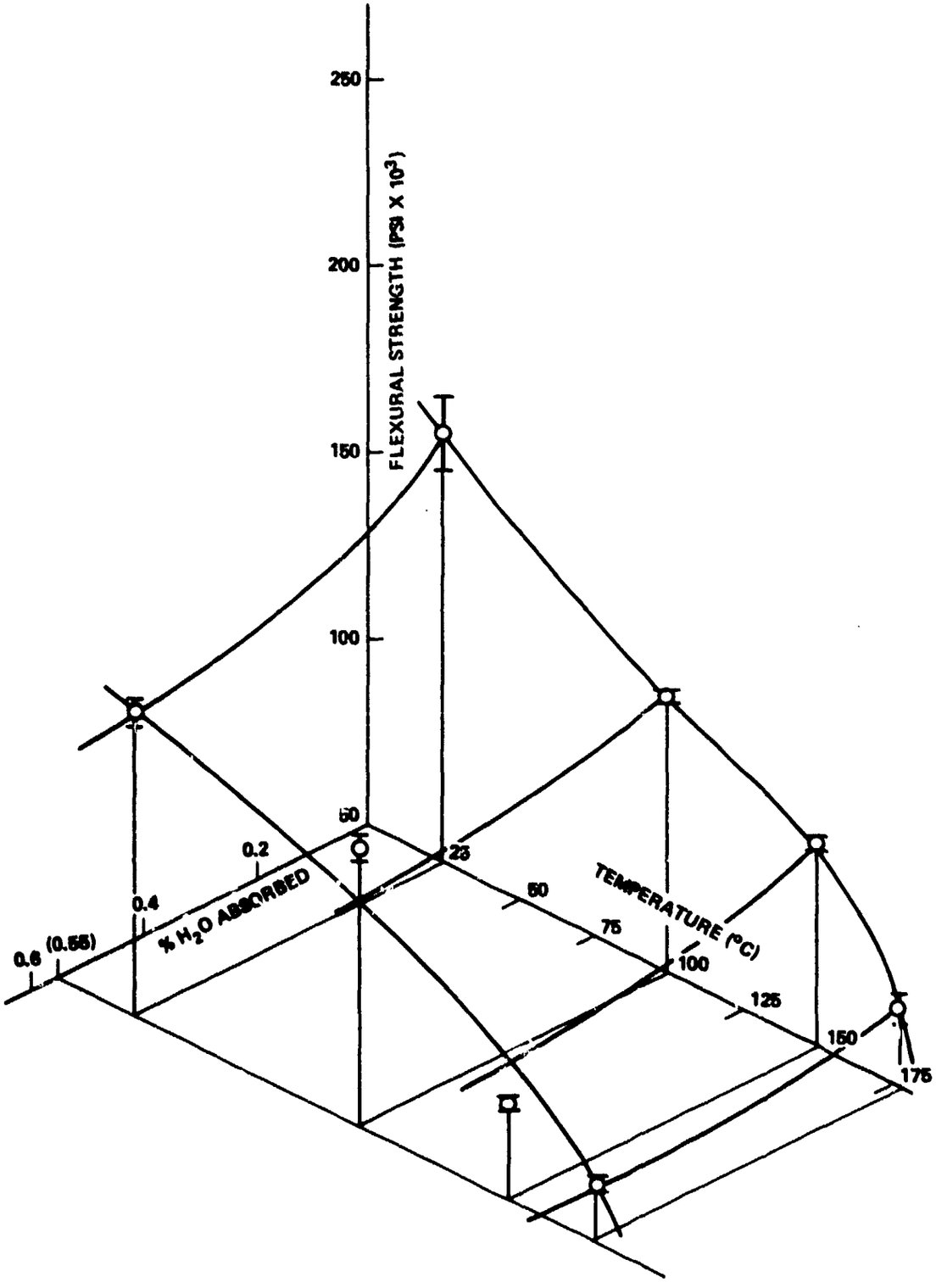

FIG. 15 FLEXURAL STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (EPON 1031 NMA/HMS) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED

