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DEVELOPMENT OF DESIGN ALLOWABLES DATA FOR ADHESIVES FOR ATTACHING REUSEABLE SURFACE INSULATION

ADDENDUM 1A TO FINAL TECHNICAL REPORT DATED OCTOBER 1972

12 February 1973

~~FOR U.S. GOVERNMENT AGENCIES AND THEIR CONTRACTORS ONLY~~

Prepared Under Contract No. NAS 9-12392

GENERAL DYNAMICS
Convair Aerospace Division
Fort Worth, Texas

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

MANNED SPACECRAFT CENTER
Houston, Texas

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DEVELOPMENT OF DESIGN ALLOWABLES DATA FOR ADHESIVES FOR ATTACHING REUSABLE SURFACE INSULATION

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TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

LIST OF TABLES (Continued)

SECTION 1

INTRODUCTION AND SUMMARY

The work described in this report was conducted under NASA, MSC Contract NAS9-12392, Amendment 2S, titled "Development of Design Allowables for Adhesives for Attaching Reusable Surface Insulation." The task consisted of conducting mechanical and thermal tests to establish design allowables data on a new room temperature vulcanizing (RTV) silicone adhesive, X3-6004, developed in cooperation with Dow Corning Corporation. Low modulus, coupled with relatively low density and good low-temperature properties of this adhesive,places it in contention as a candidate for attaching reusable surface insulation on the space shuttle.

Test methods developed and used to obtain design allowables data on the four silicone adhesives in the basic contract were used in this supplemental work to fully characterize X3-6004. In addition, a new test procedure using "dog bone" shaped specimens and an Instron extensometer was developed to obtain tensile modulus data at temperatures below -175°F, which is the glass transition temperature of the X3-6004.

Data obtained and presented herein show that the modulus values of X3-6004 are significantly lower than those of General Electric's RTV-560 and the other three adhesives characterized under the basic contract at test temperatures from 550° to -175°F. At -175°, -200° and -270°F, the modulus of X3-6004 is approximately the same as GE RTV-560 and the other three silicone adhesives included in the basic contract.

The X3-6004 adhesive exhibits good processing properties. It has a 12 percent lower density than General Electric's RTV-560. Although lower in overall strength properties as compared to the other adhesives in the program, X3-6004 has adequate adhesion to 2024T81 aluminum to compete as an adhesive for attaching reusable surface insulation. It does exhibit some tendency to revert and soften at temperatures above 350°F when in a confined area.

The new test procedure for determining modulus at -175°F and below resulted in higher modulus values than those determined using strap specimens and a photographic technique for measuring deformation under loading conditions. Whether the increased modulus is real or the result of specimen geometry could not be determined accurately because of the erratic yield behavior of the silicone elastomers when subjected to stress at temperatures below their glass transition (T_g) temperature.

SECTION 2

CANDIDATE ADHESIVE SYSTEM

The adhesive, DC X3-6004, is the product of a continued development program. Steps in its development and selection of a primer to enhance its adhesion to substrates are described in the paragraphs that follow.

2.1 DESCRIPTION OF DC X3-6004

Dow Corning's X3-6004 evolved from 77-137, an extremely low modulus silicone adhesive reportedly based on a copolymer of diphenyl and dimethyl siloxanes. Its copolymer structure imparts an inherent low glass transition (Tg) temperature to the cured elastomer. Although 77-137 exhibited low modulus, which is a desirable property for attaching low strain-to-failure external insulation blocks on the space shuttle, its strength properties were unsatisfactory. Dow Corning Corporation further developed the formulation and gave it the number X3-6000. A sample lot of X3-6000 was supplied to Convair Aerospace. The results of preliminary tests showed that X3-6000 had good strength, adhesion, and low modulus properties. Nevertheless, the base portion of the two component system was too viscous for easy processing and increased in viscosity with shelf aging. This undesirable characteristic was discussed with Dow Corning's technical personnel who confirmed the problem and attributed it to hydrogen bonding between the polymer and a specific filler ingredient in the formulation. Dow Corning reformulated the X3-6000 using different fillers. Because of the slight formulation change, Dow Corning changed the product number to X3-6004.

A production lot of X3-6004 was obtained for tests. Several small samples of the lot were mixed to check work life and handling characteristics. Work life was found to be only 30 minutes, whereas work life of the X3-6000 was 90 minutes. Hand tests of the cured samples of the X3-6004 showed it to have extremely low strength properties.

Larger batches of X3-6004 and X3-6000 were then mixed and cast into 6- by 6-inch sheets 0.070 inch thick. Specimens were cut from these sheets and tested for hardness, ultimate tensile and elongation, modulus, and tear strength as shown in Table I. Data obtained showed X3-6004, as compared to X3-6000, to be considerably lower in tensile and tear strength and only slightly

Table I PROPERTIES OF DC X3-6000, DC X3-6004 BEFORE REWORK, AND DC X3-6004 AFTER REWORK BY DOW CORNING CORPORATION (Tested at 77 $\pm$ 2^{o}F)

Test Specimen	Hardness (1) Shore A	Ultimate (2) Tensile, psi	Ultimate (2) Elongation, %	Tensile Modulus, psi		Tear, (3) lbs/inch
				@ 100% E	@ 200% E	
DC X3-6000						
1		473	700	70	109	190
2		606	820	61	91	220
3		667	870	30	61	218
Avg.	36	637	845	54	87	209
DC X3-6004 - Before rework						
1		103	300	47	70	8
2		100	280	47	74	7
3		79	220	47	70	7
Avg.	25	94	267	47	71	7
DC X3-6004 - After rework						
1		225	310	75	133	16
2		197	310	69	-	22
3		183	280	75	133	24
Avg.	30	202	300	73	133	21
RTV-560*						
1						16
2						15
3						18
Avg.						16
SLA-561*						
1						7
2						7
3						8
Avg.						7

*For comparison purposes, tear strength of GE's RTV-560 and Martin Marietta's SLA-561 were determined.

Test Methods:
(1) ASTM D-2240-68
(2) ASTM D-412-68, Die C
(3) ASTM D-624-54, Die B

lower in modulus. In view of the short work life (a minimum of 60 minutes work life is considered necessary for shop processing) and low strength, both Dow Corning and the NASA, MSC technical monitor were contacted. It was mutually agreed that the material should be returned to Dow Corning for rework. This was accomplished.

The reworked X3-6004 was returned to Convair Aerospace and retested for work life, hardness, ultimate tensile and elongation, modulus, and tear strength. Tear strength of General Electric's RTV-560 and Martin Marietta's SLA-561 was determined for comparison. Data obtained are shown in Table I. Work life was increased from 30 minutes to 90 minutes, and tear strength was increased from 7 to 21 pounds per inch (ppi) of width. Although 21 ppi tear strength is considerably below the 209 ppi exhibited by X3-6000, the forerunner of X3-6004, it compares favorably with 16 ppi for General Electric's RTV-560 and 7 ppi for Martin Marietta's SLA-561. Based on results of these tests, all test specimens required for complete characterization of X3-6004 were fabricated.

2.2 SELECTION OF PRIMER FOR PREPARING TEST SPECIMENS OF DC X3-6004

Dow Corning did not recommend a specific primer for use with X3-6004; therefore, three candidates were tested on 2024T81 aluminum substrates. The three primers evaluated were Dow Corning's 1200 and 1203 and General Electric's SE-4155. Data obtained are tabulated in Table II. Both DC-1200 and General Electric's SE-4155 produced 100 percent cohesive failures of the X3-6004 in the lap shear specimens. Based on these data, DC-1200 was selected for use on all adhesion test specimens in the program.

2.3 PROCESSING CHARACTERISTICS OF DC X3-6004

DC X3-6004 is a two-component adhesive consisting of base and catalyst. Recommended mixing ratio of the base and catalyst is 10 to 1, respectively. The catalyst is a paste and blends readily with the creamy base. In this program, the two components were accurately weighed on a flat aluminum plate placed on a torsion balance. The base and catalyst were blended and thoroughly mixed with a spatula then transferred to plastic cartridge tubes for degassing. Entrapped air bubbles were expelled by centrifuging the tubes at 1500 RPM for 10 minutes. Work life of the mixed and degassed material at room conditions is approximately 45 minutes.

Table II EFFECT OF PRIMER ON LAP SHEAR STRENGTH OF DC X3-6004, TESTED AT RT

Test Specimen Primer	Lap Shear Strength, psi	% Cohesive Failure
Dow Corning's RTV-1200		
1	130	100
2	143	100
3	144	100
Avg.	139	100
Dow Corning's RTV-1203		
1	121	95
2	115	90
3	115	90
Avg.	117	92
General Electric's SE-4155		
1	146	100
2	130	100
3	140	100
Avg.	139	100

*Specimens were 1 inch overlaps, 2024T81, .063" thick, chromic acid etched. Primers were applied in as thin coats as possible and allowed to air dry 30 minutes before bonding with X3-6004.

SECTION 3

CHARACTERIZATION OF

DC X3-6004

Physical, mechanical, and thermal property tests were conducted as described in the final report of the basic contract with the exception that some test strain rates and all tests at 600°F were deleted and an additional tension modulus test was added. A summary of the tests conducted is presented in Table III. Metal cleaning and primer application were as described in the final report.

3.1 PHYSICAL PROPERTIES

Density was determined at room temperature on five samples as shown in Table IV. The average density was 1.25 grams/cc, which is approximately 12 percent less than that of General Electric's RTV-560. The hardness of DC X3-6004 determined on cured specimens according to ASTM-D-2240-68 was 30 Shore A (Table I).

3.2 MECHANICAL PROPERTIES

3.2.1 Adhesion in Tension

Adhesion-in-tension tests were conducted on specimens consisting of two aluminum cylinders 1.125 inches in diameter bonded together with a 0.06 inch thickness of DC X3-6004. Load was applied normal to the cylinder faces. Other details of the test procedure are contained in the final report.

Ultimate tensile and elongation values are shown in Table V and Figures 1 and 2. The tensile strength increases as the temperature decreases, and a rapid increase occurs between -175°F and -200°F. Also, the data obtained at 550°F show severe deterioration of the material at this temperature.

Ultimate elongation values show an increase as the temperature is decreased to -150°F followed by a sharp drop as the temperature is further decreased. The most drastic drop occurs between -150°F and -175°F (Figure 2).

