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THERMOPHYSICAL PROPERTIES OF A LOW-DENSITY PHENOLIC-NYLON Ablation Material

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by W. T. Engelke, C. M. Pyron, Jr., and C. D. Pears

Prepared by SOUTHERN RESEARCH INSTITUTE Birmingham, Ala. for Langley Research Center

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By W. T. Engelke, C. M. Pyron, Jr., and C. D. Pears

Distribution of this report is provided in the interest of information exchange. Responsibility for the contents resides in the author or organization that prepared it.

Prepared under Contract No. NAS 1-2978 by SOUTHERN RESEARCH INSTITUTE Birmingham, Ala.

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THERMOPHYSICAL PROPERTIES OF A

LOW-DENSITY PHENOLIC-NYLON ABLATION MATERIAL

By W. T. Engelke, C. M. Pyron, Jr., and C. D. Pears Southern Research Institute

SUMMARY

Some physical properties were determined on a low-density phenolicnylon that was both nondegraded (virgin) and thermally degraded (charred). The work was performed under Task Order 4 of Contract NAS 1-2978, which was a continuation of similar studies under previous Task Orders 1 through 3. Task Orders 1 through 3 have been reported in NASA TN D-2991 (Reference 1).

The low-density phenolic-nylon was a molded composite consisting of 25% phenolic resin, 35% phenolic Microballoons, and 40% powdered nylon. The material was thermally charred in an arc tunnel.

The thermal conductivity of the virgin material from -200° F to 750° F ranged from 1. 27 x 10^{-5} Btu/sec/ft/°F (7.91 x 10^{-2} W/m/°K) to a maximum of 1. 69 x 10^{-5} Btu/sec/ft/°F (10.5 x 10^{-2} W/m/°K). These values were lower than those exhibited for the low-density phenolic-nylon evaluated under Task Order 1. The reason for the lower conductivity appears to be the weaker structure of the material evaluated under this program.

The thermal conductivity from $1000^{\circ}F$ to $5000^{\circ}F$ of the charred material increased from 11.5×10^{-5} Btu/sec/ft/°F ($0.72 \text{ W/m}/^{\circ}K$) to 76×10^{-5} Btu/sec/ft/°F ($4.73 \text{ W/m}/^{\circ}K$). During cooling the values were higher and more scattered, indicating further degradation at the higher temperatures and some shrinking and cracking of the specimens upon cooling.

The heat capacity of the virgin material increased from 0.20 Btu/lb/°F (837 J/kg/°K) at -200°F to 0.58 Btu/lb/°F (2427 J/kg/°K) at 600°F. Due to the carbonaceous character of the charred material, the heat capacity compared well with that of graphite. The values increased from 0.39 Btu/lb/°F (1632 J/kg/°K) at 1000°F to 0.57 Btu/lb/°F (2385 J/kg/°K) at 5000°F.

The coefficient of thermal expansion at 70°F for the virgin material was 23.5×10^{-6} in./in./°F (42.3 $\times 10^{-6}$ m/m/°K). The data indicated slight anisotropy of the material and also demonstrated a transformation of the material above 230°F which caused the material to contract.

The permeability of both the virgin and charred material was obtained at low pressure drops across the specimen with helium and nitrogen used as the permeating gases. The admittance of the virgin specimens ranged from $3.6 \times 10^{-4} \text{ m}^2/\text{sec}$ to $5.2 \times 10^{-4} \text{ m}^2/\text{sec}$, and the charred material exhibited values from $0.81 \text{ m}^2/\text{sec}$ to $1.87 \text{ m}^2/\text{sec}$. The flow through the charred specimen was transitional and turbulent.

The total normal emittance of the char from 1500° F to 5000° F ranged from 0.79 to 0.87.

The mechanical properties of the virgin material from -200° F to 750° F were determined. The ultimate tensile strength decreased from 570 psi $(3.93 \times 10^6 \text{ N/m}^2)$ at -200° F to below 100 psi $(0.69 \times 10^6 \text{ N/m}^2)$ at 500° F. The ultimate compressive strength decreased from 2600 psi $(17.03 \times 10^6 \text{ N/m}^2)$ at -200° F to approximately 200 psi $(1.38 \times 10^6 \text{ N/m}^2)$ at 750° F. Both tensile and ultimate strength values were lower (about one-half) than those of the phenolic-nylon evaluated under Task Order 1. The weaker structure, mentioned before, was apparently the reason for the lower values.

INTRODUCTION

This is the final report on the thermophysical property evaluation of a low-density phenolic-nylon ablation material performed for the NASA Langley Research Center under Contract No. NAS 1-2978, Task Order 4. The physical properties of the low-density phenolic-nylon in both the nondegraded (virgin) and thermally degraded (charred) forms were determined and analyzed. This investigation was performed to characterize the material for its reentry application as an ablative heat shield. The program was a continuation of similar studies performed under the previous Task Orders 1 through 3 reported in NASA TN D-2991 (Reference 1).

Specifically, the thermal conductivity, heat capacity, thermal expansion, density, permeability, and mechanical properties were determined on the virgin material; and the thermal conductivity, heat capacity, total normal emittance, density, porosity, and permeability were determined on the charred

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material. The temperature ranges were -200°F to 750°F for the evaluation of the virgin material and 1000°F to 5000°F for the charred material. Reference 1 briefly describes the apparatuses and summarizes the measurement procedures that were applied, except for the permeability measurements. Additional details concerning the test methods may be found in references cited in the discussion of data.

SPECIMEN MATERIAL

The low-density phenolic-nylon material was supplied by the NASA Langley Research Center in both the nondegraded and thermally degraded forms. The nondegraded material was supplied in a block 12 inches in diameter and 4 inches thick. The thermally degraded material was supplied in 3 inch diameter discs with the charred area approximately $\frac{1}{4}$ inch deep.

This low-density phenolic-nylon composite was molded and consisted of the following materials: (1) 40% (weight) powdered 66 nylon, DuPont's Zytel 103; (2) 25% (weight) phenolic resin, Union Carbide Corporation's BRP-5549; (3) 35% (weight) phenolic Microballoons, Union Carbide Corporation's BJO-0930. The molding was performed at a maximum pressure of 2200 psi (15.17 x 10^6 N/m²) and temperature of 340°F. The material was also post cured at a maximum temperature of 300°F. The complete molding and post curing history are shown in Figures 1 and 2.

A more thorough description of the individual constituents is included in Appendix A. The difference in composition noted between this material and the low density phenolic-nylon evaluated under Task Order 1 is the different percentages of the constituents. The material under Task Order 1 contained 50% nylon, 25% resin and 25% Microballoons, and as discussed later, was stronger and exhibited a higher thermal conductivity. Under a separate contract study, reported in Reference 2, it was concluded that the formulation and processing of the material under this present program were less than optimum for a plastic composition. The formulation was low in volume fraction of phenolic resin and the processing method caused mechanical damage to the Microballoons and large temperature gradients during processing. Thus as reported in subsequent sections, the lower mechanical strength and thermal conductivity of this material when compared to the material evaluated under Task Order 1, may be directly correlated with the formulation and processing of the ablation material.

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The thermally degraded material was produced in an arc tunnel. The three inch diameter discs of the phenolic-nylon were exposed to the following test conditions to form the chars: a stagnation enthalpy of 2800 Btu/lb (6.5 x 10^6 J/kg), stagnation pressure of 0.57 atm (5.8 x 10^4 N/m^2), thermal flux at the material surface of 140 Btu/ft²/sec (1.58 x 10^6 W/m^2), freestream Mach number of 3.0 with nitrogen gas and an exposure time of 90 seconds. The arc tunnel is that described in NASA TN D-1621 with modifications to provide for supersonic flow.

To further define the as received material, the densities of both the virgin and charred materials were obtained. For the virgin material, the bulk density was determined at room temperature by the water displacement of a paraffin coated specimen in accordance with the ASTM D311-58 procedure. The duplicate values obtained from two pieces selected at random were 36.8 lb/ft^3 (589 kg/m³) and 36.3 lb/ft^3 (582 kg/m³) providing an average density of 36.6 lb/ft^3 or 586 kg/m^3 . The density above and below room temperature was adjusted with the thermal expansion data (reported under the thermal expansion section) and is shown in Table 1. The density was not calculated above 200° F since the thermal expansion data indicated a transformation or initial degradation above this temperature which was accompanied with some weight loss as indicated in the heat capacity data. This, along with erratic expansion observed above 200° F, would make calculations meaningless.

The bulk density of the char was also determined in accordance with ASTM D311-58, and the true density was determined from a pulverized sample by employing a pycnometer. The average bulk density, determined from duplicate measurements, was 15 lb/ft³ (240 kg/m³), and the average true density was 93.6 lb/ft³ (1500 kg/m³). The volume percentage of porosity, which is the ratio of the difference between the true and bulk densities over the true density, was 84%.

A micrographic evaluation was also performed on the char to determine the average pore size and relative frequency distribution. The two specimens $(\frac{3}{32}$ in. thick) evaluated were taken from the top surface of the char. The specimens were impregnated with polyalphamethylstyrene and polished. The specimens were viewed at 100X magnification in a plane parallel to the thickness direction, and the pores were counted and measured with a calibrated eye-piece. Figure 3 is a typical photomicrograph of the char structure. The average pore diameter was 28.8 microns, and the histogram of pore diameters is shown in Figure 4. As can be seen in Figure 3, the pore structure is rather irregular with wide variation in size.

