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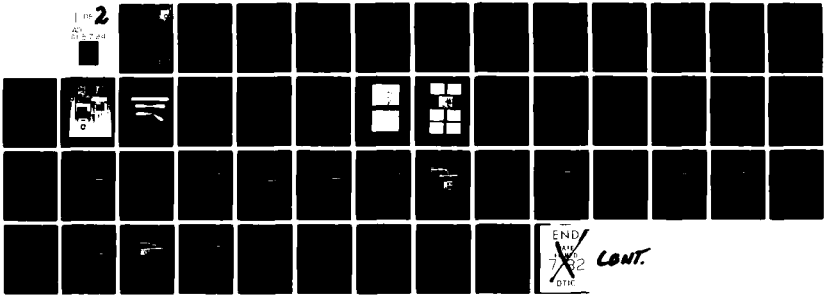

CONT.

AFWAL-TR-81-4129

EVALUATION OF REINFORCED PLASTIC RADOME MATERIALS

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NOVEMBER 1981

INTERIM TECHNICAL REPORT FOR PERIOD AUGUST 1980 - JUNE 1981

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AIR FORCE SYSTEMS COMMAND
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Mechanical Properties	Fiberglass	Thermal Conductivity	
Physical Properties	Epoxy	TGA	
Thermophysical Properties	Polyimide	Tension	
		Flexure	

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A program was conducted to measure mechanical, physical, and thermophysical properties of fiber reinforced composite radome materials in support of the Joint Army, Navy, Air Force (JANAF) laser study. Over half of the materials were found to contain voids distributed through their interiors. One was found to contain numerous microcracks. No conclusions are drawn since the objective of the effort was simply to obtain and report the data.

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PREFACE

This report covers work performed during the period from August 1980 to June 1981 under Air Force Contract F33615-80-C-5011, Project 7381. The work was administered under the direction of the Systems Support Division of the Air Force Materials Laboratory, Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, Ohio. Mr. John Rhodehamel (AFWAL/MLSE) was the Program Project Engineer.

The principal investigator on this program was James McKiernan. The major portion of the laboratory work was conducted by Messrs. Dee Pike (composite physical properties, thermal conductivity, TGA), Don Byrge (mechanical properties), Dan Miller (specific heat, heat of pyrolysis), Jim McKiernan (laminate fabrication), and Mrs. Theresa Elmore (data compilation and collation).

This report was submitted by the authors in August 1981. The contractor's report number is UDR-TR-81-75.

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SECTION 1
INTRODUCTION AND BACKGROUND

The extensive use of on-board radars in both aircraft and missiles requires an equally extensive knowledge of the capabilities and limitations of the materials and designs used in the construction of radomes. The armed services have collaborated to fabricate and evaluate these materials. The program described in this report was conducted by the University of Dayton Research Institute (UDRI) to provide materials characterization data required for radomes being evaluated under the JANAF (Joint Army, Navy, Air Force) laser study.

SECTION 2
APPROACH

The objective of this program was to determine mechanical, physical, and thermophysical properties of various types of glass reinforced composite materials used in radome construction.

2.1 MATERIALS

The Air Force Weapons Laboratory (AFWL) supplied UDRI with a series of glass reinforced composites that were representative of both flat and curved radome materials. The materials were:

(1) Cordopreg (flat)
(2) Cordopreg (curved)
(3) EG/Epoxy Type I
(4) EG/Epoxy Type II
(5) SG/Epoxy Type I
(6) SG/Epoxy Type II
(7) Quartz/Polyimide (curved)
(8) Stypole (curved)

In addition to these eight materials, UDRI also fabricated eight flat laminates of Quartz 581/Polyimide (PMR 15). Five of these laminates were shipped to AFWL for further evaluation. The remaining three were used for this program. Fabrication procedures for the Quartz 581/Polyimide are described in Appendix A.

2.2 SPECIMEN PREPARATION AND TEST METHODS

Two physical properties of each type of composite material were measured; specific gravity and laminate resin content. Three types of thermophysical property measurements were conducted on the subject materials; specific heat, thermal conductivity, and thermogravimetric analysis (TGA). One other, heat of pyrolysis, was desired. A suitable method for determining this property was not available. Appendix B discusses some of the problems in measuring heat of pyrolysis. Two types of mechanical property tests were conducted; tensile and flexure. The test matrix followed for the characterization of composite materials is listed in Table 1.

TABLE 1

MATERIAL CHARACTERIZATION TEST MATRIX

Material	Specific Gravity	Resin Content	Specific Heat	Thermal Conductivity	Thermal Gravimetric Analysis (TGA)	Heat of Pyrolysis	Tensile Properties	Flexural Properties
Cordopreg	✓	✓	✓	✓	✓	✓	✓	✓
Cordopreg (curved)	✓	✓	✓	*	✓	✓	*	*
EG/Epoxy Type I	✓	✓	✓	✓	✓	✓	✓	✓
EG/Epoxy Type II	✓	✓	✓	✓	✓	✓	✓	✓
SG/Epoxy Type I	✓	✓	✓	✓	✓	✓	✓	✓
SG/Epoxy Type II	✓	✓	✓	✓	✓	✓	✓	✓
Stypole (curved)	✓	✓	✓	*	✓	✓	*	*
Quartz/PI (curved)	✓	✓	✓	*	✓	✓	*	*
Quartz/PI (UDRI)	✓	✓	✓	✓	✓	✓	✓	✓

* Only determined on flat composite materials.

2.2.1 Laminate Physical Properties

The specific gravity of the laminates was measured in accordance with ASTM D792, Specific Gravity and Density of Plastics by Displacement, Method A-1.

The method employed to determine resin content was through ignition weight loss. The procedure used is in accordance with ASTM D2584, Ignition Loss of Cured Reinforced Resins.

2.2.2 Specific Heat

The samples were microtomed into thin pieces from which approximately 5 mg. samples were taken. Care was taken to assure that the samples tested had the same homogeneity as the overall composite. The specific heat was determined using a differential scanning calorimeter (DSC). This technique compares the rate of heat input required to maintain a constant rate of temperature rise in an unknown sample to that required to maintain the same rate of temperature rise in a known reference material. A Perkin-Elmer, Model DSC-2 instrument was used for these determinations. The specific heat of the composites was measured over the range of 100°C (212°F) to 300°C (572°F) in a nitrogen atmosphere, with a scanning rate of 10°C (18°F)/minute.

2.2.3 Thermal Conductivity

A thermal conductivity sample 2.5 inches (6.35 cm) square was machined from each flat composite panel. The technique used to determine the sample thermal conductivity is to sandwich the sample between two reference materials of known conductivity (Figure 1). These, in turn, are held firmly between a heater and a heat sink. The heat flux through this stack establishes a temperature gradient which is measured with thermocouples placed on the upper and lower surfaces of both reference plates and the specimen plate in small precisely machined grooves. Radial heat flow to and from the test stack

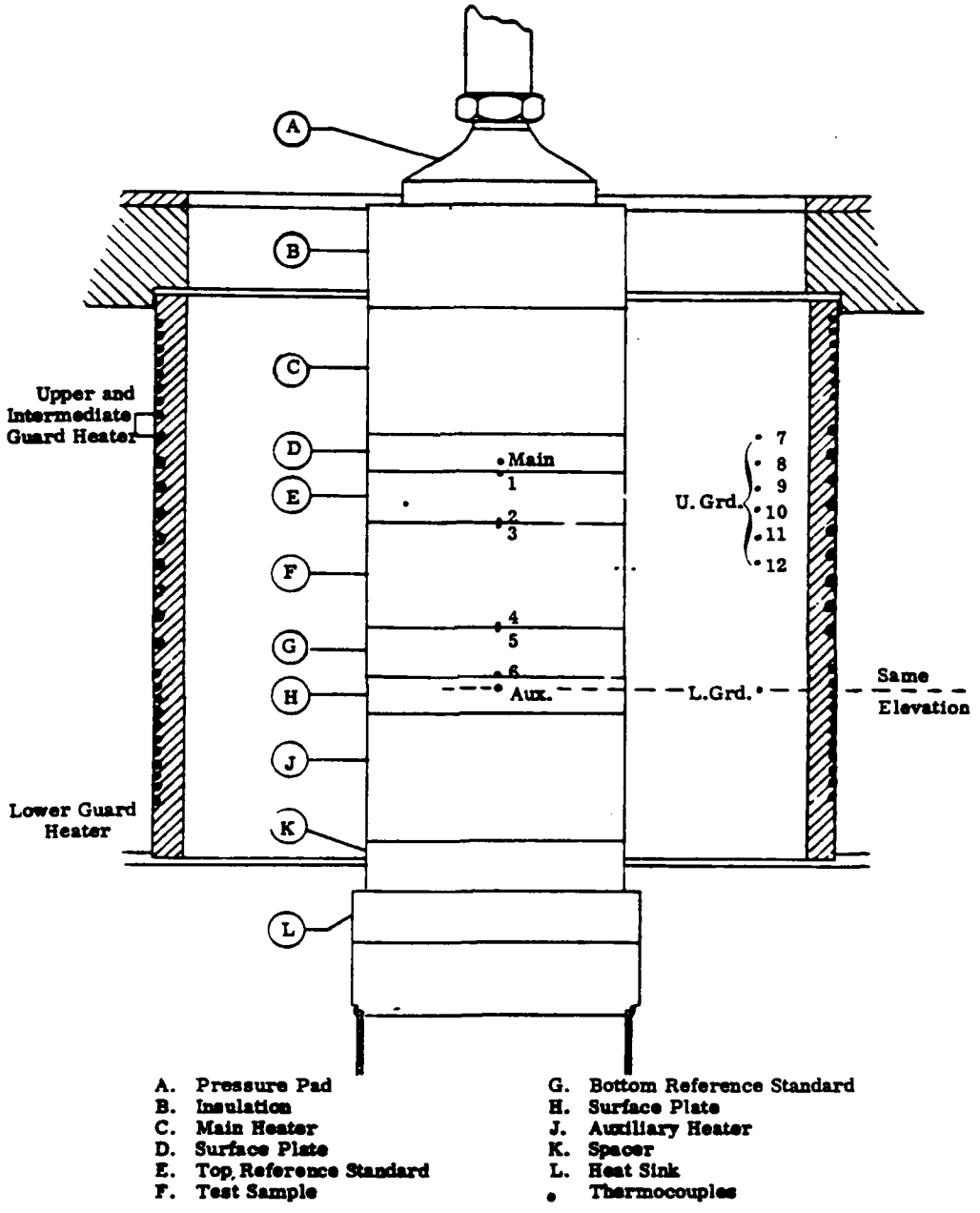


Figure 1. Cross-Sectional View of Thermal Conductivity Apparatus (from Dynatech Operating Manual).

is minimized with a cylindrical guard heater in which a linear temperature gradient, closely matching that of the test stack, is maintained. A Dynatech Model TCFCM-N20 thermal conductivity instrument was used for these measurements. Data points were taken at approximately equal temperature intervals over the range of interest and a "best-fit" curve (quadratic) was plotted through these data points.

2.2.4 Thermal Gravimetric Analysis

Thermal gravimetric analysis (TGA) required a specimen of 0.5 to 0.8 mg. in weight. Figure 2 shows the Chevenard TGA instrument used in this evaluation. A sample, of known initial weight, is suspended in a balance pan inside a furnace. The temperature is increased at a constant rate of 3°C (5.4°F)/min., and the weight is continuously recorded. As the temperature increases, the samples thermally degrade. Weight versus temperature graphs were obtained in both air and nitrogen atmospheres for each of these materials. The temperature at which the weight starts decreasing rapidly indicates the onset of material degradation.

2.2.5 Mechanical Properties

The materials were tested for tensile and flexural strength and modulus at room temperature and 350°F (177°C) for the epoxy resin systems. The polyimide resin system was tested at room temperature, 350°F (177°C), and 600°F (316°C).

2.2.5.1 Tensile Properties

Tensile specimens were machined into 0.5 inch (1.27 cm) wide by 4.5 inch (11.43 cm) long rectangular bars. A reduced section (Figure 3) 0.25 inch (0.63 cm) wide and 0.63 inch (1.6 cm) long was machined into these bars. The fabric warp direction was in the length direction of the specimens. The tests were conducted in accordance with ASTM D638, Tensile Properties of Plastics.

