

! N74 30935

BONDING OF REUSABLE SURFACE INSULATION  
WITH LOW DENSITY SILICONE FOAMS

BY

Dr. Arnold A. Hiltz; Ralph R. Hockridge; and  
Fred P. Curtis

Plastics and Advanced Composites Laboratory  
Re-Entry and Environmental Systems Division  
General Electric Company  
P. O. Box 8555  
Philadelphia, Pa. 19101

-----

Although the use of mechanical attachment and partial bond systems offer certain structural advantages in attaching Reusable Surface Insulation (RSI) to the Space Shuttle Structure, continuous bonding is regarded as the most reliable system. This is primarily due to the limited strength and strain capability of the high temperature ceramic materials requiring a soft foam pad material for strain isolation as part of the attachment system.

The foam bond attachment systems most likely to meet all the requirements, including both low and high temperature extremes are based on the Room Temperature Vulcanizing (RTV) silicone elastomers.

This paper describes approaches taken by GE-RESD to develop and evaluate reduced density, high reliable foamed bond strain isolation attachment systems for RSI. Included are data on virgin as well as on material that received 100 cycles of exposure to 650°F for approximately 20 minutes per cycle.

## 1.0 INTRODUCTION

### 1.1 Design

Typical areas of the space shuttle vehicle that may be covered with reusable ceramic insulation include leading edges, nose caps; windward body sections, wings, fins, and control surfaces. The maximum surface temperature requirements for each of these areas will define the applicable insulative material system to be used. Allowable primary structure or mounting panel temperatures will establish required insulation thicknesses.

The primary structure arrangements for these various regions of the vehicle include monocoque and semi-monocoque constructions with the RSI integrally attached.

In the integral panel concept (Figure 1), the insulation is bonded directly to the primary air frame structure, and all of the air loads are transmitted directly to this structure. Adhesive bonding makes direct attachment to the primary structure the most efficient approach.

The insulation may be bonded to standard structural materials such as aluminum, titanium, phenolic glass honeycomb, or super alloys such as Inconel. In the subject discussion, aluminum and titanium are considered as representative structural materials. The maximum operating temperature, including safety factors, is 350°F for aluminum and 650°F for titanium.

### 1.2 Requirements

#### 1.2.1 Mechanical

Use of essentially continuous attachment is deemed necessary for RSI due to the limited strength and strain capability of these high temperature ceramic materials. This negates the use of individual mechanical attachments and requires the use of a soft foam pad material for strain isolation as part of the attachment system.

Typical tensile stresses that will develop in the foam bond material system, due to the thermal gradients resulting from entry heating, are shown in Figure 2 as a function of time for several heating rates representative of a typical cross range orbiter. It may be noted from Figure 2, that the required strength is relatively low. However, the maximum stresses occur at a time period in the mission when the temperature has peaked (Figure 3).

Several material properties are critical in the design of an RSI thermal protection system. They are 1) ultimate strength in tension and shear, 2) modulus of elasticity, and 3) thermal expansion.

Orbital soak-out, particularly at the  $-250^{\circ}\text{F}$  temperature, develops high shear and tensile stresses in the bond system, since it is below its glass transition temperature.

Post-entry also imposes stresses on the bond since the RSI has become relatively cool but the bond has soaked out to the  $400\text{-}600^{\circ}\text{F}$  temperature range where strength properties are strongly influenced and reduced by temperature.

### 1.2.2 Weight

The manufacturing tolerances on the RSI and the airframe skin, and the requirements for low shear stiffness dictate a bondline thickness of at least  $0.070''$ . The densities of typical candidate adhesive systems range from about  $66$  to  $90\text{ lb/ft}^3$  which at a bondline thickness of  $0.070''$  translate into a bond weight of  $0.385$  to  $0.525\text{ lb/ft}^2$ . Reduction of the bond weight, since the bondline thickness is relatively fixed, can best be achieved by reducing its density. Fortunately, this is also the direction desired for reducing the shear stiffness of the bond. However, a decrease in density will also decrease the strength of the bond. Accordingly, a trade-off is required between bond strength and bond density.

### 1.2.3 Thermal

The adhesive system must be capable of performing its intended function for 100 missions at the maximum normal entry design bondline temperatures without excessive thermal degradation or change in properties. These temperatures are  $\sim 350^{\circ}\text{F}$  for an aluminum structure and  $\sim 650^{\circ}\text{F}$  for titanium with a factor of safety. In addition, the system must be compatible with the  $-250^{\circ}\text{F}$  temperature experienced during orbital stay. Figures 3 and 4 show typical area 1 and 2P temperature histories for various combinations of REI tile and bond thicknesses.

Area 1 and area 2P are NASA/MSC point design designations representing two extremes of shuttle heating for RSI applications. Area 1 is representative of heating which produces surface temperatures of  $1400^{\circ}\text{F}$ , and in area 2P surface temperatures of  $2300^{\circ}$  are reached.

#### 1.2.4 Space Environment

Since the space shuttle will be subjected to a hard vacuum, care must be taken to remove materials which may volatilize and recondense on colder or more polar portions of the vehicle. In the open cell silicone foam system here discussed, volatiles are removed during the post-cure cycle. In addition, the bond system must be stable to the space environment radiation.

#### 1.2.5 Ground Environment

The bond system must be stable to the effects of humidity, moisture and salt spray. It must not support the growth of fungus. It must also withstand aircraft ground handling, and be compatible with rocket and jet fuels as well as with common cleaning solvents.

### 2.0 ADHESIVE MATERIALS

Analysis of the established requirements for the RSI attachment indicate the most probable application method to be by adhesive bonding. As indicated, the use of a high shear stiffness adhesive induces high shear stress concentrations at the interface between the surface insulation and the structure, and, as a result, imposes unnecessarily high strength requirements on the bond and RSI. The use of a finite thickness flexible adhesive, on the other hand, results in the attenuation of stress concentrations and in a reduction of the shear stress requirements for both the adhesive and the insulation.

Careful evaluation of the available commercial materials with capability of meeting all the requirements led to the selection of the room temperature vulcanizing silicone rubbers (RTV). The following were selected for consideration and evaluation from the prime candidate systems indicated in Table I.

#### 2.1 PD-200 (Base)

PD-200 (Base) is a solid methyl-phenyl silicone which can be cured to a strong rubbery state at room temperature with tin soap catalysts such as T-12<sup>1</sup> or Nuocure 28<sup>2</sup>. It cures by a condensation mechanism with the elimination of volatile by-products. Venting is necessary to achieve cure in the inner portions of large bonded areas of non-porous materials.

---

<sup>1</sup> Dibutyl Tin Dilaurate, M&T Chemical Corporation.

<sup>2</sup> Tin Octoate, Tenneco Chemicals, Inc., Nuodex Division

## 2.2 PD-200

PD-200 is an open cell, methyl-phenyl silicone foam exhibiting an extremely fine cell structure which is uniformly distributed. It is formulated and chemically blown at ambient temperature and pressure utilizing open pan type molds. Initial foaming and cure requires one hour. At this point the foamed bun is slit to remove the surface skins, then step wise post-cured to 350°F over a 26 hour period to stabilize weight and dimensions. Upon completion of post cure, the foam is slit to design thickness using commercial rubber slitting equipment.

## 2.3 PD-200 (Mod)

PD-200 (Mod) is an open cell, methyl-phenyl silicone foam. In contrast to the standard PD-200, by means of proper selection of catalyst concentration and process parameters pressure, reproducible foam materials having a density range of 15 to 25 lbs/ft<sup>3</sup> have been produced. The PD-200 (Mod) is further processed (post-cured and slit) in the same manner as the standard PD-200 foam. This results in a foam system that can effect a large system weight saving over the standard PD-200.

## 3.0 BASIC ELASTOMER CHARACTERISTICS

### 3.1 PD-200 (Base)

PD-200 (Base) has been characterized by GE-RESD for use as an adhesive for re-entry vehicle application. It has also been used extensively in the production of GE Elastomeric Shield Materials (ESM).

It has excellent low temperature ( $\sim -180^{\circ}\text{F}$ ) flexural properties and high thermal stability. The ultimate strength of PD-200 (Base) as a function of temperature is shown in Figure 5. Tensile and shear moduli values as a function of temperature are shown in Figure 6, where it is seen that the modulus is relatively constant over a temperature range from 0 to +400°F.

Below -150°F, the modulus increases rapidly to the glass transition temperature of approximately -180°F. The thermal stability of the cured elastomer is shown by the thermogravimetric analysis curve of Figure 7. It is seen that PD-00 (Base) does not begin to lose weight at a significant rate until around 1100°F, and is quite stable at 600 to 650°F, the maximum bondline temperature for a titanium structure.

272<

The coefficient of thermal expansion of PD-200 (Base) is  $150 \times 10^{-6}$  in/in/ $^{\circ}$ F from  $-100^{\circ}$ F to  $600^{\circ}$ F. Its specific heat is 0.4 BTU/lb $^{\circ}$ F from  $-100^{\circ}$ F to  $600^{\circ}$ F. The thermal conductivity is shown in Figure 8.

Long term stability of the material at  $600^{\circ}$ F has been demonstrated under the SNAP-27\* Program where greater than 200 psi tensile shear strength was exhibited at  $655^{\circ}$ F on specimens aged for eight weeks at  $600^{\circ}$ F.

Cure in deep sections has been achieved by strip bonding, i.e., by leaving gaps in the bond for air entry and volatiles removal. Removal of volatiles, however, has not been a problem in the bonding of porous, low density insulation materials.

### 3.2 PD-200

PD-200 has been characterized by GE-RESO for use as a strain isolation foam system for space shuttle environment application. Its low temperature flexibility and high temperature stability are similar to the PD-200 (Base). Typical tensile and shear properties of PD-200 as a function of temperature before and after cycling are shown in Figures 9 and 10. The tensile and shear moduli values as a function of temperature are shown in Figures 11 and 12. The low temperature modulus data is shown in Figure 13. The thermal stability of the cured foam is indicated by the thermogravimetric (TGA) analysis curve of Figure 14.

The specific heat of PD-200 is shown in Figure 15, the thermal conductivity is  $2 \times 10^{-5}$  BTU/Ft-Sec- $^{\circ}$ F (Figure 16), and the thermal expansion data is shown in Figure 17. Elevated temperature stability of the PD-200 foam has been demonstrated, and is indicated by the data presented in Table II. As indicated above, the long term stability of the base material has been demonstrated in the SNAP-27 Program.