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In comparing this char with the previous degraded low-density phenolicnylon evaluated under Task Order 3, it was observed that the average pore size was larger for this material; however, the density and porosity were the same.

THERMAL CONDUCTIVITY

The thermal conductivity was determined in one direction (thickness of the supplied block) on both the virgin and charred material. For the virgin material, data were obtained from -200°F (144°K) to 750°F (673°K). The temperature range for the charred material was from 1000°F (811°K) to 5000°F (3033°K) with the data being obtained during heating and cooling cycles. The thermal conductivity of the virgin material was determined using an ASTM C177 guarded hot plate apparatus. The char specimens were evaluated in the radial inflow apparatus.

Apparatus and Procedure

ASTM guarded hot plate. - The 7-inch diameter ASTM C177 guarded hot plate apparatus employed for the thermal conductivity evaluation of the virgin material is described fully in Reference 3 and briefly below.

Basically this apparatus consisted of a central heater plate surrounded by a guard heater, each separately controlled. The guard ring was maintained at the same temperature as the central heater so that all of the heat flow was normal to the specimen surfaces. The heater plate was centrally sandwiched between layers of filler material, hot face thermocouples, the specimen, cold face thermocouples, filler material, a copper plate, and finally a cold plate to dissipate the heat. In addition to the thermocouples in contact with the specimen, thermocouples were located in the central heater and the outer copper cold plates. Intimate contact was provided at all interfaces by pressing the entire assembly together with a screw loaded frame.

Filler pads of gum rubber were used from -200°F to 150°F. Above 150°F, Fiberfrax paper was used for the filler pads with a sheet of gum rubber at the cold plate. Overlapping data were obtained at the 150°F temperature level for both filler arrangements, providing a check on contact resistance resulting from irregularities of the specimen surface.

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To obtain mean temperatures of -200°F, liquid nitrogen was circulated through the cold plate. For higher temperatures, chilled trichloroethylene and water cooling were employed. When data points were obtained below room temperature, the apparatus was enclosed in a plastic bag which was purged with dry helium to eliminate frosting and moisture condensation.

During the run on Specimen 2, the change in thickness at the higher temperature levels was monitored. This was accomplished by cutting slots in the edge of the specimens. Small pads of 5 mil stainless steel shim stock were placed above and below the specimen, and the distance between these was measured with a hole gage. Measurements were taken at each temperature level. There was some uncertainty in these measurements since the specimen degraded and collapsed more rapidly at the edge than at the center due to the greater oxidation at the edge. This uncertainty was corrected at the last point by measuring the thickness at the center of the specimen after the completion of the run. The measurements taken at the intermediate temperatures were corrected in proportion to the difference obtained on this last point between the thickness change at the edge and at the center. The uncertainties resulting from this procedure are recorded with the data. In general, the thickness changed by about 20% during the run, and this change could be measured within an uncertainty of 3% at temperatures below about 400°F and within 8% at higher temperatures where degradation of the material was more severe.

Radial inflow apparatus. - To determine the thermal conductivity of the char specimens a modified procedure of the radial inflow apparatus was employed. The basic apparatus is fully described in Reference 1 and Reference 4.

Briefly, the test section was heated radiantly in a high temperature furnace which employed a cylindrical graphite resistance heating element. The heat flowed radially inward through the specimen to a central water flow calorimeter. Water temperatures in the calorimeter were indicated by thermocouples in the water stream located one-half inch apart, axially, and heat flow through the specimen gage section was computed from measurements of water flow rate and temperature rise. Specimen temperatures were measured in two axially drilled holes located on two different radii. Below 2000°F, temperature measurements were taken with chromelalumel thermocouples; above this temperature, measurements were taken by sighting the bottom of the holes, by using an optical pyrometer and a right angle mirror device. Axial conduction in the specimen is minimized by (1) insulating the specimen on each end with graphite sleeves filled with thermatomic carbon, (2) by making the specimen length at least twice the gage length, and (3) by providing an isothermal hot zone over at least twice the specimen length.

Thermal conductivity is calculated from the standard relation

$$k = \frac{ql}{A\Delta T}$$
(1)

where k is the thermal conductivity, A is the log mean cylindrical area, and l is the radial distance over which ΔT is measured.

To measure the thermal conductivity of the char specimens, the technique described above was modified as follows: Four char strips, approximately $\frac{3}{8}$ inch wide by $\frac{3}{16}$ inch thick by 2 inches long were arranged symmetrically as shown in Figure 5. For this configuration, it was necessary that isothermals in the specimen be perpendicular to the thickness direction. This condition was achieved by placing thin strips of pyrolytic graphite at the inner surfaces of the char strips. Because of the high anisotropy of this material (the conductivity is approximately 50 times greater in the "a" direction than in the "c". or thickness, direction) the isotherms were forced to assume a square configuration. Heat flow other than through the specimen was eliminated essentially by using thermatomic carbon of extremely low thermal conductivity (approximately 0.1 Btu/hr/ft²/ $^{\circ}$ F/in.) as packing at the edges of the specimens. The space around the calorimeter was packed with graphite. Temperatures were measured at two locations in the strips, using the methods described above. This procedure was employed with good results under previous Task Orders 2 and 3, and is reported in NASA TN D-2991.1*

During each run, the thermal conductivity was determined while the specimen was heated from 1000° F to 5000° F at 500° F intervals and cooled from 5000° F to 1000° F at 1000° F intervals. This was done to determine any alterations of the char after heating to the higher temperatures.

The machining of the conductivity specimens was performed after impregnating the char with polyalphamethylstyrene to provide sufficient mechanical strength. The evaluations were made after installing the specimens in the apparatus and heat soaking them at 1000°F. At this temperature, polyalphamethylstyrene effectively vaporized, leaving no residue. This technique was also employed successfully under Task Orders 2 and 3. The impregnation of the char was required only on the thermal conductivity specimens and the photomicrographic samples.

*See **REFERENCES**

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Data and Results

<u>Virgin material.</u> - The thermal conductivity data for the virgin material are presented in Figure 6 and Table 2. As shown in the figure, the conductivity increased sharply from 1.27×10^{-5} Btu/sec/ft/°F (7.91 x 10^{-2} W/m/°K) at -250°F to 1.69×10^{-5} Btu/sec/ft/°F (10.52 x 10^{-2} W/m/°K) at -100°F, then decreased to 1.27×10^{-5} Btu/sec/ft/°F (7.91 x 10^{-2} W/m/°K) at 0°F. From 0°F to 750°F the values increased steadily to 1.49×10^{-5} (7.73 x 10^{-2} W/m/°K). These values were lower than those exhibited by the phenolic-nylon material evaluated under Task Order 1; however, the characters of the curves were very similar. The lower conductivity of this material, which exhibited the same density as the previous phenolic-nylon, may have resulted from weaker bonding (higher thermal contact resistance) between the resin and filler. This obviously resulted from the formulation and processing being less than optimum for a plastic composition as discussed previously. As discussed in a subsequent section, the lower mechanical properties exhibited by this material also indicate the weaker bonding.

<u>Charred material.</u> - The thermal conductivity values of the charred material are shown in Figure 7 and Table 3. The conductivity and mean temperature were computed by assuming a linear gradient through the specimen as has been done in the past. As shown in the figure, the values increase steadily from 11.5 x 10^{-5} Btu/sec/ft/°F (0.72 W/m/°K) at 800°F to 76 x 10^{-5} Btu/sec/ft/°F (4.73 W/m/°K) at 5000°F during the heating cycle and during cooling the values decreased from 85.6 x 10^{-5} Btu/sec/ft/°F at 4300°F to 34.7 x 10^{-5} Btu/sec/ft/°F at 1500°F. It was found that the thermal conductivity of the material during heating could be described by the equation,

k = 11.57 x 10^{-5} + 5.3 x 10^{-15} T³

(2)

where

k = thermal conductivity, Btu/sec/ft/°F

T = mean temperature of specimen, °F

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Because of the scatter in the cooling data, these are not represented by an equation but are shown as a band in Figure 7. During heating the runs on three separate specimens agreed well; however, the cooling data were scattered and higher than the conductivity during the heating cycle.

Examinations of the specimens after the run revealed that cracking and shrinkage had occurred during cooling, and the structure appeared to be more porous. These effects would tend to scatter the data and cause higher indicated conductivities due to the increased radiant (and possibly convective) heat transport. The values obtained on cooling should be treated with caution. Figure 8 illustrates the structural changes of the specimens after exposures to 5000°F. Some erosion can be observed near the ends of the strips, however, the central $\frac{1}{2}$ inch gage lengths through which the conductivity was measured appear to be undamaged.

HEAT CAPACITY

The enthalpy and heat capacity were determined on both the virgin and charred material. The data were determined from -200°F to 750°F for the virgin material and from 1000°F to 5000°F for the charred material.

Two apparatuses were used in the determinations. For the evaluations of the virgin material, a drop type adiabatic calorimeter was employed. A drop type ice calorimeter was utilized in the evaluations of the char material.

