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**MATERIALS TESTING OF THE IUS TECHROLL
SEAL MATERIAL**

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16. ABSTRACT As a part of the investigation of the control system failure on IUS-1 flight to position a Tracking and Data Relay Satellite (TDRS) in geosynchronous orbit, a study was undertaken to evaluate the techroll seal materials properties under severe flight environment conditions. This study evaluated the materials utilized in the techroll seal for possible failure modes. Studies undertaken included effect of temperature on the strength of the system, effect of fatigue on the strength of the system, thermogravimetric analysis, thermo-mechanical analysis, differential scanning calorimeter analysis, dynamic mechanical analysis, and peel test. These studies indicate that if the seal failed due to a materials deficiency, the most likely mode was excessive temperature in the seal. In addition, the seal material is susceptible to fatigue damage which could have been a contributing factor.					
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TECHNICAL MEMORANDUM

MATERIALS TESTING OF THE IUS TECHROLL SEAL MATERIAL

INTRODUCTION

Space Transportation System flight number six (STS-6) was utilized to transport the Air Force Space Division Inertial Upper Stage (IUS) propulsion system to low Earth orbit. The IUS was ejected from the shuttle bay and utilized as a propulsion system to achieve geosynchronous orbit for a Tracking and Data Relay Satellite (TDRS). IUS is a two-stage solid rocket propulsion system, built by Chemical Systems Division (CSD) of United Technologies Corp., capable of achieving geosynchronous orbit under nominal flight conditions. The IUS first stage (SRM-1), used to provide the energy required to go from low Earth orbit to geosynchronous orbit, operated according to plan producing nominal thrust and attitude conditions. The second stage (SRM-2), used to provide the thrust to circularize the geosynchronous orbit, performed nominally until approximately 75 sec into burn when a blip occurred on the thrust trace indicating a temporary increase in thrust. Approximately 5 sec later the IUS and TDRS began an uncontrolled tumble with the rocket motor continuing to provide thrust. It achieved a tumbling frequency of about 30 revolutions/min. After reprogram commands, the TDRS was successfully separated subsequent to SRM-2 burnout. The IUS propelled TDRS within 2000 miles of desired orbit and the TDRS thrusters were utilized to achieve the additional altitude.

The information, telemetered to tracking stations showing thrust and acceleration traces and nozzle actuator location, leads to the conclusion that the seal between the nozzle and motor case failed during burn of SRM-2. This seal, known as the techroll seal (TRS), is a kevlar reinforced neoprene bladder filled with silicone oil. It is protected from heat of burning propellant by an extensive thermal protection system.

The flexible joint techroll seal concept was developed to permit the use of a low weight electromechanical actuation system allowing the use of a lightweight carbon-carbon nozzle. The moveable techroll seal is a constant volume, fluid-filled bearing using a seal configured with two rolling convolutes which permit omniaxial deflection of the nozzle assembly. The techroll seal consists of two layers of Kevlar -29 fabric layered between two sheets of neoprene rubber with steel cable beads for seal retention. The seal, as shown in Figure 1, is a figure of revolution with a "U" shaped crosssection and an axial opening. One layer of Kevlar -29 fabric is sufficient to carry the loads induced during flight. The second layer is redundant and provides a generous safety factor.

The techroll seal is contained by an insulated titanium housing that allows the TRS to operate over a 44°F to 82°F temperature range during the firing mode. The seal may be subjected to other thermal environments during transportation and storage.

Figure 2 shows a crosssectional view for the techroll joint. The techroll seal is enclosed in a titanium housing and transmits load from the nosecap/throat to the nozzle ring that is attached to the motor case. The outer diameter of the TRS housing is protected by a viton thermal boot, carbon phenolic nose cap and a carbon

phenolic/silica phenolic laminate insulator. On the inner diameter, it is covered by a three-dimensional woven carbon-carbon composite (ITE) subtended by a carbon phenolic/silical phenolic fixed insulator and nozzle insulator. The techroll seal is pressurized during flight by the pressure from the burning propellant in the case acting on the nosecap/ITE material. Even pressurized to 1200 psi as occurs in flight the nozzle is able to gimble with small actuator forces acting on the nozzle.

MATERIALS EVALUATION

The techroll seal composite materials evaluated in this study were provided in two forms. Flat sheet laminate was fabricated according to the applicable sections of the specification for techroll seal manufacture of SE0780 entitled "Techroll Seal, IUS, Fabrication of." Flat molds were used to vulcanize the neoprene with the proper orientation of Kevlar cloth between them.

A rejected techroll seal manufactured to SE0780 was also provided for test material. The seal was rejected for flight hardware application due to delamination and creases in the rubber. Tests were conducted on samples from these materials designed to evaluate possible failure modes of the composite techroll seal material.

TENSILE STRENGTH VERSUS TEMPERATURE

Tests were conducted utilizing a Model 1113 Instron Universal Testing Machine with a 5000 lb capacity. Specimens of techroll seal material were exposed to test temperature for 5 min and pulled to failure in tension at a pull rate of 10 in./min.

The results of this testing, shown in Figure 3, indicates the tensile strength of the TRS material at elevated temperature. Analysis of this data indicates a rapid degradation in strength at temperatures in excess of 200°F with only 54 percent of the original room strength remaining at 500°F.

Initial materials testing revealed a slight increase in strength from 75°F to 200°F; however, additional testing indicated this effect is attributed to sample scatter and the material does not exhibit an actual strength increase. From this plot it is concluded that the TRS material should not be exposed to temperatures above 400°F even for short durations if it is to perform its load carrying function.

TENSILE STRENGTH VERSUS FATIGUE

Flexure fatigue strength of TRS material was determined on samples exposed to flexure fatigue cycling at room temperature. An MIT folding endurance tester (Fig. 4) was utilized to expose the samples to folding fatigue, under an applied tension load of 1 kg. The samples were pulled to tensile failure in an Instron Universal testing machine, Model 1113 with a 5000 lb capacity.