Table III DESIGN ALLOWABLES TEST SPECIMENS
(Specimens per Data Point)

TEST-STRAIN RATE-BONDLINE THICKNESS	TEST TEMPERATURES, °F								
	-270	-200	-175	-150	-65	RT	300	350	550
Adhesion in tension	3	3	3	3	3	3	3	3	3
Adhesion in shear	3	3	3	3	3	3	3	3	3
Poisson's Ratio, Tensile Modulus									
STRAP SPECIMENS									
0.4 in./in./min. @ R.T.				3	3	3	3	3	3
0.02 in./in./min. @ -175,-200,-270°F	3	3	3						
ROUND THROAT TEST SPECIMENS									
0.25 in./in./min. @ R.T.						5			
0.05** in./in./min. @ -175,-200,-270°F	5	5	5						
Torsional Shear Modulus									
0.4 in./in./min. 0.030 in. bondline				3	3	3	3	3	3
0.25 in./in./min.	3	3	3						
0.4 in./in./min. 0.06 in. bondline				3	3	3	3	3	3
0.25 in./in./min.	3	3	3						
0.4 in./in./min. 0.10 in. bondline				3	3	3	3	3	3
0.25 in./in./min.	3	3	3						
0.4 in./in./min. 0.25 in. bondline				3	3	3	3	3	3
0.25 in./in./min.	3	3	3						
Compression Modulus									
0.4 in./in./min.				3	3	3	3	3	3
0.007** in./in./min.	3	3	3						
Thermal Cycling-Effect of 5 different thermal cycles									
Band shaped specimens						18*			
Adhesion in tension						15			
Double lap shear (Torsion)									
Shear modulus to 25% failing load						18*			
Ultimate tensile shear strength						18*			
Constant Strain-Stress Relaxation									
Band shaped specimens									
0.4 in./in. strain at R.T.			3			3			
0.005 in./in. strain at -200°F & below	3	3							
Density						3			
Thermal Expansion	3 specimens tested over -290 to 600°F temperature range								
Specific Heat	3 specimens tested over -130 to 600°F temperature range								
Thermal Conductivity	1 specimen tested over -290 to 500°F temperature range								
Work Life						Report			
Durometer Hardness						Report			
Total Specimens 327									

* Includes 3 controls (no thermal cycling)

** Strain rates were varied to achieve satisfactory stress-strain relationship.

Table IV

Density of DC X3-6004 at Room Temperature

ASTM-D-297, 17(C)

	1.2465 grams/cc
	1.2482 grams/cc
	1.2471 grams/cc
	1.2471 grams/cc
	1.2722 grams/cc
Average	1.2522 grams/cc

Table V ADHESION IN TENSION OF DC X3-6004, CYLINDER SPECIMENS

Temperature & Specimen No.	Tensile, PSI	Elongation, %	Cohesive Failure,%
550°F			
1	3	37	100
2	2	48	100
3	1	67	100
Avg	2	51	100
350°F			
1	67	94	100
2	69	62	99
3	67	71	100
Avg	68	76	100
300°F			
1	85	86	100
2	89	111	100
3	88	127	100
Avg	88	108	100
RT			
1	152	176	99
2	161	176	100
3	143	160	100
Avg	152	170	100
-65°F			
1	262	290	100
2	255	233	100
3	255	260	100
Avg	257	261	100
-150°F			
1	815	349	100
2	752	411	100
3	660	321	10
Avg	742	360	70
-175°F			
1	965	13	98
2	965	19	99
3	910	-	98
Avg	947	16	98
-200°F			
1	3640	12	99
2	2740	6	100
3	4360	5	98
Avg	3580	8	99
-270°F			
1	5350	14	50
2	5050	18	50
3	4580	17	50
Avg	4993	16	50

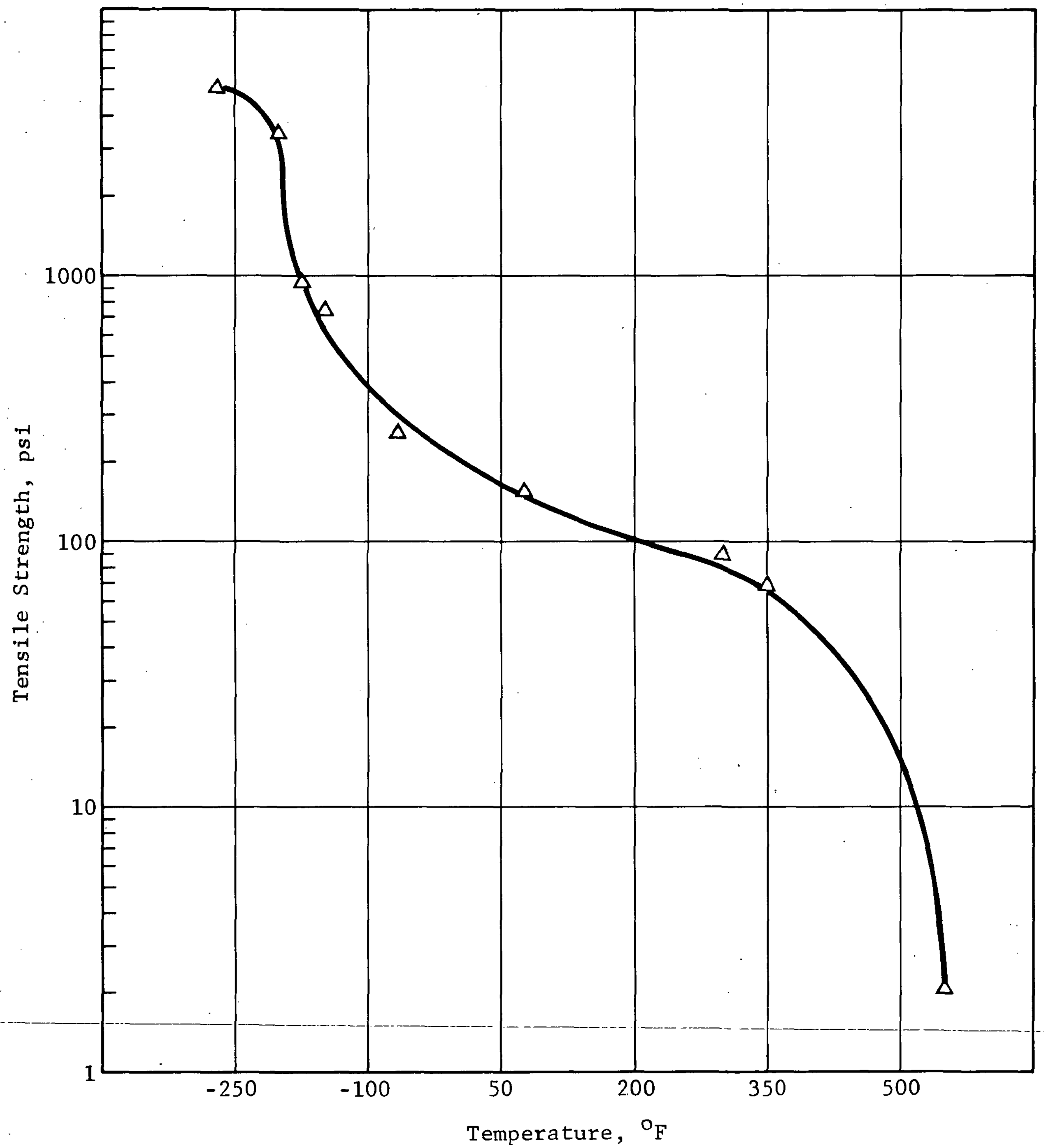

Figure 1 Tensile Strength Vs. Temperature, DC X3-6004

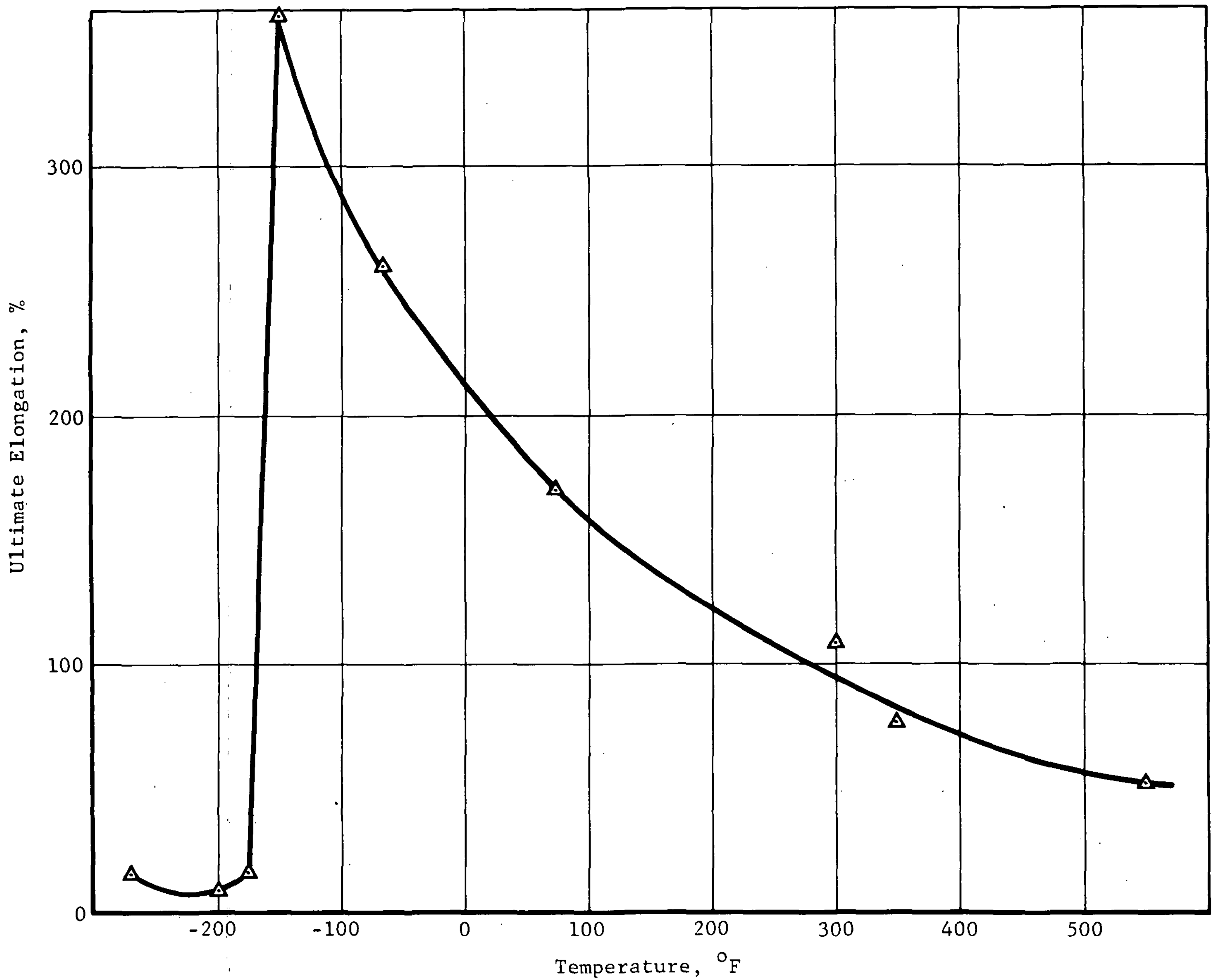

Figure 2 Ultimate Elongation Vs. Temperature, DC X3-6004

3.2.2 Adhesion in Shear

Shear properties were determined on single overlap shear specimens as described in the final report.

Shear values obtained are shown in Table VI and Figure 3. As can be seen, the shear strength increases as the temperature decreases from 550°F to -175°F; this is followed by a slight decrease in shear strength as the temperature decreases below -175°F. Also, as noted in Table VI, all specimens tested at temperatures below -175°F exhibited 100 percent adhesive failure, whereas all specimens tested above this temperature failed cohesively. This adhesive mode of failure may account for the lower values obtained at temperatures below -175°F. The shear values obtained at 550°F indicate severe deterioration of the material at this temperature.