Apparatus and Procedure

Adiabatic calorimeter. - This apparatus is fully described in Reference 3 so that only the major features are discussed here. Briefly, the enthalpy of a specimen at a particular temperature was determined by dropping the heated or cooled specimen into a cup which was maintained adiabatic. Enthalpy was determined from the weight of the specimen and the temperature change of the cup. The calorimeter cup was placed in an insulated container which was immersed in a bath of ethylene glycol. Adiabatic conditions were maintained by heating or cooling the bath. The specific heat was calculated from the slope of the enthalpy versus temperature curve. This slope was determined by averaging both a graphical solution and an analytical solution in which the enthalpy curve was fitted by a least squares approach, and the resulting equation differentiated to obtain the specific heat.

The temperature range of the adiabatic calorimeter equipment was extended from -50° F to -250° F. This was accomplished by cooling the specimen in a cooling chamber specially designed for inserting within the standard cold box. The cooling chamber is constructed of two concentric

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cylinders in which the specimen was inserted within the central cylinder, and liquid nitrogen was placed in the annulus between the cylinders. With this system, temperatures of -250° F were readily obtained. A continuous dry helium purge was maintained eliminating any "frosting" on the specimen.

Ice calorimeter. - The drop type ice calorimeter employed to determine the enthalpy of the degraded material is fully described in Reference 4.

This calorimeter employed a cup surrounded by an ice mantle. The enthalpy determinations were made by dropping the heated specimen into the apparatus and measuring the volume of ice melted as the specimen cooled to 32° F. Specific heat was calculated from the slope of the enthalpy versus temperature curve.

Data and Results

<u>Virgin material.</u> - The enthalpy and heat capacity data of the virgin material are included in Figure 9 and Table 4. The heat capacity values increased from 0.20 Btu/lb/°F (837 J/kg/°K) at -200°F to 0.58 Btu/lb/°F (2427 J/kg/°K) at 600°F, with the values above 600°F becoming erratic due to the excessive degradation of the material. The data up to 600°F agreed well with the previous values obtained on the low-density phenolic-nylon evaluated under Task Order 1. In comparison with the heat capacity of phenolic-carbons, graphites, and silicas evaluated here previously, the phenolic-nylon exhibited higher values. This was expected since the heat capacity of nylon is significantly higher than that of carbon, graphite, or silica.

<u>Charred material.</u> - The enthalpy and heat capacity data for the charred phenolic-nylon are included in Figure 10 and Table 5. As shown in the figure, the heat capacity increased from 0.39 $Btu/lb/^{\circ}F$ (1632 J/kg/ $^{\circ}K$) at 1000°F to 0.57 $Btu/lb/^{\circ}F$ (2385 J/kg/ $^{\circ}K$) at 5000°F. This compares favorably with the heat capacity obtained on the degraded phenolic-nylon evaluated under Task Order 3, which remained a constant 0.52 $Btu/lb/^{\circ}F$ (2175 J/kg/ $^{\circ}K$) from 1200°F to 5000°F. As was expected, due to the carbonaceous character of this material, the values obtained also compared well with those of graphite (0.4 $Btu/lb/^{\circ}F$ at 2000°F to 0.55 $Btu/lb/^{\circ}F$ at 5000°F for CFZ).

The scatter of the enthalpy data was relatively high due to the small mass of the specimens; therefore, four specimens were evaluated in order to reduce the uncertainty.

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THERMAL EXPANSION

The thermal expansion of only the virgin material was determined from -200° F to 750° F employing the precision quartz tube dilatometer.

Apparatus and Procedure

A complete description of the quartz tube dilatometers is included in Reference 3. Briefly, the specimen was placed in the bottom of a quartz cylinder and a quartz rod placed on the specimen extended to the open end of the cylinder. The thermal expansion was indicated by a precision dial gage mounted to the cylinder with its stylus bearing on the rod. The specimen, which was heated in a tube type furnace, was instrumented with thermocouples at three locations along its length to minitor temperatures and temperature gradients. From calibration with standards such as "A" nickel, the precision of the measurements has been confirmed within 0.0001 inch scatter at any level and the total uncertainty within + 5%.

The cryogenic temperatures were obtained by two methods for Specimens 1 and 2. The first was performed by immersing the quartz dilatometer in a dewar filled with liquid nitrogen. The second utilized a coil with several orifices drilled on the inside diameter which provided a surrounding spray of the liquid and gaseous nitrogen on the outside of the quartz tube. The second method provided better control of the temperatures desired.

Data and Results

The thermal expansion data are shown in Figure 11 and Tables 6 and 7. Duplicate data were obtained in the direction perpendicular to the 4 inch thickness defined as "ab" direction, and singular data from room temperature to 750°F were obtained in the thickness direction defined as the "c" direction. The determination in the thickness direction was not required under the contract but was performed in order to determine if any anisotropy of expansion existed in this material.

As can be seen from the figure, in the "ab" direction the material expanded steadily from -200°F to 230°F at which point contraction occurred and continued until 750°F. The coefficient of expansion in the "ab" direction at room temperature was 23.5×10^{-6} in./in./°F (42.3×10^{-6} m/m/°K).

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The expansion in the "c" direction, also shown in Figure 11, differed from that in the "ab" direction above 150° F, being about 20% greater at 230° F. Above 230° F, contraction in the "c" direction occurred in a manner similar to the behavior in the "ab" direction; however, above the cure temperature (340° F) of the material, erratic data were obtained.

The difference in the observed expansions in the "ab" and "c" directions may have been due to some anisotropy in the material, but more likely these differences, particularly above the cure temperature of **340°F**, were the result of degradation of the material. We have noted with previous expansion evaluations on reinforced plastics that erratic and unrepeatable data are usually obtained above the material's cure temperature due to the spurious thermal motions associated with degradation. More evaluations, which were beyond the scope of this contract, would be required to fully define the anisotropy that does exist.

In comparing the above values with those obtained on the phenolic-nylon evaluated under Task Order 1, the prior phenolic-nylon exhibited expansions about 10% higher over the range from -300° F to room temperature. From room temperature to about 150°F the curves for the two materials were almost identical. Above 150°F the prior phenolic-nylon also behaved erratically but did not begin to contract until about 400°F.

PERMEABILITY

The permeability on both the virgin and charred materials was determined at room temperature.

Apparatus and Procedure

The apparatus employed for both virgin and charred material is completely described in Appendix B. The procedure simply provides a pressure drop across the specimen by mounting and sealing the specimen between two chambers at different pressures. The flow of the permeating gas through the specimen is monitored downstream from the specimen. The measurement of the flow, specimen mean pressure, and pressure drop across the specimen provided the data required to investigate the permeability of the material. These data can be utilized in various equations to provide permeability coefficients which are applicable for various flow conditions. We chose to determine the admittance (K_{mv}) defined in the equation

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$$K_{mv} = \frac{P_m Q_m}{\Delta P} \frac{L}{A}$$

where

 K_{my} = admittance, cm²/sec

 P_m = mean pressure in specimen

- $Q_m = flow rate through specimen, defined at mean pressure cm³/sec$
- L = thickness of specimen, cm

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A = cross sectional area of specimen exposed to flow, cm^2

 ΔP = pressure drop through specimen

This admittance value is applicable to the combined Poiseuille (viscous) and Knudsen (molecular) flows.

Permeating gases of helium and nitrogen were used in this investigation. Gas flow rates through the virgin material were measured with a bubble flowmeter; the higher flow rates through the more porous char material were monitored with a precision Rotameter made by Fischer and Porter Company. Pressure differentials across the specimen ranged from 1.5 to 9.8 inches of water. Two thicknesses (approximately 0.25and 0.43 inches) of the virgin phenolic-nylon and only one thickness (0.25 inch) of the char were evaluated.

Data and Results

The pressure drop across, flow through, and mean pressure of both the virgin and charred specimens are shown in Tables 8 and 9. The calculated admittance is also included in the table to facilitate a comparison of the data obtained under the various conditions for the two materials.

<u>Virgin material.</u> - It was observed that for the virgin material the admittance did not change (within the limits of uncertainty of the equipment) with the two purge gases or with the variation in pressure differentials across the specimen. However, some increase in values was noted for the thicker specimens. The average values of the thinner Specimens 1, 4, and 5 were 4.15×10^{-4} , 3.88×10^{-4} , and 4.10×10^{-4} m²/sec, respectively; whereas, the average values of the two thicker specimens were 4.29×10^{-4} and 5.07×10^{-4} m²/sec, respectively.

<u>Charred material.</u> - For the degraded material, the admittance was higher due to the cracked and porous structure and varied depending on the permeating gas used and pressure gradient across the specimen. Nitrogen provided lower values (approximately 0.8 to $1.0 \text{ m}^2/\text{sec}$) than those exhibited with helium (approximately 1.4 to $1.9 \text{ m}^2/\text{sec}$), and there also was a drop of 20% in the admittance as the pressure drop across the specimen was increased from 1.5 to 3.0 inch of water.

It is important to know the type of flow that occurs within the char in studying the effectiveness of transpirational cooling during ablation. Due to the size variations of the pores and channels within the material, the type of flow will naturally vary between turbulent, transitional, and laminar within the different passages. The data obtained on this char do indicate that most of the flow was either transitional or turbulent. This was demonstrated by the fact that the helium provided higher flow rates through the char when compared with nitrogen. In a flow regime of mostly turbulent flow, the volume flow rate is inversely proportional to the square root of the gas density, and for laminar flow the flow rate is inversely proportional to the viscosity. This can be seen from Darcy's equation (valid here due to the low pressure drop) and was also illustrated by Creutz. Since there is a wide variation in density (nitrogen 0.072 lb/ft³; helium 0.010 $1b/ft^3$) and only a slight difference in viscosity (nitrogen 1770 x 10⁷ poise; helium 1960×10^7 poise) of the two gases, the higher flow of the helium indicated that very little laminar flow occurred.