Figure 5 shows the relationship of strength to folding fatigue cycles for both flat laminate and actual TRS material. It is noted that the rate of decrease is rapid to 1000 cycles where the decrease begins to moderate. The difference in the rate of

strength deterioration between flat sheet material and techroll seal material is attributed to the difference in the lay-up configuration and the bond strength between layers of Kevlar. The flat sheet material retained 47 percent of its original strength after 1000 cycles while the techroll seal material had 57 percent remaining after 1000 cycles.

THERMOGRAVIMETRIC ANALYSIS (TGA)

Thermogravimetric analysis was conducted both in air and nitrogen utilizing a Dupont Model 1090 system with the Model 951TGA attachment. Additional TGA tests were run in vacuum utilizing a Mettler Model TA-2.

Figures 6, 7, and 8 graphically represent the TRS material weight loss as a function of temperature. Table 1 compares the reaction in vacuum, air, and N₂. These data indicate that the TRS material is stable over the range of temperatures it is expected to encounter during flight. If the TRS temperature exceeds 75°C, the neoprene begins to decompose with a rapid increasing rate of weight loss beginning at 285°C. The Kevlar fibers begin to chemically deteriorate at 350°F with the rate dependent on the atmospheric environment. The rate of weight loss is greater in vacuum than in air or nitrogen up to 350°F, while the air environment results in the greater weight loss above this temperature. Large weight loss rates at the higher temperatures is attributed to oxidation of the polymer.

THERMOMECHANICAL ANALYSIS (TMA)

Thermomechanical analysis was conducted on the TRS composite material utilizing a Dupont 1090 system in conjunction with its Model 943 TMA accessory. The TMA results from this study, shown on Figure 9, indicate uniform properties over the temperature range of -33°C to 100°C. The mechanical expansion is relatively constant until a temperature is encountered that results in decomposition of the test material. The abrupt change in coefficient of expansion is a result of a rapid change in modulus of the neoprene at its glass transition temperature (t_g). It is believed that the expansion rate of the composite is controlled by the neoprene below its t_g and by the Kevlar cloth above that temperature.

DIFFERENTIAL SCANNING CALORIMETER (DSC)

Differential scanning calorimeter analysis was conducted on the neoprene in an attempt to understand the chemical changes that occur in the rubber as a function of exposure temperature. The DSC tests results were obtained utilizing the Dupont 1090 System with the Model 910 DSC attachment. The resulting data shown in Figure 10 indicate that no reactions are apparent in the 0 to 100°C range. Two endothermic reactions are noted between -45°C and 0°C. The reaction at -45°C is associated with the glass transition temperature of the neoprene rubber.

DYNAMIC MECHANICAL ANALYSIS (DMA)

Dynamic Mechanical Analysis obtained from the use of a Depont 1090 system with a Model 982 DMA accessory on TRS material provides a plot of vibration damping as a function of temperature. The damping feature generally indicates a materials change on a molecular scale. The resulting data exhibited in Figure 11 indicate only one reaction between the temperature range of -100 to +40°C. The large decrease in E and increase in damping capacity beginning at approximately -40°C is related to the glass transition of the neoprene.

PEEL STRENGTH

Bond strengths of the various layers of the TRS composite material in a peel mode were obtained utilizing a Model 1113 Instron Universal Testing Machine. The results from the peel test are shown in Table 2. These data indicate a very weak bond between the layers in the composite. This is a well documented problem with Kevlar fibers. A strength in the range of 15 lb/in. would be considered a desirable value for these bonds. The data obtained are relatively consistent between the flat sheet and the material cut from a rejected techroll seal except for the bond between the Kevlar sheets. This is expected, since the seal used was rejected for flight use due to delamination.

SCANNING ELECTRON MICROSCOPE ANALYSIS (SEM)

Samples of techroll seal material were subjected to 2000 cycles of flexure fatigue under a 1 kg tension load. The samples were separated at each laminate to expose the surface of the Kevlar cloth. Individual fibers were extracted from the Kevlar and were coated with carbon to allow SEM analysis. This testing was conducted to attempt to identify the damage mechanism of TRS material exposed to cyclic flexure fatigue. The SEM photomicrograph, shown in Figure 12, indicates that the Kevlar fibers fragment axially under compressive loading. Additionally, the presence of "knees" is detected (Fig. 13). This is a result of a fibers failing in a buckling mode.

CONCLUSIONS

The testing of the Inertial Upper Stage techroll seal materials substantiated the following general property characterizations:

- 1) Kevlar fibers tend to degrade when subjected to compressive loading.
- 2) Kevlar cloth tends to have low bonding strength to other materials.
- 3) The techroll seal composite system will degrade rapidly when subjected to over design use temperatures.