3.2.3 Torsional Shear Modulus

Shear modulus tests were conducted on double overlap shear specimens as shown in Figure 4 loaded in torsion. Bond thicknesses were 0.03, 0.06, 0.10, and 0.25 inch. Tests were conducted using a strain rate of 0.4 in./in./min. at -150°F and above and 0.25 in./in./min. at -175°F and below. Details of the test setup and procedure are fully described in the final report.

In general, the data, as shown in Table VII and graphically in Figure 4, indicate that the shear modulus values are independent of bondline thickness at -150°F and above but that they increase as bondline thickness increases when tested at temperatures below -150°F. Also, the shear modulus increases as the temperature decreases to -200°F; this is followed by a slight decrease at -270°F. As shown, severe deterioration of the material occurs at 550°F.

3.2.4 Tensile and Compression Modulus

A photographic method was employed for determining tensile and compression modulus values using specimens bench marked with 0.02-inch-diameter dots. The tensile modulus specimens were 0.1 inch thick, 12 inches long, and 2.5 inches wide (strap specimens), and the compression modulus specimens were parallelepipeds 1.5 inches square by 4 inches high. Photographs were taken at

Table VI SINGLE OVERLAP SHEAR STRENGTH OF DC X3-6004

Temperature & Specimen No.	Shear Strength, psi	Cohesive Failure, %
550°F		
1	1	100
2	1	100
3	1	100
Avg	1	100
350°F		
1	47	100
2	33	100
3	35	100
Avg	38	100
300°F		
1	61	100
2	62	100
3	65	100
Avg	63	100
RT		
1	130	100
2	120	100
3	150	100
Avg	133	100
-65°F		
1	230	100
2	230	100
3	257	100
Avg	239	100
-150°F		
1	920	100
2	890	100
3	950	100
Avg	920	100
-175°F		
1	1350	100
2	1414	100
3	1509	100
Avg	1425	100

Table VI

Single Overlap Shear Strength of DC X3-6004 (Cont'd)

Temperature & Specimen No.	Shear Strength, psi	Cohesive Failure, %
$-200^{\circ}F$		
1	1145	0
2	1267	0
3	1786	0
Avg	1399	0
$-270^{\circ}F$		
1	1103	0
2	1091	0
3	1118	0
Avg	1104	0

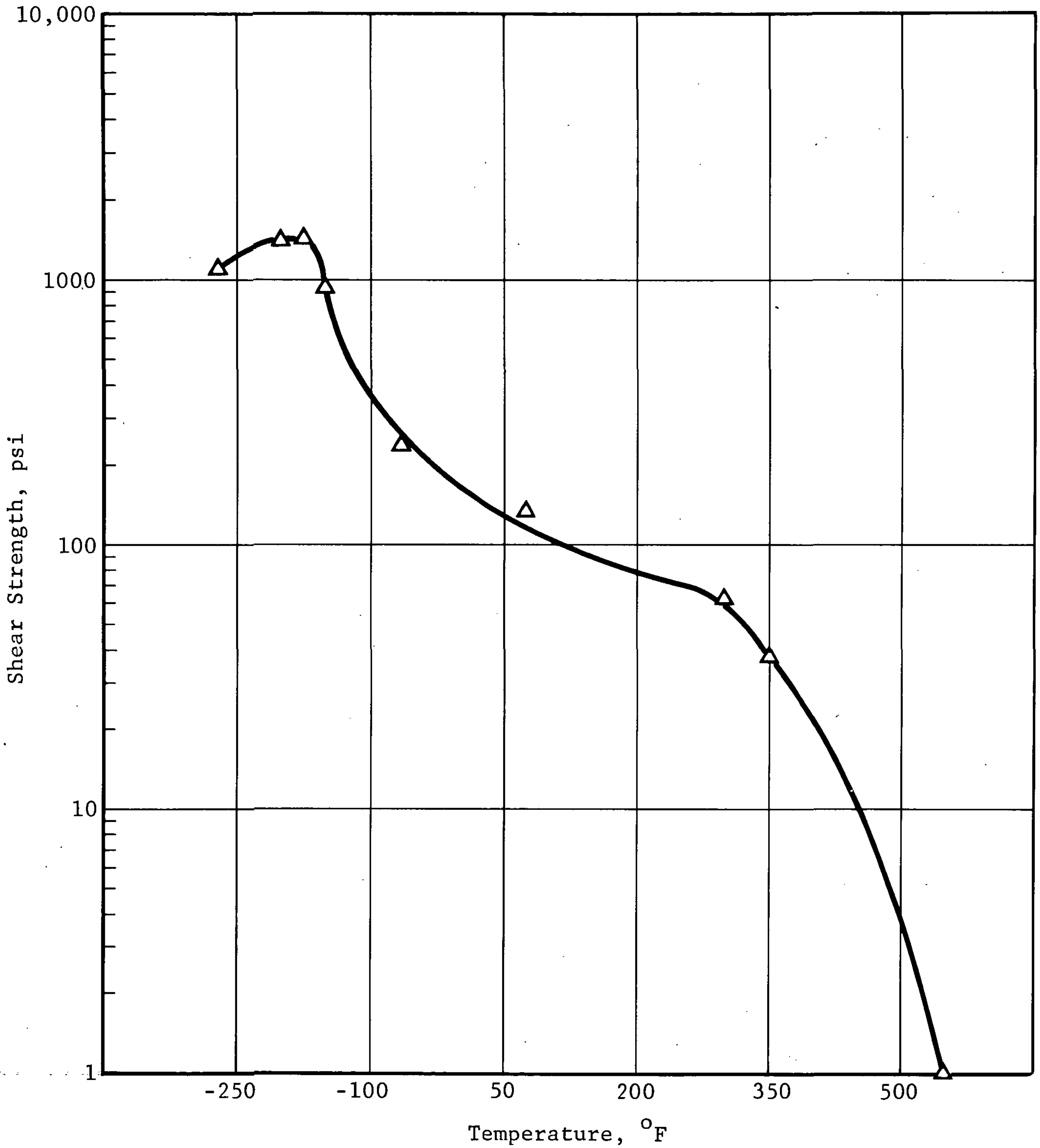

Figure 3 Shear Strength Vs. Temperature, DC X3-6004

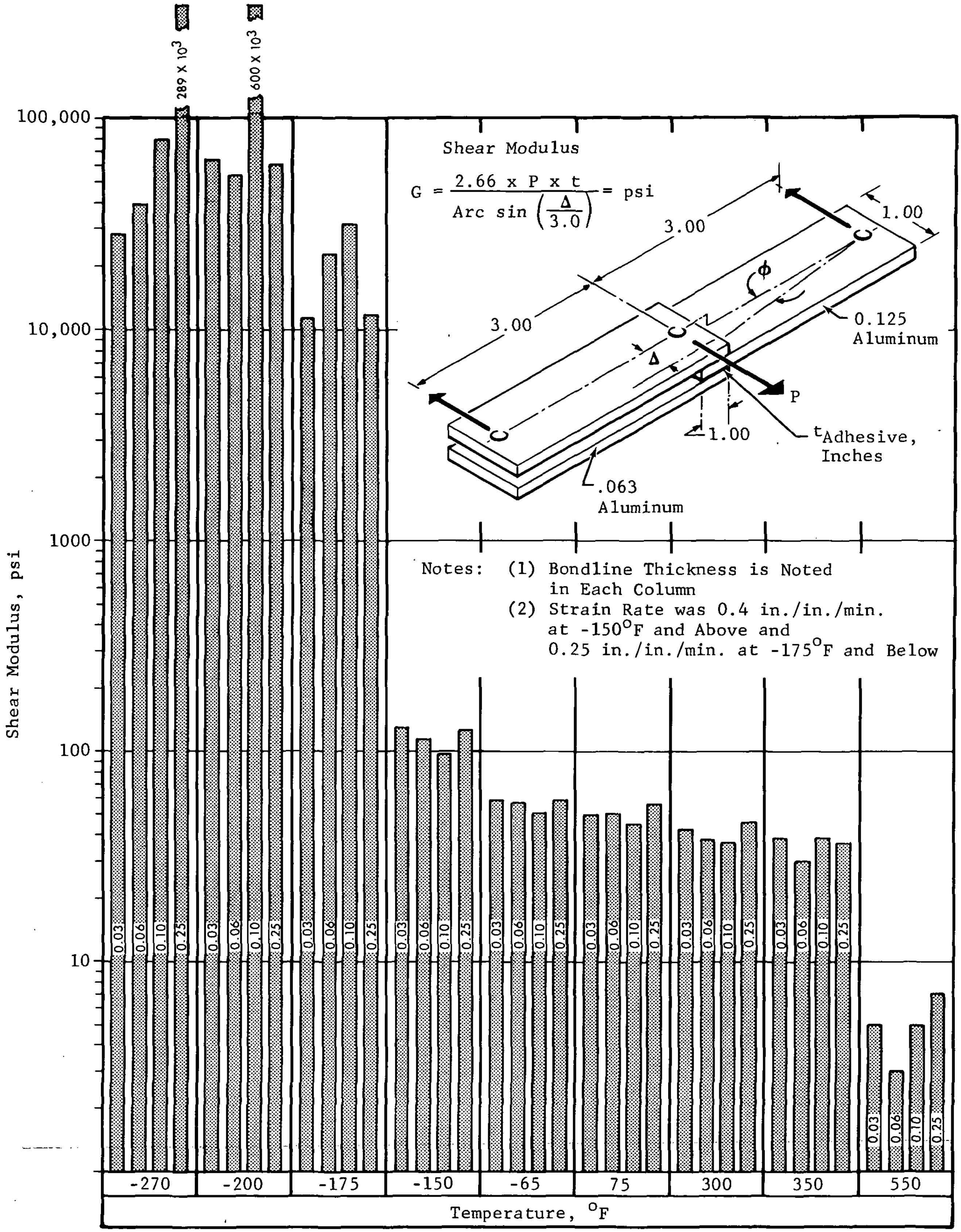

Figure 4 Shear Modulus of DC X3-6004 at Various Bondline Thicknesses and Temperature

Table VII SHEAR MODULUS OF DC X3-6004, psi
(Double Lap Torsion Test)

SPECIMEN	TEMPERATURE, °F -270	-200	-175	-150	-65	RT	300	350	550
0.03 Bond Line									
1	12.2×10^3	63.8×10^3	16.9×10^3	125	44	52	41	37	2
2	23.6×10^3	16.1×10^3	10.9×10^3	128	72	46	47	41	5
3	48.1×10^3	114.6×10^3	7.8×10^3	137	57	52	42	38	7
Avg	27.9×10^3	64.8×10^3	11.9×10^3	130	58	50	43	39	5
0.06 Bond Line									
1	13.4×10^3	44.6×10^3	12.0×10^3	117	51	46	40	28	3
2	45.4×10^3	57.9×10^3	29.5×10^3	116	58	53	39	31	2
3	57.0×10^3	48.7×10^3	27.4×10^3	109	63	53	37	31	4
Avg	38.6×10^3	50.4×10^3	23.0×10^3	114	57	51	38	30	3
0.10 Bond Line									
1	100.5×10^3	1.4×10^6	27.5×10^3	101	49	47	35	32	5
2	88.1×10^3	0.1×10^6	31.7×10^3	99	56	46	35	44	6
3	51.6×10^3	0.2×10^6	38.9×10^3	90	48	45	41	41	4
Avg	80.1×10^3	0.6×10^6	32.7×10^3	97	51	46	37	39	5
0.25 Bond Line									
1	349×10^3	67.9×10^3	12.3×10^3	129	67	57	46	30	6
2	401×10^3	44.4×10^3	11.2×10^3	116	55	56	50	42	8
3	119×10^3	73.4×10^3	11.5×10^3	140	53	57	42	40	6
Avg	289×10^3	61.9×10^3	11.7×10^3	128	58	57	46	37	7

NOTE: Strain rate was 0.4 in./in./min. at -150°F and above and 0.25 in./in./min. at -175°F and below.

incremental loadings which were later measured to determine modulus and Poisson's ratio in tension and compression. Strain rates used are shown in Table VIII. A detailed discussion of the test method is contained in the final report.