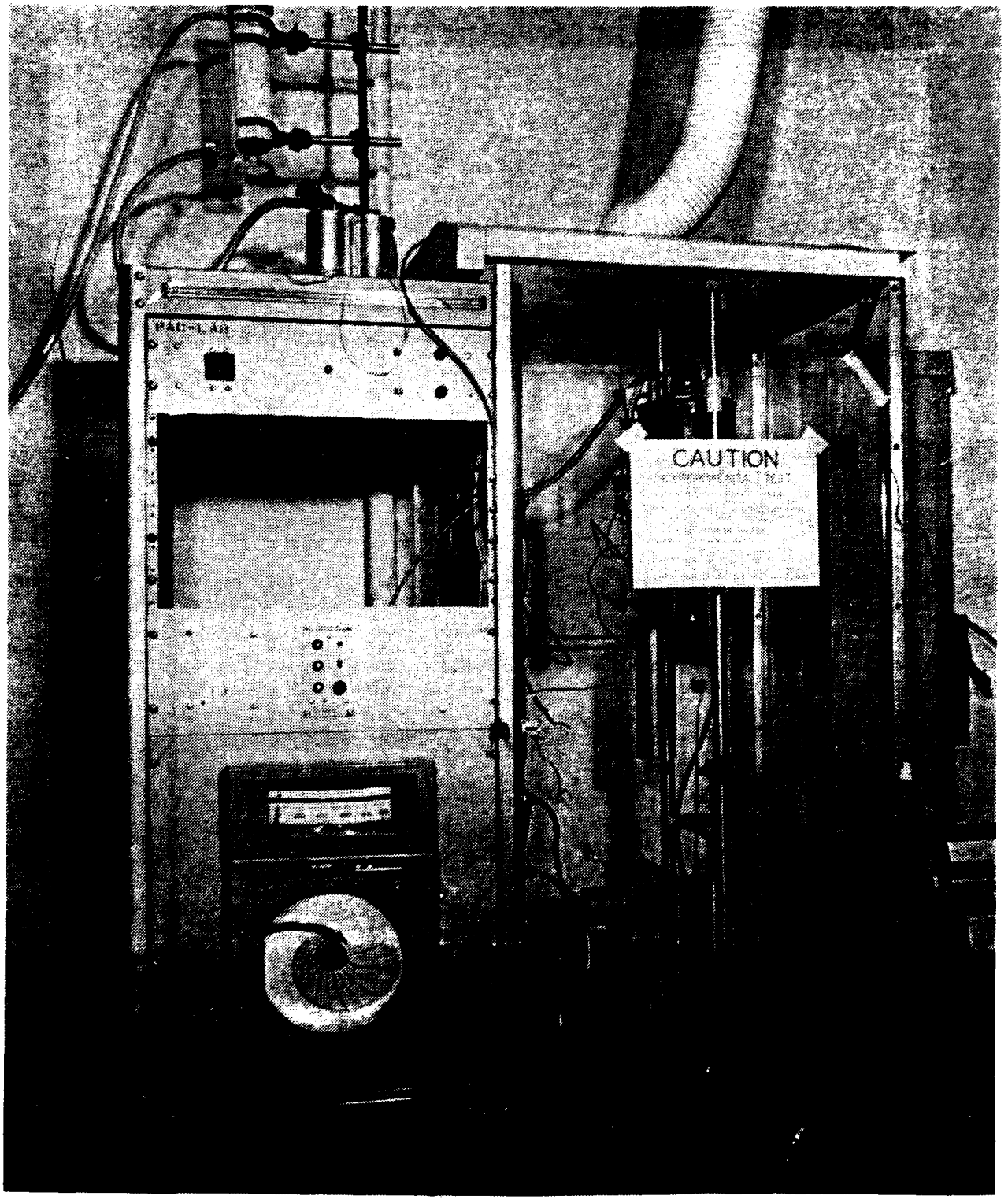


Figure 2. Chevenard Thermal Gravimetric Analyzer.

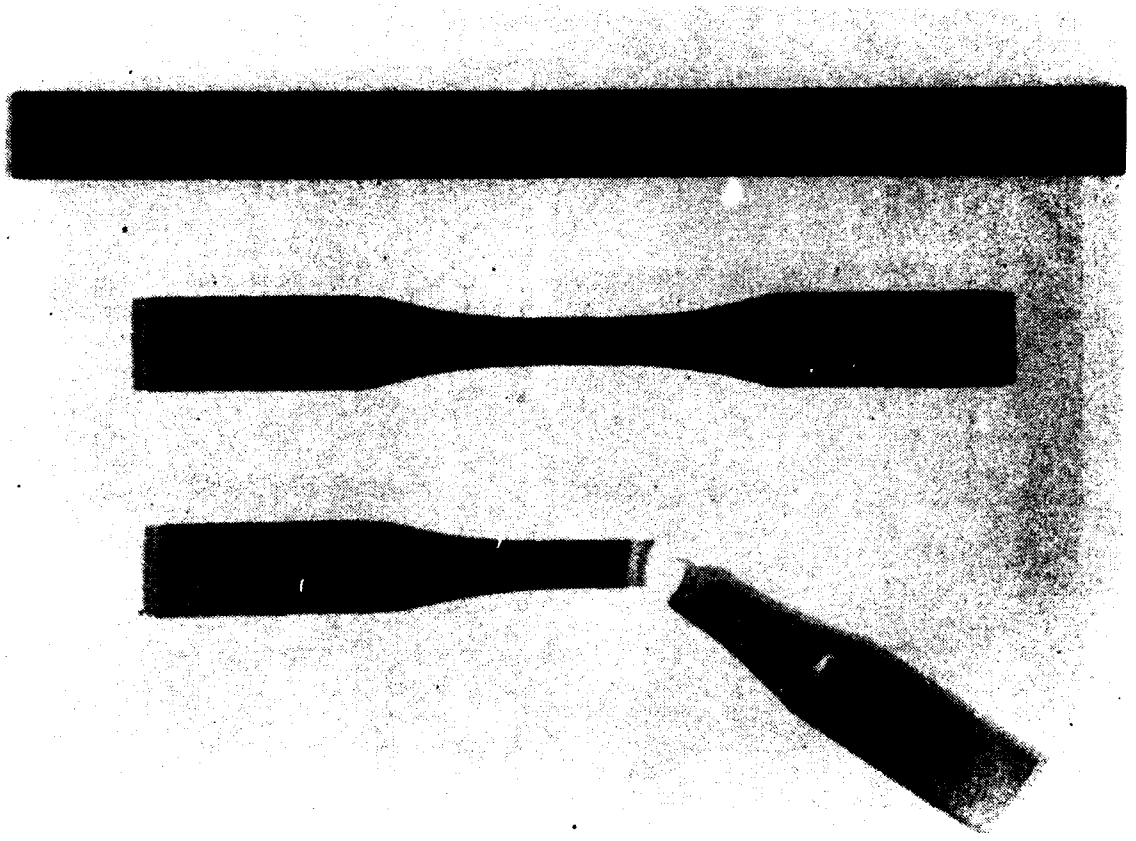


Figure 3. Tensile Specimens (Before and After Testing).

2.2.5.2 Flexural Properties

Flexure specimens were machined into 0.5 inch (1.27 cm) wide by 2.5 inch (6.35 cm) long rectangular bars. The fabric warp direction was in the lengthwise direction of the test specimens. The tests were conducted in accordance with ASTM D790, Flexural Properties of Plastics and Electrical Insulating Materials, Method I (three point loading) utilizing a 16 to 1 span to depth ratio.

SECTION 3
DISCUSSION OF RESULTS

3.1 LAMINATE PHYSICAL PROPERTIES

Table 2 presents the specific gravity and resin content values obtained on the various reinforced composite materials used in this program. There was a substantial difference in the specific gravity of the AFWL supplied curved polyimide/quartz panel and that of the UDRI fabricated flat polyimide/quartz laminate. Photomicrographs were obtained on these two laminates and are presented in Figure 4. It is clear from the photomicrographs that the AFWL supplied curved panel was of very poor quality and has a very high void content while the UDRI fabricated flat panel was essentially void-free.

Figure 5 presents 150X photomicrographs of the seven materials not illustrated in Figure 4 which were tested in this program. The following observations can be made from these photographs.

(a) Both the flat and curved cordopreg laminates were void-free and of apparent high quality. The white areas in the photos probably indicate the presence of some sort of particulate filler.

(b) The stypole curved laminate was void-free but contained numerous cracks.

(c) The SG/epoxy laminates both contained some voids, with the Type II material having numerous small voids uniformly distributed throughout, and the Type I material having fewer voids. In both cases the porosity is within the fiber bundles rather than being concentrated between the plies. This would indicate that the most probable source of the porosity is the release and entrapment of a volatile resin constituent during processing.

(d) The EG/epoxy laminates both exhibit some porosity also, although the Type II material is very nearly void-free.

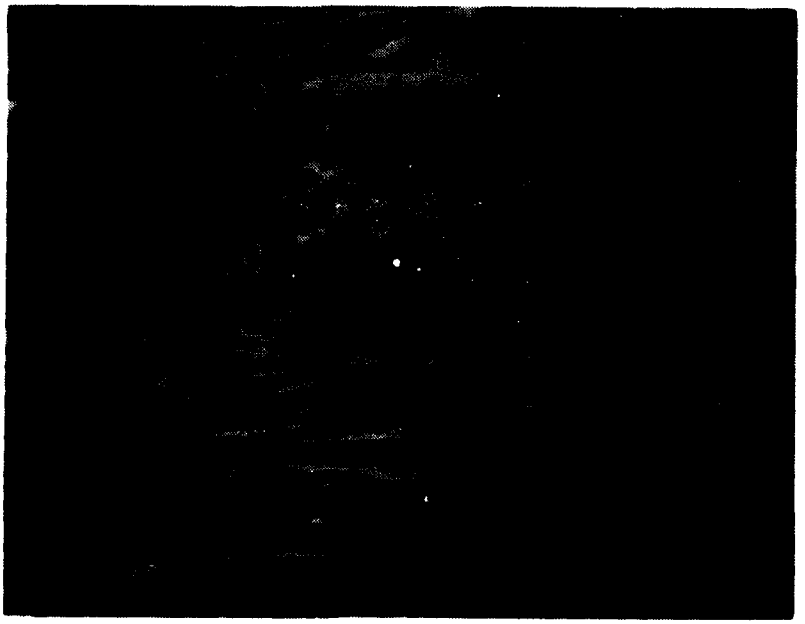
TABLE 2

PHYSICAL PROPERTIES[1] OF JANAF LAMINATES

Materials	Specific Gravity	% Resin Content by Wt.
Cordo (flat)	1.97	34.15
Cordo (curved)	1.93	34.93
EG/Epoxy Type 1	1.83	36.48
EG/Epoxy Type 2	1.94	31.86
SG/Epoxy Type 1	1.90	31.93
SG/Epoxy Type 2	1.90	29.38
Stypole	2.00	33.95
Polyimide/Quartz (curved)	1.60	26.65
Quartz 581/Polyimide (flat - UDRI)	1.82	27.82

[1]Average of three specimens.

(a) AFWL Supplied Curved Panel, Specific Gravity = 1.60



(b) UDRI Fabricated Flat Panel, Specific Gravity = 1.82

Figure 4. Photomicrographs of Quartz/Polyimide Laminates.

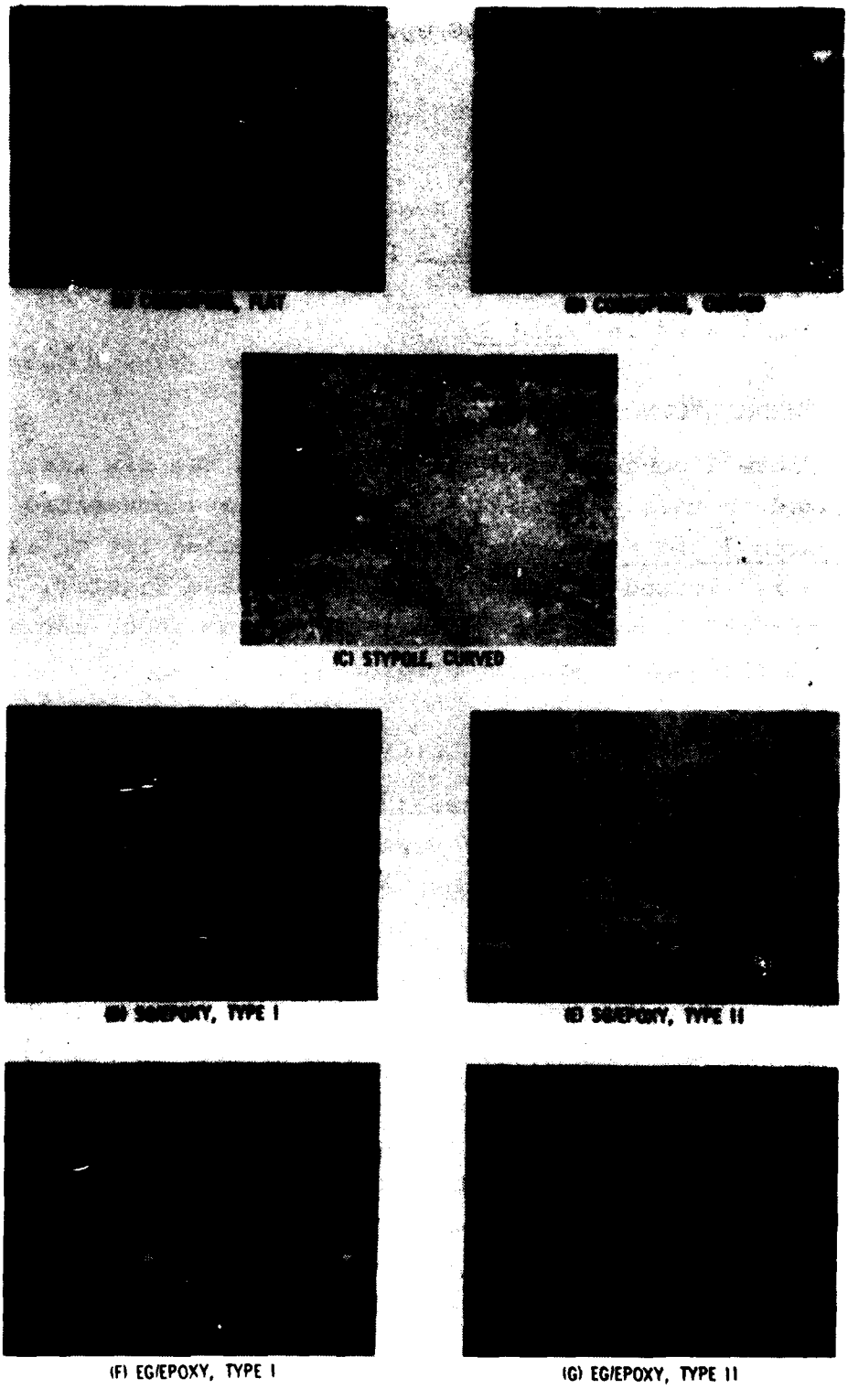


Figure 5. Laminate Photomicrographs.