The same conclusion can also be drawn by applying the data to the following relation offered by Creutz:

 $Q\alpha(P\Delta P)$

where

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Q = flow of gas through the sample (corrected for Knudsen and slip flow rates)

- P = mean pressure of the specimen
- ΔP = pressure drop across the specimen
- δ = exponent which is 0.5 for turbulent and 1.0 for laminar flow

Using the data shown in Table 9, an approximate value of δ can be obtained by plotting Q vs (P Δ P) on logarithmic coordinates and determining the slope, which is the exponent δ . This was done, without correcting for Knudsen or slip flow (which would be relatively small at these large flows), and the slopes were about 0.9 for helium and 0.7 for nitrogen. This value indicates the flow to be transitional. Therefore, for the pressure drops between 1.5 to 3.0 inches of water, the flow should be considered transitional or turbulent for this char.

TOTAL NORMAL EMITTANCE

The total normal emittance of the degraded material from 1500°F to 4000°F was determined. This was performed by comparing the irradiance from the specimen to that from a cavity-type blackbody maintained at the same temperature.

Apparatus and Procedure

A complete description of the apparatus and procedure employed for the emittance determinations is included in Reference 4. Briefly, the specimen was heated in an induction furnace and its irradiance was monitored by a 160-junction thermopile calibrated against a cavity type blackbody to about 5200°F. An optical pyrometer was employed to monitor temperature of the specimen. The assumption of graybody emittance and the use of the Wein and Stefan-Boltzmann equations permitted an iterative calculation of true temperature and emittance.

Temperatures below $1500\,^{\circ}$ F were not obtained since the irregular surface of the char and its weak structure inhibited the proper contact required for a surface thermocouple. Temperatures above $4000\,^{\circ}$ F were not obtained due to the destruction of the tungsten heating discs under the specimen. The high heat flux and low conductivity of the char created a very high temperature gradient across the specimen which accounted for the melting of the tungsten disc while the specimen surface temperature was about $4000\,^{\circ}$ F.

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The specimens were prepared by carefully cutting the discs $(\frac{1}{2}$ inch diameter by $\frac{3}{16}$ inch to $\frac{1}{8}$ inch thick) from the charred material. The top surface of the char was unaltered from the as received condition and no resin impregnant was used.

Data and Results

The total normal emittance data are shown in Figure 12 and Table 10. The emittance increased slightly from 0.79 at 1550°F to 0.87 at 3300°F and then decreased to 0.79 at 4000° F. These values were, over most of the temperature range, slightly lower than those obtained on the low-density, phenolic-nylon char evaluated under Task Order 3. The significant difference between the emittances of the two materials occurred between the temperatures of 3000° F and 3500° F, where the emittance of the char under Task Order 3 decreased due to the formation of a white residue. The same residue was noted on Specimen 1 (see remarks in Table 10); however, its effect on emittance was negligible for this char since the amount of residue formed on the present specimens was probably less. It was noted during the runs that the "speckled" appearance of the specimen viewed through the optical pyrometer was not as noticeable for these runs when compared with the runs performed under Task Order 3 indicating less residue formation. Apparently the residue was formed by impurities within the char as discussed in Reference 6.

Extra specimens were evaluated for emittance to obtain a representative mean due to the higher than normal data scatter which resulted from the varying surface structure and the gray to black color variation. The variation in the porous surface structure apparently caused different degrees of subsurface radiation.

MECHANICAL PROPERTIES

On the nondegraded material, the tensile and compressive strength properties in one direction were obtained from -200° F to 750° F. The specific properties determined were ultimate strength, yield strength at 0.2% offset, Poisson's ratio, Young's modulus, total elongation or compression, and stress-strain curves.

Apparatus and Procedure

The apparatus and procedures employed for the mechanical property determinations are completely described in Reference 3. The specimens were loaded in a Tinius-Olsen universal testing machine. Curves of stress versus strain and lateral versus axial strain were continuously plotted to failure on X-Y recorders. Strains were measured axially and laterally in one direction. Triaxial strains were recorded during the initial tests until it was confirmed that the material was essentially isotropic for this property. Temperatures were obtained by programmed heating above 70°F using tungsten filament radiation lamps controlled by a Powerstat. For cooling, the specimen was surrounded by cotton sprayed with liquid or gaseous nitrogen at a rate sufficient to provide the desired temperature. The load was programmed to provide a given stress rate for failure at approximately 5 minutes, thus the stress rates differed at different temperatures. The strain in the direction of loading was measured by dual "strain gage" clip-on extensometers mounted on lugs clamped to the specimen. The strain in the lateral direction (perpendicular to the loading direction) was measured by mechanical extensometers in contact with the specimen which transmitted the lateral motion to a differential voltage transformer.

This type of instrumentation provided precision recordings of the load and the biaxial strains, continuously, to failure of the specimen. The Young's modulus, ultimate strength, yield strength, Poisson's ratio, and total elongation or compression were obtained from the curves.

The tensile specimens employed were $8\frac{1}{4}$ inches long containing a gage section $2\frac{1}{2}$ inches long by $\frac{1}{2}$ inch wide by $\frac{1}{4}$ inch thick. The compressive specimens were $\frac{1}{2}$ inch by $\frac{1}{2}$ inch in cross section by 1 inch in the loading direction.

Data and Results

<u>Tensile properties.</u> - All the tensile property values are shown in Figures 13 and 14 and Table 11. Data were obtained to only 600° F since burning of the specimens at higher temperatures caused premature breaking. The tensile stress-strain curves, which are direct tracings from the X-Y recordings, are included in Figures C1 through C22 of Appendix C. Yield strength was not obtained on all runs in tension since fracture occurred in most cases before the 0.2% offset.

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As can be seen from Figures 13 and 14, the tensile strength and elastic modulus decreased with an increase in temperature, with a sharp drop occurring between 200°F and 300°F. The ultimate strength decreased from 570 psi $(3.93 \times 10^6 \text{ N/m}^2)$ at -200°F to below 100 psi $(0.69 \times 10^6 \text{ N/m}^2)$ at 500°F. These values were about 50% lower than values obtained on the low-density phenolic-nylon evaluated under Task Order 1. The lower elastic modulus, yield strength and total elongation indicate the significantly weaker structure of this material when compared with the previous lowdensity phenolic-nylon. The weaker structure obviously resulted from the formulation and processing being less than optimum for this material.

Considerable scatter was observed in the ultimate strength values between $-200^{\circ}F$ and $200^{\circ}F$, and in elastic modulus and Poisson's ratio at $-200^{\circ}F$. This scatter may have resulted from several factors, such as variations in stress rates (recall that these were varied to maintain the loading period constant at about five minutes) or in the geometry of the specimens which may have resulted in nonuniformities in stress and strain. Unfortunately, time and funds did not permit an evaluation of these parameters; however, such a study will be performed on a low-density phenolicnylon of similar composition under a follow up task order (Task Order 4 under Contract No. NAS 1-5448).

The sharp drop in the strength and modulus above 200°F correlates with the contraction at 230°F observed in the thermal expansion measurements. Apparently the material undergoes a transformation above 200°F, even though this temperature is below the cure temperature. Above the cure temperature deterioration of the material contributed to the decrease in strength. Typical photographs of the specimens after the run illustrating the deterioration are shown in Figure 15. Also illustrated are the types of fracture that occurred. The relatively flat breaks obtained did indicate good tensile failure for most of the specimens.

<u>Compressive properties.</u> - All compressive property values are shown in Figures 16 and 17 and Table 12. The compressive stress-strain curves are included in Figures C23 through C48 of Appendix C. Some of these curves are direct tracings of the X-Y recorder; however, the others were plotted since the lateral strains were obtained on a recording oscillograph.

As shown in Figures 16 and 17, the strengths and moduli decreased steadily with an increase in temperature. The ultimate strength decreased from 2600 psi $(17.93 \times 10^6 \text{ N/m}^2)$ at -200°F to approximately 200 psi $(1.38 \times 10^6 \text{ N/m}^2)$ at 750°F. As was the case with the tensile properties, these values were considerably lower (again by approximately one-half) than

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those exhibited for the previous phenolic-nylon evaluated under Task Order 1. As did the tensile strengths, the compressive strength values exhibited considerable scatter below 200°F. The elastic modulus and Poisson's ratio values were also scattered, though not as much as the values measured in tension. Further studies are needed to isolate variations in the test methods from those inherent in the material.

DISCUSSION OF RESULTS AND A COMPARISON WITH PROPERTIES OF PRIOR PHENOLIC - NYLON

Some correlation of the properties can be made from a review of the data. For purposes of comparison, comprehensive plots of the properties of the virgin low-density phenolic-nylon are presented in Figures 18 and 19. Properties of the prior low-density phenolic-nylon, evaluated under Task Order 1 of Contract No. NAS 1-2978, are also shown.

Reference to Figure 18 shows that the thermal conductivity of the phenolic-nylon evaluated in this task order ranged from 7 to 23 percent lower than that of the phenolic-nylon evaluated under the prior Task Order 1. As discussed earlier, the lower conductivity may have resulted from the high percentages of filler and poorer bonding between resin and filler in this material. The characters of both curves were similar, both exhibiting a sharp peak in the vicinity of -100° F. The reason for this peak, which is about 30 percent higher than the values on either extremity, is not known.