Tension and compression modulus values for DC X3-6004 are shown in Table VIII and Figures 5 and 6. Although compression modulus values are somewhat higher than the tensile modulus values, the plot of modulus versus temperature for both properties are very similar. The modulus values increase as temperature decreases, and a sharp increase occurs between -65°F and -200°F. The low tensile modulus value obtained at 550°F indicates deterioration of the material at this temperature. Because of the severe deterioration occurring at 550°F and the length of time (30 minutes to 1 hour) required to stabilize the compression blocks at temperature, compression modulus tests could not be conducted at 550°F.

3.2.5 Tensile Modulus Determinations Using Round Throat Specimens

Near and below the T_g temperature of the silicone adhesives investigated, the specimen deformation is so small that accurate tensile modulus values cannot be obtained from the strap specimens. Therefore, an additional test method using a round throat specimen was attempted to determine tensile modulus on DC X3-6004.

The specimen is a round throat "dog bone" shaped molded specimen with aluminum doublers bonded on each end for load introduction (Figure 7). The specimen was loaded using an Instron test machine, and strain was determined with a 2-inch gauge length Instron extensometer attached to the throat of the specimen. Tests were conducted at room temperature, -175°F, -200°F, and -270°F. Tensile modulus was calculated as follows:

$$\text{Modulus} = \frac{P/A}{\Delta/2}$$

where: P/Δ = slope of load deformation curve, lbs./in.

A = cross section area of specimen, sq. in.

2 = gauge length of extensometer, in.

TABLE VIII

TENSILE AND COMPRESSION MODULUS OF DC X3-6004

psi

STRAIN RATE, in/in/min	TEMPERATURE, ^{o}F								
	550	350	300	R T	-65	-150	-175	-200	-270
	TENSILE MODULUS								
0.4	28	92	94	85	115	1790			
0.4	30	95	90	103	122	321			
0.4	29	79	-	100	109	8947			
AVG	29	89	92	96	115	3686			
0.02							2099	61873	73045
0.02							17222	46262	112358
0.02							102027	94745	94406
AVG							40449	67627	93270
	COMPRESSION MODULUS								
0.4		88	151	160	193	568			
0.4		112	146	186	238	446			
0.4		116	137	188	252	395			
AVG		105	145	178	228	470			
0.1							629		
0.1							1048		
0.1							1015		
AVG							897		
0.01								37793	
0.05								15056	
0.05								197382	
AVG								83410	
0.007									291891
0.007									219035
0.007									106279
AVG									205735