The same observation regarding void location and probable source as was made for the SG/epoxy laminates would seem to apply to these as well.

3.2 SPECIFIC HEAT

The specific heat data measured for each material are presented in Table 3. Figure 6 presents a least-squares "best-fit" quadratic curve for each of the sets of specific heat data listed in Table 3.

3.3 THERMAL CONDUCTIVITY

Thermal conductivity was measured on the six flat laminates evaluated in this program. These results are presented graphically in Figures 7 and 8. A quadratic least-squares fit of each set of data is plotted for each material in these figures. Table 4 lists thermal conductivity values taken from these curves for each material at various temperatures.

3.4 THERMAL GRAVIMETRIC ANALYSIS (TGA)

TGA curves were obtained on all nine materials evaluated in this program. The weight versus temperature graphs obtained in both air and nitrogen atmospheres are presented in Figures 9 through 26.

3.5 MECHANICAL PROPERTIES

Mechanical properties were obtained on the six flat laminates characterized in this investigation. These materials were tested for tensile and flexural strength and modulus at room temperature, 350°F (177°C) and 600°F (316°C), depending on the resin matrix in the laminates. The tensile and flexural properties of these materials are presented in Tables 5 and 6, respectively. Not only are there very marked differences between the various materials, the relative degree of degradation at elevated temperatures also varies quite significantly from material to material.

TABLE 3

SPECIFIC HEAT[1] OF JANAF LAMINATES

Temp. (°C)	Cordopreg (flat)	Cordo Precurved	EG/Epoxy Type 1	EG/Epoxy Type 2	SG/Epoxy Type 1	SG/Epoxy Type 2	Stypole	Quartz PI (curved)	Quartz 581 PI (UDRI)
100	0.199	0.217	0.207	0.300	0.234	0.221	0.157	0.123	0.234
125	0.228	0.255	0.209	0.275	0.234	0.224	0.151	0.129	0.242
150	0.262	0.269	0.196	0.307	0.238	0.223	0.155	0.138	0.256
175	0.275	0.279	0.225	0.315	0.243	0.234	0.168	0.145	0.261
200	0.290	0.297	0.234	0.326	0.251	0.239	0.167	0.151	0.270
225	0.303	0.312	0.234	0.329	0.258	0.247	0.170	0.165	0.275
250	0.321	0.331	0.232	0.335	0.249	0.251	0.177	0.158	0.279
275	0.322	0.334	0.256	0.337	0.263	0.253	0.178	0.186	0.284
300	0.331	0.340	0.266	0.348	0.271	0.254	0.201	0.198	0.289[2]

[1]Units are (cal/gm-°C). All tests were run in a nitrogen atmosphere.

[2]This data point was extrapolated.

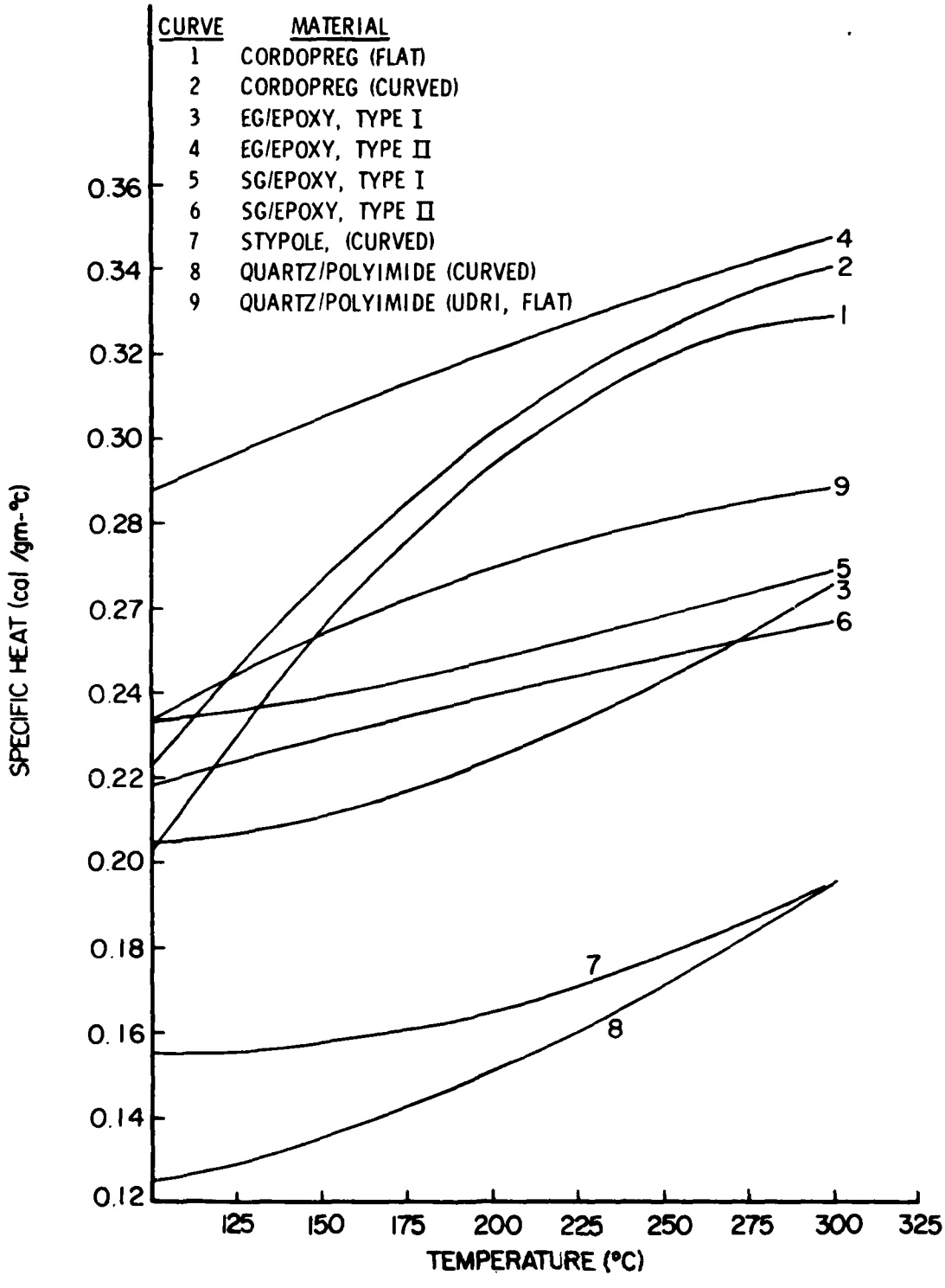


Figure 6. Specific Heat Results.

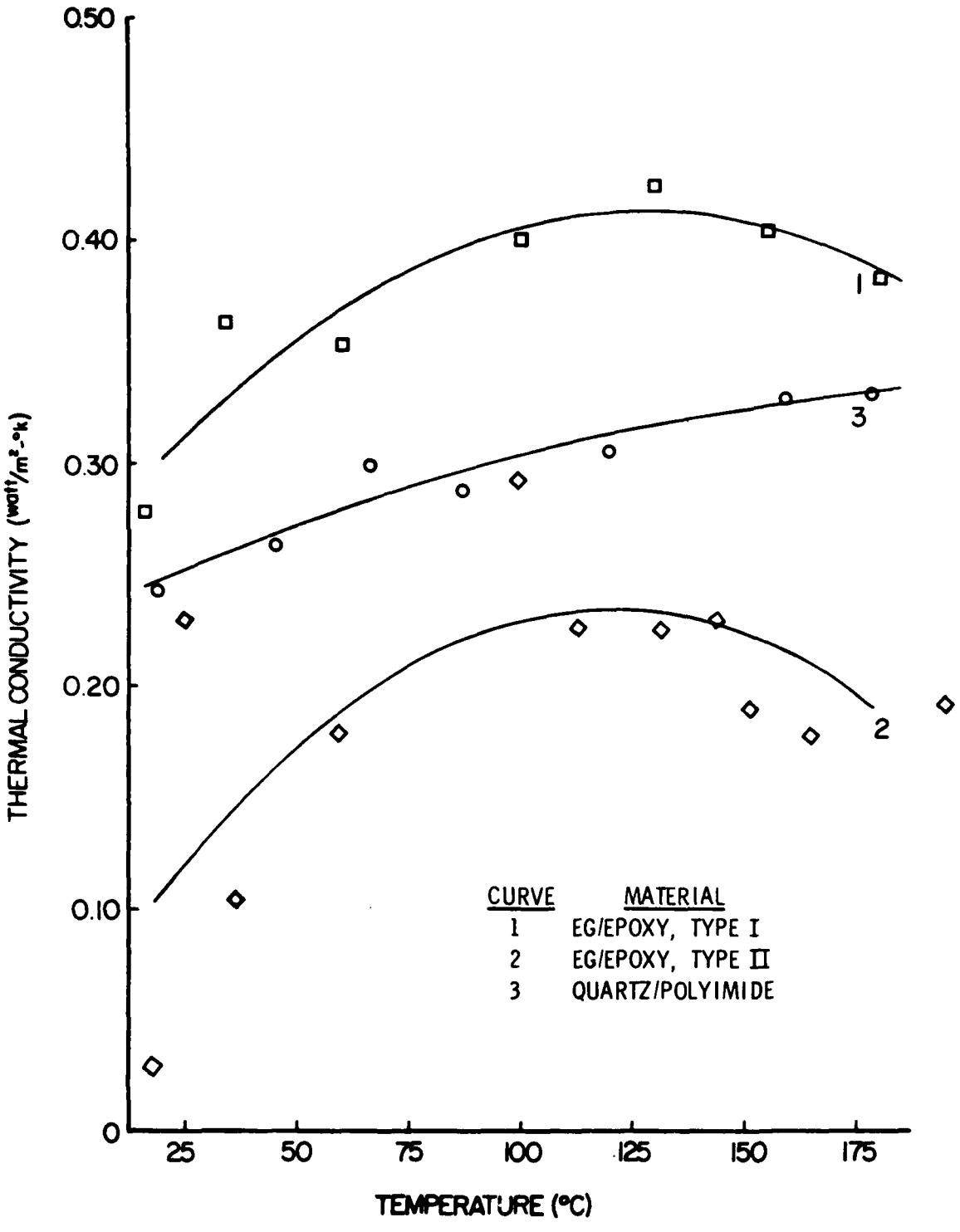


Figure 7. Thermal Conductivity Curves on EG/Epoxy Type 1 and 2 and Quartz 581/PMR-15 Laminates.

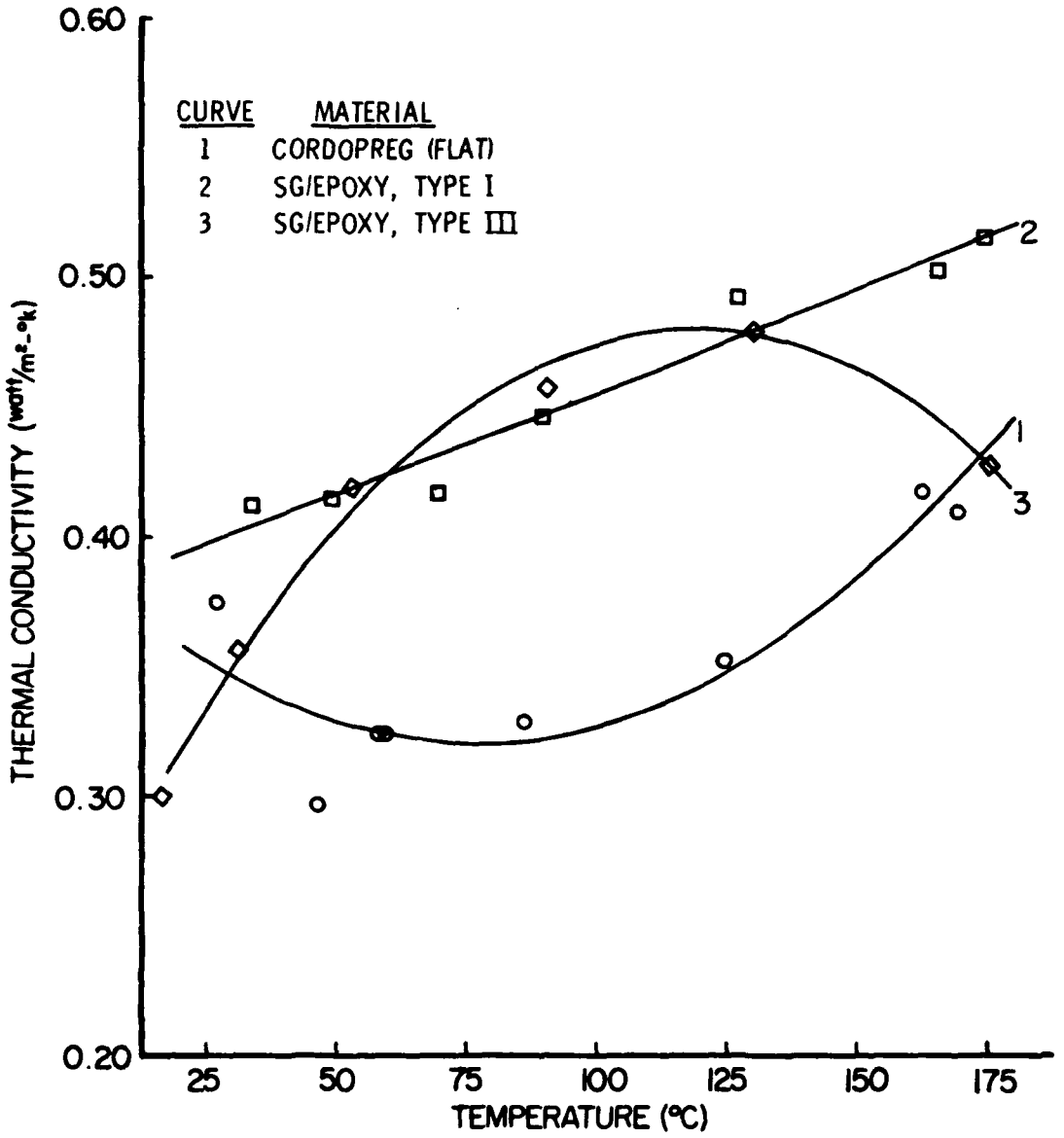


Figure 8. Thermal Conductivity Curves on SG/Epoxy Type 1 and 2 and Cordopreg Laminates.