In contrast to PD-200 (Base) where cure in deep section requires the strip bonding approach, bonding of the PD-200 foam has not been a problem due to its porous nature.

### 3.3 PD-200 (Mod)

PD-200 (Mod), a recent modification of the well characterized PD-200 foam system, is currently being extensively evaluated. The PD-200 (Mod), in a density range less than 20 lb/ft $^3$ , can offer considerable advantage in weight reduction and in strain isolation. As can be seen in Figure 18, 1.0 inch of foam at 28 PCF results in a tile thickness of 1.7 inch and a weight of 4.4 lb/ft $^2$  (Curve A), while use of a 15 PCF foam (Curve B) results in a weight of 3.9 lb/ft $^2$ , a weight reduction of 0.5 lb/ft $^2$ .

\*

Space Nuclear Auxiliary Power Unit

#### 4.0 ADHESIVE APPLICATION TO RSI SYSTEMS

During the development of the GE Reusable External Insulations (REI), it was necessary to bond samples to substrates (mostly aluminum) for evaluation against the Space Shuttle Orbiter environments of:

- a) Moisture
- b) Re-entry thermal simulation at representative local pressures
- c) Orbital cold soak exposure
- d) Airframe load strain compatibility

The specimen geometry consisted of a 4" x 8" x 1" REI panel bonded to an aluminum plate. Four inches of exposed aluminum on each end served for gripping the panel in a Universal Testing Machine. Each specimen was exposed to a thermal test cycle, and some specimens were also exposed to a -170°F cold soak prior to the heat exposure. Design of the specimens was such that the stress at the center of the REI face was equivalent to the maximum that would occur on an infinite size end. Earlier tests of this nature utilized solid PD-200 (Base). These tests indicated that the bond modulus (shear stiffness) of the solid adhesive was unacceptable for REI Mullite materials.

Consequently, the adhesive was changed from a solid bond to a foam bond system utilizing PD-200 and PD-200 (Base). Subsequent testing indicated the bond modulus to be acceptable for REI Mullite as a result of the strain isolation properties of the PD-200.

The PD-200 foam bond system was applied in two stages. The foam sheet was first bonded to the REI panel with PD-200 (Base), cured, then bonded to the precleaned, primed structure. Such an assembly has successfully survived exposure to a -250°F thermal cycle environment.

## 6.0 SUMMARY AND CONCLUSIONS

The room temperature vulcanizing silicone elastomers meet all the requirements for the attachment of reusable external insulation to the space shuttle vehicle. Of these flexible silicones, PD-200 (Base), especially when used in a reduced density configuration, such as PD-200 or PD-200 (Mod), could fulfill all the requirements. The adhesive systems based on PD-200 are usable up to approximately 650°F. Reduction of the density by chemical foaming provides a reduced weight and a lower modulus strain isolation bond system offering considerable advantage for the shuttle reusable surface insulation TPS.

### ACKNOWLEDGEMENT

The authors are indebted to Jim Brazel for the thermal property data, to Dave Lowe for the mechanical property measurements, and to Jim Kreitz, Jr., for his bonding development contributions.

Portions of the work reported in this paper were sponsored by NASA Contracts NAS 1-10533 and NAS 9-12084.



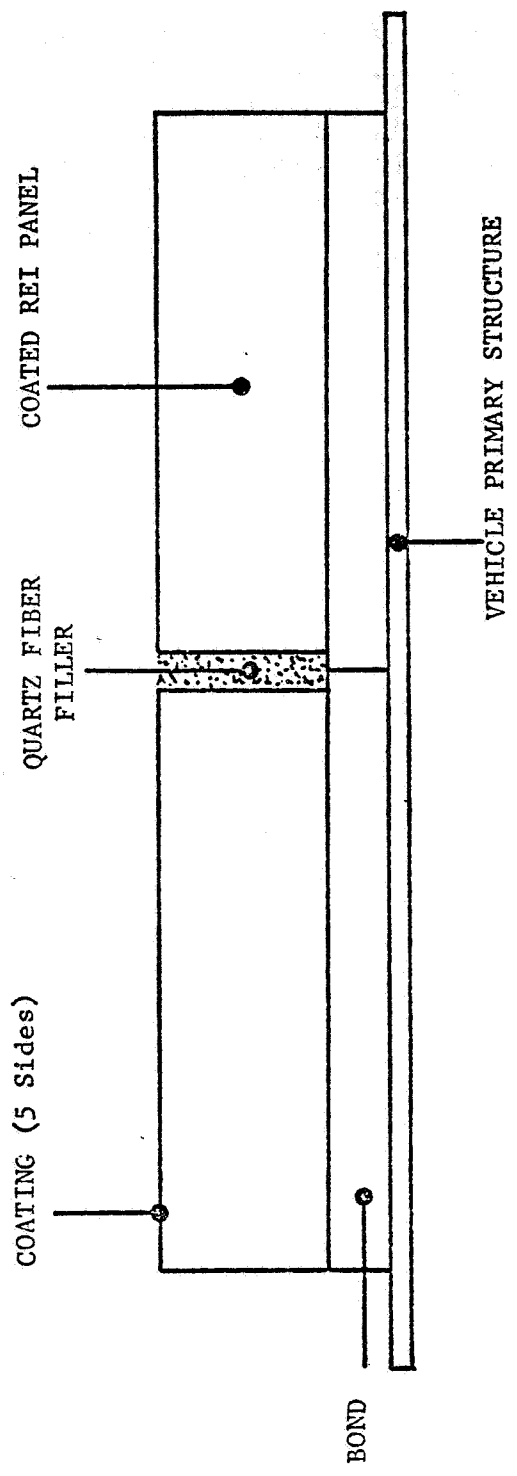
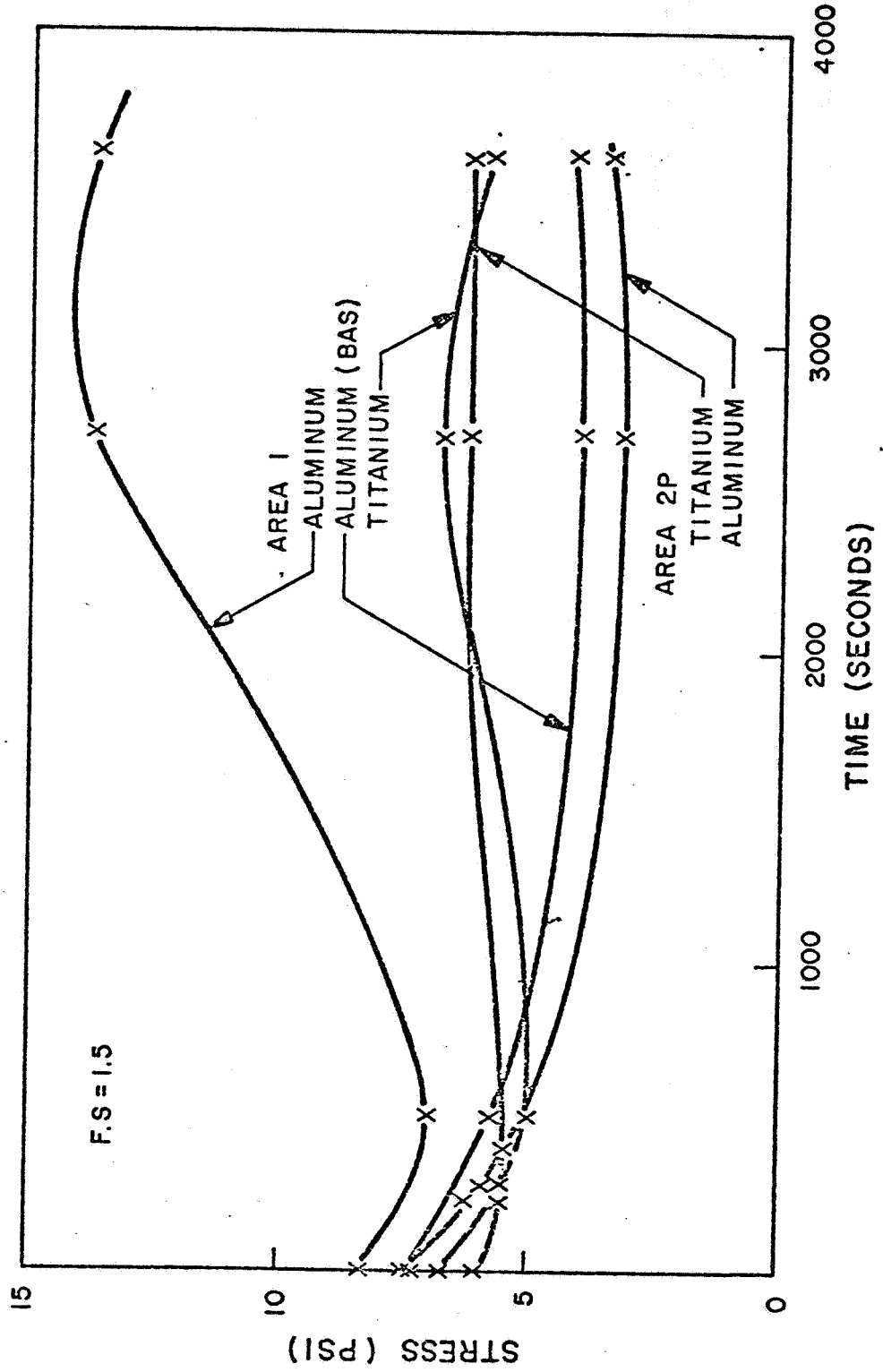