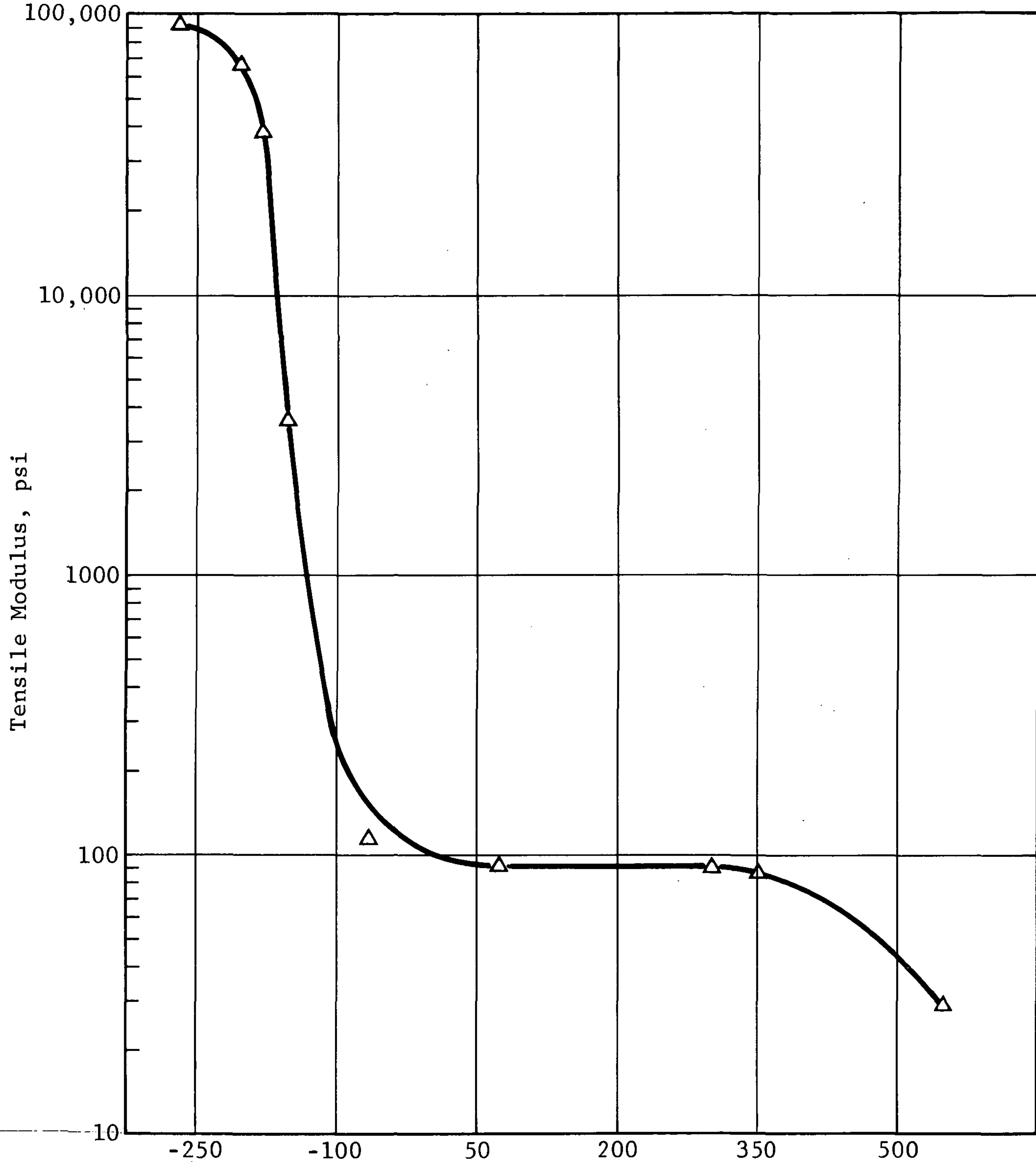

Figure 5 Tensile Modulus Vs. Temperature, DC X3-6004

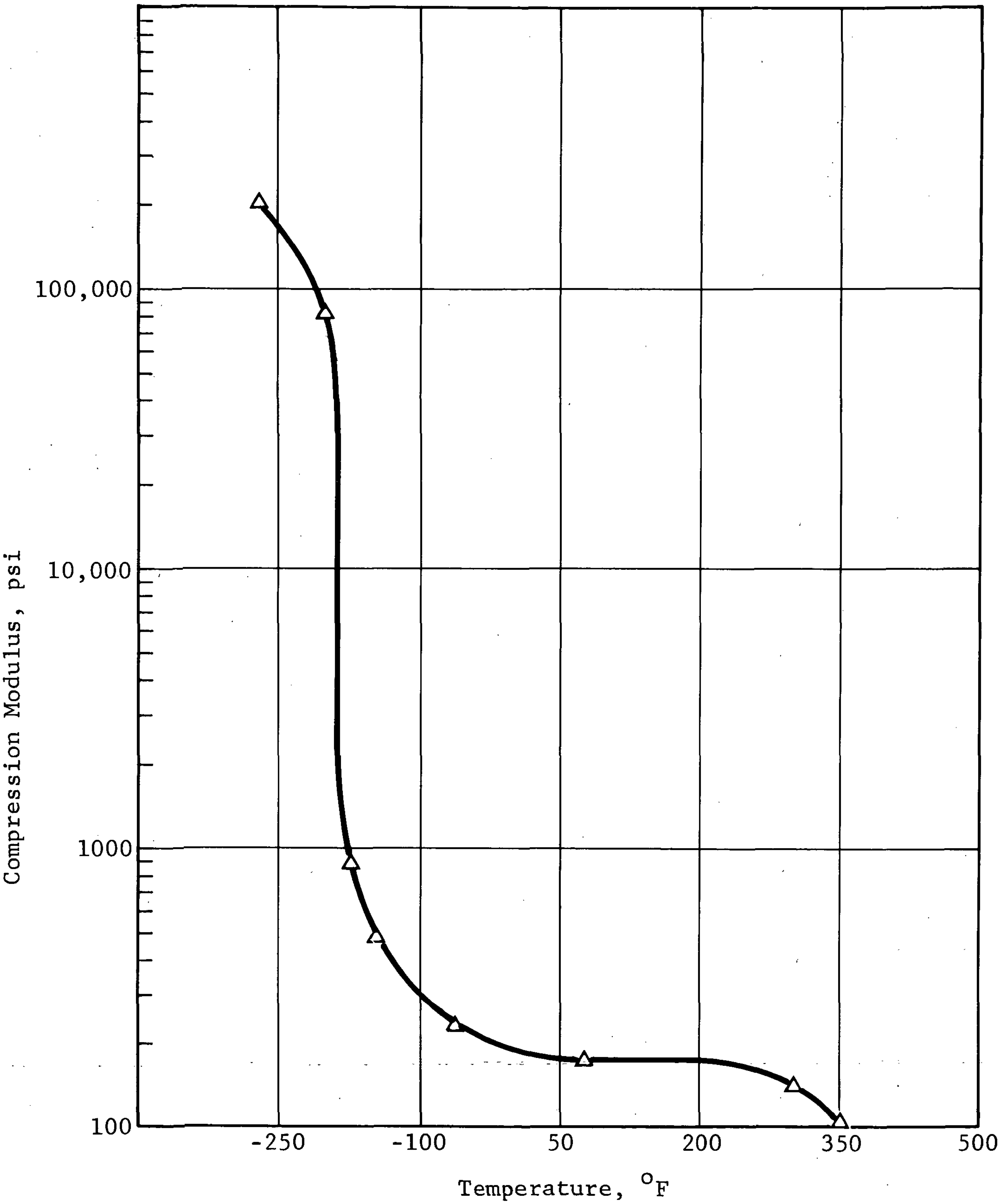

Figure 6 Compression Modulus Vs. Temperature, DC X3-6004

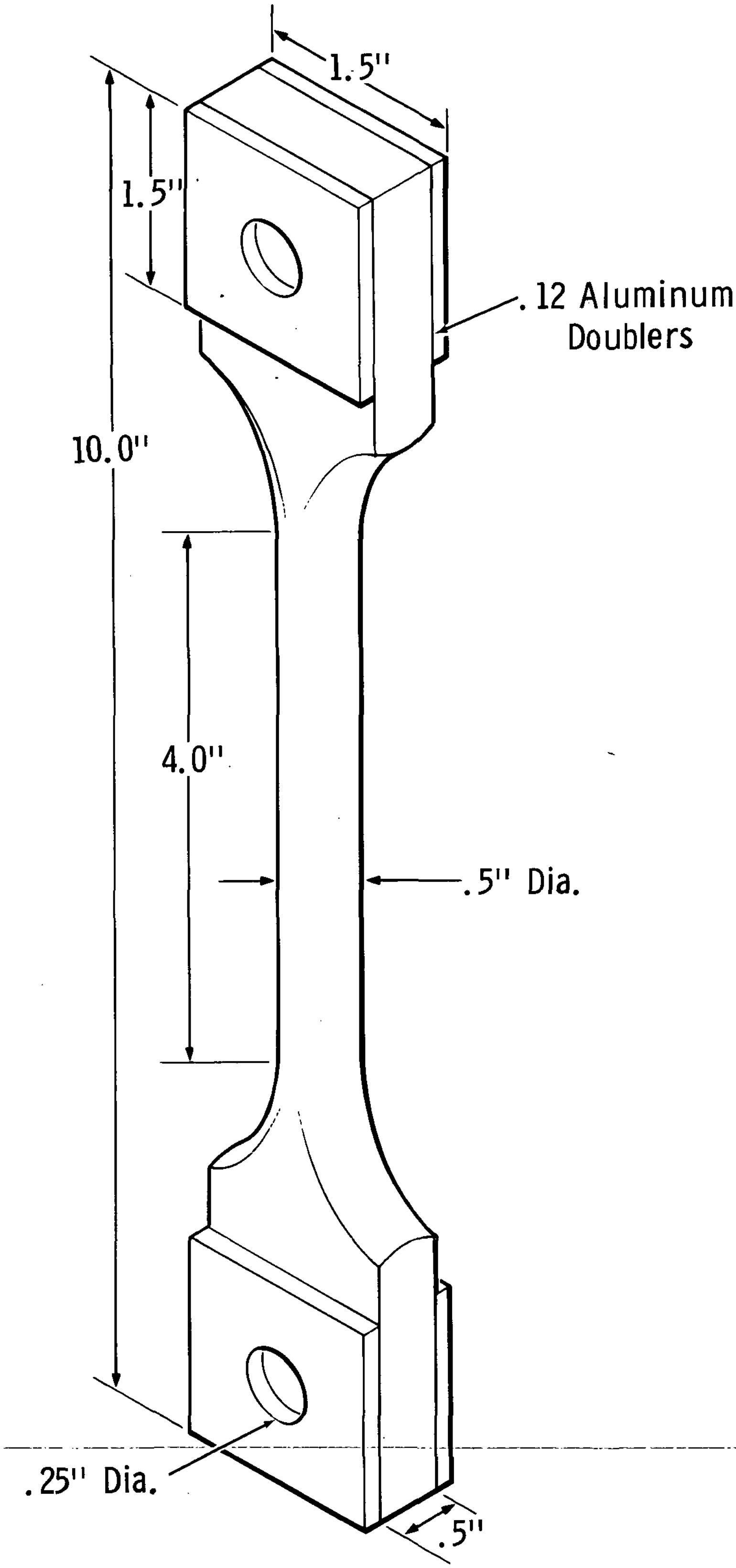

Figure 7 Round Throat Tensile Modulus Specimen

In order to determine if slippage of the extensometer occurred during test, the specimens were unloaded and the two load deformation curves compared.

In addition to the tests conducted on DC X3-6004, GE RTV-560 was tested at room temperature and -200^{o}F.

The data obtained are shown in Table IX. The modulus values obtained at room temperature are approximately one-half those obtained with the strap specimens, whereas the modulus values obtained at -175^{o}F and below are considerably greater than those obtained with the strap specimens. As can also be seen from Table IX, strain rate, particularly at -175^{o}F, has a great effect on the modulus values obtained. At the low strain rate, the relaxation of the specimen is believed to prevent accurate modulus determinations (see Table XIII for constant-strain/stress-relaxation properties).

During the preliminary test setup, it was noted that when tested at low strain rates at -200^{o}F and -270^{o}F shrinkage of the specimen occurred upon application of load. This was more noticeable with GE RTV-560 than with DC X3-6004 and is apparently the same phenomenon discussed in Section 6 of the final report.

In Table X, a comparison is shown of tensile modulus values obtained by the various test methods utilized. Tensile modulus values calculated from the slope of the initial loading curve of the constant strain or band test specimens are included in this table. With the exception of the room temperature modulus values, the highest values were obtained from the round throat specimens followed by the band specimens and the strap specimens. In addition to the accuracy of measurement, it also appears that the specimen geometry has a great effect on measured modulus values.

3.2.6 Poisson's Ratio

Poisson's ratio was determined from measurements taken during the tensile modulus testing of the strap specimens and during compression modulus testing. Details of the measurement techniques and calculations are contained in the final report.

The data obtained, Table XI, show that the Poisson's ratio of DC X3-6004 is close to 0.5, which is similar to the ratio of other adhesives tested under the basic contract. Values of Poisson's ratio computed using measured moduli and the assumption

TABLE IX

TENSILE MODULUS OF DC X3-6004 USING ROUND THROAT SPECIMENS

TEMPERATURE & SPECIMEN NO.	STRAIN* RATE, in/in/min	DC X3-6004	RTV-560
R T			
1	0.25	52	168
2	0.25	63	170
3	0.25	58	162
4	0.25	52	
5	0.25	55	
AVG		56	167
-175°F			
1	0.025	$10.1\text{x}10^3$	
2	0.025	$10.3\text{x}10^3$	
AVG		$10.2\text{x}10^3$	
-175°F			
1	0.25	$136.2\text{x}10^3$	
2	0.25	$44.75\text{x}10^3$	
3	0.25	$74.53\text{x}10^3$	
4	0.25	$126.2\text{x}10^3$	
5	0.25	$144.3\text{x}10^3$	
AVG		$105.2\text{x}10^3$	
-200°F			
1	0.005	$583.7\text{x}10^3$	
2	0.005	$790.8\text{x}10^3$	
3	0.005	$597.4\text{x}10^3$	
4	0.005	$695.8\text{x}10^3$	
5	0.05	$728.3\text{x}10^3$	$587.9\text{x}10^3$
6	0.05		$854.7\text{x}10^3$
7	0.05		$614.8\text{x}10^3$
AVG		$679.2\text{x}10^3$	$685.8\text{x}10^3$
-270°F			
1	0.05	$687.9\text{x}10^3$	
2	0.05	$959.3\text{x}10^3$	
3	0.05	$758.1\text{x}10^3$	
4	0.05	$806.5\text{x}10^3$	
5	0.05	$777.7\text{x}10^3$	
AVG		$797.9\text{x}10^3$	

* Strain rate based on elongation in 4 inch round section of specimen (Figure 7).

Table X

COMPARISON OF TENSILE MODULUS VALUES OF DC X3-6004
OBTAINED USING TEST SPECIMENS OF VARIOUS SHAPES

Temperature	Strap Specimen	Round Throat Specimen	Band Specimen
R.T.			
High	103	63	111
Low	85	52	105
Avg.	96	56	108
$-175^{o}F$			
High	$102.0x10^3$	$144.3x10^3$	$49.9x10^3$
Low	$2.1x10^3$	$44.7x10^3$	$38.4x10^3$
Avg.	$40.4x10^3$	$105.2x10^3$	$45.6x10^3$
$-200^{o}F$			
High	$94.7x10^3$	$790.8x10^3$	$510.5x10^3$
Low	$46.3x10^3$	$583.7x10^3$	$397.5x10^3$
Avg.	$67.6x10^3$	$679.2x10^3$	$467.2x10^3$
$-270^{o}F$			
High	$112.4x10^3$	$959.3x10^3$	$629.0x10^3$
Low	$73.0x10^3$	$687.9x10^3$	$497.2x10^3$
Avg.	$93.3x10^3$	$797.9x10^3$	$564.0x10^3$

TABLE XI
POISSON'S RATIO IN TENSION AND COMPRESSION
OF DC X3-6004

STRAIN RATE, in/in/min	TEMPERATURE, °F								
	550	350	300	R T	-65	-150	-175	-200	-270
	TENSION								
0.4	0.380	0.412	0.428	0.416	0.436	0.558			
0.4	0.450	0.434	0.419	0.445	0.448	0.507			
0.4	0.429	0.429	-	0.430	0.417	0.842			
AVG	0.420	0.425	0.424	0.430	0.434	0.636			
0.02							0.577	1.193	1.231
0.02							1.220	2.204	0.744
0.02							0.288	1.056	0.096
AVG							0.695	1.484	0.690
	COMPRESSION								
0.4		0.608	0.686	0.687	0.659	0.810			
0.4		0.659	0.706	0.715	0.649	0.757			
0.4		0.654	0.666	0.698	0.686	0.768			
AVG		0.640	0.686	0.700	0.665	0.778			
0.1							0.749		
0.1							0.892		
0.1							0.981		
AVG							0.874		
0.01								0.175	
0.05								0.412	
0.05								0.100	
AVG								0.229	
0.007									1.687
0.007									1.722
0.007									0.308
AVG									1.239

of perfectly elastic behavior in an isotropic continuum $G = \frac{E}{2(1+\mu)}$ are shown in Table XII.

Examination of the data shows that at very low temperatures, the apparent stiffness increases suddenly by several orders of magnitude and volume decreases (strain of negative magnitude in both length and width). This behavior suggests that the material is complex and that residual material stresses caused by freezing are released when load is applied. The observed strains are a composite of the strains caused by the externally applied loads and the strains caused by release of internal (thermal) stresses.

There is also evidence that the material is complex at temperatures above freezing. As shown, DC X3-6004 is stiffer in compression than in tension. This behavior is analogous to that of a reinforced material where tension loads are borne by the reinforcement while compression loads are borne by all parts of the material. Since there is more material effective in compression, the stiffness is greater.