TABLE 4

THERMAL CONDUCTIVITY OF LAMINATES TESTED
IN JANAF PROGRAM

Temperature (°F)	Temperature (°C)	Thermal Conductivity (w/m-°K) EG/Epoxy Type 1	EG/Epoxy Type 2	SG/Epoxy Type 1	SG/Epoxy Type 2	Cordopreg (flat)	Quartz/polyimide (UDRI)
77	25	0.312	0.120	0.397	0.334	0.351	0.252
122	50	0.355	0.172	0.416	0.401	0.328	0.271
167	75	0.385	0.209	0.435	0.448	0.319	0.289
212	100	0.405	0.229	0.455	0.474	0.326	0.303
257	125	0.412	0.234	0.474	0.479	0.347	0.315
302	150	0.408	0.223	0.495	0.464	0.383	0.324
347	175	0.393	0.197	0.515	0.428	0.433	0.330

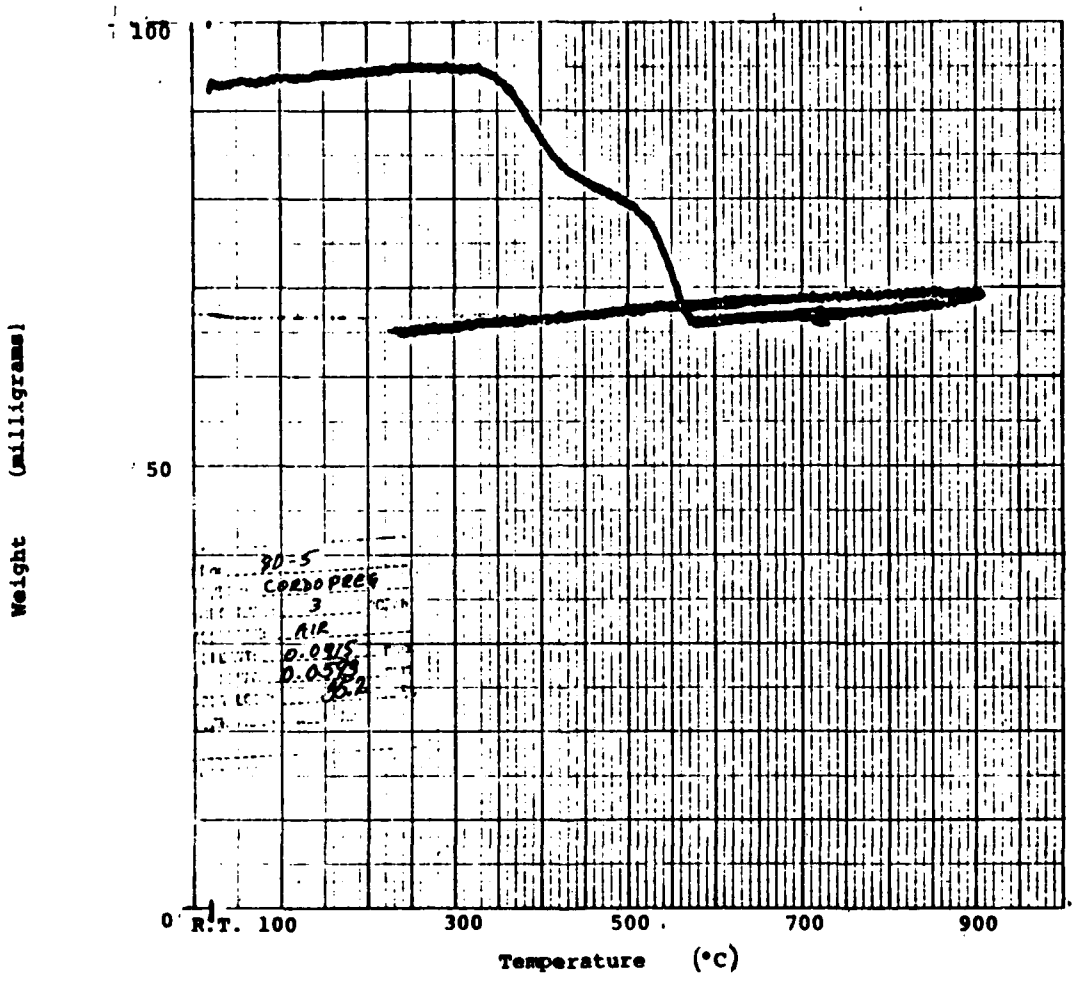


Figure 9. TGA in Air on Cordopreg (flat).

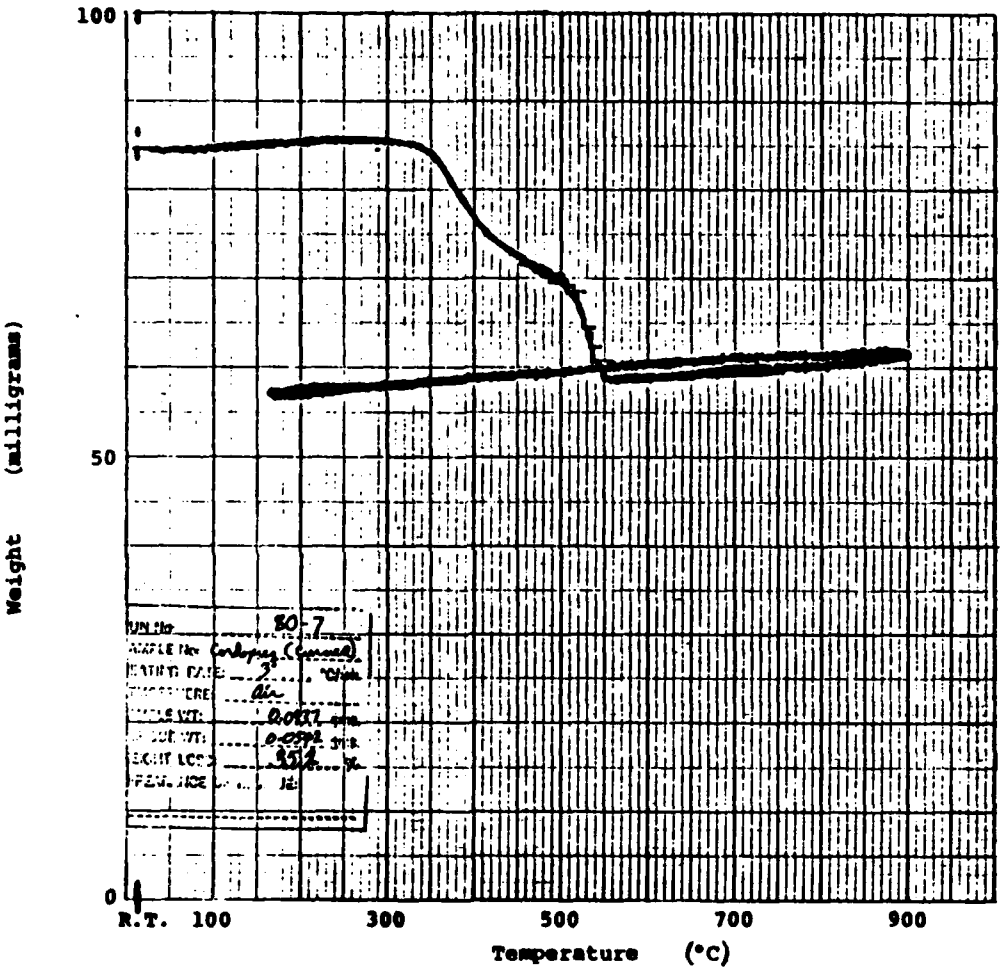


Figure 10. TGA in Air on Cordopreg (curved).

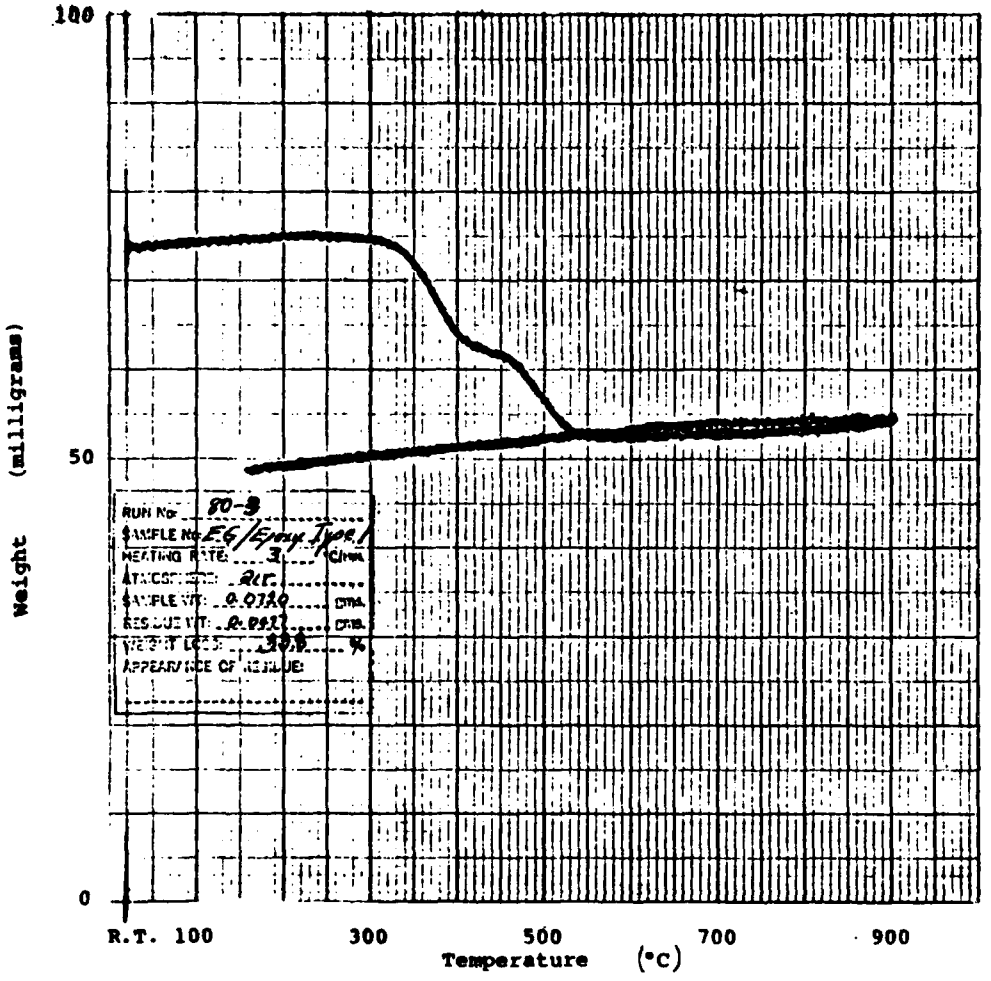


Figure 11. TGA in Air on EG/Epoxy Type 1.