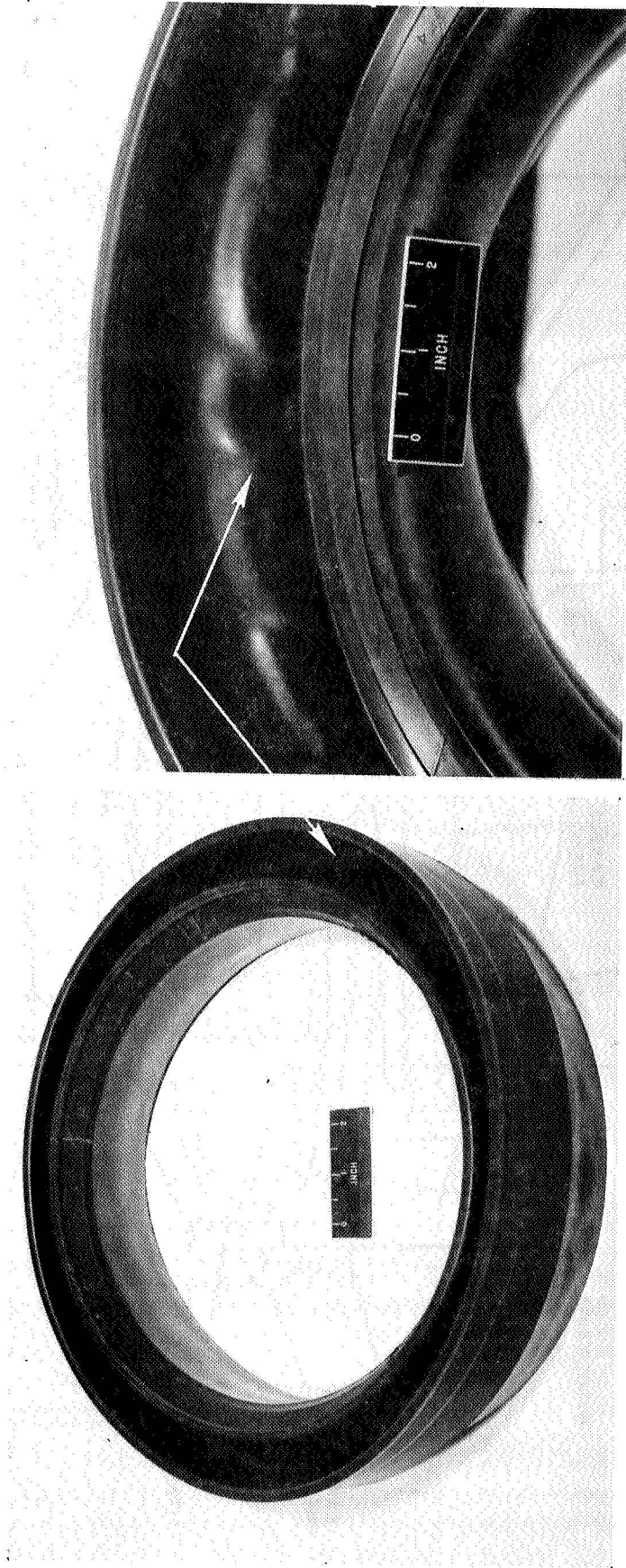


Figure 1. Techroll seal.

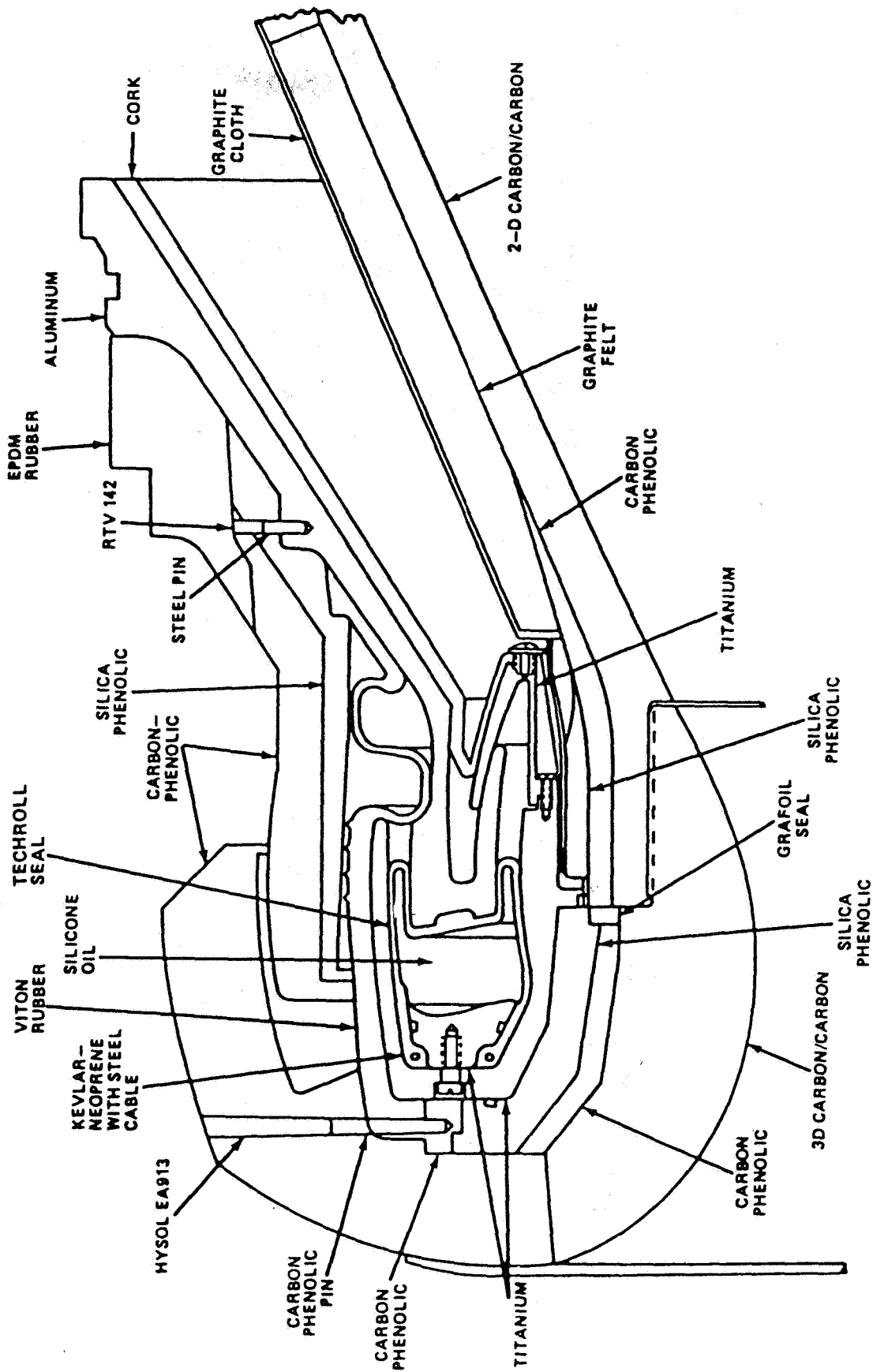


Figure 2. SRM-2 nozzle joint.