FIGURE 1  
TPS INSTALLATION

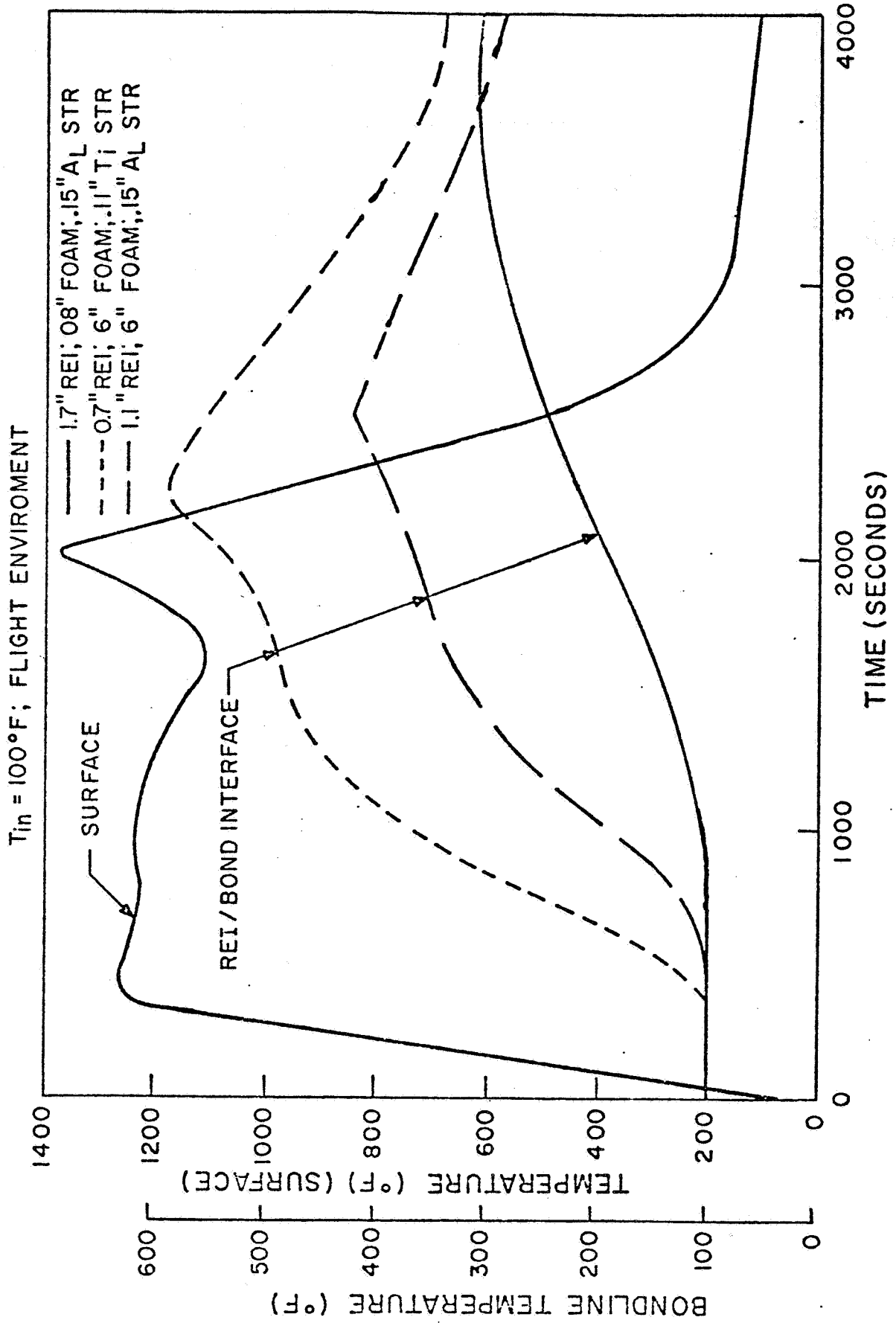
# BOND TENSILE STRESS HISTORIES

FIG. 2



# AREA I PROTOTYPE PANEL TEMPERATURE HISTORIES

FIG. 3



# AREA 2P PROTOTYPE PANEL TEMPERATURE HISTORIES

$T_{in} = 100^\circ F$ ; FLIGHT ENVIRONMENT

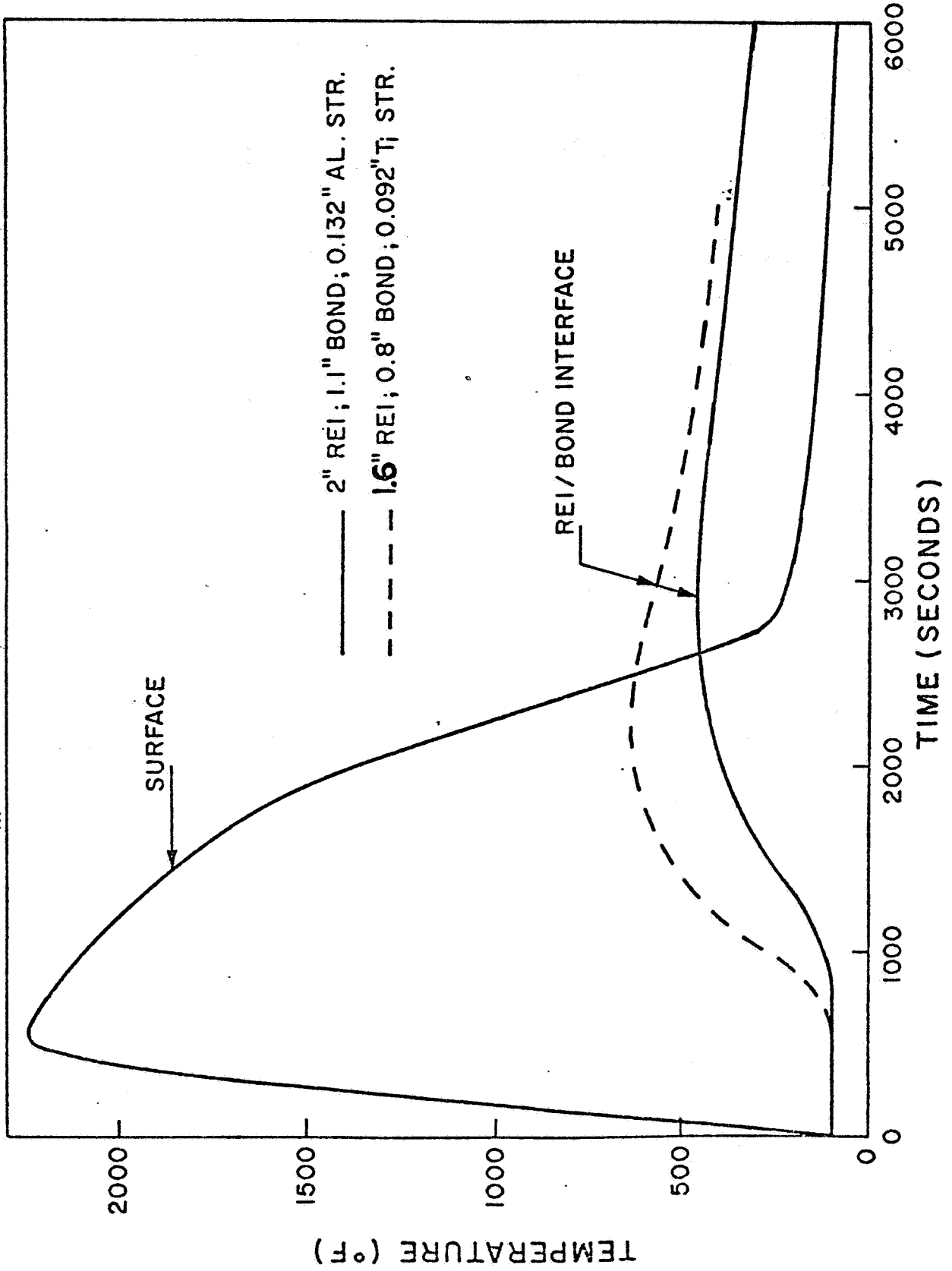


FIG. 4

FIGURE 5

ULTIMATE STRENGTH OF PD-200 (BASE)

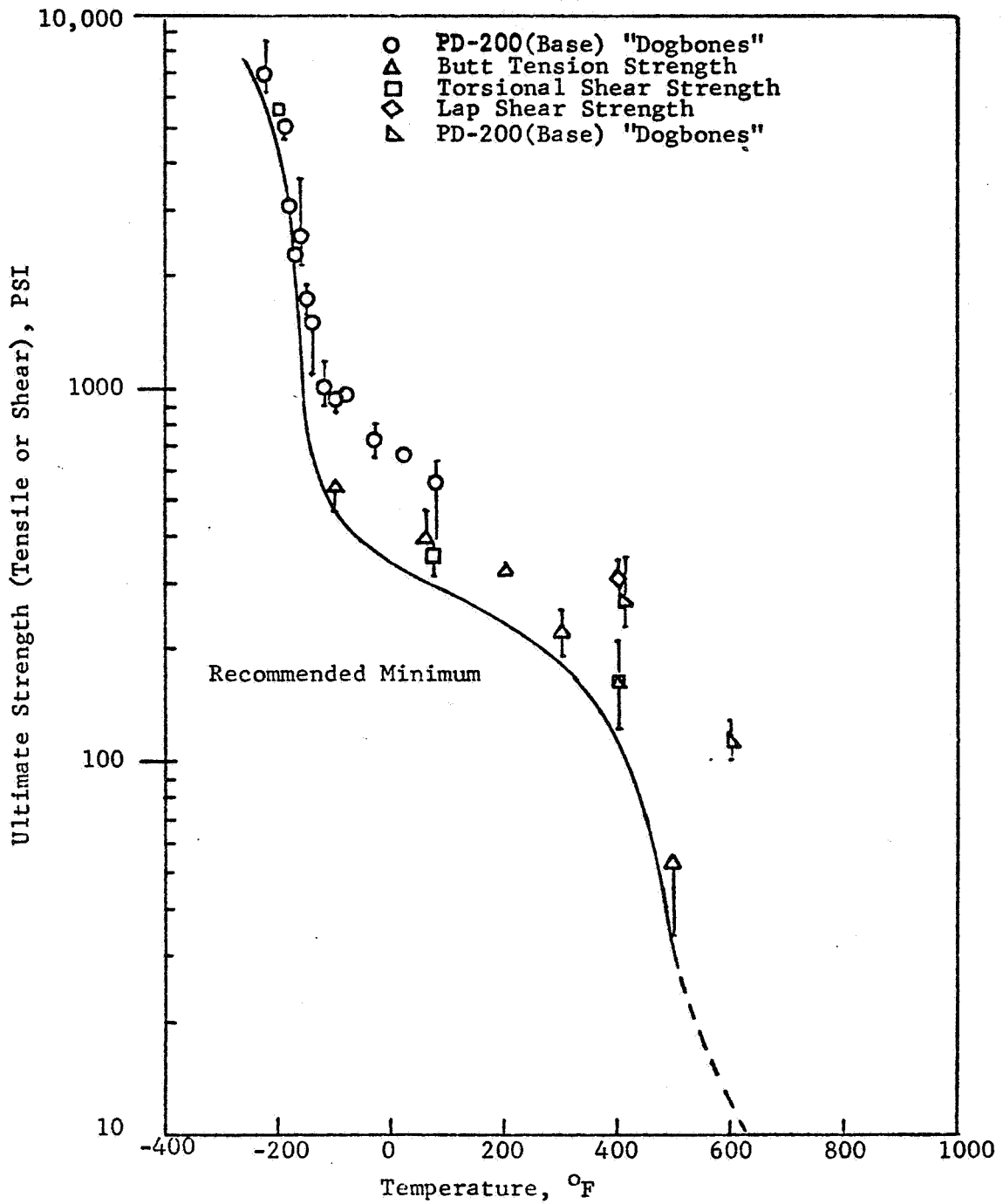


FIGURE 6

ELASTIC MODULI OF PD-200 (BASE)