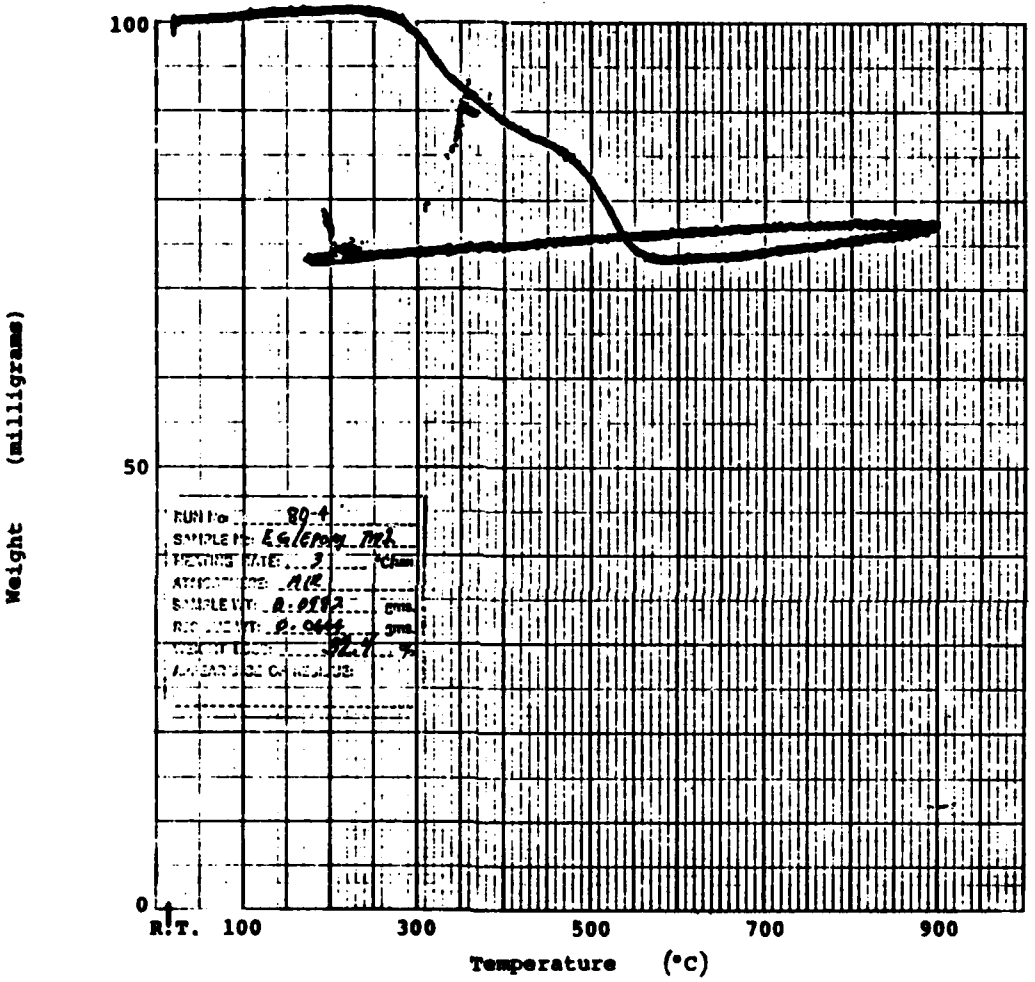


Figure 12. TGA in Air on EG/Epoxy Type 2.

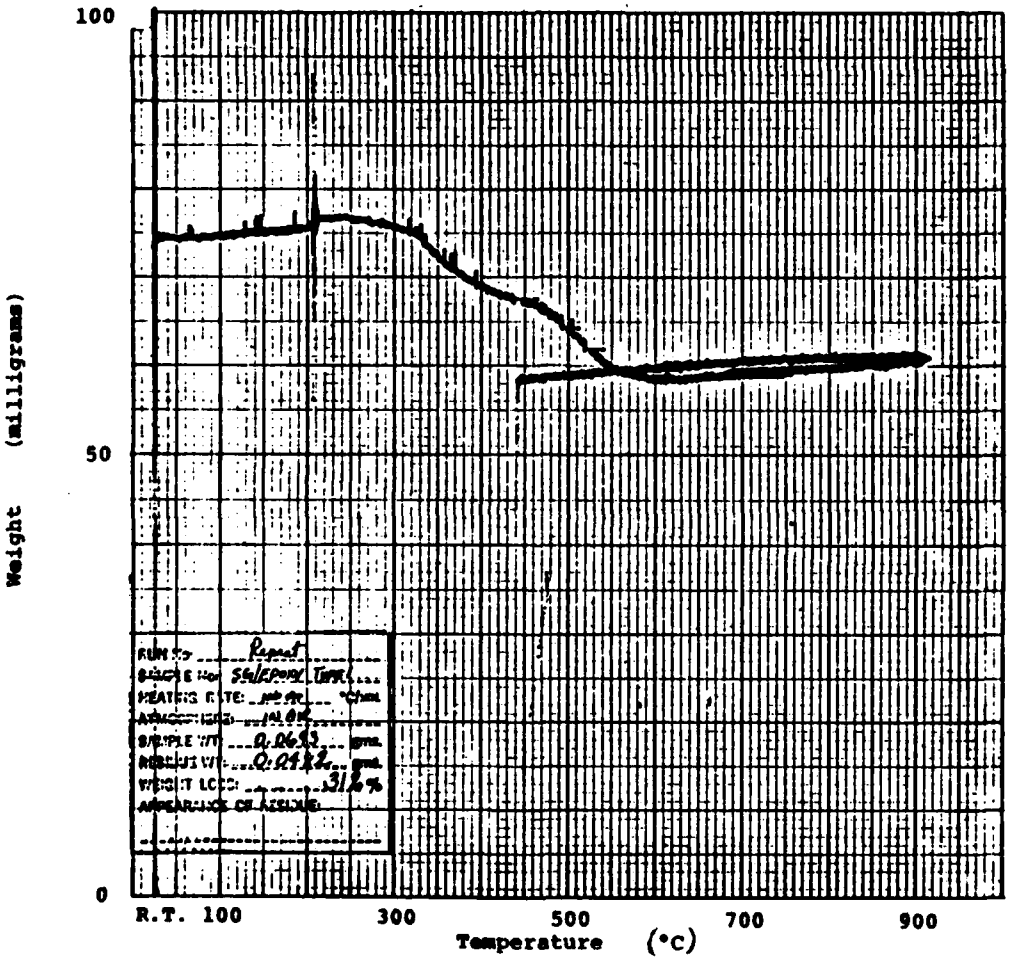


Figure 13. TGA in Air on SG/Epoxy Type 1.

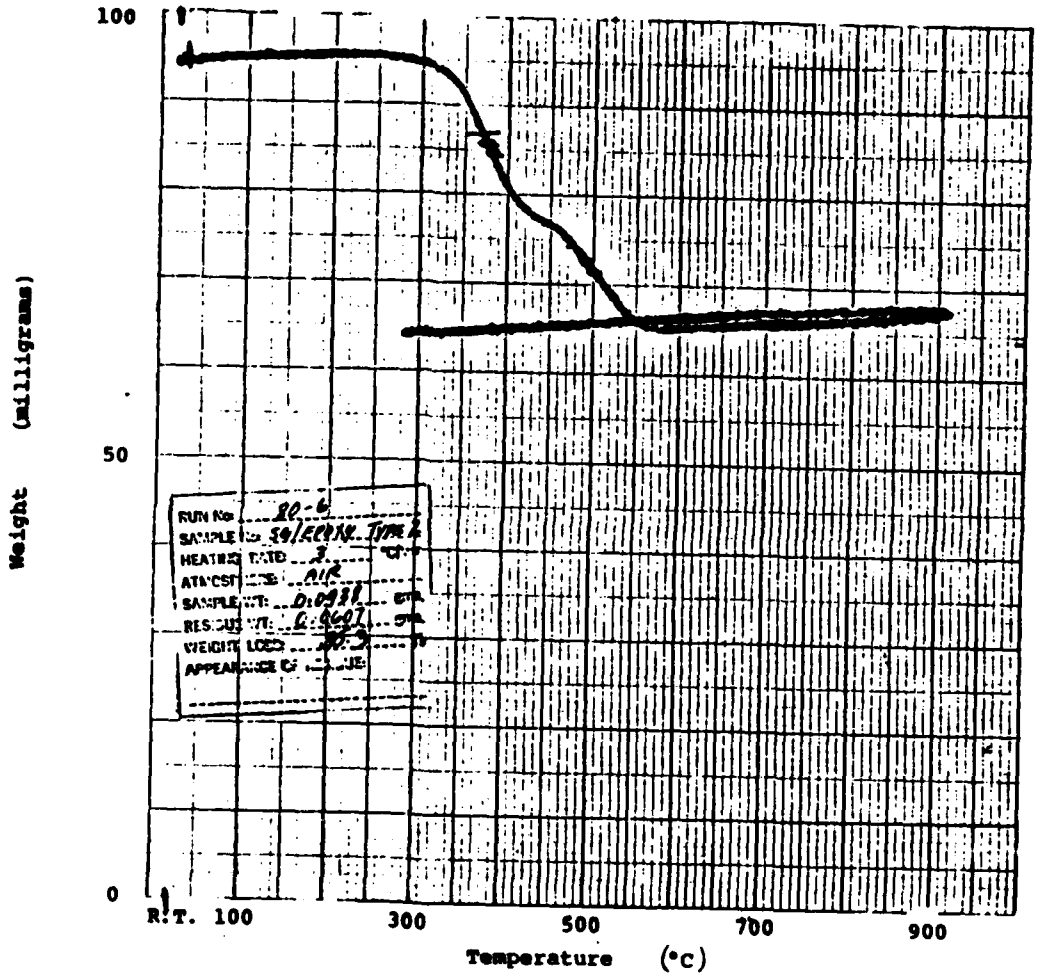


Figure 14. TGA in Air on SG/Epoxy Type 2.

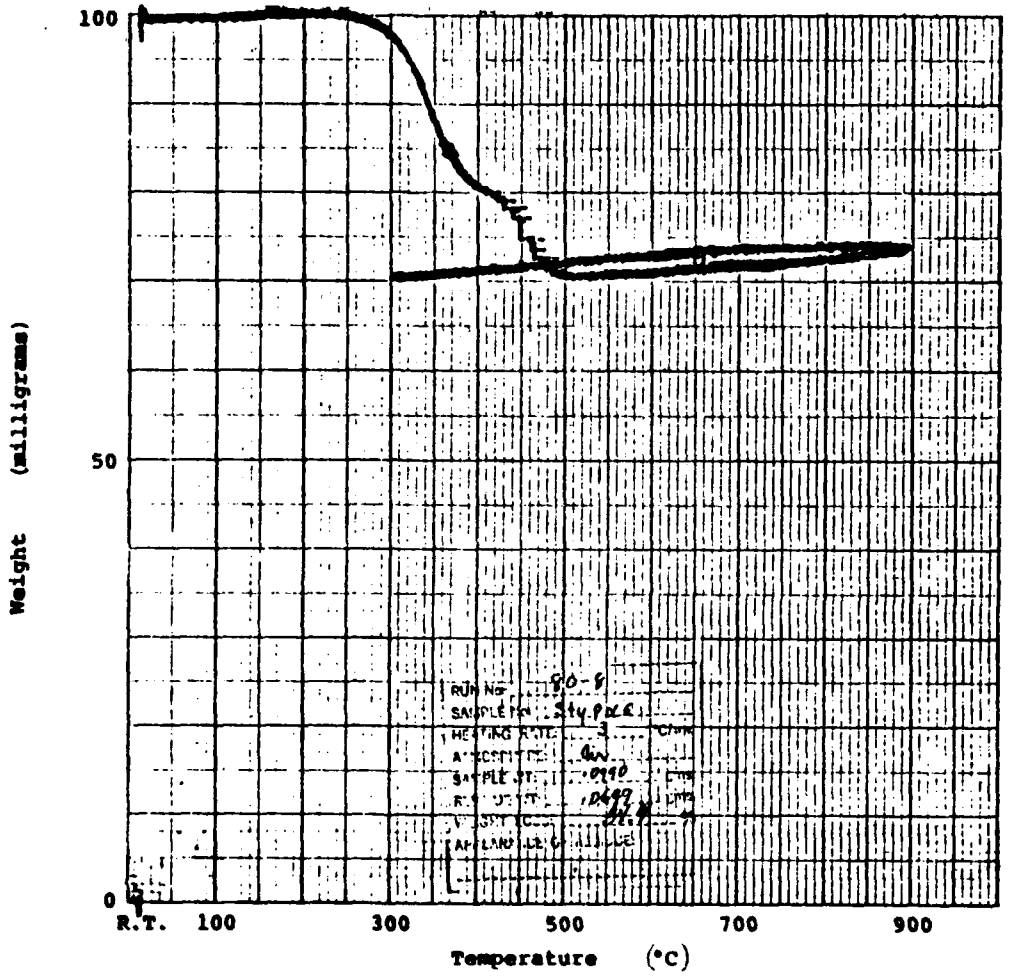


Figure 15. TGA in Air on Stypole (curved).

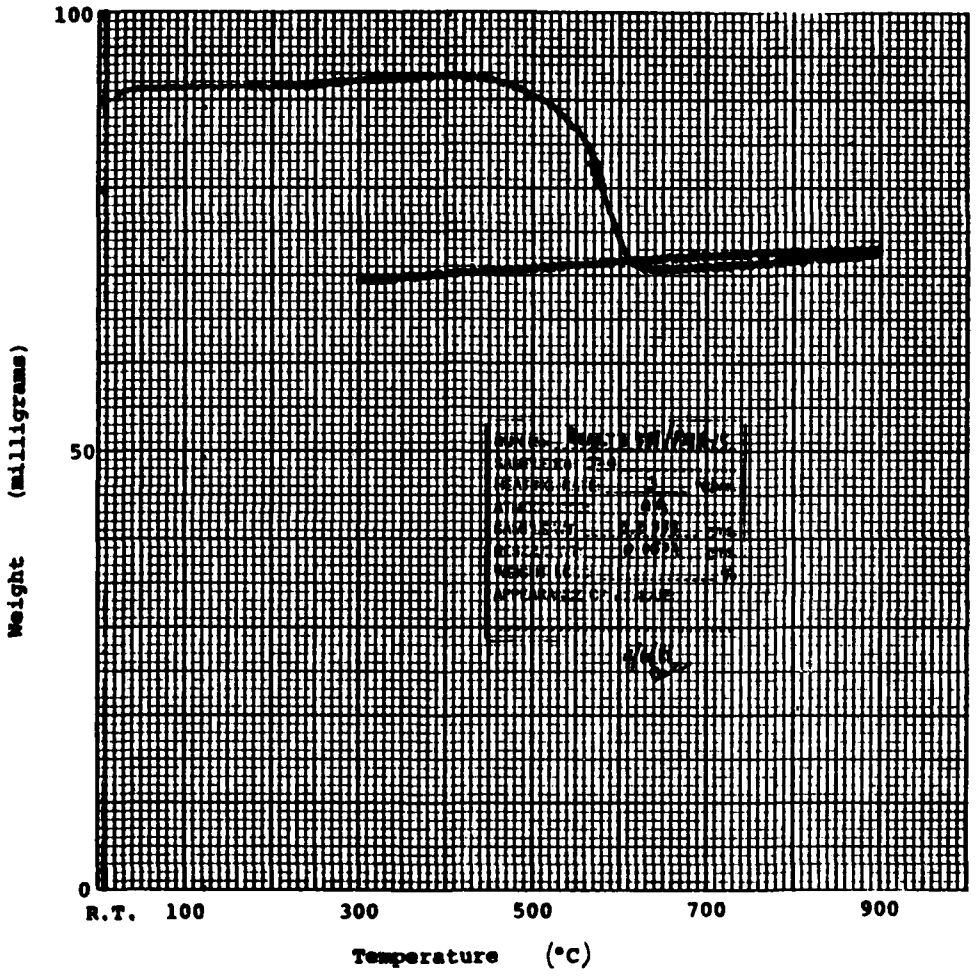


Figure 16. TGA in Air on Quartz 581/PMR-15 (flat).