The enthalpies of the current and prior materials were essentially the same.

The thermal expansion of the current phenolic-nylon was lower than that of the prior material over the temperature range from -300° F to 150° F. Both materials exhibited erratic behavior above 150° F.

Figure 19 shows composite plots of the mechanical properties of the current and prior (Task Order 1) phenolic-nylons. The ultimate tensile and compressive strengths of the current material were about 50 percent lower at -200° F than those of the prior material but decreased less with temperature, so that at 600°F the two materials exhibited about the same strengths. The tensile modulus exhibited a rather sharp decrease at 200°F (recall that thermal expansion exhibited a reversal near 200°F). The moduli were lower than those of the prior material from -200° F to 200°F and about the same at higher temperatures.

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Figure 20 shows a composite plot of the thermal properties of the low-density phenolic-nylon char. For comparison, data are presented for a high-density and a low-density phenolic-nylon char evaluated under prior Task Orders 2 and 3, respectively, of Contract No. NAS 1-2978.

Observe that the emittance of this char remained more nearly constant with temperature than that of the prior low-density char, but was slightly lower over most of the temperature range.

The thermal conductivity of this char was lower than that of either of the prior chars. As expected, the characters of the three curves were similar with the values increasing rapidly with temperature due to radiation. The differences in the conductivities of this and the prior low-density char can probably be attributed to differences in microstructures, even though the densities and porosities of the two materials were about the same. The current material was more heterogeneous, containing areas of relatively dense char separated by large voids (see the prior Figure 3). The median pore size, which is probably more representative of the relatively dense areas than the mean pore size, was 18.2 microns. Reference to Figure 4 shows that the majority of the pores are clustered around this size. On the other hand, the prior low-density char was more uniform in appearance, exhibiting few large voids. The average pore size of the prior material was also larger at 24.2 microns. At higher temperatures, radiant heat transfer would be larger through the char having the larger pore size, other factors being equal. At lower temperatures differences in conductivities could result from such factors as degree of graphitization of the matrix. X-ray diffraction patterns, not obtained in these programs, might detect such differences.

The current low-density char also exhibited the lowest enthalpy of the three chars, although the heat capacities of the two low-density chars were about the same. The heat capacity of the high-density char increased more with temperature, being about 20 percent greater at 4000°F than that of the low-density chars.

Southern Research Institute Birmingham, Alabama February 24, 1967

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Figure 1. Molding conditions for the low-density phenolic-nylon (data supplied by NASA, Langley Research Center)



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Figure 2. Post cure of low-density phenolic-nylon (data supplied by NASA, Langley Research Center)



Figure 3. Photomicrograph of typical pore structure of the low-density phenolic-nylon char (100X)



Figure 4. Histogram of pore diameters in the low-density phenolic-nylon char



Figure 5. Strip specimen configuration for thermal conductivity evaluation in the radial inflow apparatus

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Thermal conductivity in 10⁻⁵ - Btu/sec/ft/°F

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Figure 7. Thermal conductivity of low-density phenolic-nylon char







Figure 9. Enthalpy and heat capacity of the virgin low-density phenolic-nylon



Figure 10. Enthalpy and heat capacity of the low-density phenolic-nylon char





Temperature - °K

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Figure 13. Ultimate tensile strength versus temperature for the virgin low-density phenolic nylon


Figure 14. Initial elastic modulus and initial Poisson's ratio loaded in tension versus temperature for the virgin low-density phenolic-nylon

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gage areas showing fracture and thermal degradation of material at 400°F, 600°F, and 700°F Photographs (about 3X) of virgin low-density phenolic-nylon tensile specimens' Figure 15.



Figure 16. Yield and ultimate compressive strength versus temperature for the virgin low-density phenolic-nylon



Figure 17. Initial elastic modulus and initial Poisson's ratio loaded in compression versus temperature for the virgin low-density phenolic-nylon



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Tempe	rature	Den	sity 3
°F	°K	kg/m ³	lb/ft
-200	144	596	37.2
-100	200	592	36, 9
0	255	589	36.8
75	297	586	36.6
100	311	585	36.5
200	366	579	36.1

DENSITY VARIATION WITH TEMPERATURE OF THE VIRGIN LOW-DENSITY PHENOLIC-NYLON

*Calculated from density values measured at room temperature and measured coefficients of thermal expansion.

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Average specimen mean temperature - °F	Total heat input watts	Average specimen ΔT °F	Specimen thermal conductivity in 10 ⁻⁵ Btu/sec/ft/°F	Specimen thermal conductivity W/m/°K	Time at temperature hour
Specimen No. 1					
Gum Rubber Filler	4 00	49.0	1 95	0.070	E 0
-241.0	4.02	43.0	1.20	0.078	5.U
-01.0	0.90	02 1	1.10	0.000	5.0
-13.5	5 20	45 0	1 28	0.009	5.0
110.0	0.20	10.0	1.20	0.000	0.0
Fiberfrax- Gum					
Rubber Filler			.[
182.3	9.08	87.2	1.17	0.073	4.0
359.4	27.80	230.6	1.36	0.085	4.5
398.8	16.25	136.7	1.34	0.084	5.5
547.0	25.17	195.2	1.45	0.091	4.8
606.0	30.45	223.0	1.54	0.096	14.0
821.8	44. 78	293.5	1.53	0. 095	6.5
Specimen No. 2		~			
Gum Rubber Filler					
-202. 2	4.70	39.9	1.35	0.084	6.0
-101.6	6.52	48.1	1.55	0.097	5.3
-20.1	5.86	80. 7	1.35	0.084	4.5
117.0	5. 42	46. 2	1.34	0.084	18.0
Fiberfrax - Gum			a series and		
Rubber Filler			1	1	
168.5	9.07	76.2	1.36	0.085	6.0
324.8	26.23	201.8	1.43	0.089	18.5
362.7	16.73	129.3	1.42	0.088	6.5
490. 7	24.48	170.0	1.49	0.093	16.0
592.4	30.63	209.3	1.47	0.092	7.5
594.8	30. 52	211.7	1.45	0.090	22.7
714.8	37.28	259.5	1.31	0.082	8.3
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THERMAL CONDUCTIVITY OF THE VIRGIN LOW-DENSITY PHENOLIC-NYLON

Central diameter = 4.0 inches

Specimen thickness

Specimen 1 = 0.2493 inch prior to run 0.2216 inch after run Specimen 2 = 0.2529 inch prior to run (no change observed to $325^{\circ}F$)

 $= 0.2439 \text{ inch at } 324.8^{\circ}\text{F} \pm 3\% \\ = 0.2421 \text{ inch at } 362.7^{\circ}\text{F} \pm 3\% \\ = 0.2289 \text{ inch at } 490.7^{\circ}\text{F} \pm 7\% \\ = 0.2221 \text{ inch at } 592.4^{\circ}\text{F} \pm 8\% \\ \end{bmatrix}$

Thickness estimated from monitoring thickness

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change by measuring the gap spacing between

the hot and cold face of the specimen.

= 0.2217 inch at 594. $8^{\circ}F + 8\%$ = 0.2015 inch at 714. $8^{\circ}F - measured after run with micrometer.$

THE THERMAL CONDUCTIVITY OF LOW-DENSITY PHENOLIC-NYLON CHAR, USING THE RADIAL INFLOW APPARATUS

	Notes	اسو اسو اسو اسو			9 9 9 9	శాశా బిబిబి	ళ శాశా లో లో లో
ivity	Watts/meter/°K	0.816 0.852 0.852 0.846 0.839	0. 882 0. 895 0. 845 0. 864	1. 036 1. 008 1. 018	1. 687 1. 658 1. 658	1.875 1.889 1.846	2. 031 2. 091 2. 091
thermal conduct	in 10 ⁻⁵ Btu/sec/ft/°F	13.1 13.7 13.6 13.5	14.2 14.4 13.5 4	16.3 16.3 16.3	28. 1 26. 6 26. 2	30.1 30.3 29.6	34. 0 33. 6 33. 6
Specimen	Btu/hr/ft ² /°F/in.	5.66 5.81 5.87	6. 12 6. 21 5. 86 5. 99	7.19 6.99 7.06	11.7 8.11 8.11	13.0 13.1 12.8	145
Mean temperature	H.	1068 1068 1068 1070	1540 1545 1545 1546	2115 2115 2115	2805 2805 2805	3275 3275 3275	3670 3670 3670
Total heat removed by 1" colorimeter	gage length Btu/hr	130 136 135	188 192 182	258 250 253	342 336 339	428 429 421	502 493 493
	gage length F	710 712 712	951 956 958 961	1107 1107 1107	834 834 834	934 934 460 34	968 968 968 968
Specimen	outer lace temperature °F	1,11,1	2110 2110 2110 2110	2800 2800 2800	3425 3425 3425	3970 3970 3970	4390 4390 4350
	Time	On 7:45 9:00	Up 9:10 9:50	Up 10:15 11:00	11:45	Up 1:30 1:45	Up 2:10 2:45
	SRI run number	Run 1 on Specimen 1 (5-12-66) Cal. A-5	****				

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THE THERMAL CONDUCTIVITY OF LOW-DENSITY PHENOLIC-NYLON CHAR USING THE RADIAL INFLOW APPARATUS