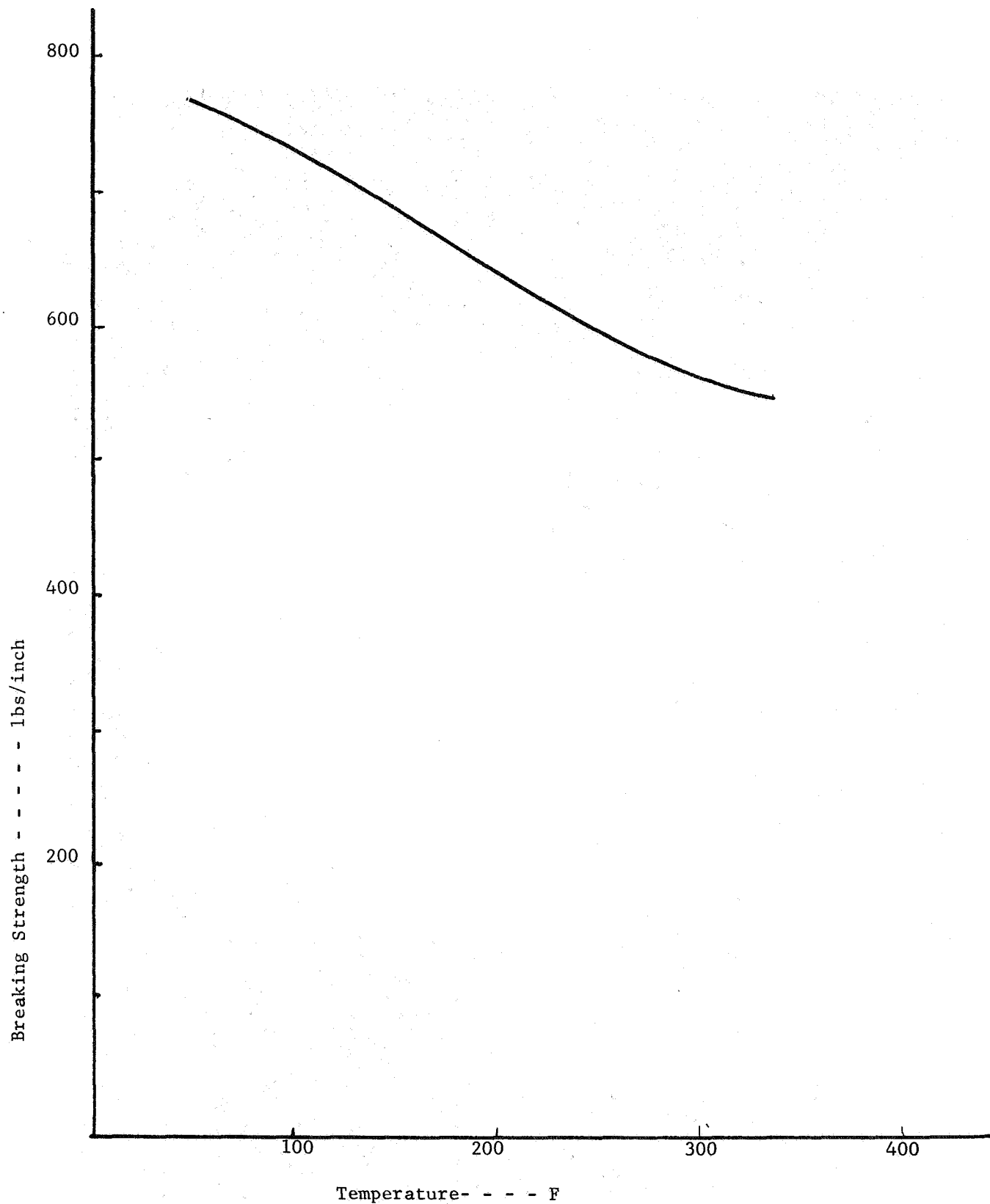


Figure 3. Tensile strength of flat sheet Techroll seal composite material at temperature.