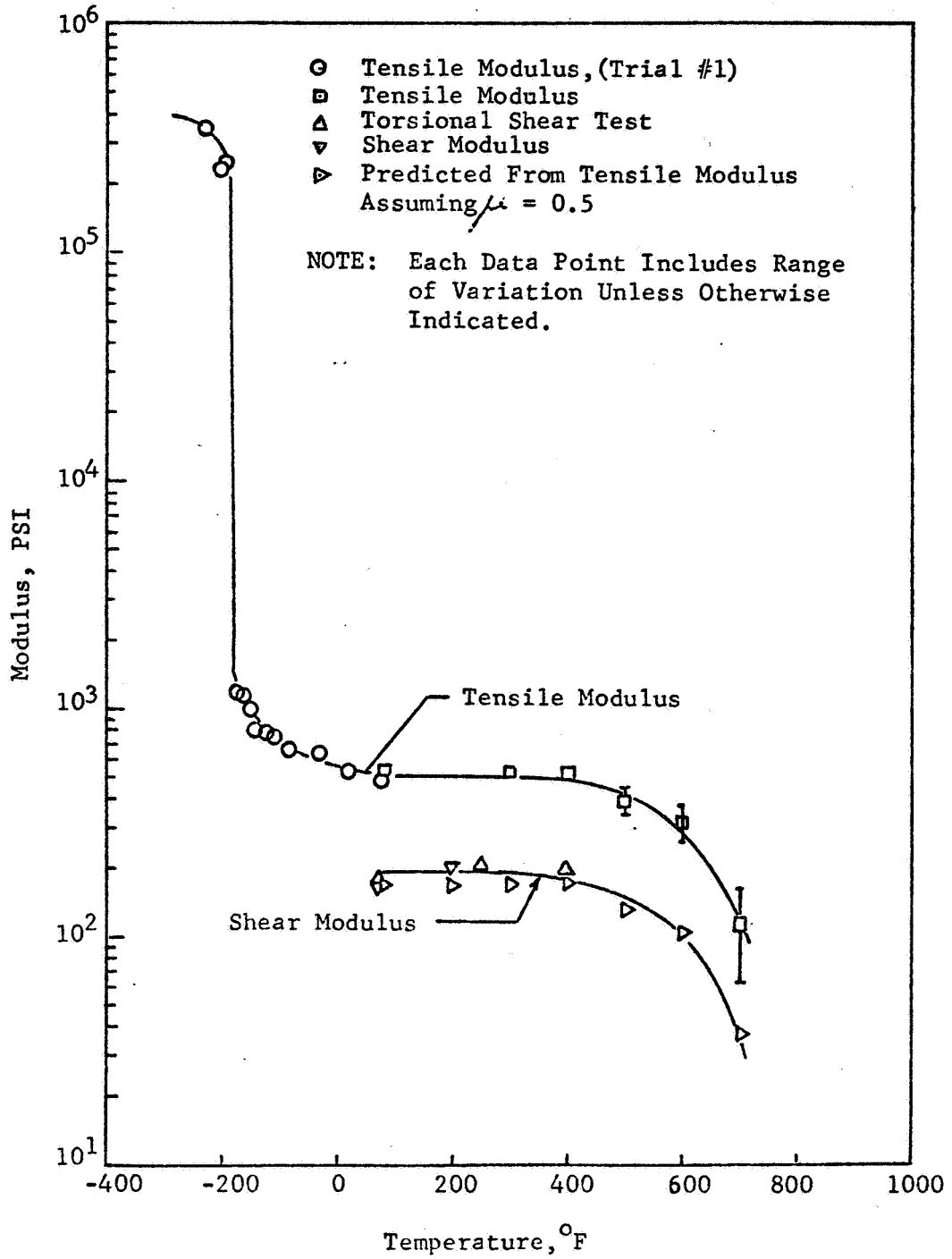


FIGURE 7

TGA ANALYSIS OF PD-200 (BASE)

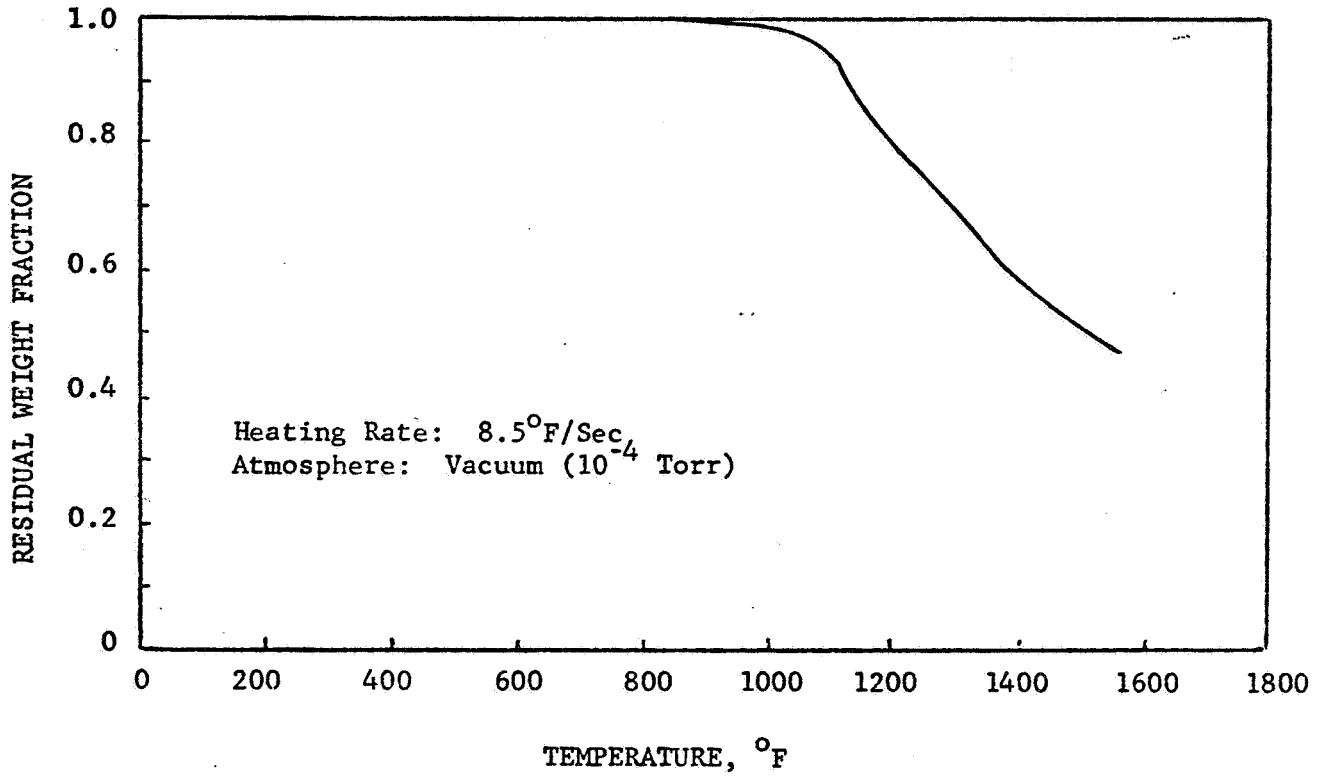
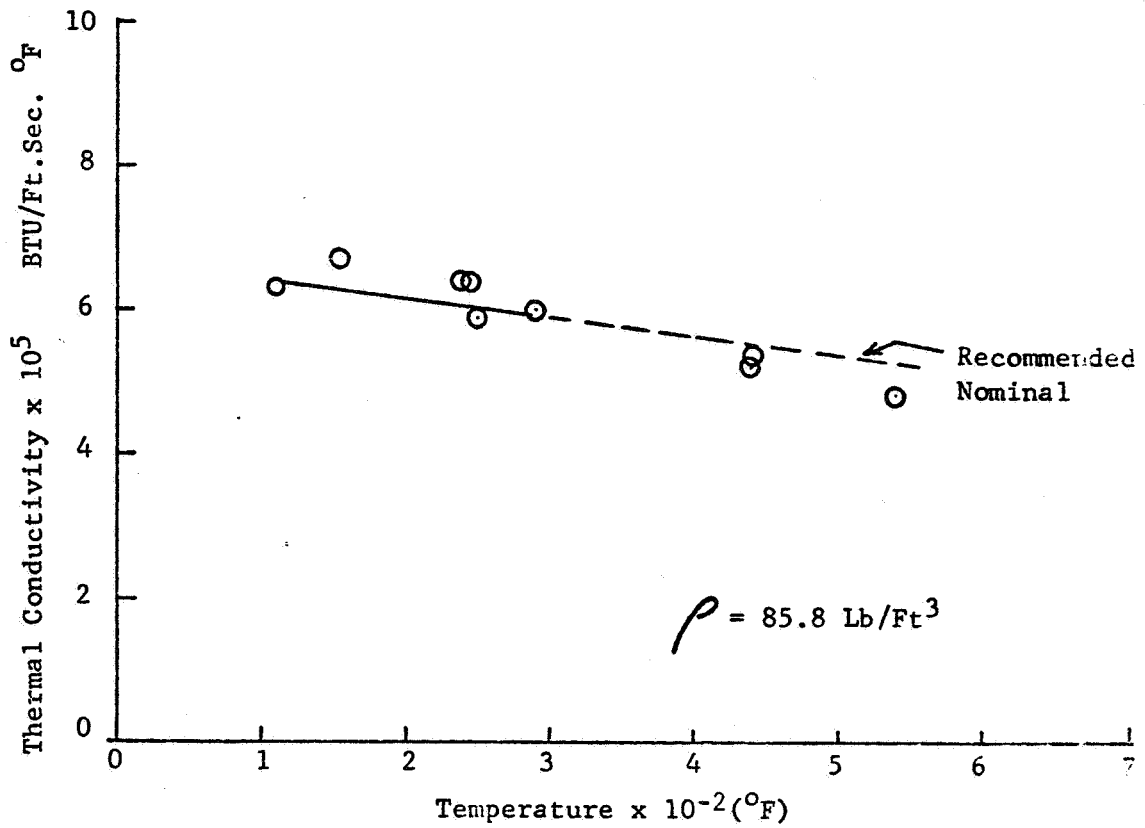