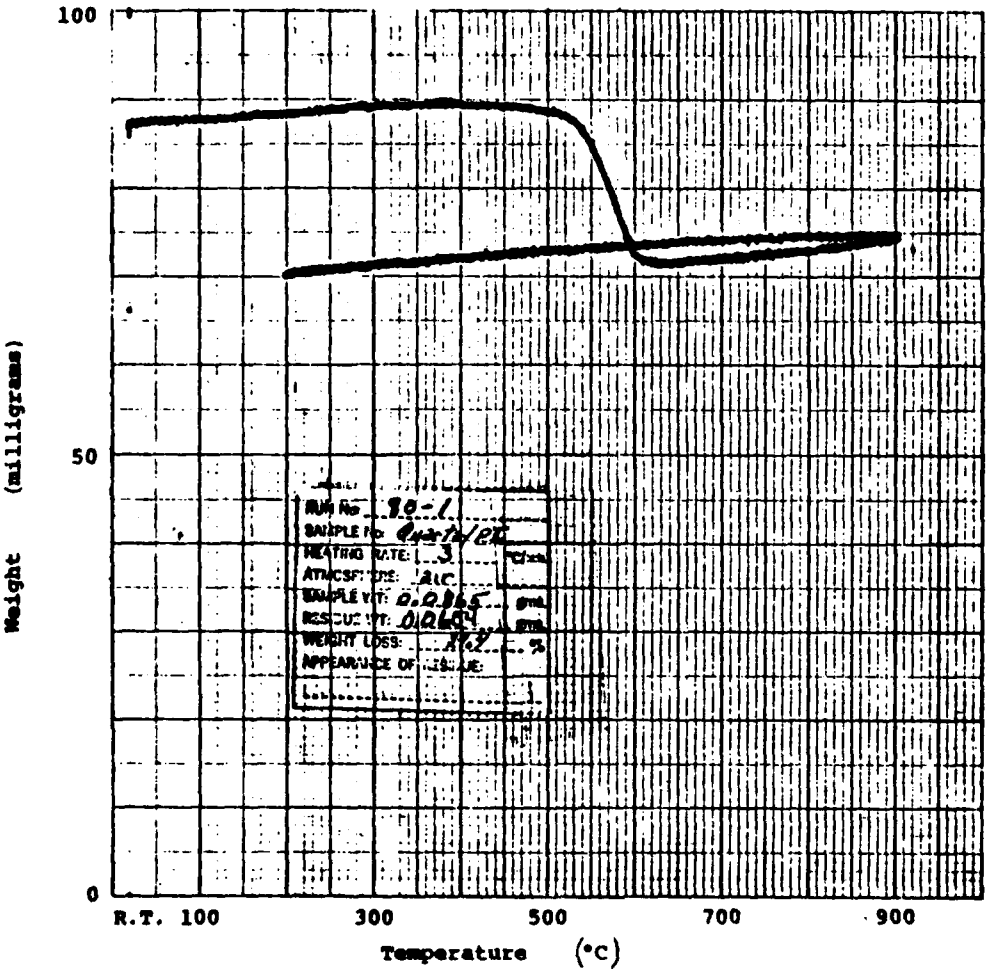


Figure 17. TGA in Air on Quartz 581/Polyimide (curved).

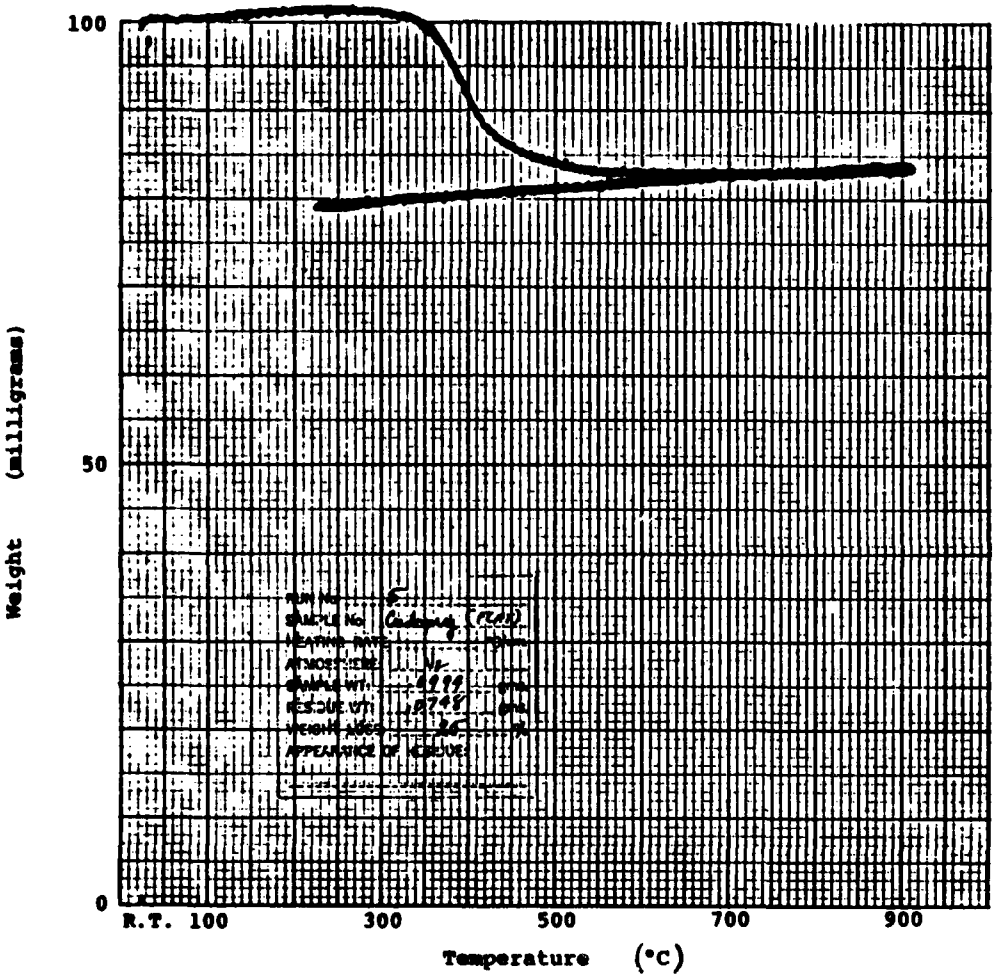


Figure 18. TGA in Nitrogen on Cordopreg (flat).

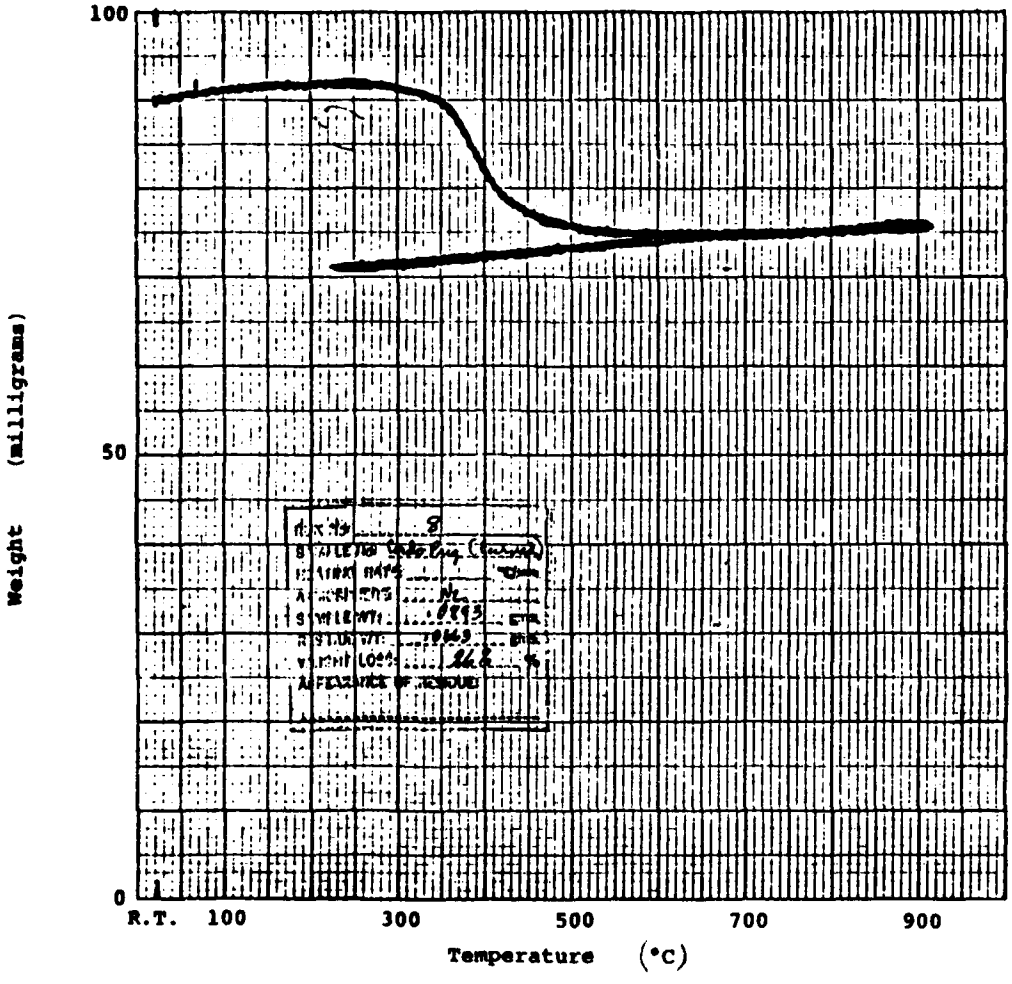


Figure 19. TGA in Nitrogen on Cordopreg (curved).

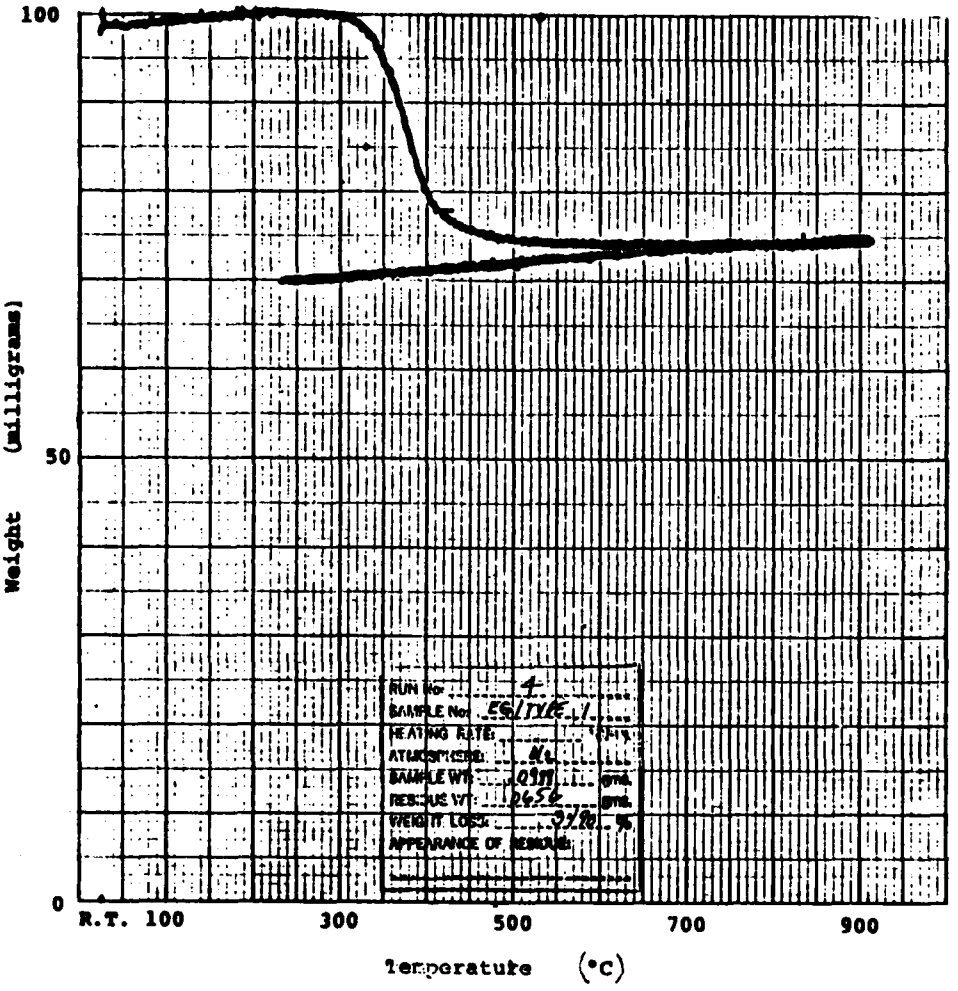


Figure 20. TGA in Nitrogen on EG/Epoxy Type 1.