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ctivity	Watts/meter/°K	2. 653 2. 682 2. 480	2 . 912 2. 956 2. 942	4. 744	2,278 2,235 235	2 485 2 581 2 581 2 581 2 581	2.120 2.134 2.076
in thermal condu	in 10 ⁻⁵ Btu/sec/ft/°F	42.6 30.8 30.8 30.8	46. 7 47. 4 47. 1	73. 7	36. 6 37. 4 83. 8 8. 8	40.0 41.4 41.9	34. 0 34. 2 33. 3
Specime	Btu/hr/ft /°F/in.	18.4 17.2 17.2		32.9	15.05	17.3 17.9 18.1	14. 7 14. 8 14. 4
	Mean temperature of specimen °F	4140 4140 4140	4475 4475 4475	4925	3360 3360 3360 3360	3080 3080 3080 3080	2163 2161 2156
Total heat removed by	r calorimeter gage length Btu/hr	568 573 529	607 614 612	672	438 438 438 430 430	365 374 378 381	282 284 276
	4.1. across gage length °F	878 878 878	52 57 58 8 8 8 8 8 8 8 8	280	044 440 440	8 8 8 8 8 8 8 8 8 8 8 8	547 547 547
Specimen	outer lace temperature °F	4795 4795 4795	0110 5110 5110	5355	3930 3930 3930 3930	3525 3525 3525 3525	111
	1 I I B	Up 3:05 3:55	Up 4:25 5:10	Up 5:35 6:00	Down 6:50	Down 7:17 7:45	Down 8:09 9:20
	SRI run aumber	Run 1 on Specimen 1 (continued)					

THE THERMAL CONDUCTIVITY OF LOW-DENSITY PHENOLIC-NYLON CHAR USING THE RADIAL INFLOW APPARATUS

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ductivity	Watts/meter/°K	0. 676 0. 688 0. 672 0. 658	0. 799 0. 790 0. 782 0. 756	0. 900 0. 885 0. 906 0. 906	1.269 1.257 1.246 1.257	2. 379 2. 365 2. 336 2. 336
nen thermal con	in 10 ⁻⁹ Btu/sec/ft/°F	10.9 11.0 10.8 10.8	12.8 12.5 12.5 12.1	संसं भारत है। संसं संसं संस का स	20.2 20.3 20.5 20.5 20.5 20.5 20.5 20.5 20.5 20.5	38. 2 37. 5 37. 5
Specit	Btu/hr/ft ^a /°F/in.	4. 68 4. 66 4. 56	5.55 5.45 5.45 5.45 5.45 5.45 5.45 5.45	6.24 6.23 28 28 28 28 28 28 28 28 28 28 28 28 28	8.80 8.72 8.64 8.72	16.5 16.2 16.2
	Mean temperature of specimen °F	814 816 816 818	1347 1341 1358 1356	1776 1781 1785 1788	2515 2515 2515 2555	3375 3375 3375 3375
Total heat removed by	r calorimeter gage length Btu/hr	97.9 99.7 95.8	169 168 168 168	231 232 232 232	320 315 316	390 388 384 385
	AT across gage length °F	585 586 589 589	8 8 8 8 5 8 8 5 0 5 8 8 5 0 5 8 8 5 0 5 8 8 5 0 5 8 8 5 8 5	1035 1035 1036 1038	1000 1000 1000	650 650 650 650
Specimen	outer face temperature °F		1890 1890 1890 1890	2475 2475 2475 2475	3250 3250 3250 3250	3850 3850 3850 3850
	Time	On 10: 00 11: 20	Up 11: 30 12: 20	UP 12:45 1:30	UP 1: 45 2: 45	UP 3: 50 3: 30
	SRI run number	Run 1 on Specimen 2 (5-10-66) Cal. A-5				

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THE THERMAL CONDUCTIVITY OF LOW-DENSITY PHENOLIC-NYLON CHAR USING THE RADIAL INFLOW APPARATUS

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ductivity	Watts/meter/°K	3.028 3.014 2.884 3.043	4, 225 4, 110 4, 110	0. 805 0. 816 0. 820	1.280 1.249 1.259	1.357 1.360 1.384	, ,
nen thermal con	in 10 ⁻⁵ Btu/sec/ft/°F	48. 6 48. 4 46. 3 48. 8	67.8 67.8 66.0 70.4	12.9 13.1 13.2	50.6 50.9 50.9	21.8 21.8 22.2	
Specin	Btu/hr/ft ² /°F/in.	21.0 20.9 21.1	29.3 29.3 30.4 5	5.58 5.68 69 68 89 89 89 89 89 89 89 89 89 80 80 80 80 80 80 80 80 80 80 80 80 80	8.88 8.66 8.73	9.41 9.43 9.60	
-	Mean temperature of specimen °F	3935 3935 3935 3935	4610 4610 4610 4610	1289 1289 1292	2120 2120 2120	2470 2470 2470	
Total heat removed by	r calorimeter gage length Btu/hr	472 469 470 470	629 629 611 652	153 155 156	268 262 264	30 4 30 4 30 4	
	AT across gage length °F	616 616 616 616 616	2888 2888 2888 2888 2888 2888 2888 288	832 831 834	000 6 6 6	942 942 942	
Specimen	outer face temperature °F	4385 4385 4385 4385	5040 5040 5040	1800 1800 1800	2760 2760 2760	3140 3140 3140	-
	Time	Up 4:00 4:55	90:	On 7: 50 9: 35	Up 9: 50 11: 00	UP 11: 40 12: 45	
	SRI run number	Run 1 on Specimen 2 (continued)	· · · · · · · · · · · · · · · · · · ·	Run 1 on Specimen 3 (5-25-66) Cal. A-5			

THE THERMAL CONDUCTIVITY OF LOW-DENSITY PHENOLIC-NYLON CHAR USING THE RADIAL INFLOW APPARATUS

	Notes		रू के के इ. के के	क क क क क ल क क	శా శా శా లే లే లే లే	क क क क लैल ले ले
iivity	Watts/meter/°K	1.903 1.903 1.903	3.389 3.345 3.374	4. 586 4. 614 4. 571 4. 629	5. 422 5. 328 5. 364 5. 364	3. 446 3. 446 3. 446 3. 446 3. 446
thermal conduct	in 10 ⁻⁵ Btu/sec/ft/°F	30.6 30.6 30.9	54. 4 53. 7 54. 2	73.6 74.1 74.3 74.3	87.0 84.7 86.1 86.1	55.3 55.6 55.8 55.3 55.3 55.3 55.5 55.5 55.5 55.5
Specimen	Btu/hr/ft /°F/in.	13.2 13.2 13.2	888 899 899	31.8 31.7 31.7 21.1	37.6 37.8 37.3 37.3	23.9 24.0 23.1
	Mean temperature of specimen °F	3270 3270 3270	4330 4330 4330	4970 4970 4970	4245 4245 4245 4245	3325 3325 3325 3325
Total heat removed by	7" calorimeter gage length Btu/hr	405 404 403	518 512 516	656 656 64 66 4	512 505 507	373 377 374 374
	∆T across gage length °F	912 912 912	657 657 657	615 615 615 615	405 405 405 405	466 466 466 466
Specimen	outer face temperature °F	3920 3920 3920	4795 4795 4795	5405 5405 5405 5405	4535 4535 4535 4535 4535	3 6 5 5 3 6 5 5 3 6 5 5 3 6 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5
	Time	UP 1: 00 1: 45	Up 2: 45 3: 20	Up 4:02	Down 4: 30 5: 26	Down 5: 56 6: 50
	SRI run number	Run 1 on Specimen 3 (continued)				

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TABLE 3 - Concluded

THE THERMAL CONDUCTIVITY OF LOW-DENSITY PHENOLIC-NYLON CHAR USING THE RADIAL INFLOW APPARATUS

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	Notes	4444 లేలేలేలే	સ ન ન ન જે છે છે છે	~ ~ ~ ~ ~	
tivity	Watts/meter/°K	3. 576 3. 576 3. 735 634 634	3.115 3.106 3.100 3.187	2.163 2.105 2.183 2.130	
thermal conduc	in 10 ⁻⁵ Btu/sec/ft/°F	88.57. 88.08 8.0.08	50.0 47.5 51.2	48 48 14 10 14 10 14 10 14 10 14 10 14 10 14 10 14 10 14 10 14 10 14 10 14 10 14 10 14 14 14 14 14 14 14 14 14 14 14 14 14	
Specimen	Btu/hr/ft ² /°F/in.	25. 2 8 25. 2 8 25. 2 8	21.6 21.5 22.1	15.0 14.6 15.0	
	Mean temperature of specimen °F	3025 3025 3025 3025 3025	2455 2455 2455 2455	1422 1422 1422 1422	
Total heat removed by	ralorimeter gage length Btu/hr	3320 3320 3320 3320 322	266 268 268	159 157 158	
	AT across gage length °F	380 0 380 0 380 0 380 0	0 00 00 00 00 00 00 00 00 00 00 00 00 00	318 318 318 318	· · · · · · · · · · · · · · · · · · ·
Specimen	outer face temperature °F	3300 3300 3300 3300 3300 3300 3300 330	2710 2710 2710 2710	1770 1770 1770 1770	
	Time	Down 7: 05 8: 00	Down 8: 25 9: 00	Down 9: 18 10: 00	Off 10, 17
	SRI run number	Run 1 on Spectmen 3 (continued)	· · · · · · · · · · · · · · · · · · ·		