FIGURE 8

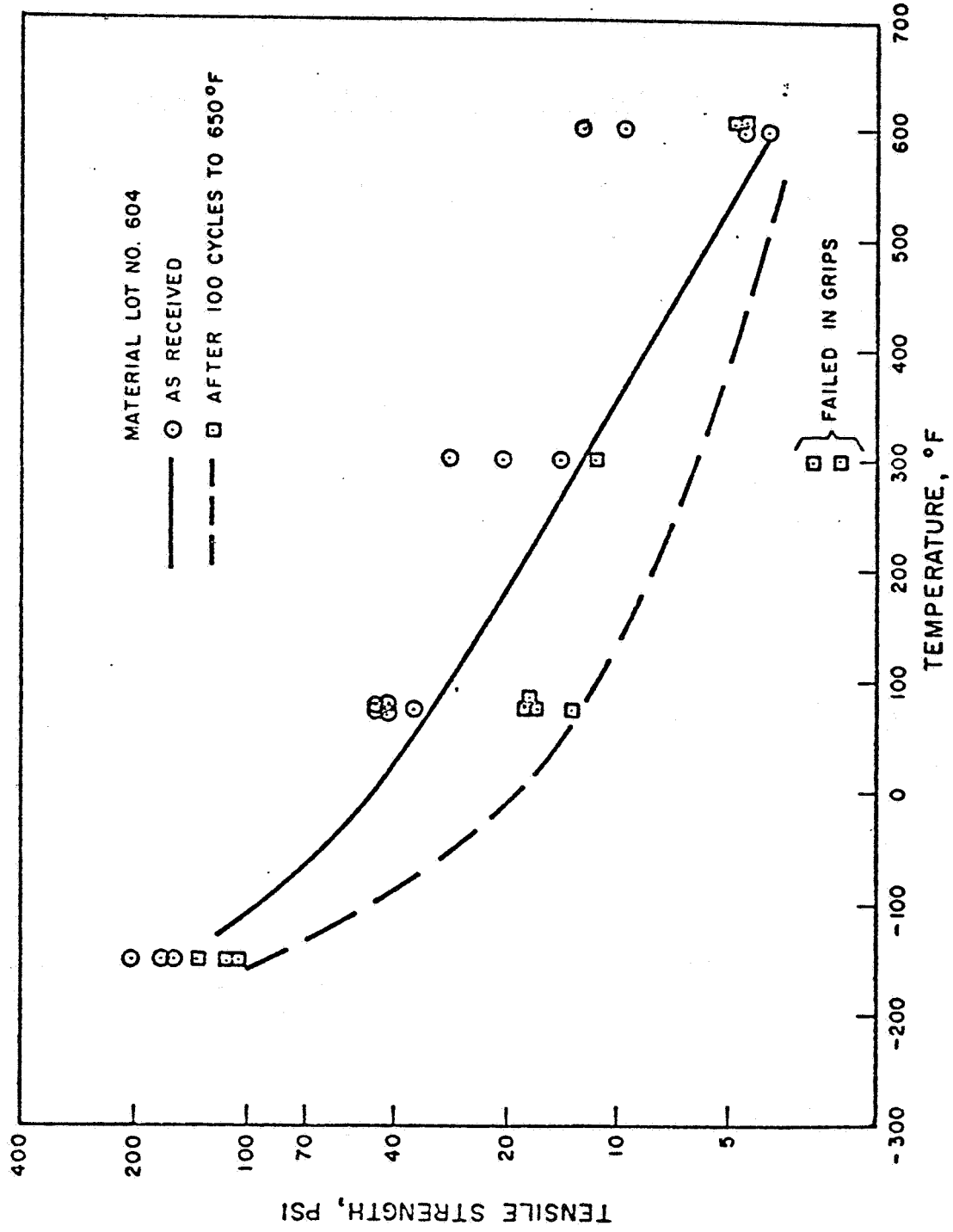
THERMAL CONDUCTIVITY OF PD-200 (BASE)