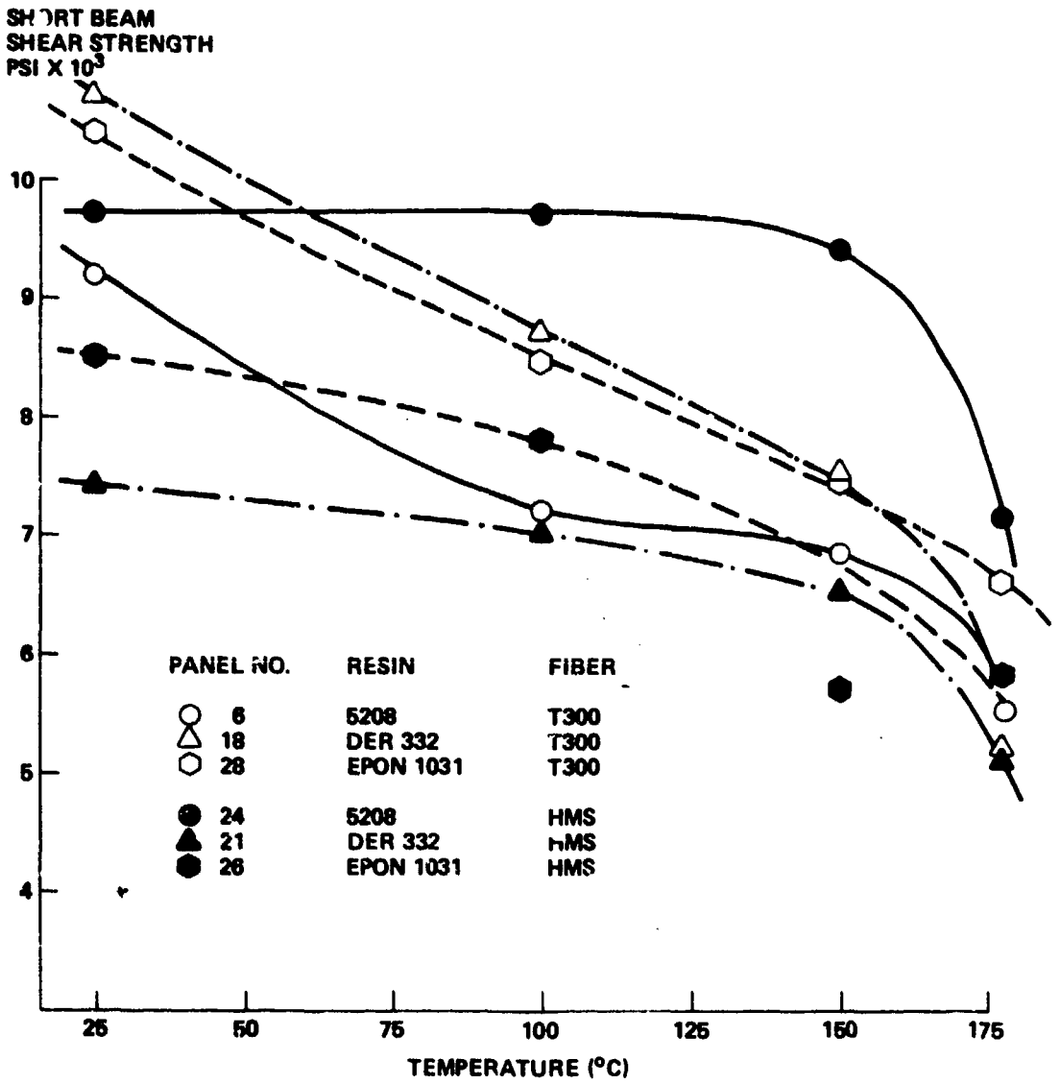

FIG. 16 SHORT BEAM SHEAR STRENGTH OF CARBON FIBER REINFORCED COMPOSITES AS A FUNCTION OF TEMPERATURE. (BEFORE EXPOSURE)

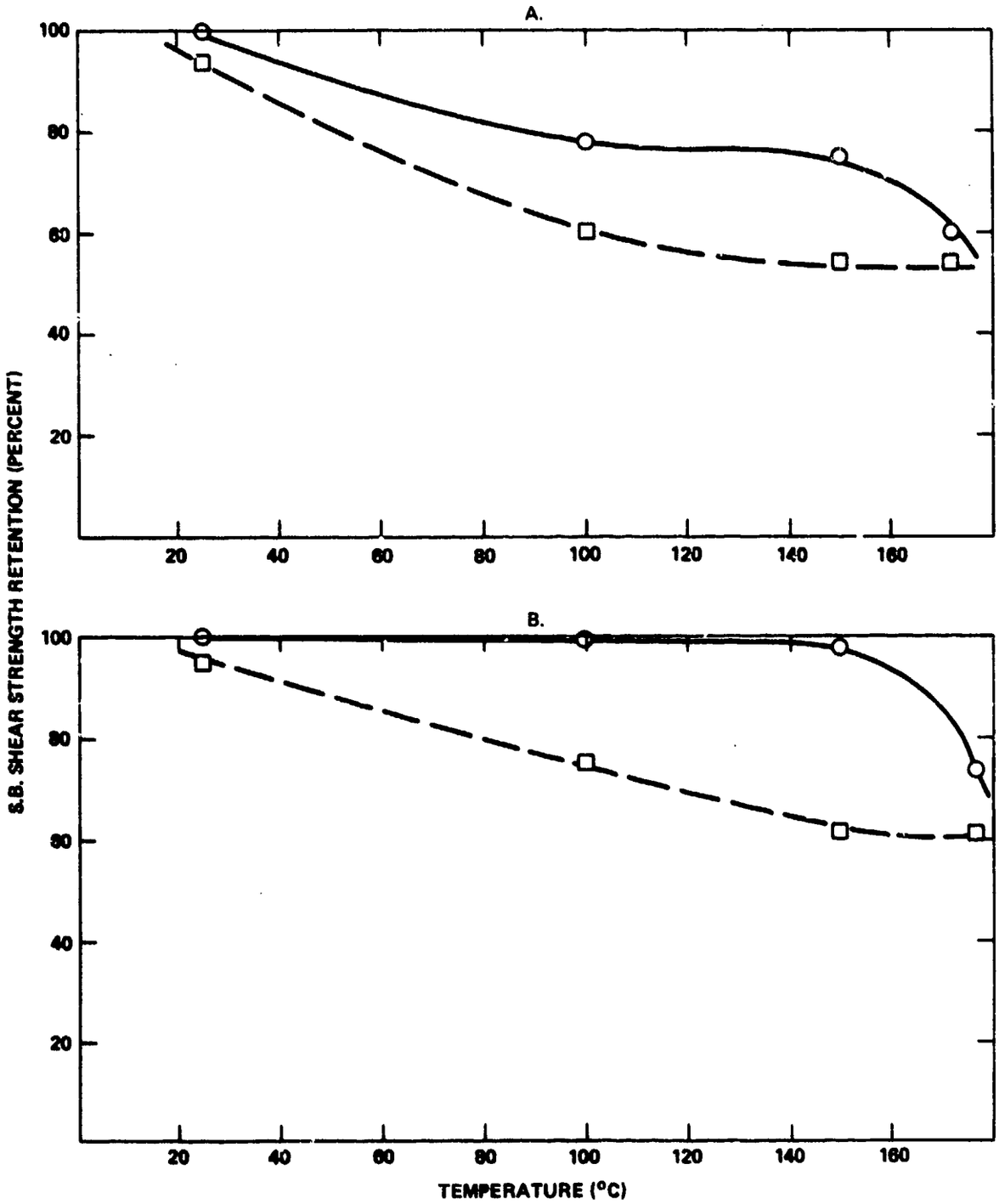

FIG. 17 PERCENT S.B. SHEAR STRENGTH RETENTION OF (A) NARMCO 5208/T300 EPOXY COMPOSITE, (B) NARMCO 5208/HMS EPOXY COMPOSITE BEFORE AND AFTER HUMIDITY EXPOSURE