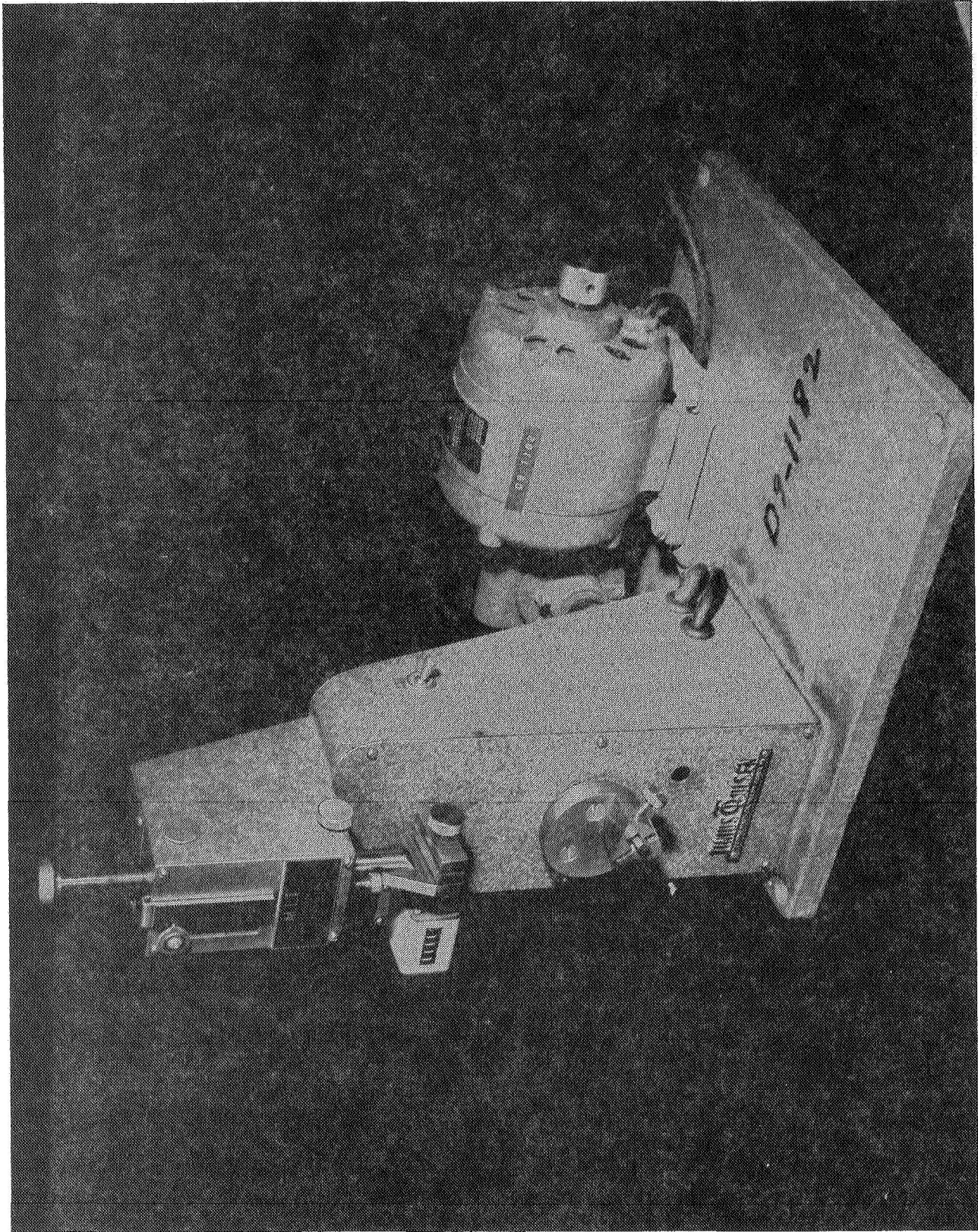


Figure 4. MIT folding endurance tester.

STRENGTH AFTER CYCLIC LOADING

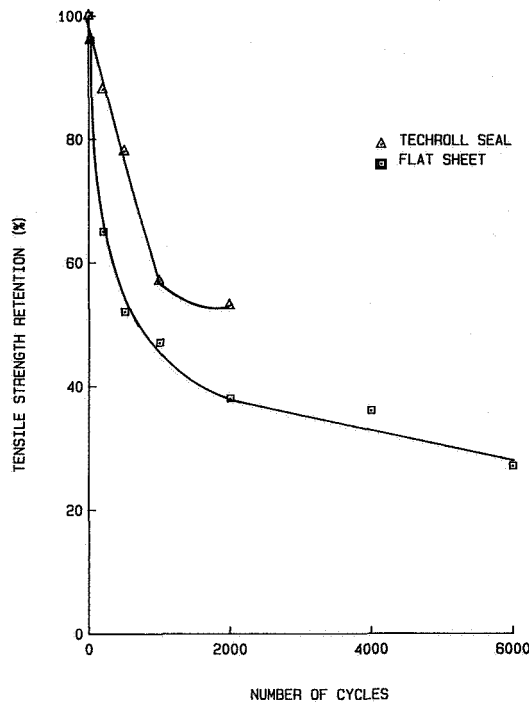


Figure 5. Percent unflexed tensile strength retained versus number of flexed cycles.

Sample: 2ND STAGE TECHROLL SEAL
 Size: 21.36 mg
 Rate: 20DEG/MIN 50ML/MIN AIR
 Program: TGA Analysis V1.0

TGA

Date: 10-May-83 Time: 9:07:49
 File: IUS.01 #6 TGA DATA
 Operator: HUNDLEY
 Plotted: 27-Jul-83 10:19:34

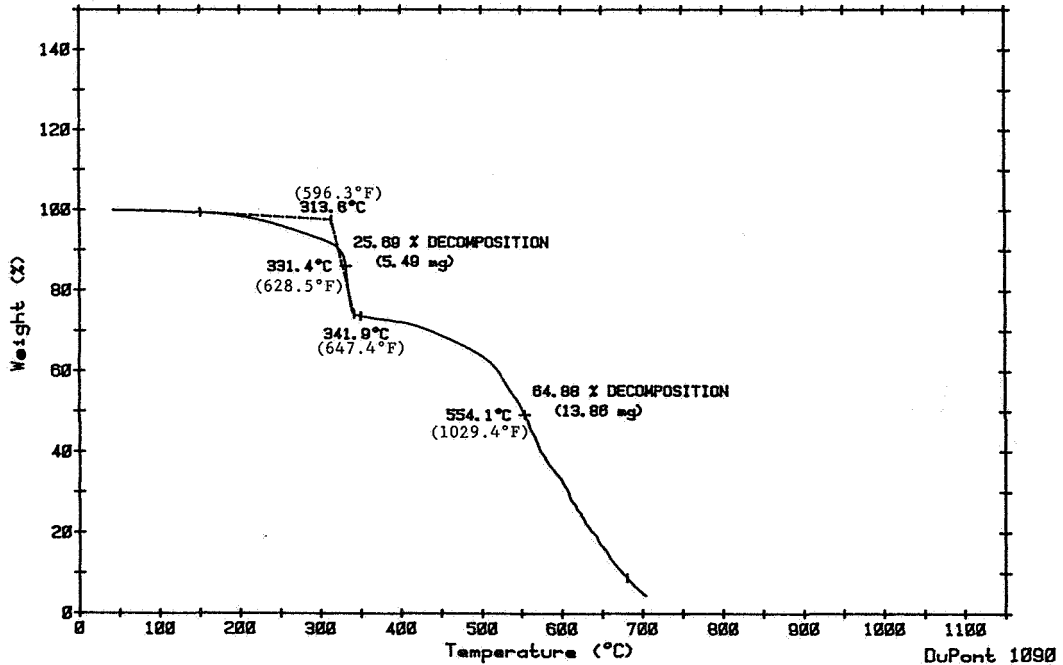


Figure 6. Thermogravimetric analysis of Techroll seal material in air atmosphere.