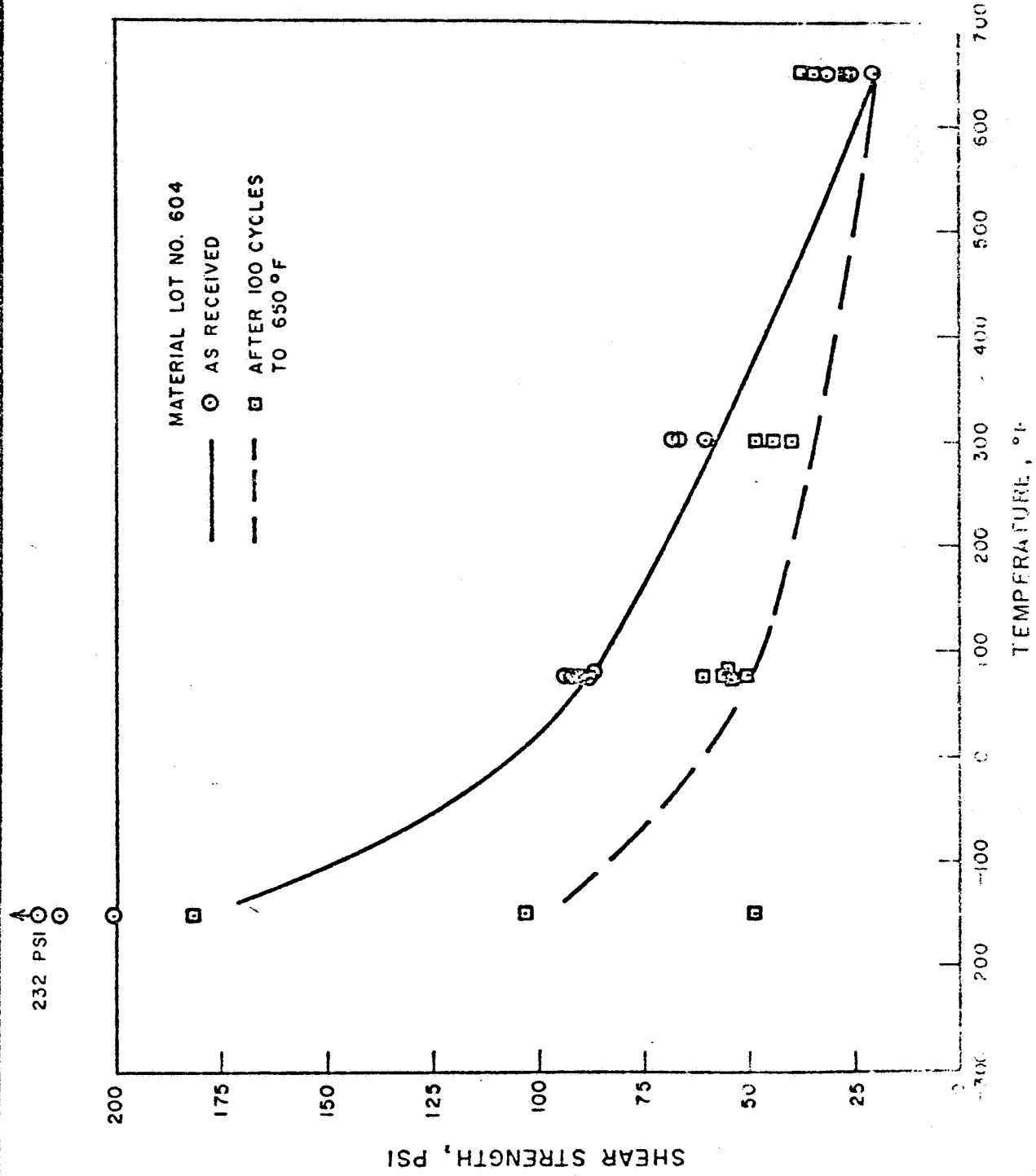
# TENSILE STRENGTH OF PD200

FIG. 9



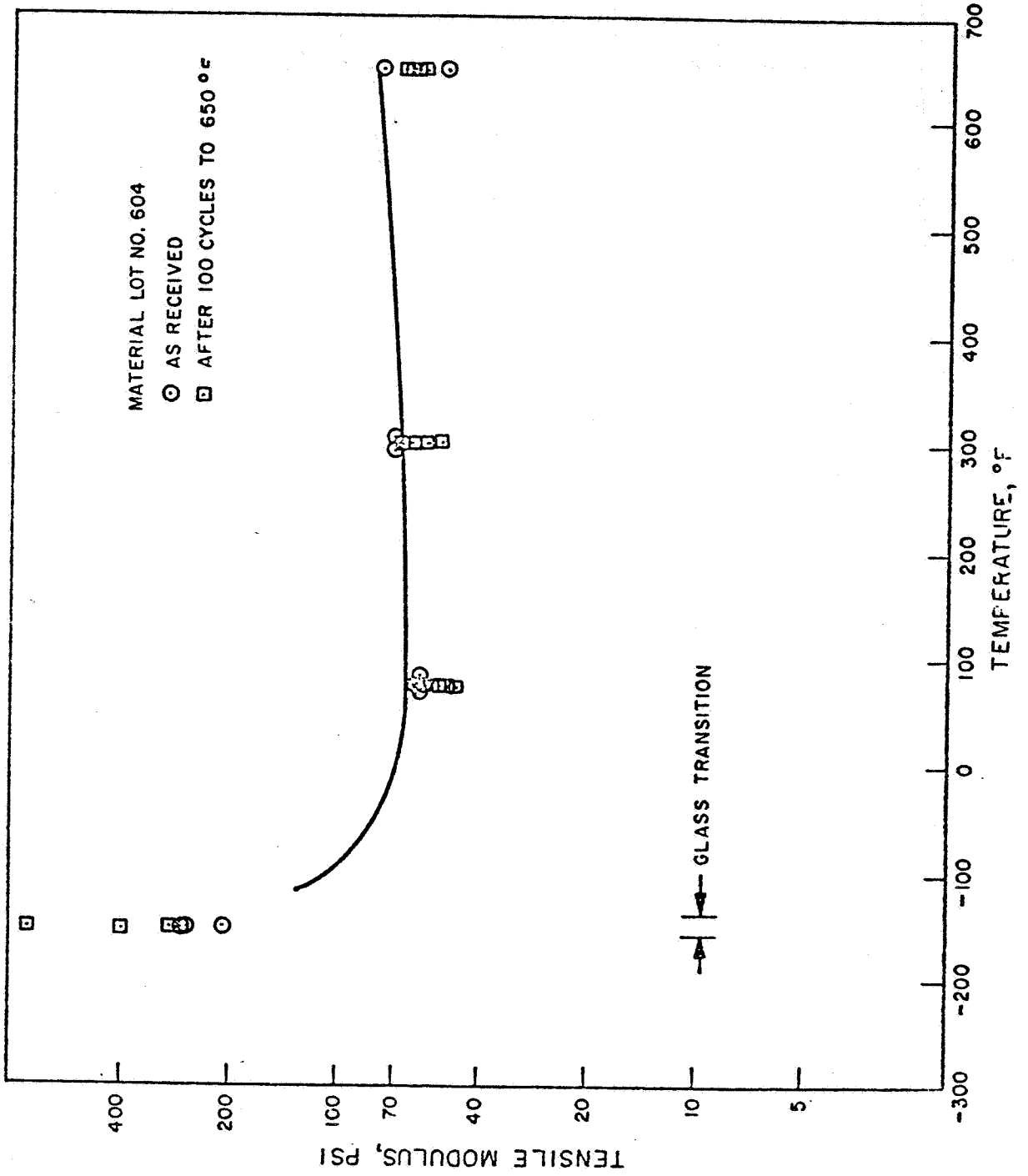
# SHEAR STRENGTH OF PD200

FIG. 10



# TENSILE MODULUS OF PD200

FIG. II