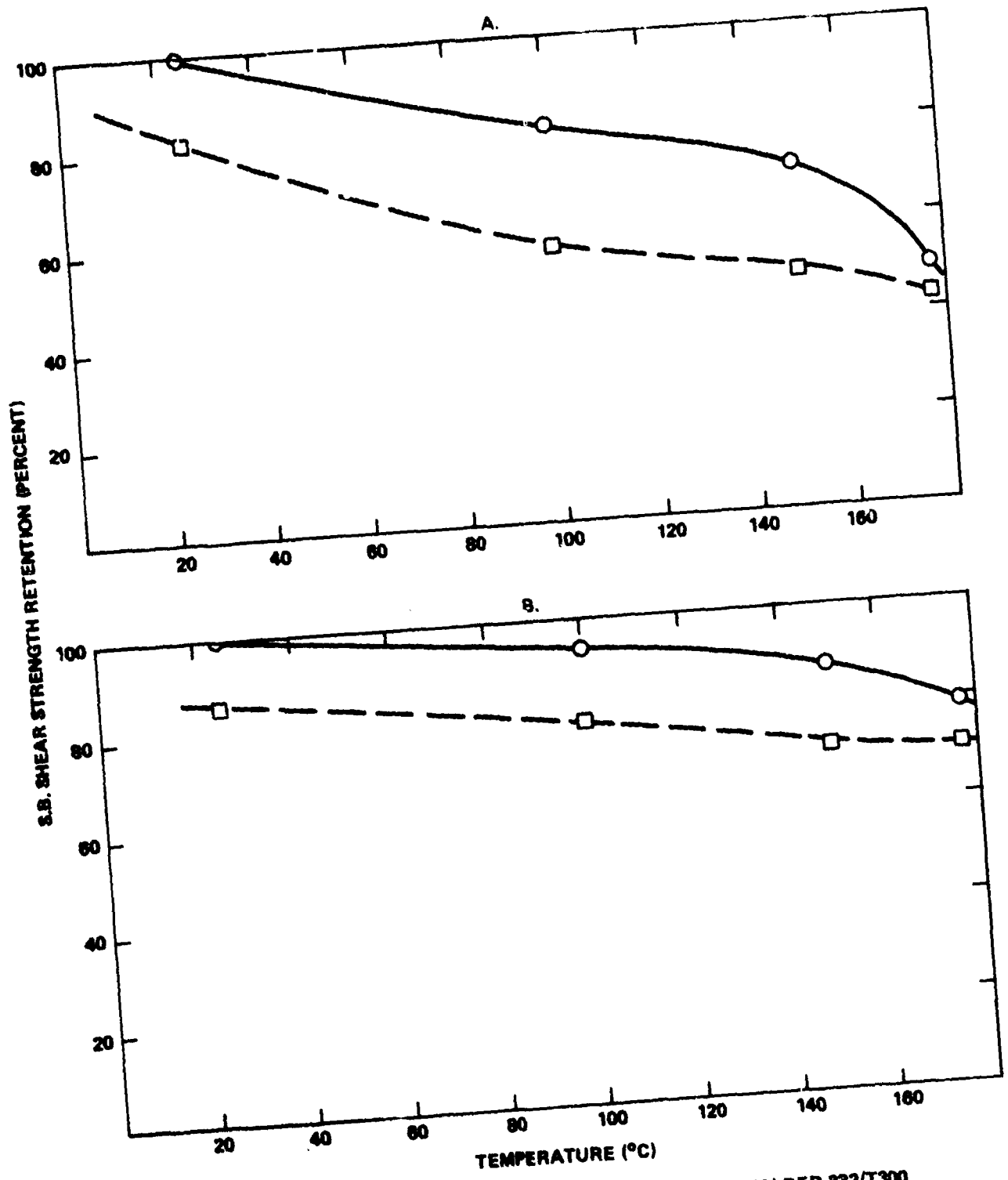

FIG. 18 PERCENT S.B. SHEAR STRENGTH RETENTION OF (A) DER 332/T300 EPOXY COMPOSITE, (B) DER 332/HMS EPOXY COMPOSITE BEFORE AND AFTER HUMIDITY EXPOSURE