Sample: 2ND STAGE TECHROLL SEAL
Size:
Rate: 20DEG/MIN 50ML/MIN GN2

TGA

Date: 12-May-83 Time: 9:17:33

Operator: HUNDLEY

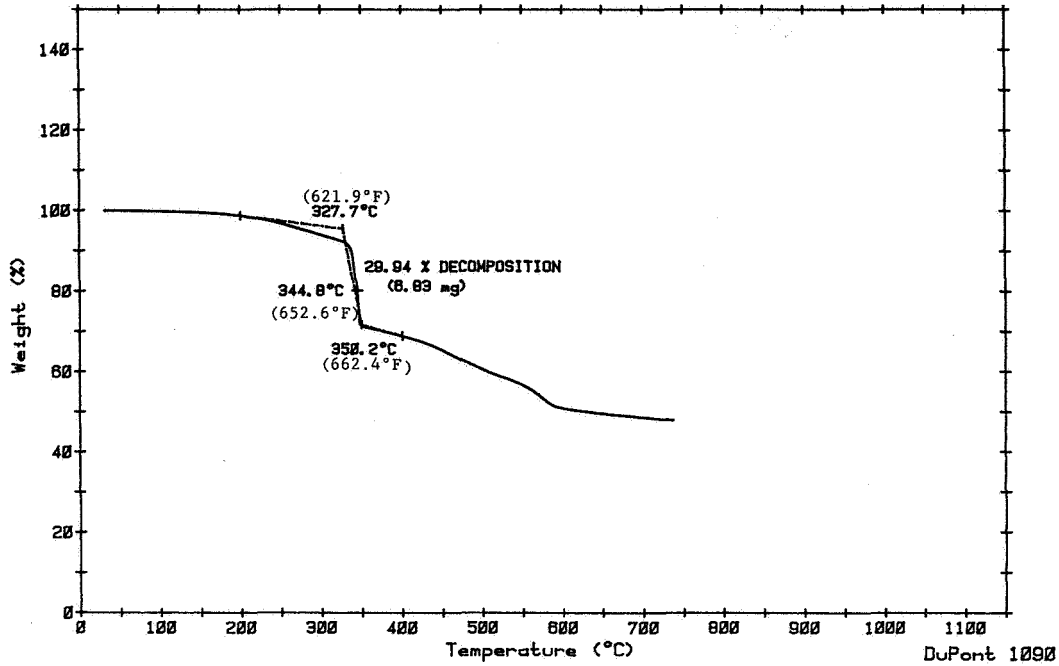


Figure 7. Thermogravimetric analysis of Techroll seal material in N₂ atmosphere.

Sample: 2ND STAGE TECHROLL SEAL
Rate: 6 DEG/MIN, 20 MICRON VACUUM

TGA

METTLER MODEL TA-2

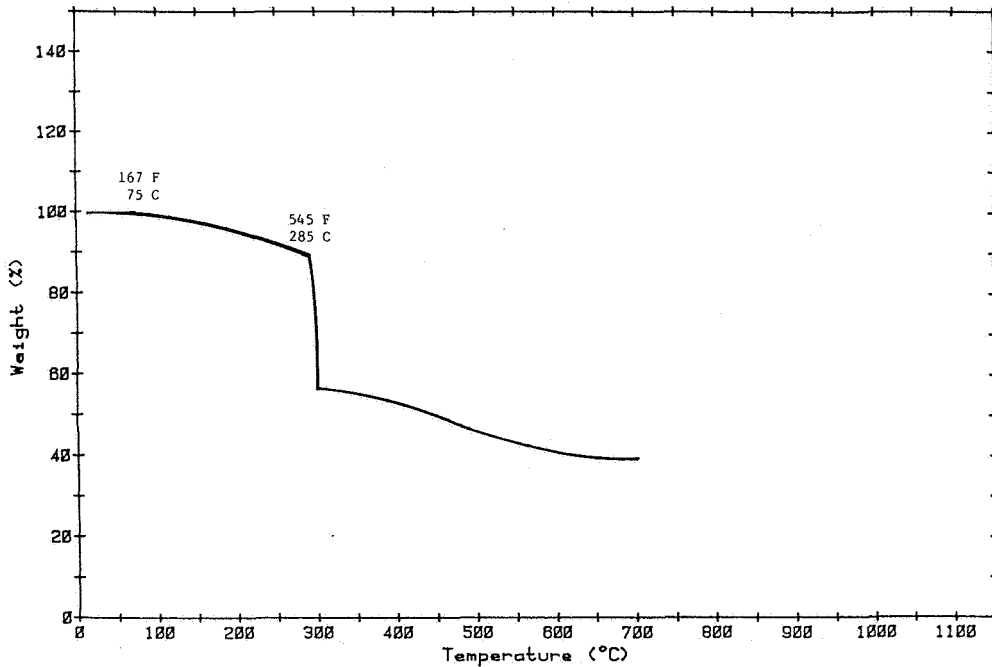


Figure 8. Thermogravimetric analysis of Techroll seal material in vacuum.