# SHEAR MODULUS OF PD200

FIG. 12

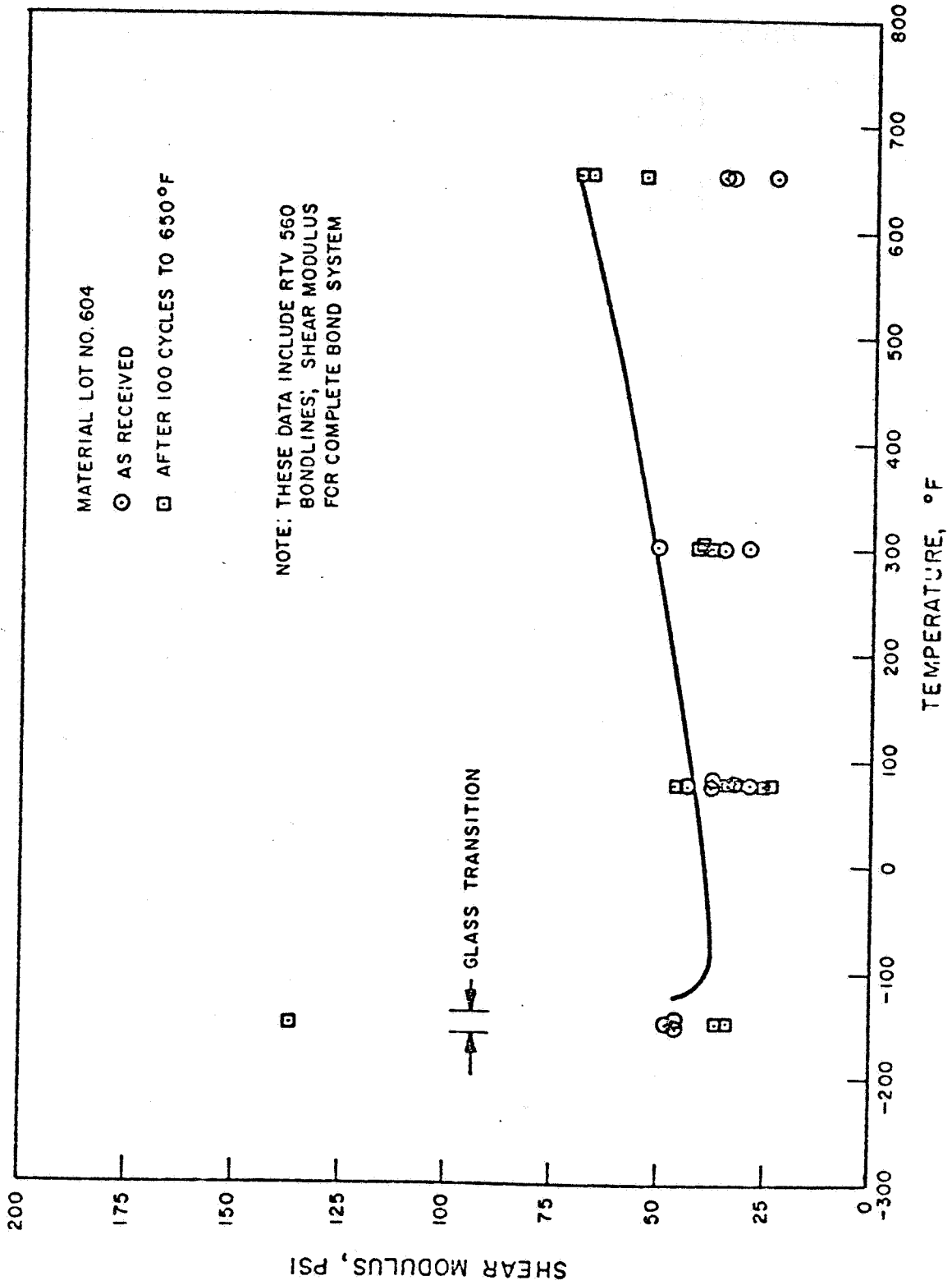
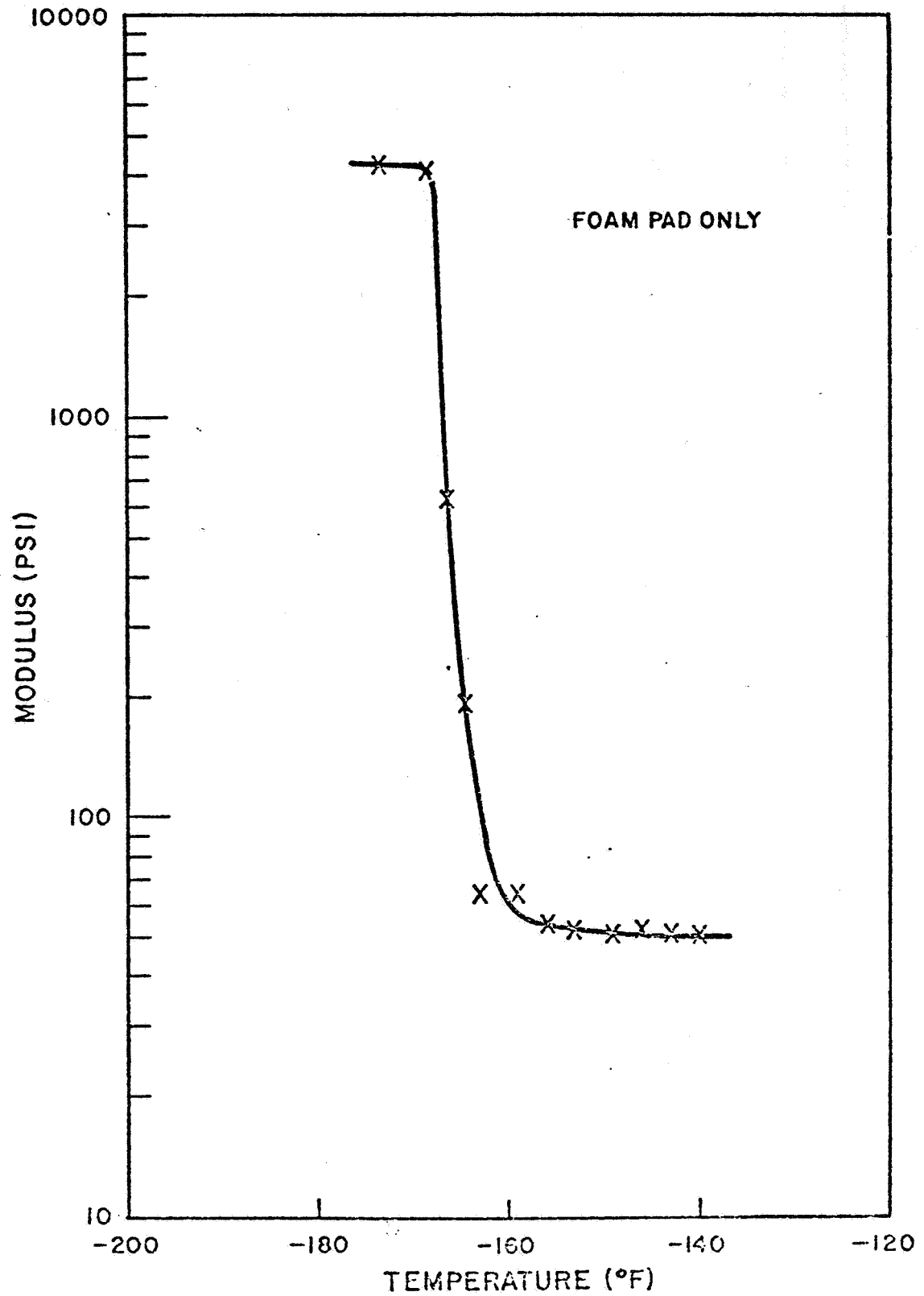
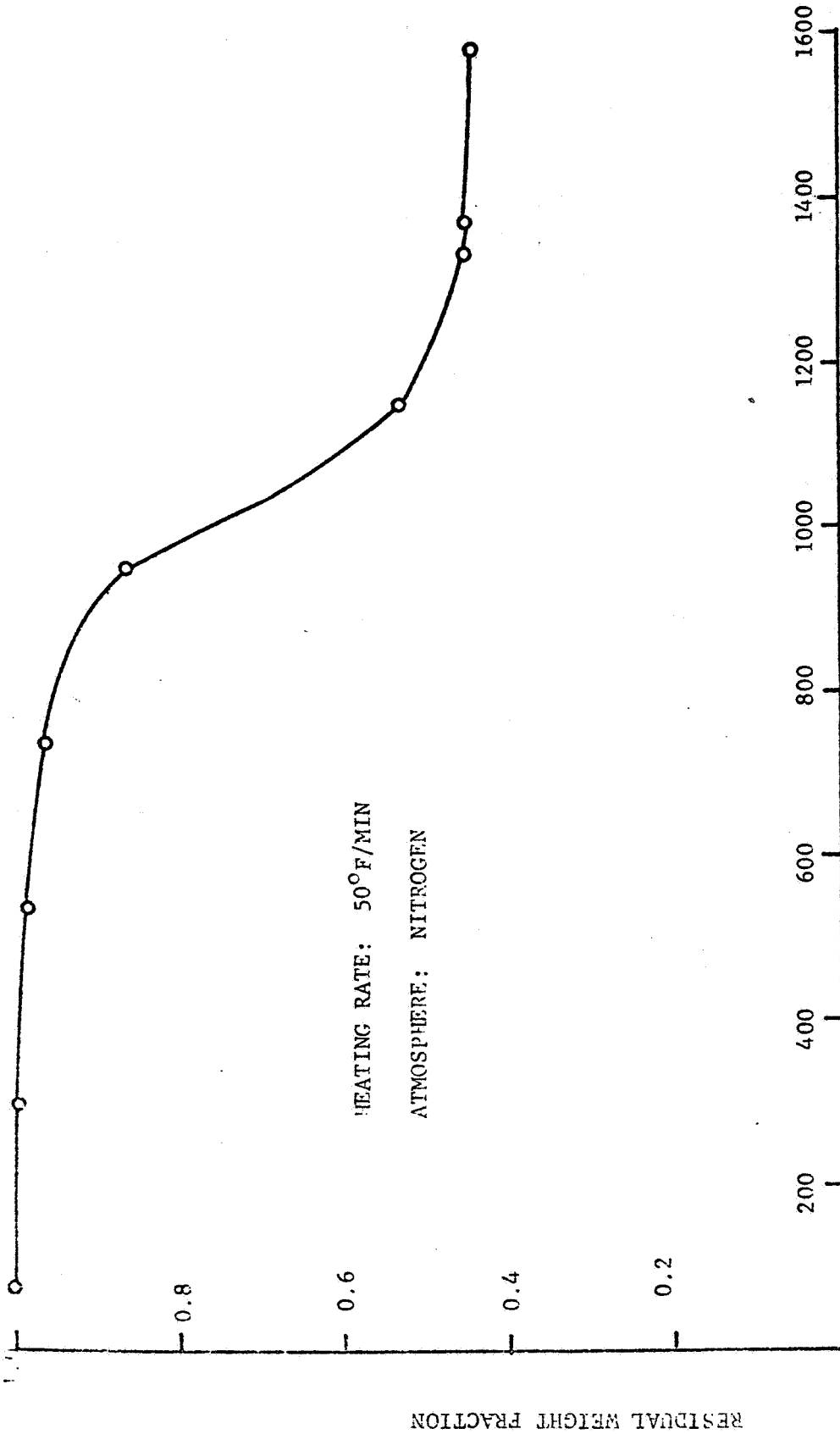