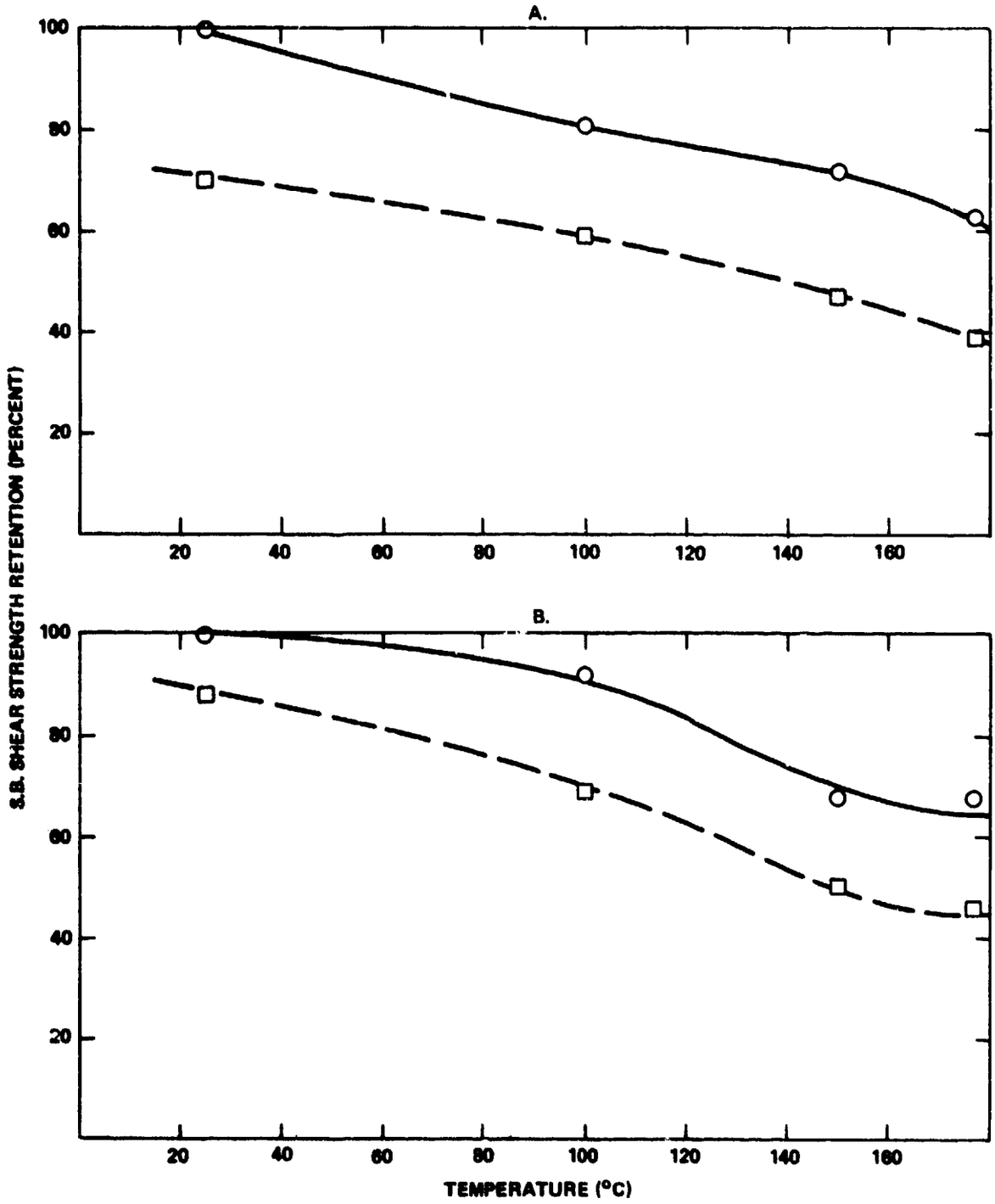

FIG. 19 PERCENT S.B. SHEAR STRENGTH RETENTION OF (A) EPON 1031/T300 EPOXY COMPOSITE, (B) EPON 1031/HMS EPOXY COMPOSITE BEFORE AND AFTER HUMIDITY EXPOSURE

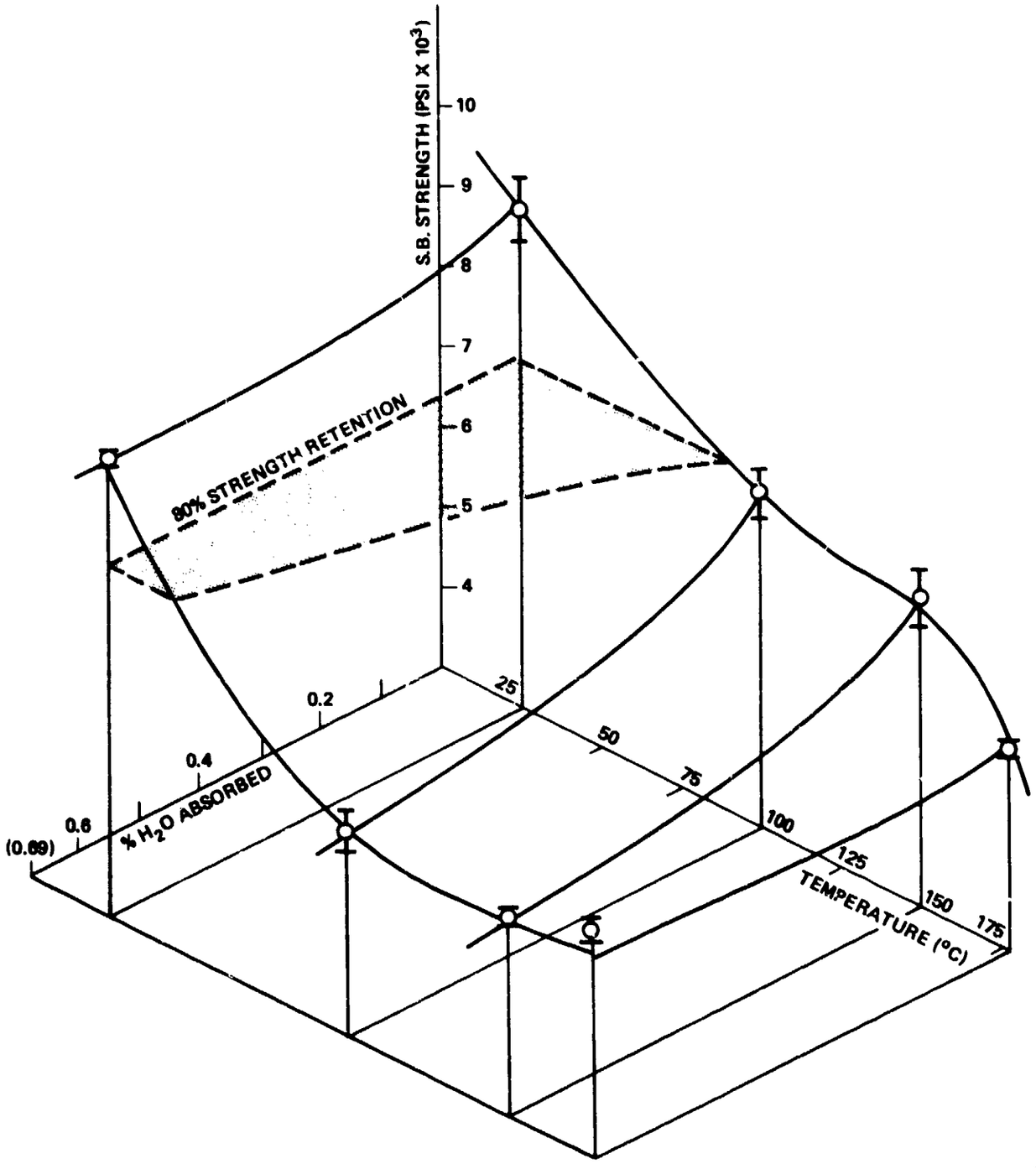

FIG. 20 SHORT BEAM SHEAR STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (NARMCO 5208/T300) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED.