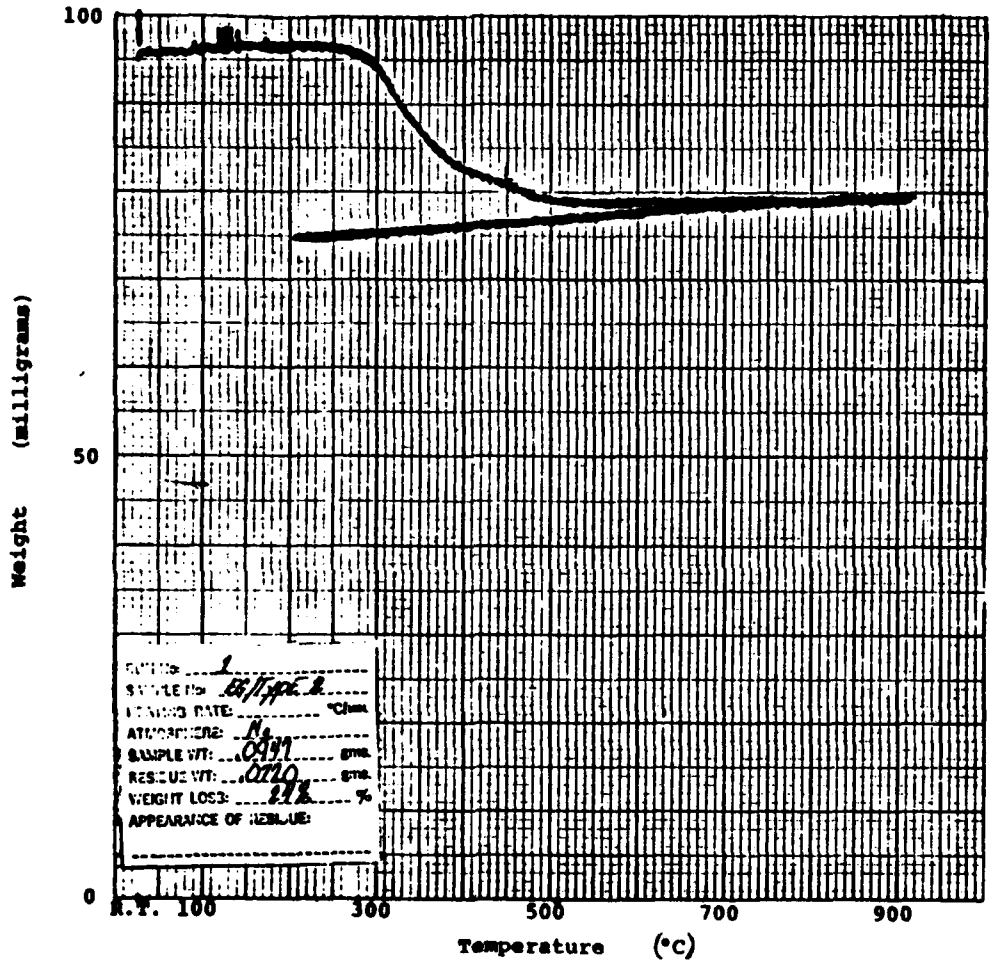


Figure 21. TGA in Nitrogen on EG/Epoxy Type 2.

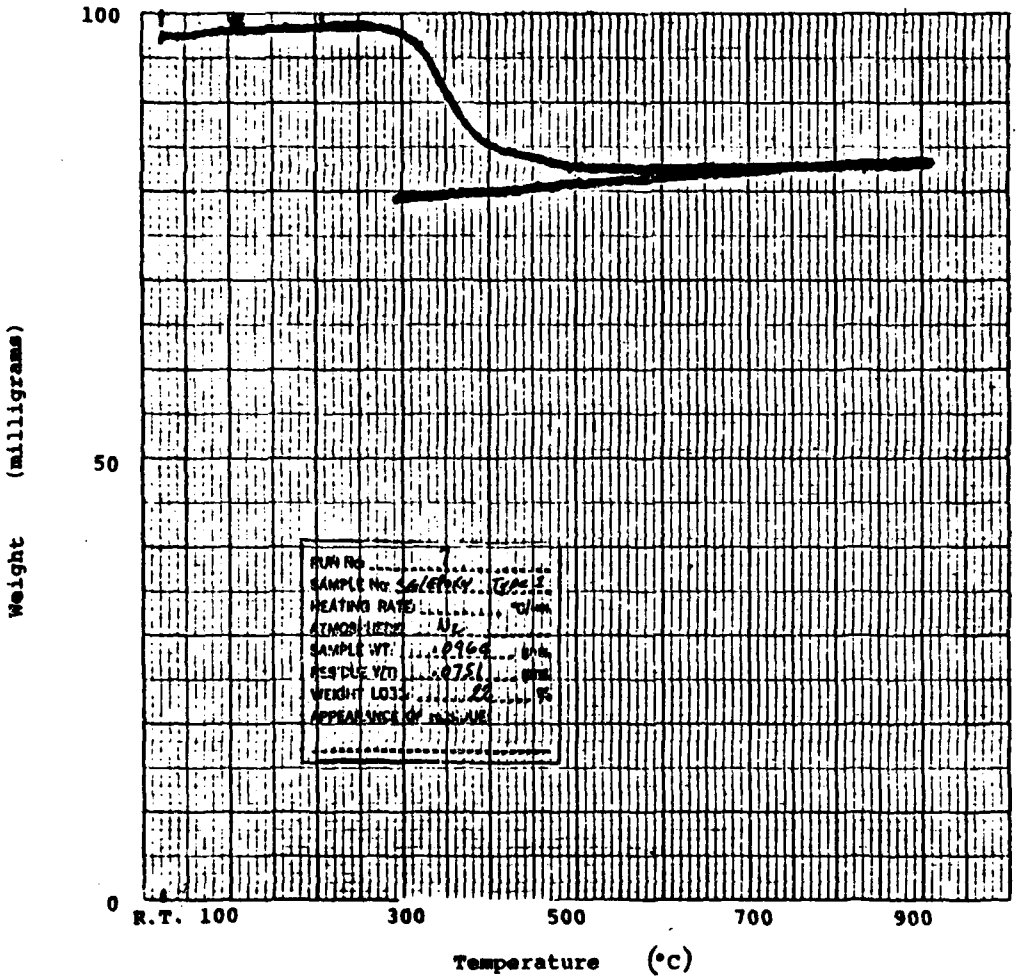


Figure 22. TGA in Nitrogen on SG/Epoxy Type 1.

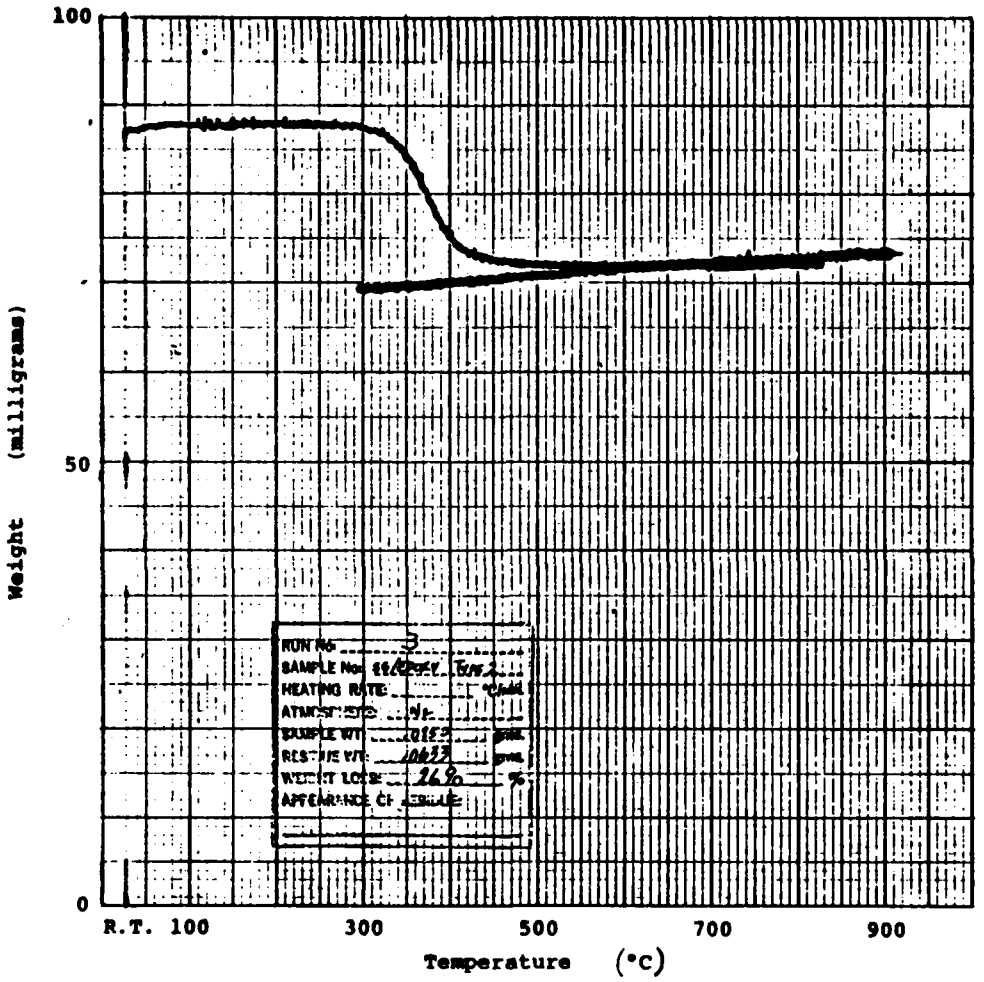


Figure 23. TGA in Nitrogen on SG/Epoxy Type 2.

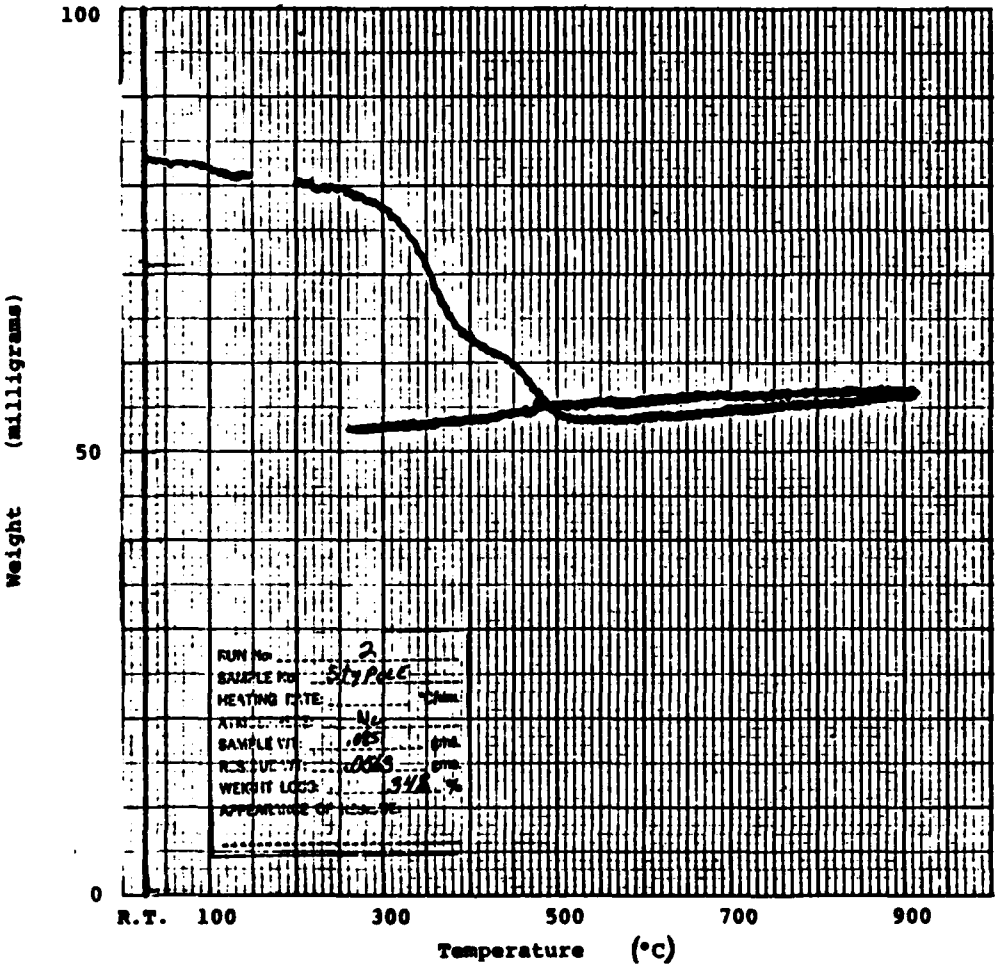


Figure 24. TGA in Nitrogen on Stypole (curved).