In summary, data indicate that the material is a complex mechanical arrangement of membranes and is not perfectly elastic, isotropic, and homogeneous. Also, the apparent complexity of this material, the drastic change in properties at cryogenic temperatures, and, particularly, the apparent closeness of Poisson's ratio to 0.5 indicate that analysis techniques based on theories of elasticity are not advisable.

3.2.7 Constant-Strain/Stress-Relaxation

Stress-relaxation tests were conducted using molded band specimens with an inside diameter of 4 inches and a cross section dimension of 0.125 inch thickness by 0.25 inch width.

The specimens were subjected to strains of 0.1 in./in. (10% elongation) at room temperature and -175°F and 0.004 in./in. (0.4% elongation) at -200°F and -270°F. Tests were conducted using a Scott 2K test machine, and stress was continuously recorded until relaxation was complete. During initial loading of the specimens, a strain rate of 0.4 in./in./min. was used at room temperature and -175°F, and a rate of 0.005 in./in./min. was used at -200°F and -270°F. Three specimens were tested at each temperature.

Constant strain test results are shown in Table XIII. Stress relaxation versus time for a typical specimen at each temperature

TABLE XII

POISSON'S RATIO COMPUTED FROM AVERAGE TENSILE, COMPRESSION, AND SHEAR MODULI*
DC X3-6004

TEMPERATURE, ^{o}F	TENSILE MODULUS	COMPRESSION MODULUS	SHEAR MODULUS	POISSON'S RATIO TENSION	POISSON'S RATIO COMPRESSION
550	29	--	5	1.900	--
350	89	105	36	0.236	0.458
300	92	145	41	0.122	0.768
RT	96	178	51	- 0.059	0.745
- 65	115	228	56	0.027	1.036
-150	3686	470	117	14.752	1.009
-175	40449	897	17.2×10^3	0.176	-0.974
-200	67627	83410	194.3×10^3	- 0.826	-0.785
-270	93270	205735	108.9×10^3	- 0.572	-0.055

$* \; G = \dfrac{E}{2(1+\mu)}$ where: G = Shear Modulus
E = Tensile or Compression Modulus
μ = Poisson's Ratio

TABLE XIII

SUMMARY OF CONSTANT STRAIN TESTS ON BAND SPECIMENS
psi

TIME \ COND. & SPEC. NO.	10%E @ RT			10%E @ -175°F			0.4%E @ -200°F			0.4%E @ -270°F		
	19	20	21	22	23	24	25	26	27	28	29	30
Initial	14.2	13.3	13.3	561.4	588.0	650.9	1632	1915	1947	2174	2250	1982
15 sec	13.2	12.0	12.3	131.6	184.6	140.4	1491	1791	1807	2160	2250	1974
30	13.2	12.0	12.0	82.5	116.2	117.5	1407	1662	1693	2148	2250	1965
45	12.4	11.5	12.0	77.2	94.0	112.3	1370	1573	1596	2139	2248	1956
60	12.4	11.5	12.0	75.4	92.3	105.3	1365	1513	1526	2125	2246	1947
75	-	-	-	-	-	-	-	1477	1474	2118	2245	1939
90	12.4	11.5	12.0	73.7	90.6	100.0	1333	1419	1435	2104	2243	1935
105	-	-	-	-	-	-	-	-	-	2096	2241	1933
120 sec	12.4	11.5	11.8	70.2	85.5	96.5	1316	1393	1421	2087	2239	1926
15 min	12.4	11.3	11.1	43.9	51.3	50.9	929.8	1034	1070	2070	2232	1912
30	12.4	11.2	11.1	40.4	49.6	43.9	736.8	888.9	926.3	2052	2232	1904
45	11.5	11.0	11.1	35.1	47.9	42.1	635.1	829.1	798.2	2021		1833
60	11.5	10.8	10.6		46.2	38.6	578.9	760.7	736.8	2009		1786
75	11.5	10.6	10.4		44.4	35.1	526.3	726.5	684.2	1991		1754
90	11.5	10.4	10.4		42.7		508.8	714.5	649.1	1969		1740
105	11.5	10.4	10.3				487.7	700.9	628.1	1957		1737
120	11.0	10.3	10.3				452.6	687.2	614.0	1953		
135	11.0	10.3					447.4	666.7	605.3	1948		
150							438.6	649.6	596.5	1930		
165									587.7			
180									578.9			
195												
210												
225												
240												
300												
360 min	11.0	10.3	10.3	35.1	42.7	35.1	438.6	649.6	578.9	1930	2232	1737
% Stress Relaxation	22.5	22.7	23.1	93.8	92.7	94.6	73.1	66.1	70.3	11.2	0.8	12.4

is shown in Figure 8. In general, the greatest stress relaxation occurs during the first two minutes following loading and is essentially complete one hour after loading. The greatest percent relaxation occurs around the glass transition temperature of the material--90 percent relaxation at -175^{o}F and 70 percent relaxation at -200^{o}F as compared with 22 percent at room temperature and 12 percent at -270^{o}F. The data obtained for DC X3-6004 is similar to that obtained for the other four adhesives tested under the basic contract.

3.3 THERMAL PROPERTIES

The thermal properties of DC X3-6004 adhesive were determined using laboratory procedures described in the final report for thermal expansion, thermal conductivity, and specific heat. However, a test procedure is described herein for conducting thermogravimetric analysis, which was not reported in the final report. This was an added test to obtain comparative weight loss versus temperature data on all five adhesives.

3.3.1 Thermal Expansion

The thermal expansion properties of DC X3-6004 adhesive were determined with a Perkin Elmer TMA apparatus over the temperature range of -275^{o}F to 600^{o}F. The size of the specimen tested, the techniques used, and the data reduction methods were as described in the final report. The resulting data are shown in Table XIV and are graphically displayed in Figure 9.

The resulting values for the thermal expansion and expansivity for DC X3-6004 adhesive are somewhat higher than that for RTV-560 and RL-1973 sponge adhesives, but they are a little lower than that for the SLA-561 adhesive. Furthermore, the glass transition point is between -150^{o} and -200^{o}F and is shown graphically in Figure 9 to be at -175^{o}F. This graphical display shows thermal degradation starting at about 550^{o}F, and the value for expansion is shown as a dotted line above 550^{o}F.

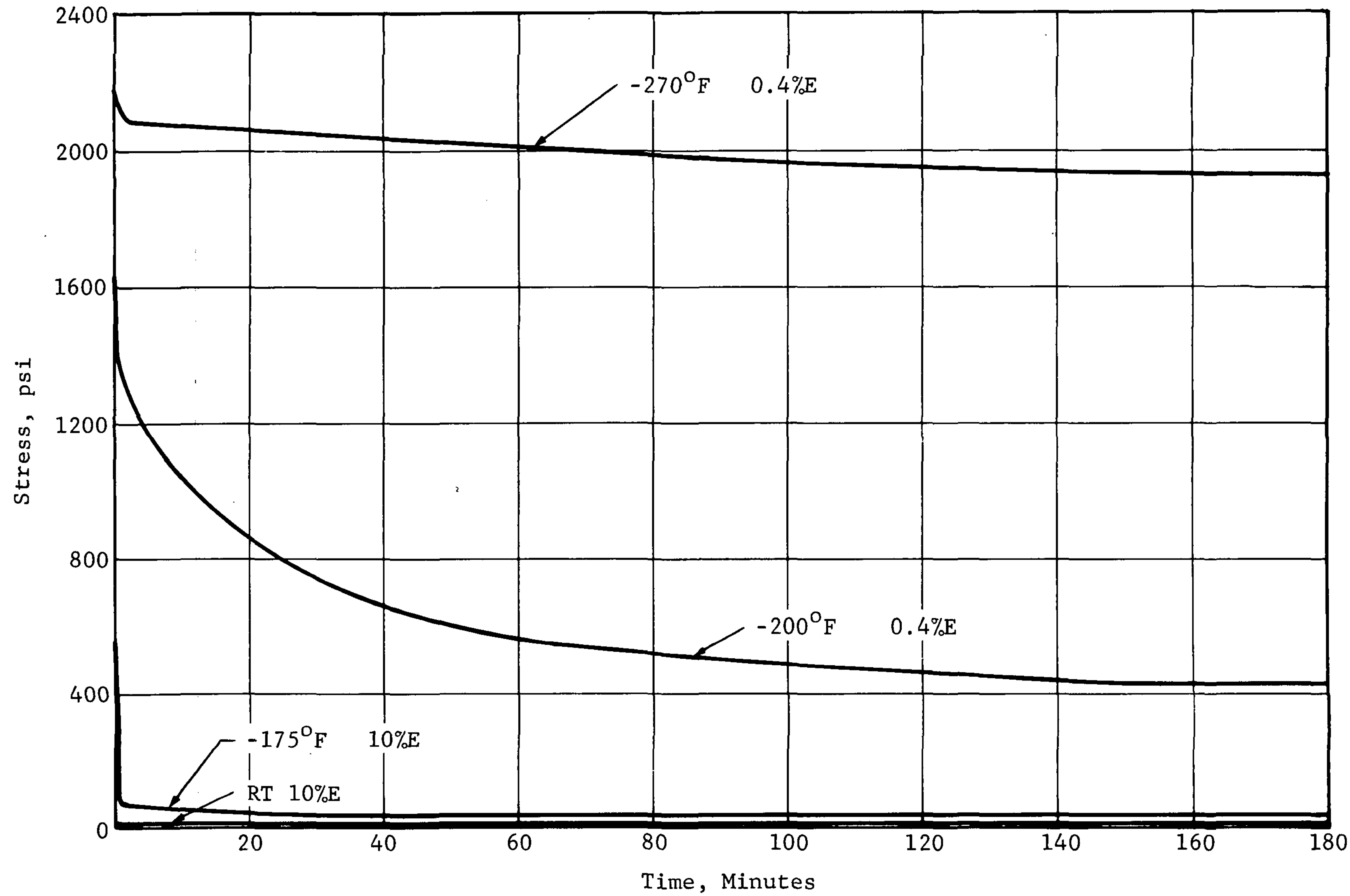

Figure 8 Stress Relaxation at Constant Strain, DC X3-6004

TABLE XIV

SUMMARY OF LINEAR THERMAL EXPANSION, DC X3-6004 (NORMALIZED TO 75°F)

INSTRUMENT: PERKIN ELMER CORP., MODEL TMS-1
PROCEDURE: ASTM E-228, PROCEDURE B

Temperature, °F	Expansion $\Delta L/L_{75°F}$, inch/inch	Expansivity* $\alpha_{75° \text{ to } T°F}$, inch/inch°F
-275	-3.825×10^{-2}	10.93×10^{-5}
-250	-3.719	11.44
-225	-3.617	12.06
-200	-3.533	12.84
-175	-3.406	13.62
-150	-3.029	13.46
-125	-2.712	13.56
-100	-2.399	13.71
- 75	-2.073	13.82
- 50	-1.707	13.66
- 25	-1.374	13.74
0	-1.025	13.66
25	-0.663	13.26
50	-0.353×10^{-2}	14.12×10^{-5}
75	0	0
100	0.369×10^{-2}	14.76×10^{-5}
125	0.686	13.72
150	1.058	14.10
175	1.427	14.27
200	1.746	13.97
225	2.080	13.87
250	2.429	13.88
275	2.785	13.93
300	3.072	13.65
325	3.369	13.48
350	3.662	13.32
375	3.956	13.19
400	4.208	12.94
425	4.511	12.89
450	4.795	12.79
475	5.040	12.60
500	5.245	12.34
525	5.471	12.16
550	5.798	12.21
575	7.496	15.78
600	10.201×10^{-2}	19.43×10^{-5}