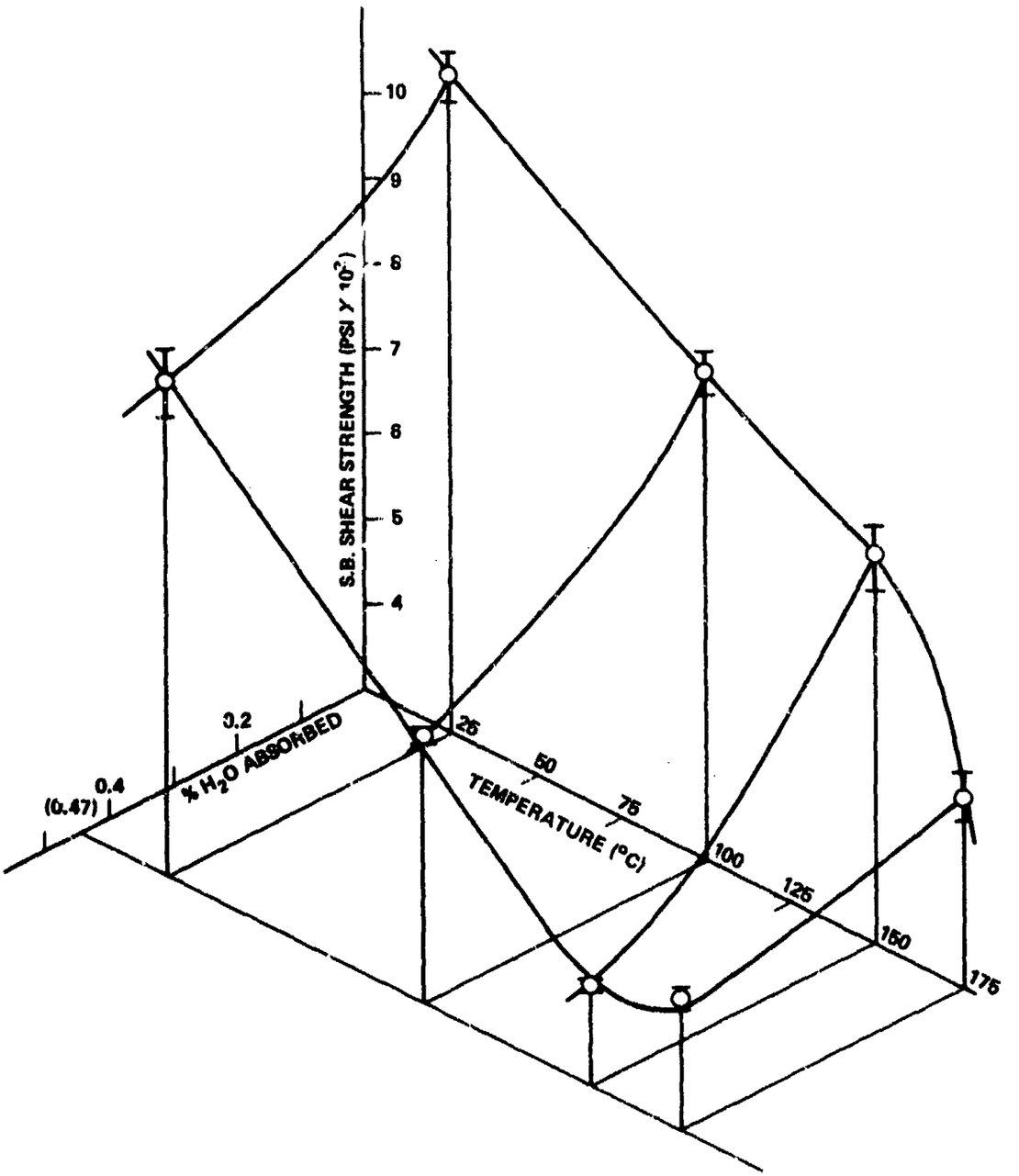

FIG. 21 SHORT BEAM SHEAR STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (DER 332 DADS/T300) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED.

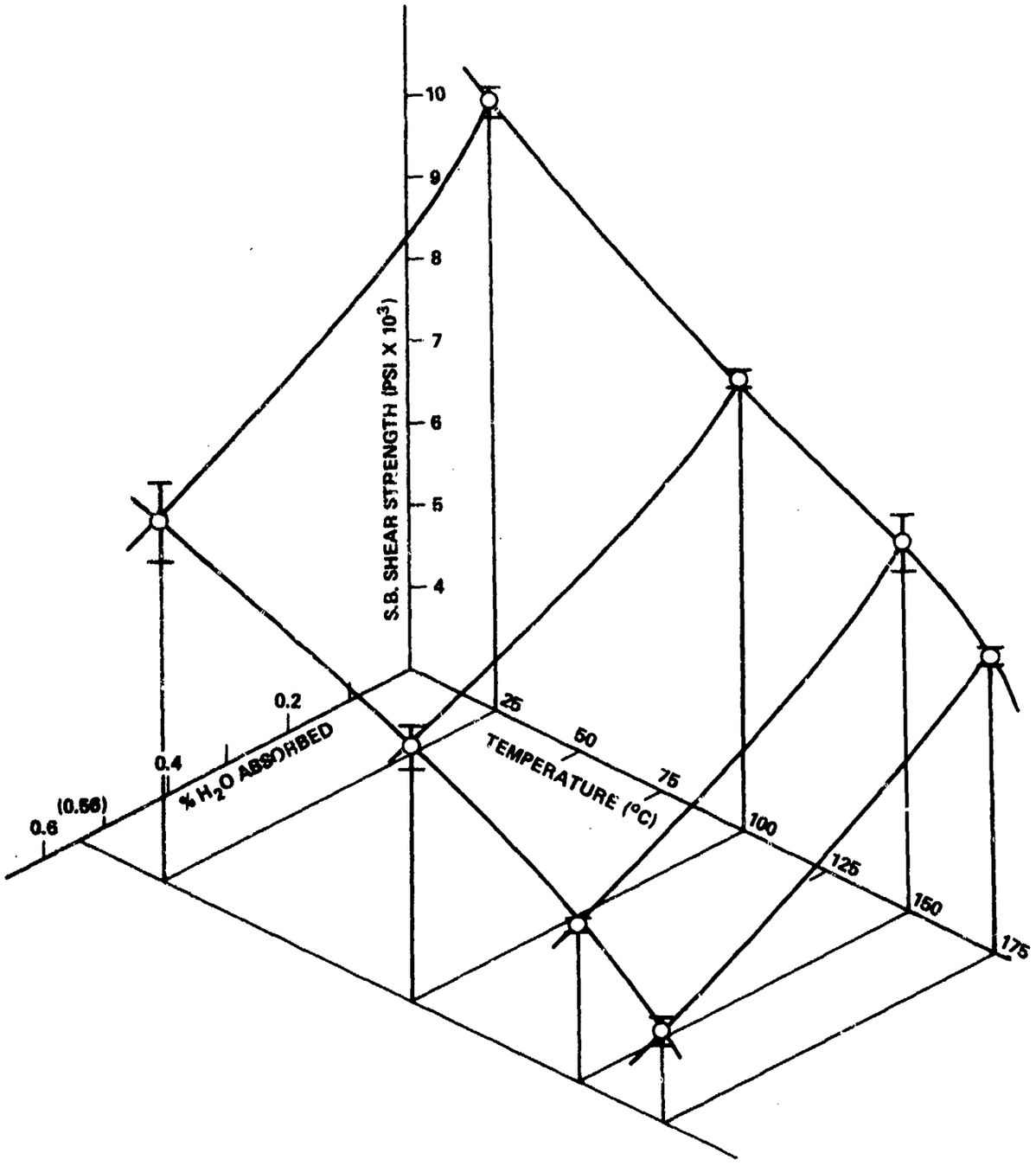

FIG. 22 SHORT BEAM SHEAR STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (EPON 1031 NMA/T300) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED.