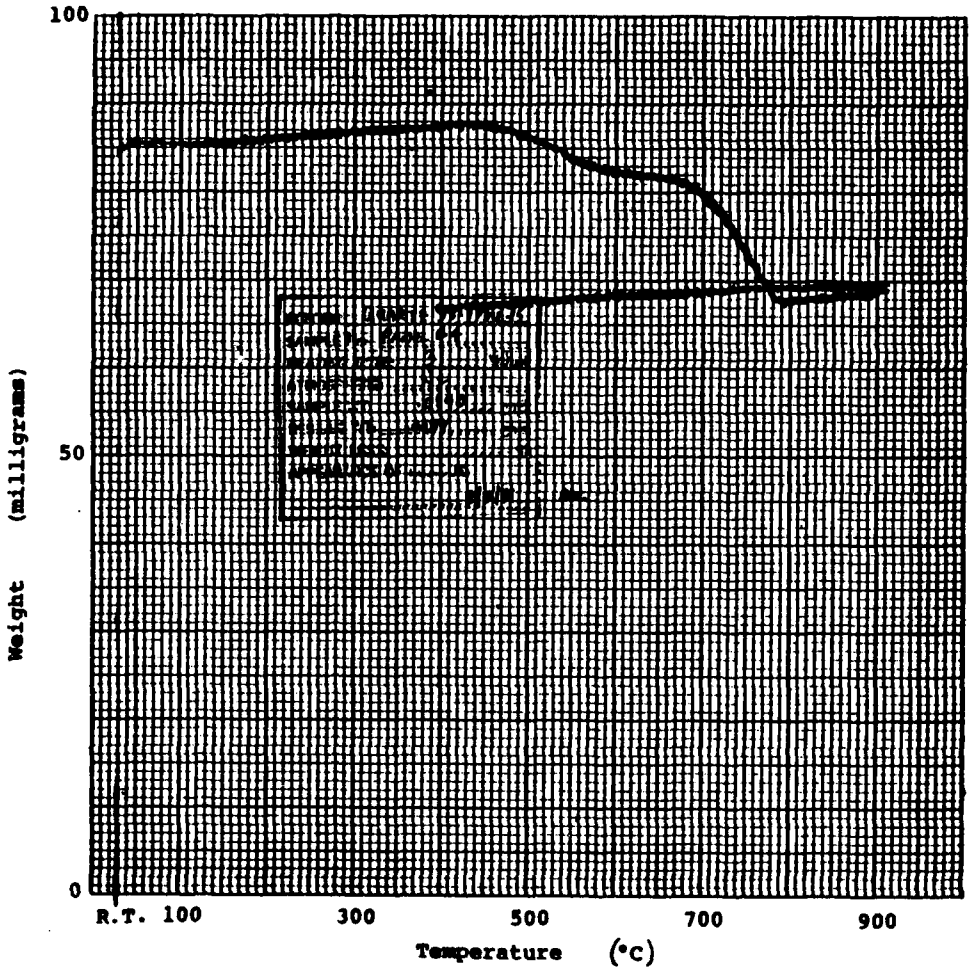


Figure 25. TGA in Nitrogen on Quartz 581/PMR-15 (flat).

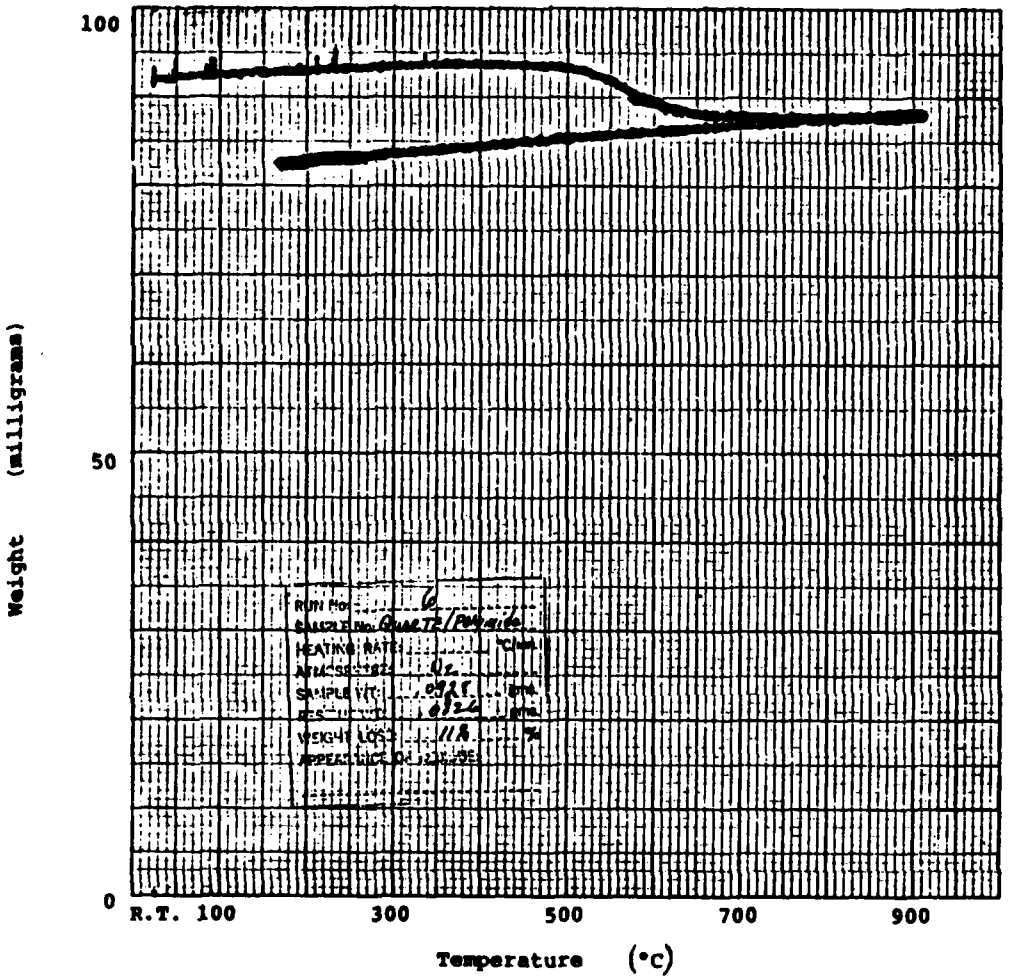


Figure 26. TGA in Nitrogen on Quartz 581/Polyimide (curved).

TABLE 5

TENSILE PROPERTIES OF JANAF LAMINATES[1]

Material	72°F(22°C)				350°F(177°C)				600°F(316°C)	
	Ult. Strength		Initial Modulus		Ult. Strength		Initial Modulus		Ult. Strength	
	psi (10^3)	MPa (10^2)	psi (10^6)	MPa (10^4)	psi (10^3)	MPa (10^2)	psi (10^6)	MPa (10^4)	psi (10^3)	MPa (10^2)
Cordopreg (flat)	35.1	2.42	2.07	1.43	15.4	1.06	1.31	0.90	---	---
EG/Epoxy Type 1	92.8	6.40	3.89	2.68	67.0	4.60	4.40	3.00	---	---
EG/Epoxy Type 2	88.1	6.07	4.13	2.85	67.3	4.64	3.28	2.25	---	---
SG/Epoxy Type 1	147	10.1	5.83	4.02	90.3	6.22	4.56	3.14	---	---
SG/Epoxy Type 2	135	9.30	5.50	3.80	20.5	1.41	4.24	2.92	---	---
Quartz 581/ PMR-15	92.8	6.40	2.65	1.83	81.8	5.63	2.50	1.72	74.0	5.1

[1]Values represent an average of three specimens for all materials except EG/Epoxy, Type 1 and SG/Epoxy, Type 2, in which cases the values represent an average of only two specimens.

TABLE 6

FLEXURAL PROPERTIES OF JANAF LAMINATES[1,2]

Material	72°F(22°C)				350°F(177°C)				600°F(316°C)			
	Ult. Strength		Initial Modulus		Ult. Strength		Initial Modulus		Ult. Strength		Initial Modulus	
	psi (10^3)	MPa (10^2)	psi (10^6)	MPa (10^4)	psi (10^3)	MPa (10^2)	psi (10^6)	MPa (10^4)	psi (10^3)	MPa (10^2)	psi (10^6)	MPa (10^4)
Cordopreg (flat)	52.7 T	3.6	2.5	1.7	16.7 C	1.2	0.84	0.58	8.5	0.59	0.95	0.65
EG/Epoxy Type 1	176.9 C&S	12.0	4.7	3.2	12.8 C&S	0.88	0.93	0.64	5.3	0.37	0.50	0.34
EG/Epoxy Type 2	153.8 T&S	11.0	5.6	3.6	74.1 C&S	5.1	2.63	1.8	N/A		N/A	
SG/Epoxy Type 1	215.0 C&S	15.0	5.2	3.5	98.0 C&S	6.8	3.50	2.4	N/A		N/A	
SG/Epoxy Type 2	207.5 C&S	14.0	5.1	3.5	17.5 S	1.2	1.23	8.5	N/A		N/A	
Quartz 581/ PMR-15	110.0 T	7.6	2.7	1.8	103.0 T&C	7.1	2.4	1.7	82 C	5.6	2.4	1.7

[1]Values represent an average of three specimens.

[2]Letters below strength values indicate failure mode.

T - tensile failure on bottom
C - compressive failure on top
S - shear failure along midplane

SECTION 4
SUMMARY

Mechanical, physical, and thermophysical properties required for radome materials being evaluated under the JANAF laser study have been measured. Nine different composite systems were tested during the investigation. These materials contained either glass or quartz reinforcement and either epoxy or polyimide matrix resins.

"As received" materials in this study represented those laminates processed by an industry which was still experimenting with the composite problems of excessive voids, resin-starved areas, quality-control etc. As the technology of composite materials handling and processing matured, those problems were resolved to the point that composite laminates today can be processed to contain little or no porosity or as having void content of less than 0.5 percent.

APPENDIX A

CURE AND POSTCURE SCHEDULE FOR QUARTZ 581/PMR-15 FLAT LAMINATES[1]

Cure Cycle

1. Apply partial vacuum (approximately 10 inch Hg).
2. Heat to 325°F (163°C) at 5°F (2.8°C)/minute.
3. Apply full vacuum and hold at 325°F (163°C) for 30 minutes.
4. Heat to 490°F (254°C) at 5°F (2.8°C)/minute and apply 200 psi.
5. Continue heating to 550°F (288°C) and hold at 550°F (288°C) for 1 hour.
6. Cool to below 150°F (65°C) under full vacuum and pressure, before removing from Autoclave.

Postcure

1. Heat to 650°F (343°C) at 5°F (2.8°C)/minute and hold for 4 hours.
2. Heat to 700°F (371°C) at 5°F (2.8°C)/minute and hold for 2 hours.
3. Cool slowly to below 150°F (65°C) (usually overnight) before removing from circulating air oven.

[1]Prepreg obtained from U.S. Polymeric.

APPENDIX B
HEAT OF PYROLYSIS ON RADOME MATERIALS

A considerable amount of time was spent trying to measure the heat of pyrolysis on microtomed samples of the radome materials in this program. Those efforts were unsuccessful for several reasons, listed below.

(1) The pyrolysis reaction occurs over a broad temperature range (from 225°C to above 550°C);

(2) The reaction is not uniform in nature (this is probably due to the nature of the sample, i.e., instead of one peak, many peaks are generated);

(3) The sample undergoes weight loss during reaction (calculations are based on a constant weight of sample);

(4) The energies measured are small because over 60 percent of the sample is nonreactive reinforcement; and,

(5) The sample gives off volatiles during the reaction, causing not only the change in weight mentioned above, but more importantly, causing deposits to form on the furnace surface. This caused the characteristics of the furnace to change and thereby influenced the data. In addition, these deposits proved very difficult to clean off the furnace.

One possible solution may be to put a sample in a volatile sample pan and using a high heating rate (i.e., 80°C/min) increase the temperature to 490°C. The heat evolved at this temperature would be measured as a function of time. Data from this experiment may be used with TGA data to calculate a realistic rate. There is no assurance, however, that this method will provide valid data.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SUPPLEMENTAR

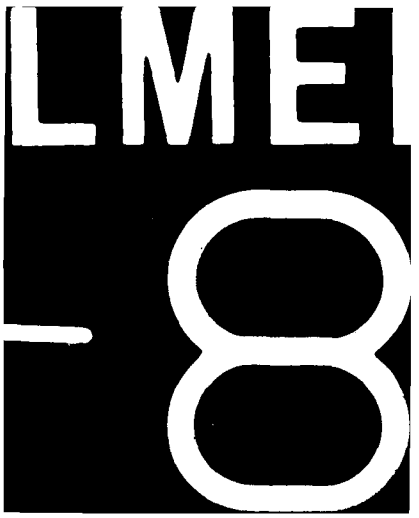
INFORMATION

ADDENDUM TO INTERIM TECHNICAL REPORT

AFWAL-TR-81-4129

The original report entitled, "Evaluation of Reinforced Plastic Radome Materials", neglected to specify how long the specimens tested for mechanical properties at elevated temperature were held or "soaked" at temperature prior to testing. This information should have been included in Paragraph 2.2.5, page 6, as the last sentence. This sentence should read, "All specimens tested at elevated temperature were held at the test temperature for 30 minutes before testing to insure thermal equilibrium."

This information should also be added as a footnote to the test columns for 350°F and 600°F in Tables 5 and 6, pages 38 and 39 respectively.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