$*\alpha_{T1 \text{ to } T2} = (\Delta L/L)_{T2} - (\Delta L/L)_{T1} \div T2-T1$

example: $\alpha_{0 \text{ to } 350°F} = [(3.662)-(-1.025)] \times 10^{-2} \div 350$
$= 13.39 \times 10^{-5}$ inch/inch°F

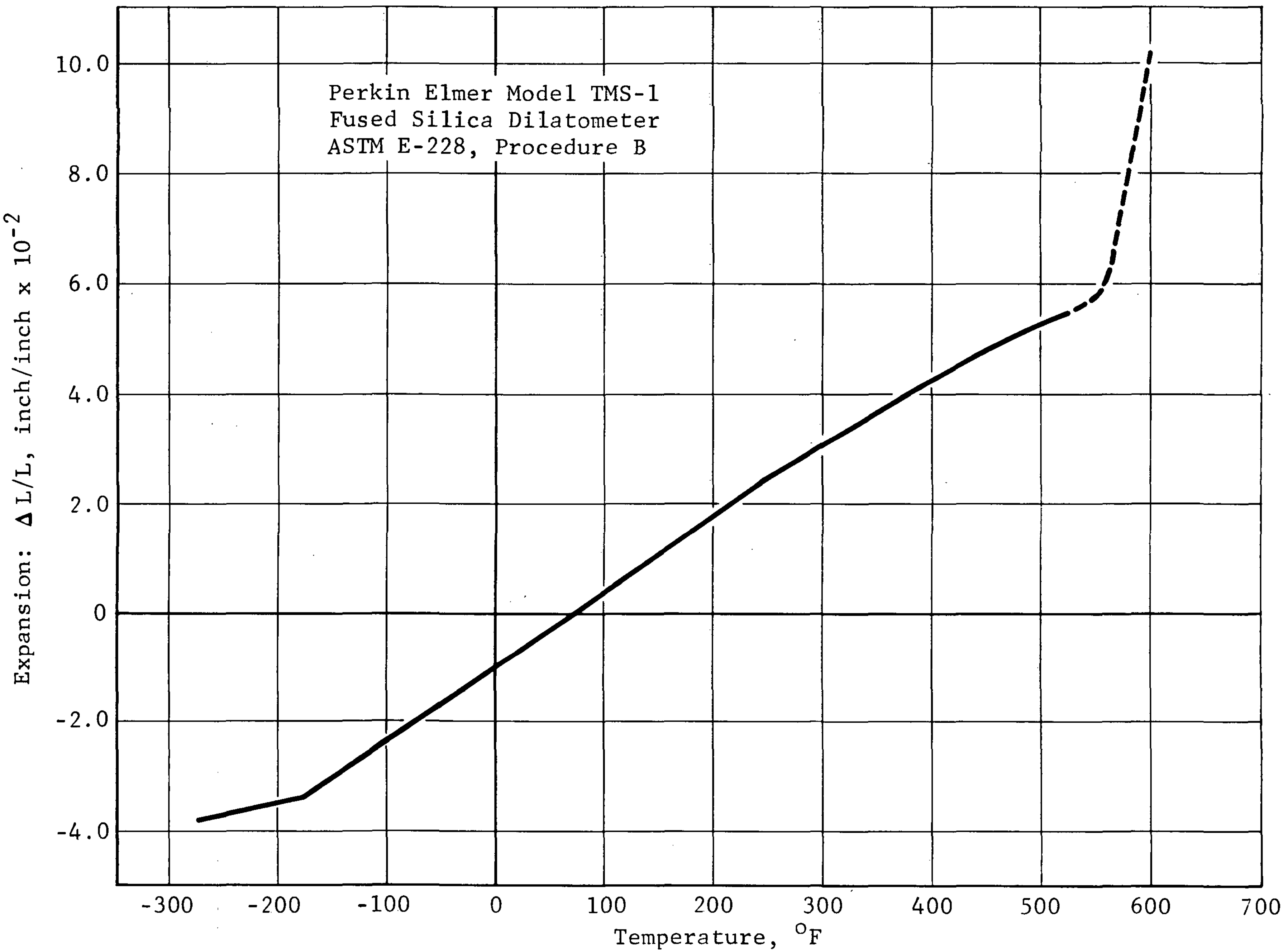

Figure 9 Linear Thermal Expansion Vs. Temperature, DC X3-6004

3.3.2 Thermal Conductivity

Thermal conductivity was measured per ASTM C-177 on the DC X3-6004 adhesive over a mean temperature range of -238°F to 485°F. The cold side temperature, hot side temperature, the mean temperature, and the resultant value of K are shown in Table XV. These test results are lower than those obtained earlier for RTV-560, but they are somewhat higher than the conductivity values for DC 93-046, SLA-561, and RL-1973 adhesives. A graphic representation of thermal conductivity is presented in Figure 10.

3.3.3 Specific Heat

The value for specific heat of DC X3-6004 was determined on the Perkin Elmer DSC instrument exactly as described in the final report. Values were obtained over a temperature range of -130°F to 550°F. Specific heat data are reported in Table XVI, and the values for specific heat of DC X3-6004 were of the same order of magnitude as those reported earlier for the other three adhesive systems.

3.3.4 Thermogravimetric Analysis

The Perkin Elmer TGS-1 is a thermogravimetric analyzer used to measure the weight of a sample as a function of its temperature or as a function of time at a constant temperature. Samples weighing from 0.1 to 200 mg are suspended from an electrobalance sensitive to 0.01 microgram. A low mass furnace is placed around the suspended sample, and temperatures from 75 to 1800°F can be programmed at rates from 0.56° to 280°F per minute. Analyses may be made in various atmospheres at normal or reduced pressures.

Applications of thermogravimetric analysers include qualitative and quantitative analyses, kinetic studies of decomposition reactions, determinations of thermal stability, studies of absorption phenomena, and quantitative measurements of moisture and residual solvent content.

To obtain the thermogravimetric analysis data reported herein, a thermal degradation profile was determined for each of the five adhesive materials. Samples weighing approximately 5 mg were heated at a rate of 36°F per minute from 75 to 1382°F in a nitrogen atmosphere. One comparison analysis was conducted in air. Sample

Table XV

THERMAL CONDUCTIVITY TEST RESULTS

Sample DC X3-6004 Procedure ASTM C-177

Thickness 0.25"

Average Cold Side Temp. °F	Average Hot Side Temp. °F	Mean T. °F	K $\frac{\text{BTU-in.}}{\text{°F-ft}^2\text{-hr.}}$
-308	-268	-288	0.51
-301	-243	-272	0.58
-281	-190	-236	0.95
-256	-161	-209	1.42
-151	+ 54	- 49	1.56
- 14	+ 20	+ 3	1.59
+ 81	+188	+135	1.59
+187	+299	+243	1.53
+281	+501	+391	1.44
+446	+524	+485	1.40

Table XVI

SUMMARY OF SPECIFIC HEAT VS TEMPERATURE
DC X3-6004

Temperature, °K	°F	Specific Heat, Calories/gram°K
183	-130	0.22
219	- 65	0.24
300	80.6	0.32
442	300	0.35
450	350	0.35
561	550	0.37

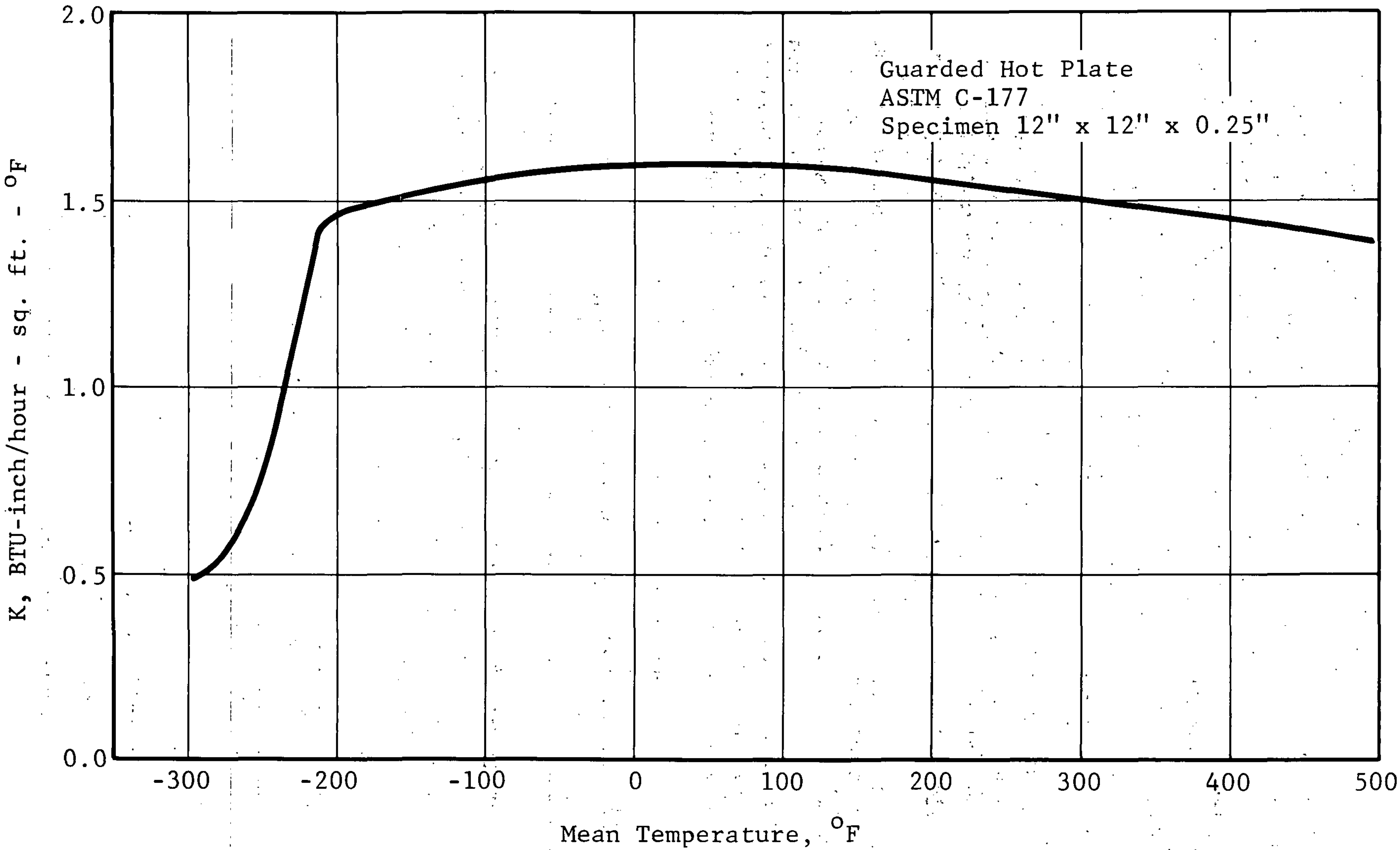

Figure 10 Thermal Conductivity Vs. Mean Temperature, DC X3-6004

weight changes were recorded at a sensitivity producing 25.4 cm of chart displacement for a 0.4 mg weight loss. Observations of four thermal events were recorded; these events were the temperature at which the initial loss of weight began, the temperature at the onset of the major weight loss indicating the beginning of thermal decomposition, the temperature at which the sample ceased to lose weight, and the percent total weight loss. Since no thermogravimetric data were obtained on the three adhesives for the final report, all five adhesives including DC X3-6004 were tested. The resulting data are summarized and compared in Table XVII.