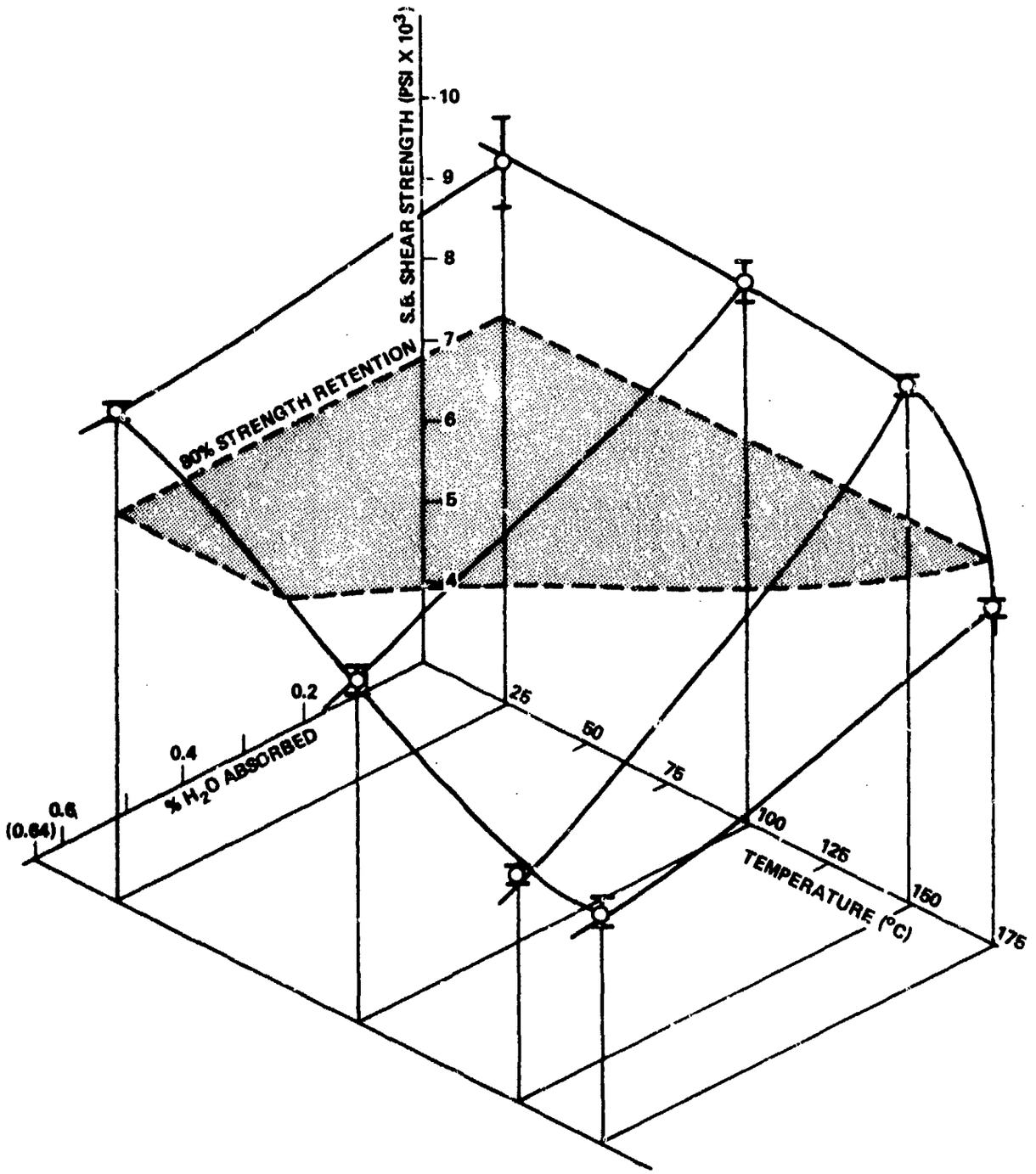

FIG. 23 SHORT BEAM SHEAR STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (NARMCO 5208/HMS) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED.