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Notes:
ΔT measured with thermocouples, in front and back holes.
ΔT measured with thermocouples, in left and right side holes.
ΔT measured with optical pyrometer, using left and right side holes. Average of several ΔT readings used.
Mean temperature calculated as follows:
Mean temperature calculated as follows:
Specimen 1, Tmean = Tof - 0.734 ΔT (°F)
Specimen 3, Tmean = Tof - 0.714 ΔT (°F)
Specimen 3, Tmean = Tof - 0.710 ΔT (°F)

Only one ΔT measured before heater element failed. No cooling data taken with specimen 2. Heater element failed. Conductivity calculated from the following: ທ່ອ່ກ່

K = Factor X

8. Factors were the following: (for calculation in Btu/hr/ft/°F/in.) 막

Front to Back Side to Side

28.45 29.46 29.76 30.91 28.03 30.34 Specimen 1 Specimen 3 Specimen 3

ENTHALPY OF THE VIRGIN LOW-DENSITY PHENOLIC-NYLON

Enthalpy above 85°F reference Btu/lb	74.49 221.07 264.44 277.13	31.63 110.13 208.45 225.23 292.09	38 63 59. 80 140. 08 137. 98 1973. 98 265. 69 334. 92	-52.88 -49.15 -32.80	-10.81	208. 45 287. 50	339.98	314. 39	- 70. 85 -97. 67 -88. 48
Enthalpy h = $\frac{K}{W_s} (t_s - t_1)$ Btu/lb	77.02 214.09 259.16 277.94	35.97 111.47 219.08 219.19	41.68 65.54 141.26 173.38 197.38 256.98 332.26	-53.68 -45.68 -28.91	-9.27	211.71 286.96	339. 23	312.98	-68, 44 -99, 64 -89, 03
Final wt. of sample gm	3. 9390 3. 7900 3. 2331 2. 3043	3. 6146 3. 5746 3. 5640 3. 1762 1. 7240	3.1770 3.1522 3.1522 3.1448 3.0760 2.9064 2.5250 1.7790	3. 4985 3. 5312 3. 5400	2. 3381	2.1381 1.4977	2. 3138	2. 7425	3. 0784 3. 0569 3. 0560
Initial wt. of sample gm	4. 0201 3. 8568 3. 7900 3. 2331	3. 6863 3. 6146 3. 5746 3. 5640 3. 1762	3. 2346 3. 1770 3. 1592 3. 1448 3. 0760 2. 9064 2. 5250	3. 4870 3. 4985 3. 5312	2. 3259	2. 3647 2. 3471	4. 3460	4. 9902	3. 0953 3. 0784 3. 0569
Heating time to temperature min	125 35 40 75	4 2 0 2 5 9 3 0 5 5 9	103554255 1035547 10055547	73 4 0	09	90 31	30	37	20 20 20
Initial sample temperature °F	274 533 628 722	171 340 542 562 790	182 250 378 448 495 623 780	- 72 - 21 - 21		512 716	767	789	-197 -223 -262
Change in cup temperature °F	2.52 6.74 5.32 5.32	1.05 3.31 6.05 5.78 4.17	1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1	-1.56 -1.34 -0.85	-0.18	3.76 3.57	6.52	7.13	-1.75 -2.53 -2.26
Final cup temperature •F	78.60 99.17 95.87 83.09	75.95 81.87 94.00 98.04 87.17	77. 36 77. 36 82. 56 86. 30 89. 39 90. 56	87. 39 73. 30 72. 45	72.77	78.30 86.39	86.56	88. 22	75.39 91.17 87.13
Initial cup temperature °F	76.08 92.43 88.91 77.77	74. 90 78. 56 87. 95 83. 00 83. 00	76.28 77.32 81.87 91.48 85.65	88.95 74.64 73.30	72.90	74. 54 82. 82	80.04	81.09	77. 14 93. 70 89. 39
Specimen number and run number	Specimen 1 Run 1 3 5	Specimen 2 Run 1 3 4 5	Specimen 3 Run 1 3 4 4 5 5 7 7	Specimen 4 Run 1 3	Specimen 5 Run 1	Specimen o Run 1 Specimen 7 Run 1	Specimen 8 Run 1	Specimen 9 Run 1	Specimen 10 Run 1 3 3

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ENTHALPY OF THE LOW-DENSITY PHENOLIC-NYLON CHAR

Specimen	·			· · · · · · · · · · · · · · · · · · ·
number	Drop	Initial	Final	Enthalpy from drop
and run	temperature	weight	weight	temperature to 32°F
number	۴F	grams	grams	Btu/lb
Specimen 1				
Run 3	1025	1.165	1.095	101.5
6	2050	1.245	1.235	642.8
7	2550	1.290	1.275	784.5
8	3075	1.310	1.240	1305.9
9	3505	1.245	1.245	1415.5
10	4075	1.240	1.235	1645.4
11	4555	1.275	1.250	1549.5
Specimen 2				
Run 5	1985	1.280	1.265	502.0
6	2540	1.205	1.195	916.7
8	3500	1.250	1.230	1316.5
11	4920	1.290	1.230	2452.4
12	5055	1.220	1.210	2296.1
Specimen 3				
Run 2	2520	1.230	1.215	823.2
3	3055	1.255	1.230	1381.1
4	4520	1.265	1.230	1923. 2
Specimen 4				
Run 1	1497	1.235	1.255	227.7
4	3020	1.195	1.185	1018.2
5	4140	1.180	1.185	1473.7
6	5025	1.230	1.205	2358.3
n de la companya de la compa		1	L	

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THERMAL EXPANSION IN THE "ab" (PERPENDICULAR TO THE THICKNESS) DIRECTION OF THE VIRGIN LOW-DENSITY PHENOLIC-NYLON

Unit elongationCorrectedcorrection forspecimendilatometerunitnmotion110 ⁻³ in. /in.	0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0	0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0
Observed unit elongation 10 ⁻³ in./ii	50,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5,	22222222222222222222222222222222222222
Observed total elongation 10 ⁻³ in.		0.0 1.1.1.8.8 1.1.1.8.8 1.1.1.4.9 1.1.1.8.8 1.1.1.4.8.8 1.1.1.4.8.8.8 1.1.1.4.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.
Average temperature °F	73. 5 - 73. 5 - 87. 0 - 87. 0 - 301. 6 - 301. 7 - 301. 7	78.0 - 225.0 - 266.0 - 266.0 - 266.0 - 266.0 - 266.0 - 266.0 - 266.0 - 266.0 - 266.0 - 203.5 - 24.6 - 24.6
Temperature No. 3 °F	73. 5 - 21. 0 - 21. 0 - 247. 5 - 247. 5 - 60. 0 139. 5 - 60. 0 139. 5 234. 5 234. 5 238. 7 504. 0 516. 5	78.0 - 231.0 - 266.0 - 266.0 - 266.0 - 111.0 - 111.0 - 20.5 - 111.0 - 266.0 - 266.0 - 266.0 - 20.5 -
Temperature No. 2 *F	73. 5 -35. 0 -35. 0 -35. 0 -35. 6 -325. 6 -325. 6 139. 5 139. 5 133. 7 255. 0 255. 0 255. 0 255. 0 264. 0 616. 5 616. 5 ttom of scale	78. 0 - 286. 0 - 223. 7 - 233. 7 - 235. 0 - 233. 7 - 235. 0 - 235.
Temperature No. 1 °F	73. 5 - 73. 5 - 35. 0 - 35. 0 - 331. 8 - 331. 8 - 331. 8 - 331. 8 - 333. 6 504. 0 516. 5 888. 6 516. 5 Reached bo	1222 5 1222 5 1222 5 1222 5 1232 5 1233 1235 1235 1235 1235 1235 1235 1235
Time	9:00 9:20 9:20 9:20 9:45 9:45 11:15 11:42 11:42 11:42 11:42 11:42 11:50 12:50 1:15	12:25 12:46 12:45 12:45 1:15 1:15 1:2:45 1:2:0 1:2:0 1:2:0 1:2:0 1:2:0 1:2:0 1:2:0 1:2:0 2:15 2:15 2:15 2:2:2:2 2:2:2:2:2 2:
Specimen	Specimen 1 Initial length 2.9976 Final length 2.8731	Specimen 2 Initial length 3.0070 Final length specimen broke

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THERMAL EXPANSION IN THE "c" OR THICKNESS DIRECTION OF THE VIRGIN LOW-DENSITY PHENOLIC-NYLON

and the

Corrected specimen unit elongation 10 ⁻³ in./in.	,
Unit Elongation correction for dilatometer motion 10 ⁻⁴ in. / in.	0.000000000000000000000000000000000000
Observed unit elongation 10 ⁻³ in./in.	
Observed total elongation 10 ⁻³ in.	-140.5
Average temperature °F	75. 0 252. 3 26. 3 26. 0 26. 0 257. 2 257. 2
Temperature No. 3 °F	75.0 75.0 92.3 125.5 148.5 203.5 2044 148.5 599.0 659.0 653.0 653.0 653.0 653.0 75.0
Temperature No. 2 아F	75. 0 92. 7 92. 7 126. 5 149. 0 201. 3 234. 0 234. 0 234. 0 234. 0 256. 0 238. 5 481. 0 549. 0 549. 0 549. 0 541. 0 541. 0 541. 0 711. 5 711. 5
Temperature No. 1 °F	75. 0 75. 0 92. 0 125. 5 149. 5 238. 0 341. 0 352. 5 388. 0 375. 7 388. 0 390. 5 4401. 0 4401. 0 628. 0 628. 0 628. 0 628. 0 632. 0 750. 0 750. 0
Time	3:25 3:25 3:25 3:26 4:55 5:24 5:26 5:25 5:25 5:25 5:25 5:25 5:25 5:25
Specimen	Specimen 1 Initial length 3. 0117 Final length 2. 8684 2. 8684