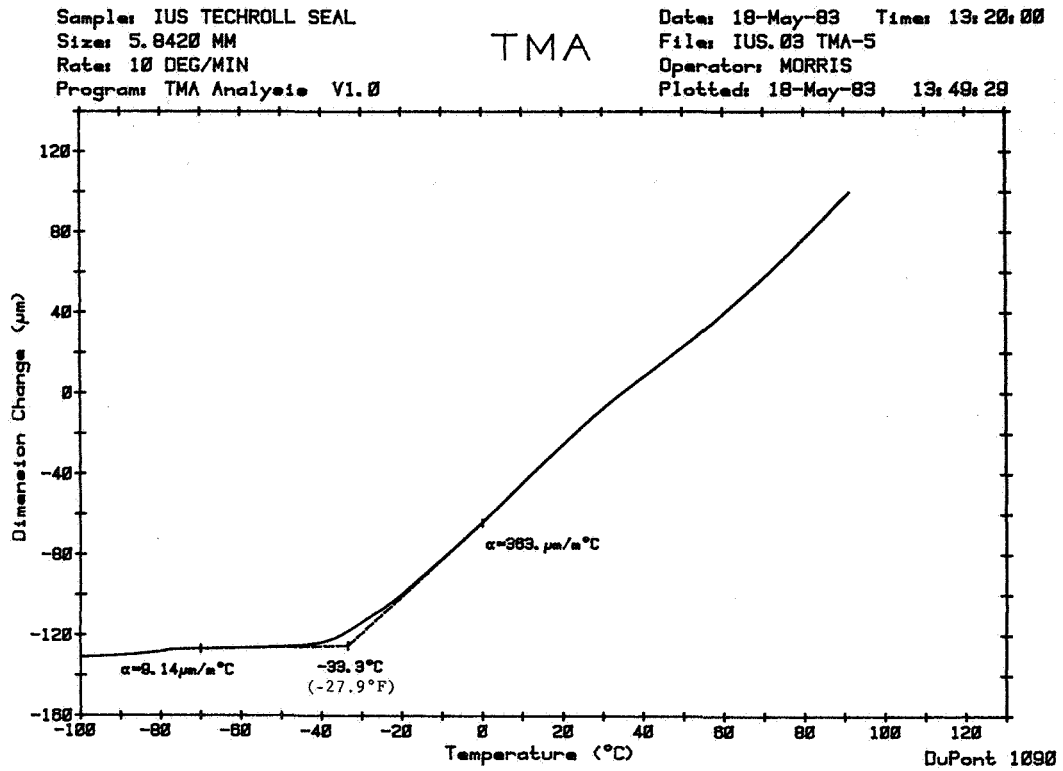


Figure 9. Thermomechanical analysis of Techroll seal material.

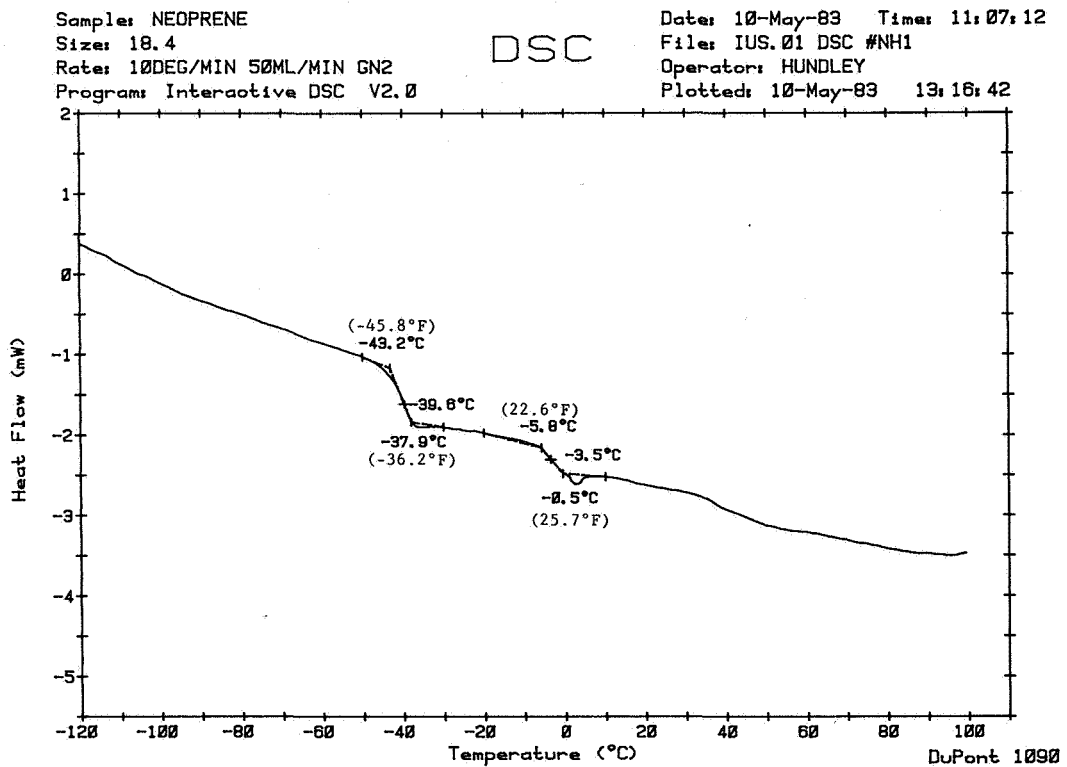


Figure 10. Differential scanning calorimeter analysis of neoprene.

Sample: TECHROLL SEAL
Size: 10.00X13.56X1.41 MM
Rate: 5 DEG/MIN
Program: Advanced DMA V1.0

DMA

Date: 16-May-83 Time: 14:23:57
File: TECHROLL.03 DMA DATA #4
Operator: LEDBETTER
Plotted: 17-May-83 7:06:09