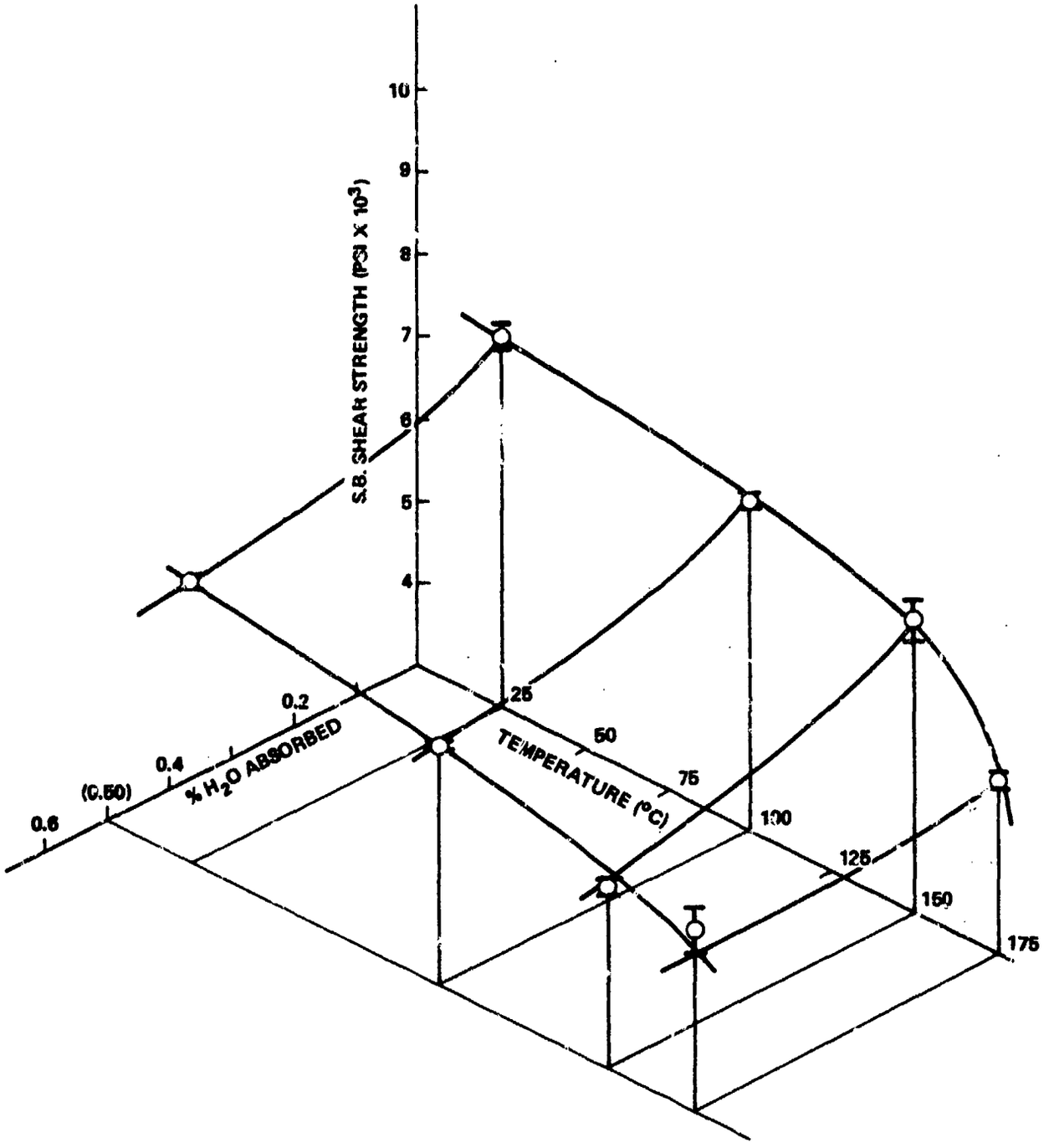

FIG. 24 SHORT BEAM SHEAR STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (DER 332 DADS/HMS) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED.

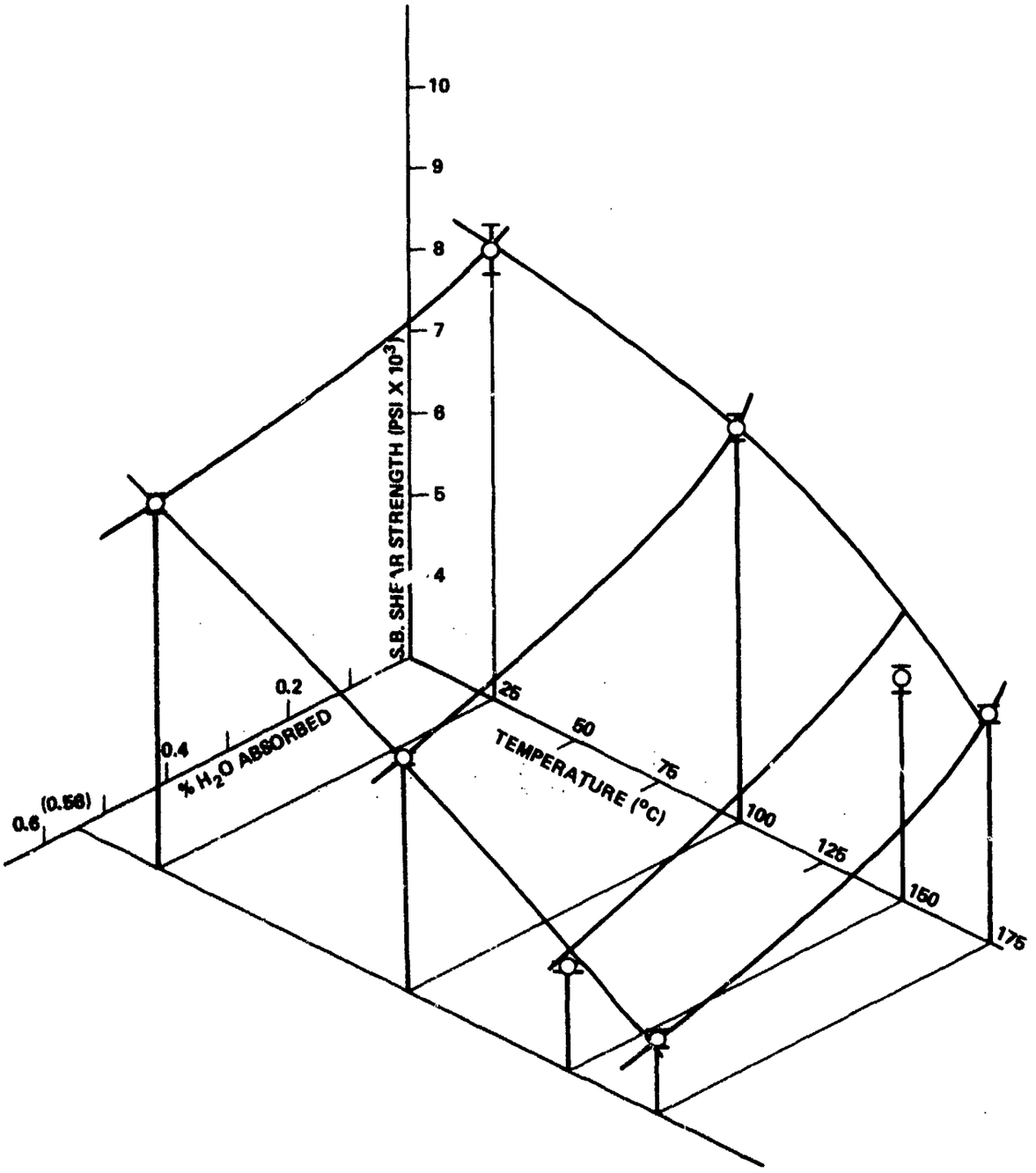

FIG. 25 SHORT BEAM SHEAR STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (EPON 1031 NMA/HMS) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED.