It was observed that there were two distinct weight loss rates for each of the adhesives tested. When each sample was increased in temperature at the start of each test, no weight was lost until a certain temperature was reached. (This is reported in Table XVII as "Temperature of Initial Weight Loss.") As the temperature was increased further weight was lost at a rather low rate until a second temperature was reached. (This temperature is identified in the table as "Temperature at Start of Major Weight Loss.") Above this temperature, weight was lost at a much higher rate in terms of milligrams per degree. The total percent weight loss observed at the end of the test at the maximum temperature is presented in the last right-hand column in Table XVII.

3.4 EFFECTS OF THERMAL CYCLING ON MECHANICAL PROPERTIES

Tensile strength, tensile modulus, ultimate elongation, adhesion in tension, shear strength, and shear modulus were determined at room temperature before and after exposure to the following thermal cycles:

1. R.T. (77^{o}F), -290^{o}F., R.T.
2. R.T., 500^{o}F, R.T.
3. R.T., -290^{o}F, 500^{o}F, R.T.
4. R.T., 350^{o}F, R.T.
5. R.T., -290^{o}F, 350^{o}F, R.T.

Table XVII THERMOGRAVIMETRIC ANALYSIS OF FIVE RTV SILICONE ADHESIVES

Perkin-Elmer TGS-1, 77 to 1382°F at 36°F/Min.

Adhesive	Atmosphere	Temp. of Initial Weight Loss	Temp. at Start of Major Wt. Loss	Temp. Final Wt. Loss	Total Wt. Loss, %
DC X3-6004	N_2	329°F	815°F	1067°F	61.2
DC X3-6004	Air	329	797	1067	60.3
MMC-SLA-561	N_2	329	689	1094	30.1
DC 93-046	N_2	311	815	1031	77.3
RL-1973 Sponge	N_2	293	797	923	37.7
RTV-560	N_2	338	887	1247	52.2

Time at temperature was five minutes, and three specimens were tested at each condition. Specimens were allowed to return to room temperature before a subsequent temperature exposure.

Adhesion-in-tension specimens (cylinder adhesion) were two aluminum cylinders 1.125 inches in diameter bonded together with a 0.06-inch thickness of DC X3-6004. Load was applied perpendicular to the cylinder faces. Band specimens, as described previously, were used to determine tensile properties. Shear modulus was determined on double overlap shear specimens loaded in torsion to approximately 25 percent of failing load. These specimens were then loaded to failure in tension to determine ultimate shear strength. Complete details of the test procedure are contained in the final report.

As shown in Table XVIII, the only effects resulting from thermal cycling occurred in those cycles requiring exposure to 500°F.

In the cylinder adhesion test, a decrease in both tensile strength and elongation occurred after the 500°F exposures whereas with the band specimens only a decrease in modulus was observed. In the shear tests, both a decrease in shear strength and modulus was noted.

The test adhesive in the cylinder adhesion test and shear tests is confined between aluminum. It was noted during testing of DC X3-6004, that when the material is exposed to elevated temperatures in a confined condition considerably more degradation occurs than when it is exposed to air during exposure. This accounts for the decrease in cylinder adhesion tensile strength and shear strength as compared with the minor decrease in tensile strength evidenced by the band specimens.

Table XVIII EFFECTS OF THERMAL CYCLING ON DC X3-6004

Environmental Conditioning & Specimen No.	Cylinder Adhesion			Band Specimens			Double Lap Shear		
	Tensile, psi	Elongation, %	Cohesive Failure,%	Tensile, psi	Elongation, %	Tensile Modulus,psi	Shear, psi	Cohesive Failure,%	Shear Modulus,psi
Control									
1	152	176	99	116	175	108	106	100	49
2	161	176	100	114	177	106	108	100	41
3	143	160	100	151	231	118	111	100	42
Avg	152	170	100	127	194	111	109	100	44
RT, -290°F, RT									
1	151	186	100	112	165	103	106	100	53
2	169	183	100	148	218	110	96	100	51
3	140	167	100	153	239	113	92	100	49
Avg	153	179	100	137	207	109	98	100	51
RT, 500°F, RT									
1	10	84	100	119	207	71	19	100	17
2	11	115	100	117	191	82	21	100	20
3	30	88	100	129	211	79	21	100	17
Avg	17	96	100	121	203	77	20	100	18
RT, -290°F, 500°F, RT									
1	28	53	100	109	193	66	22	100	21
2	32	38	100	123	224	71	19	100	18
3	Failed During Handling			115	206	69	22	100	19
Avg	30	46	100	116	208	69	21	100	19
RT, 350°F, RT									
1	148	184	100	133	209	100	91	100	39
2	148	190	100	111	174	91	106	100	33
3	156	189	100	148	233	106	102	100	43
Avg	151	188	100	131	205	99	100	100	38
RT, -290°F, 350°F, RT									
1	156	193	100	121	221	100	85	100	42
2	169	208	100	131	206	103	100	100	38
3	149	172	100	114	182	98	98	100	38
Avg	158	191	100	122	203	100	94	100	39

SECTION 4

CONCLUSIONS AND RECOMMENDATIONS

4.1 CONCLUSIONS

The conclusions drawn from the results of this supplemental program are presented below.

1. At temperatures above its glass transition point of -175°F, DC X3-6004 exhibits modulus values significantly lower than the four silicone adhesives characterized under the basic contract, but at -175°, -200°, and -270° its modulus is approximately the same as the other four silicone adhesives.

2. Strength properties of DC X3-6004 are lower than those of the other three solid silicone adhesives tested and reported in the final report of the basic contract, but its adhesion to 2024 T81 aluminum is adequate to compete as an adhesive for attaching reusable surface insulation.

3. DC X3-6004 is not as thermally stable as the other phenyl silicone adhesives tested in the program. Specimens thermally cycled to 500°F lost 80 to 90 percent of their room temperature adhesive strength. The band-shaped specimens in the same thermal cycle test lost only 5 to 10 percent of their strength. This difference is indicative of the thermal reversion tendency of X3-6004 when subjected to 500°F in a confined area such as a metal-to-metal overlap joint.

4. Constant-strain/stress-relaxation tests show that DC X3-6004 behaves similar to the other silicone adhesives. At its glass transition temperature (-175°F), DC X3-6004 loses over 90 percent of the stress required to hold a constant 10 percent strain, whereas at room temperature, stress relaxation is only 22 to 23 percent. At -200° and -270°F, specimens could only be strained 0.4 percent. Stress-relaxation was approximately 70 percent at -200° and 12 percent at -270°F.

5. Thermal expansion and specific heat properties of DC X3-6004 fall in line with those of the other silicone adhesives. However, its thermal conductivity falls in between that of General Electric's RTV-560 and the other three adhesive systems.

6. The new test procedure developed in this program for determining tensile modulus using molded "dog bone" shaped specimens and an Instron extensometer gives modulus values approximately one half those obtained with the strap specimens at room temperature. In contrast, at -175^{o}F and below, the new method resulted in considerably higher modulus values.

4.2 RECOMMENDATIONS

Findings brought to light by tests conducted in this supplemental program and data obtained and observation made during work on the basic contract prompt the following recommendations.

1. Observations of room temperature vulcanizing silicone rubber specimens being subjected to loading at temperatures below their glass transition points have revealed that specimens do not yield uniformly. The yielding apprently occurs at stress points in the cold stiffened specimens, and the specimen softens at these points. This causes anomalies to occur in other areas of the specimen; sometimes even shrinkage occurs. The specimen no longer acts as a homogeneous material. This phenomenon was discussed in Section 6 of the final report. Further investigations should be made of this phenomenon in order to provide a better understanding and explanation for designers who use these materials at extremely low temperature.

2. DC X3-6004 possesses many desirable properties that make it a candidate for attaching reusable surface insulation; however, it is deficient in resistance to reversion at temperatures above 350^{o}F. It is recommended, therefore, that the manufacturer, Dow Corning Corporation, develop curing catalysts for the copolymer base that will mitigate this undesirable characteristic.

APPENDIX

QUALITY CONTROL DOCUMENT

The quality control document shown here was received with the DC X3-6004 tested in this program. It certifies that the material complies with all applicable specification requirements and that test reports are on file at the vendor's facility.

DOW CORNING CORPORATION, MIDLAND, MICHIGAN 48640, U.S.A.

SHIP TO

D.C. ORDER NO. → DAE8511342

CUSTOMER ORDER NO.
522756

3094707
GENERAL DYNAMICS CORP
STRS GRP 172 X SHED C
FT WORTH ,TX 76101
A E MCDONALD BG 80

CERTIFICATE OF COMPLIANCE

IT IS HEREBY CERTIFIED THAT THE ARTICLES LISTED BELOW COMPLY WITH ALL APPLICABLE SPECIFICATION REQUIREMENTS.

TEST REPORTS ARE ON FILE SUBJECT TO EXAMINATION.

BY A B Lewis

A. B. LEWIS
MANAGER. CORPORATE QUALITY CONTROL

QUANTITY	CONTAINER SIZE	PRODUCT DESCRIPTION	DOW CORNING LOT NO.	FREIGHT CLASS	WARNING CODE
000040	LB	8032055 X3-6004 ADHESIVE			
		PKGD AS 36.4 LB BASE & 3.64 LB			
		CAT (CAT. IN 2 x 1.82 lb cans)			
	EA	~~0001300~~ TRANSPORTATION ALLOWANCE			

VC
Record
4B
T-4089
158
2
11-3
2 Ctn

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DOW CORNING CORPORATION
MIDLAND, MICHIGAN

BY R. G. VanSumeren

R. G. VANSUMEREN
SUPERVISOR, CUSTOMER SERVICE

SEND ALL INQUIRIES REGARDING THIS SHIPMENT TO:
YOUR NEAREST DOW CORNING CUSTOMER SERVICE CENTER

OR TO: DOW CORNING CORPORATION
P.O. BOX 1592
MIDLAND, MICHIGAN 48640, U.S.A.

ACCOUNT NO.	PACKED BY	DATE	CHECKED BY	DATE		
			Ray	11/1/72		

CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS	CONTAINERS
	1	1		1-6 gal									
GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.	GROSS WT.
	6 gal #												
	1 ct												

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