FIG. 13  
LOW TEMPERATURE MODULUS  
OF PD200





TEMPERATURE °F

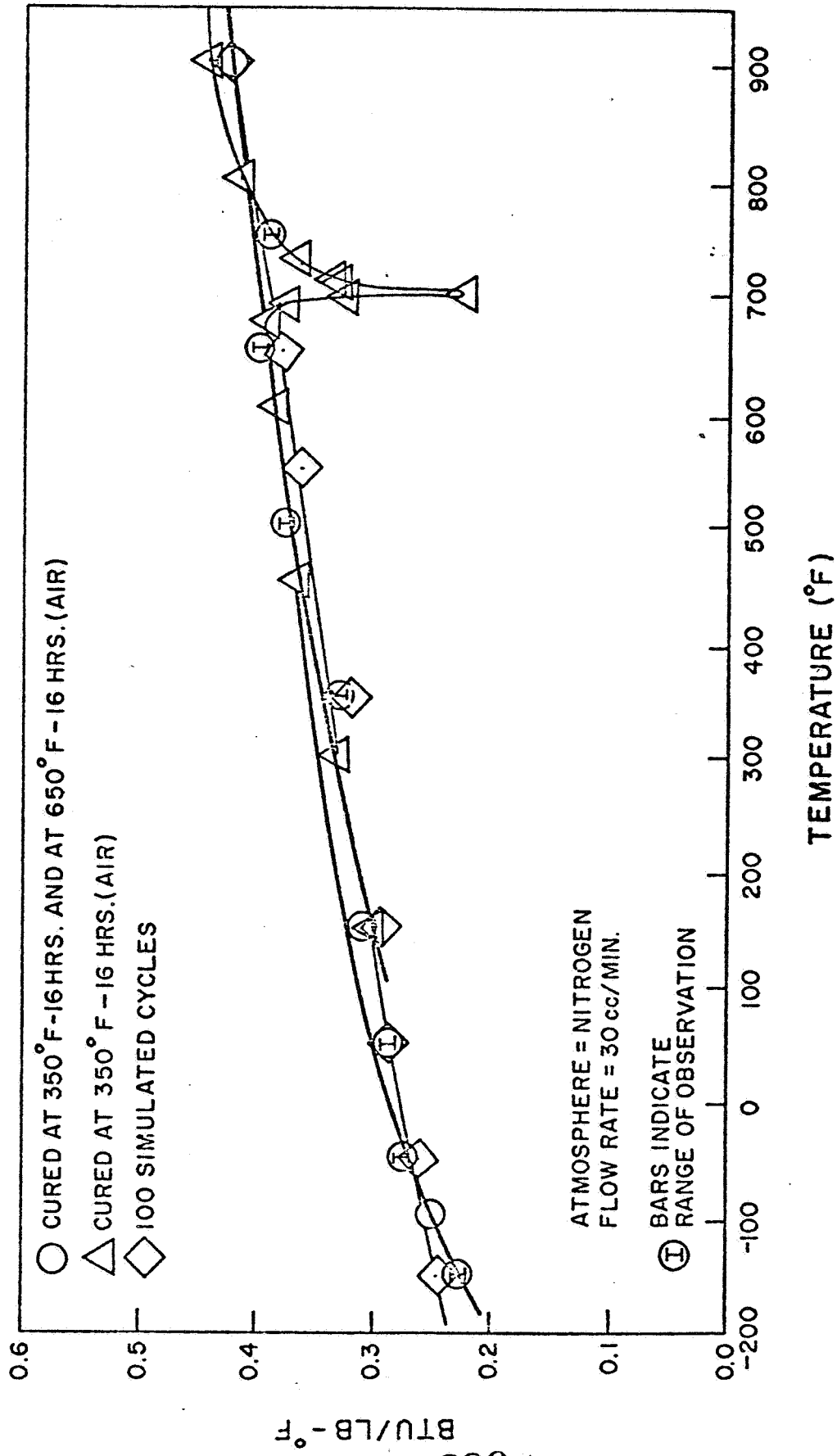
FIGURE 14

TGA OF PD - 200

RESIDUAL WEIGHT FRACTION

# SPECIFIC HEAT OF PD-200 (LOT#602)

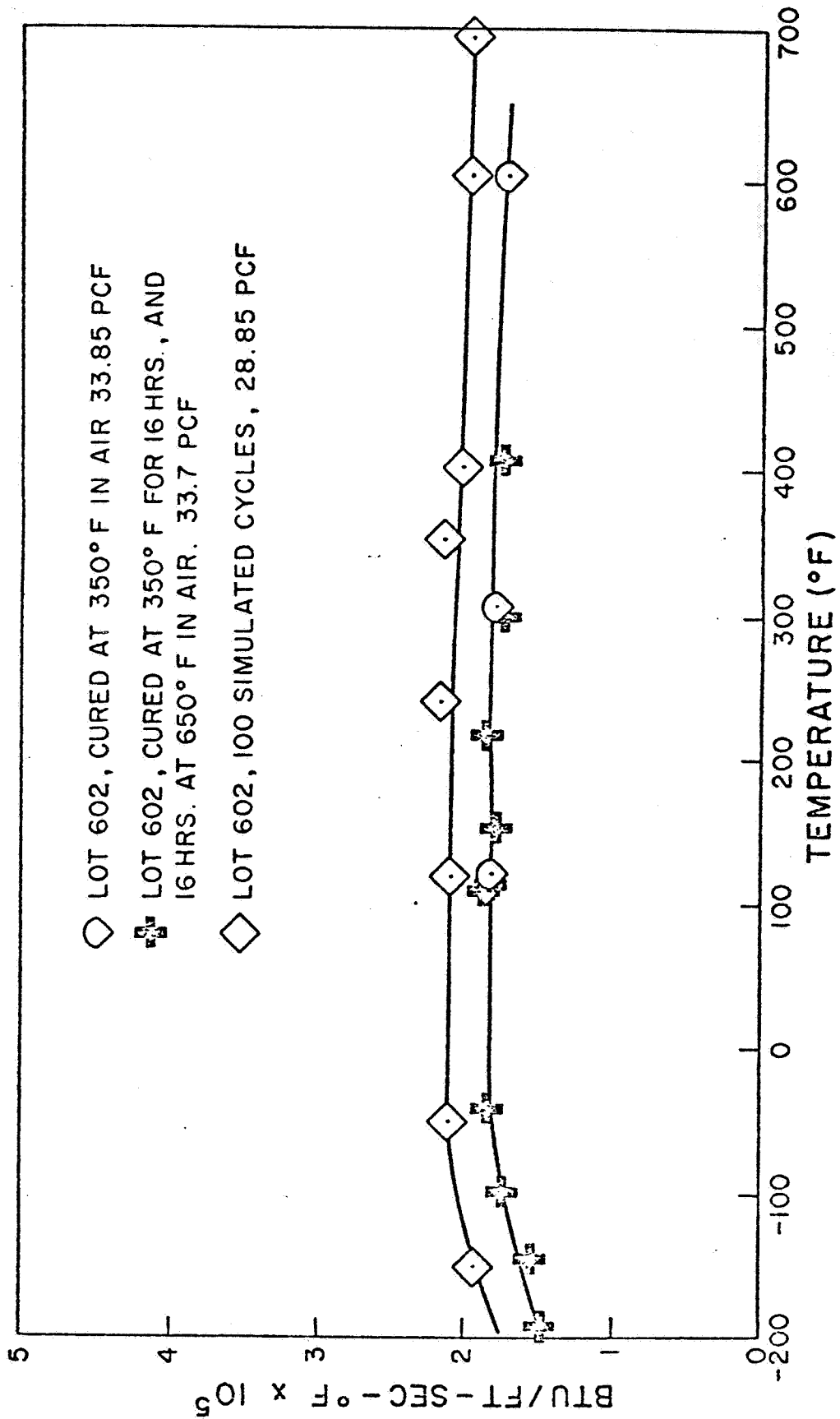
FIG. 15



C-4

# THERMAL CONDUCTIVITY OF PD-200

FIG. 16





# THERMAL EXPANSION OF PD200

FIG.17

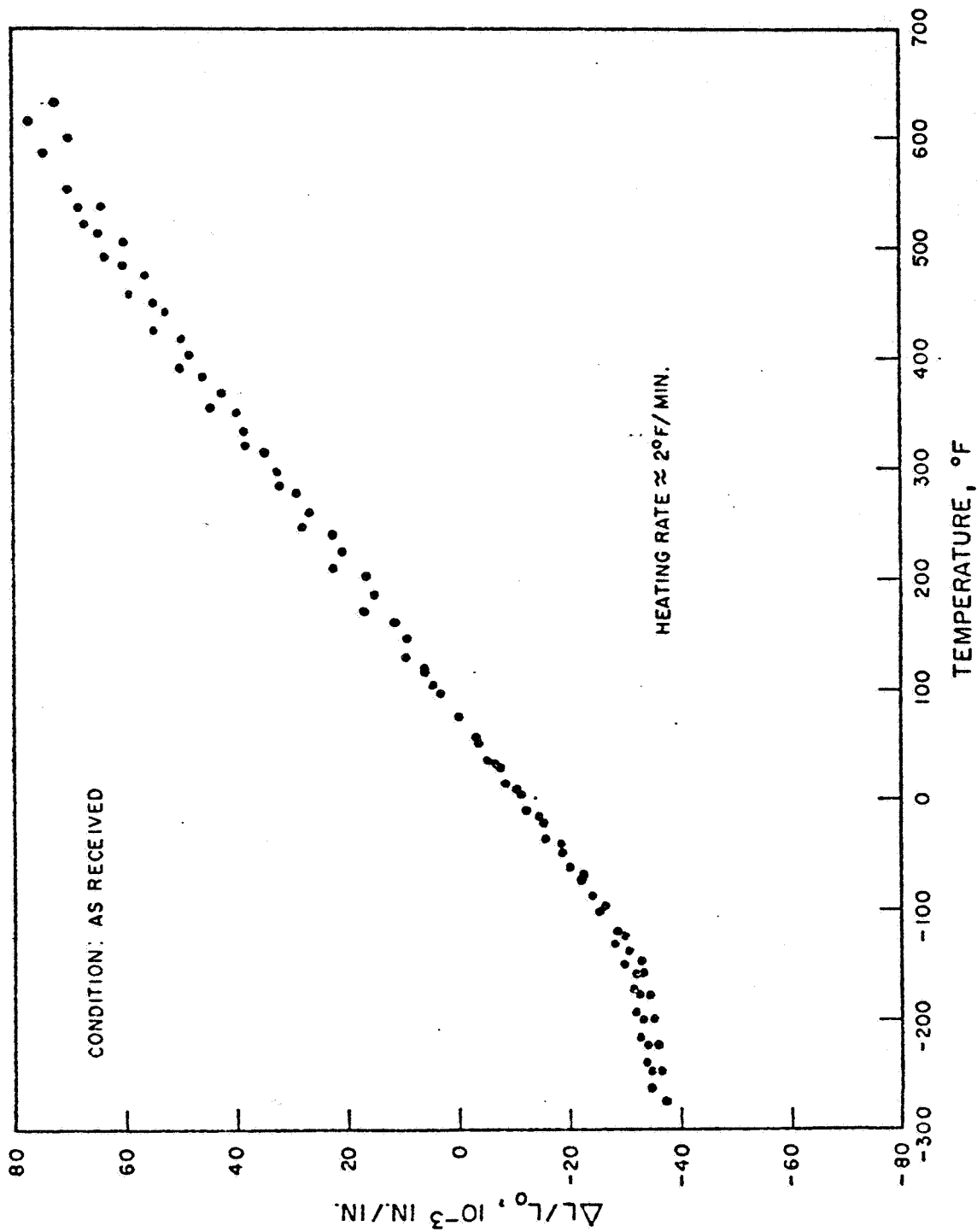


FIG. 18

# EFFECT OF VARYING PD 200-F28 THICKNESS AREA 2-PERTURBED

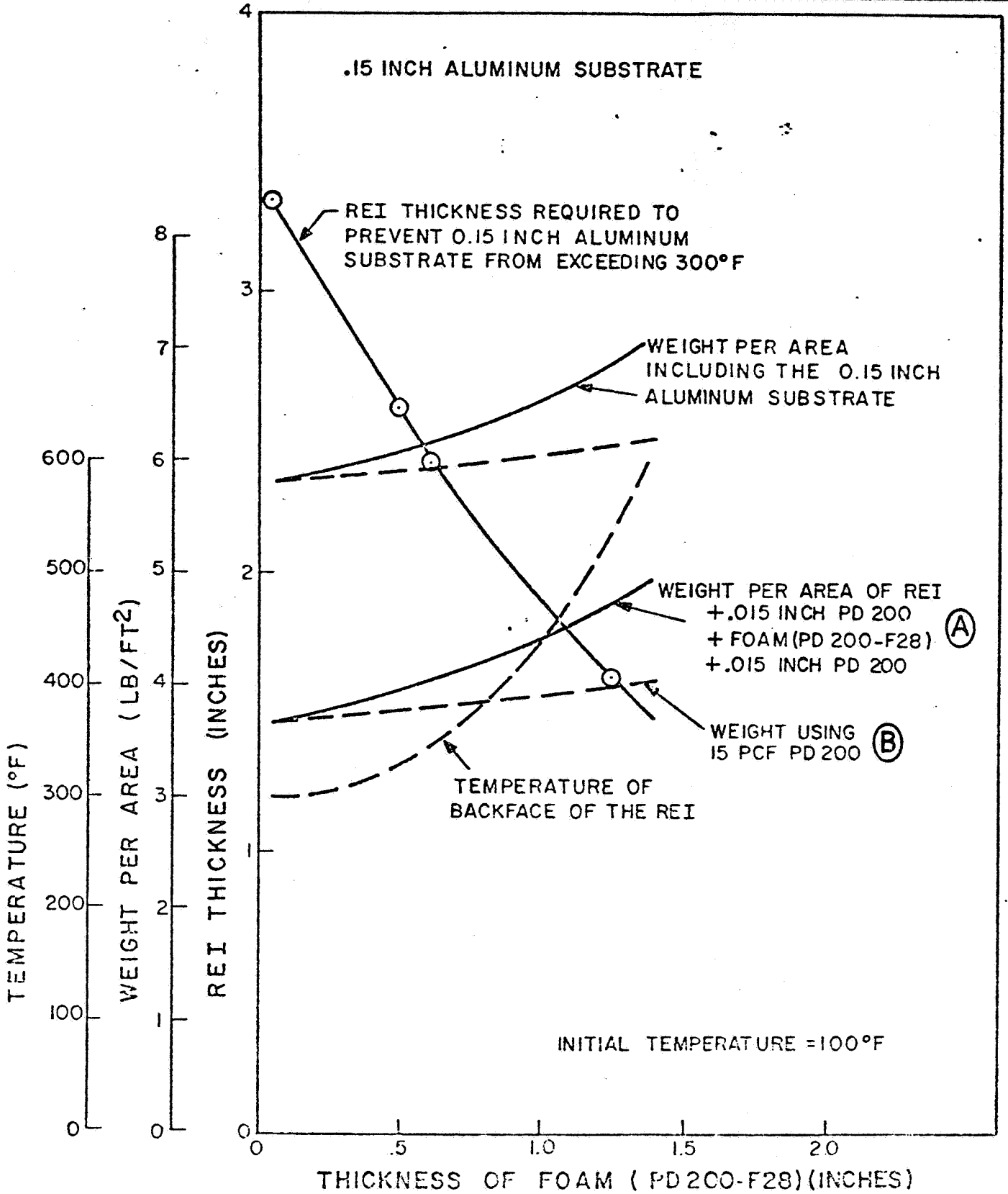


TABLE I     RTV SILICONE ADHESIVE CANDIDATES

<u>COMPOUND</u>	<u>CHEMICAL TYPE</u>	<u>POLYMERIZATION MECHANISM</u>	<u>GLASS TRANS. TEMP.</u>	<u>STRENGTH</u>	<u>DECOMPOSITION TEMPERATURE</u>
RTV 630	Methyl Vinyl Silicone	Addition	-80°F	High	High
PD-195	Methyl Phenyl Vinyl Silicone	Addition	-180°F	High	High
PD-200 (Base)	Methyl Phenyl Silicone	Condensation	-180°F	Lower	Lower
RTV 511	Methyl Phenyl Silicone	Condensation	-180°F	Lower	Lower
RTV 602	Methyl Phenyl Silicone	Condensation	-80°F	Lower	Lower
DC 93-046	----	----	-85°F	Lower	Lower

26  
294

TABLE II

ROOM TEMPERATURE LAP SHEAR STRENGTH OF PD-200 ADHESIVE

AFTER 16 HOUR THERMAL SOAK EXPOSURES

Test Number	Exposure Temperature, °F			
	<u>75</u>	<u>450</u>	<u>550</u>	<u>625</u>
1	94.5	67.1	49.3	33.7
2	83.3	58.6	46.3	31.3
3	82.6	70.0	52.5	31.8
4	84.2	63.5	56.4	32.0
5	83.2	62.2	41.8	32.6
$\bar{X}$	85.6	64.3	49.3	32.3
S.D.	5.0	4.4	5.6	0.9
Failure Mode	All Cohesive	All Cohesive	All Cohesive	All Cohesive

Specimens: 1.0 x 1.0 inch lap joint  
.063 aluminum adherents  
.090 inch thick PD-200  
RTV 560 bond ~ .005 inch thick (both sides)