SHORT BEAM SHEAR STRENGTH OF NARMCO 5208/HMS COMPOSITE, TESTED AT 125° C AFTER EXPOSURE TO 80% RH AT 75° C

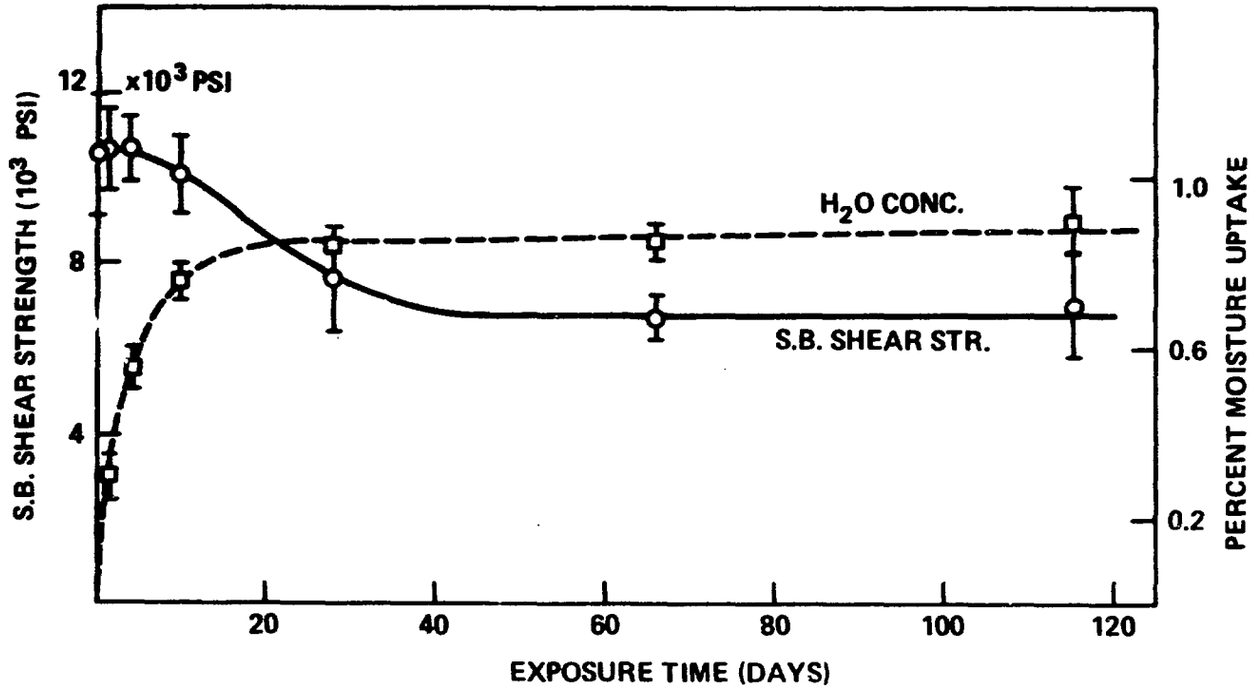

FIG. 26 SHORT BEAM SHEAR STRENGTH OF NARMCO 5208/HMS COMPOSITE, TESTED AT 125° C AFTER EXPOSURE TO 80% RH AT 75° C

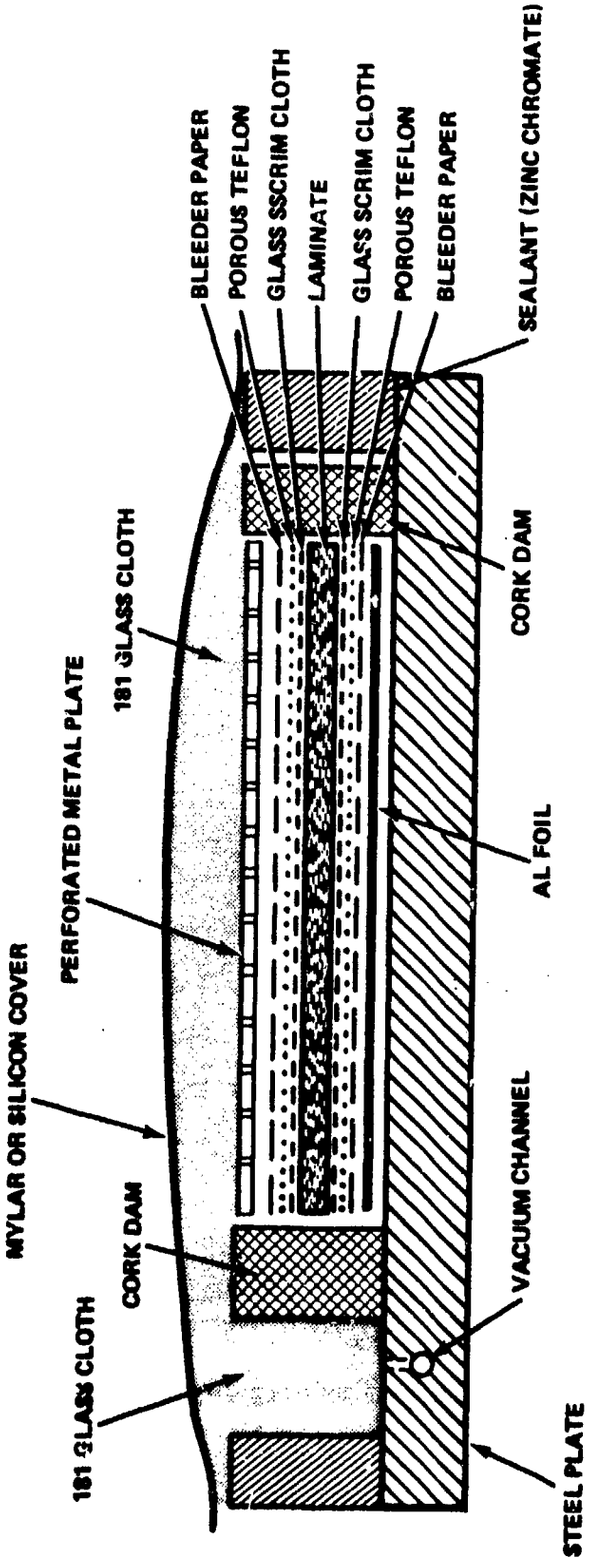

FIG. 27 LAY-UP FOR THE COMPOSITE PREPARATION BY APPLICATION OF VACUUM AND EXTERNAL PRESSURE

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