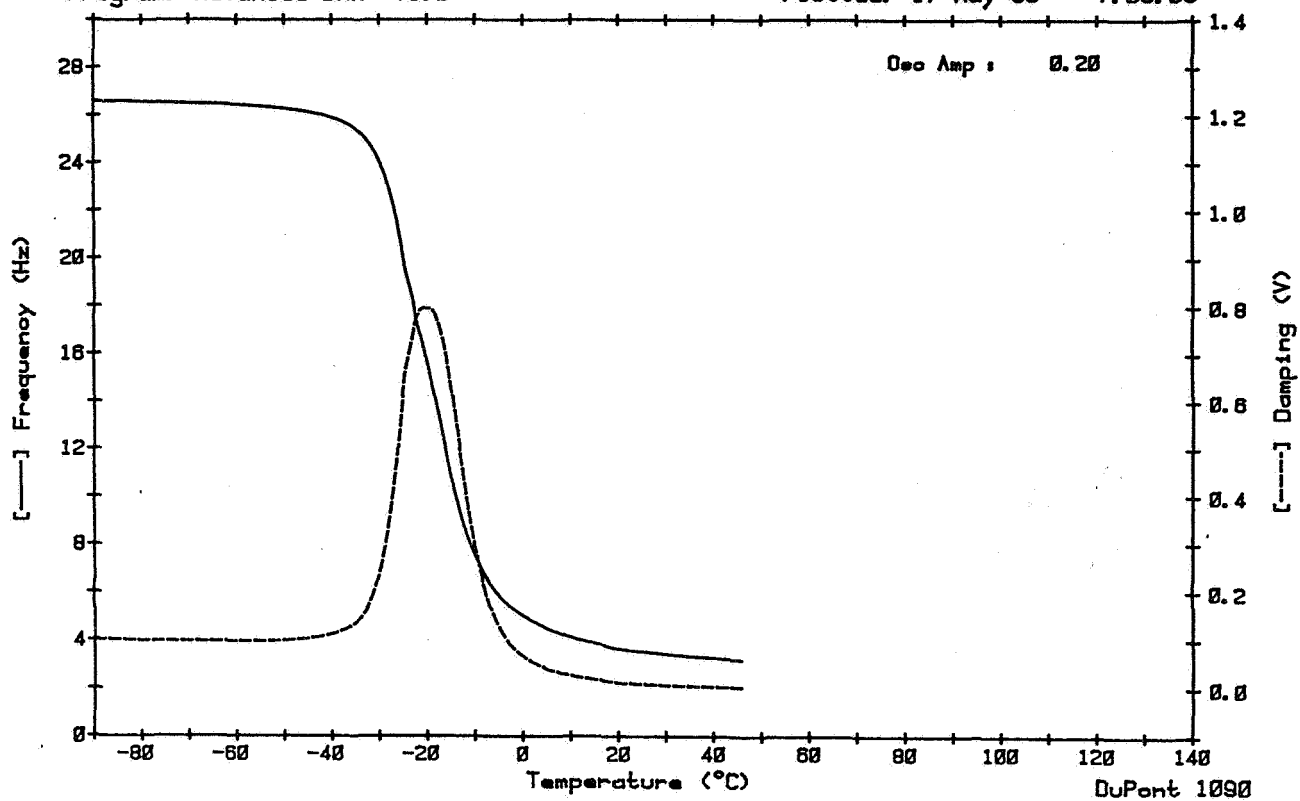


Figure 11. Dynamic mechanical analysis of Techroll seal material.



Figure 12. Kevlar fibers (3700X)

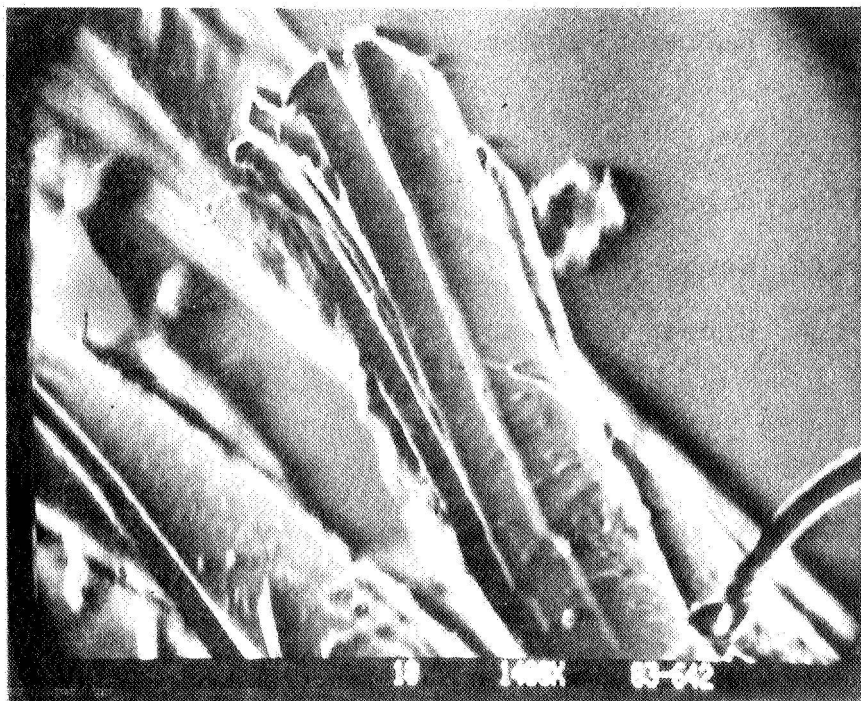


Figure 13. Kevlar fibers (400X)

TABLE 1. COMPARISON OF THERMALGRAVIMETRIC ANALYSIS OF TECHROLL SEAL MATERIAL IN DIFFERENT ENVIRONMENTS.

<u>Atmosphere</u>	<u>Temperature of First Weight Loss (°C)</u>	<u>Neoprene Decomposition Temperature (°C)</u>	<u>Total Weight Loss (%)</u>
Vacuum	75	285	57
Air	150	314	95
N ₂	200	328	52

TABLE 2. PEEL STRENGTH OF TECHROLL SEAL COMPOSITE

180 Degree Peel Test

<u>Flat Sheet</u>	<u>Peel Strength (lb/in.)</u>
0.010 in. Rubber to Kevlar	2.0
0.010 in. Kevlar to Kevlar	4.5
0.035 in. Rubber to Kevlar	4.0
<u>Flat Roll Seal</u>	
0.013 in. Rubber to Kevlar	3.0
0.013 in. Kevlar to Kevlar	0 - 4.0
0.040 in. Rubber to Kevlar	4.0

Inherently low bond strength between Kevlar and Neoprene Observed.

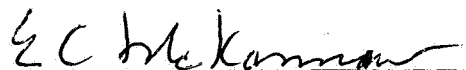
Large areas of delamination present in TRS.

APPROVAL

MATERIALS TESTING OF THE IUS TECHROLL SEAL MATERIAL

By Ronald Nichols and William B. Hall

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.



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