Specimen number		Pressure drop across	Mean pressure	Flow rate of gas	Admittanc K _m v	e
and	Purge	specimen	of specimen	gage	cm ² /sec or	ft ² /sec
thickness	gas	in. of water	in. of water	cm ³ /sec	10 ⁻⁴ m ² /sec	
					······································	
Specimen 1	nitrogen	2.50	408.05	.1100	4. 2078	.00453
0.263 inch		5.50	409.55	. 2411	4.1922	.00451
		5.60	409.60	. 2450	4.1839	. 00450
		9.75	411.68	. 4214	4.1333	.00445
		9.75	411.68	. 4184	4.1038	.00442
	helium	2.50	408.05	. 1062	4.0624	.00437
		2.80	408.20	. 1210	4.1327	. 00445
		5.60	409.60	. 2465	4. 2095	. 00453
		5.30	409.60	. 2381	4. 2962	. 00462
		9.60	411.60	. 4077	4.0614	. 00437
[9.60	411.60	. 4120	4.1042	.00442
Specimen 2						
0.4218 inch	helium	2.60	408.10	. 0739	4.371	.00470
		2.70	408.15	. 0782	4.454	.00479
		5.60	409.60	. 1613	4.430	.00477
		5.60	409.60	. 1647	4. 523	. 00487
		9.60	411.60	. 2740	4.390	.00472
		9.70	411.65	. 2809	4.454	.00479
1	nitrogen	2.50	408.05	. 0733	4.509	. 00485
		2.70	408.15	. 0703	4.004	. 00431
		5, 50	409.55	. 1563	4.371	.00470
		5.60	409.60	.1468	4.032	.00434
		9.60	411.60	. 2483	3.978	.00428
		9.60	411.60	. 2475	3.965	.00427
Encodercen 2	1					
0 4334 inch	nitnogo-	2 65	409 19	0019	4 005	00500
0. 4004 Inch	murogen	2.00	400.15	. 0812	4.900	.00528
		5.00	400,10	. 0001	5.045	.00543
		0.00	409.03	. 1798	5.094	.00548
		0.10	409.00	.1808	5.033	.00542
		9.00	411.00	. 3003	5.007	.00539
	holi	9.70	411.00	. 3080	5.093	.00548
	nenum	4. JU 9. 50	400.00	. 0805	0.104	.00555
		4. JU 5. 75	400.UD	.0/93	5.017	.00546
1. A.		5.75	409.68	. 1852	5.166	.00555
		5.75	409.68	. 1855	5.164	.00556
		9.40	411.50	. 2907	4.950	. 00533
		9.55	411.58	. 3067	5.141	. 00553

PERMEABILITY OF VIRGIN LOW-DENSITY PHENOLIC-NYLON AT ROOM TEMPERATURE

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TABLE 8 - Concluded

PERMEABILITY OF VIRGIN LOW-DENSITY PHENOLIC-NYLON AT ROOM TEMPERATURE

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		<u> </u>										<u>.</u>				<u> </u>						<u>,</u>				<u></u>	
e	ft²/sec		. 00438	. 00432	. 00427	. 00427	. 00427	. 00420	. 00412	. 00409	. 00409	. 00410	. 00402	. 00397		. 00437	. 00456	. 00435	. 00445	. 00439	. 00436	. 00436	. 00432	. 00446	. 00453	. 00432	. 00441
Admittan K _m v	cm ² /sec or 10 ⁻⁴ m ² /sec		4.067	4.017	3.964	3.964	3.967	3.905	3.825	3.801	3.800	3.807	3.736	3.689		4.065	4.238	4.041	4.139	4.076	4.050	4.052	4.017	4.141	4.208	4.012	4.097
Flow rate of gas at bubble	gage cm³/sec		. 1131	.1223	. 2392	. 2392	. 4098	. 4098	. 1227	.1240	. 2314	. 2380	. 3717	. 3831		. 1190	.1428	. 2433	. 2538	. 4255	. 4273	.1164	. 1176	. 2493	. 2557	. 4166	. 4255
Mean pressure	of specimen in. of water		408.08	408.20	409.58	409.58	411.55	411.63	408.28	408.30	409.60	409.68	411.38	411.58		408.13	408.33	409.53	409.58	411.53	411.58	408.10	408.13	409.53	409.55	411.50	411.50
Pressure drop across	specimen in. of water		2.55	2.80	5.55	5.55	9.50	9.65	2.95	3.00	5.60	5.75	9.15	9.55		2.65	3.05	5.45	5.55	9.45	9.55	2.60	2.65	5.45	5.50	9.40	9.40
	Purge gas		helium						nitrogen)						nitrogen)					helium				<u> </u>	
Specimen number	and thickness	specimen 4	0.249 inch												Specimen 5	0.250 inch	-										

Exposed area of specimen = 2.52 cm

Note:

PERMEABILITY OF THE LOW-DENSITY PHENOLIC-NYLON CHAR AT ROOM TEMPERATURE

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and Purge specimen flowmeta thickness gas inch of water inch of water flowmeta Specimen 1 nelium 1.50 401.25 400.3 Specimen 2 nitrogen 1.50 401.55 400.4 Specimen 2 1.50 401.50 401.0 0.250 inch nitrogen 1.50 401.50 401.4 0.250 inch nitrogen 1.50 402.69 401.4 1.50 402.69 401.4 401.4 401.4 0.250 inch nitrogen 1.50 402.59 401.4 1.50 404.54 402.1 402.1 402.1 3.00 404.54 401.4 402.1 401.4 1.50 402.29 401.4 402.1 401.4 1.50 402.29 401.4 401.5 401.4	s pressure of	at	of gas	Kmν	
Specimen 1 helium 1.50 401.25 400.3 0.250 inch helium 3.00 402.60 400.4 0.250 inch 1.50 401.50 400.4 3.00 402.69 401.4 0.250 inch 1.50 401.50 401.4 Specimen 2 1.50 402.69 401.4 0.250 inch 1.50 402.69 401.4 1.50 404.54 401.4 401.4 1.50 404.54 401.4 401.4 1.50 404.54 401.4 401.4 1.50 404.54 401.4 401.4 1.50 404.54 401.4 401.4 1.50 402.59 401.4 402.1 3.00 402.26 401.4 401.4 1.50 403.59 401.5 401.5	specimen fl er inch of water inc	owmeter ch of water	at flowmeter cm ³ /sec	cm ² /sec or 10 ⁻⁴ m ² /sec	ft ² /sec
3.00 402.60 400.5 nitrogen 1.50 401.50 400.4 Specimen 2 3.00 403.30 401.0 Specimen 2 1.50 403.30 401.4 0.250 inch 1.50 402.69 401.4 1.50 402.69 401.4 3.00 404.54 401.4 1.50 404.54 401.4 1.50 404.54 401.4 3.00 403.59 401.4 1.50 404.54 401.4 1.50 402.29 401.4 1.50 403.59 401.4 1.50 403.59 401.4 1.50 403.59 401.4	401.25	400.3	122	12,219	13.2
Specimen 2 3.00 403.30 401.0 Specimen 2 0.250 inch 1.50 402.69 401.4 0.250 inch 1.50 402.59 401.4 3.00 404.54 402.1 3.00 404.54 402.1 helium 1.50 402.24 401.4 401.4 3.00 404.54 401.4	402.60 401.50	400.5 400.4	233	11, 66 4 10, 150	12.6 11.0
Decument nitrogen 1.50 402.69 401.4 0.250 inch 1.50 402.59 401.4 3.00 404.54 402.1 3.00 404.54 402.1 helium 1.50 402.59 401.4 3.00 404.54 402.1 3.00 404.54 402.1 1.50 402.25 401.4 1.50 402.24 401.4	403.30	401.0	163	8, 186	8.8
1.50 402.59 401.4 3.00 404.54 402.1 3.00 404.54 402.1 1.50 404.24 402.1 3.00 404.24 402.1 3.00 403.29 401.4	402.69	401.4	88	9, 883	10.7
3.00 404.44 402.1 helium 1.50 402.24 401.4 3.00 403.59 401.5	402.59 404.54	401.4 402.1	98 163	9, 881 8, 208	10.7 8.9
helium 1.50 402.24 401.4 3.00 403.59 401.5	404.44	402.1	161	8, 086	8.7
3.00 403.59 401.5	402.24	401.4	115	11, 552	12.5
	403. 59	401.5	213	10, 668	11.6

Note:

Exposed area of specimen = 1.69 cm^2

Time	Observed temperature	Radiometer output millivolta	True temperature	Emittanco	Bomonks
Time	F	initivorts	F	Emittance	Remarks
Specimo	en 1				ч.
3:53	1468	0. 198	1551	0.77	Attempted to run at
3:58	1463	0.206	1540	0.81	higher temperature
4:05	1561	0.256	1649	0.82	(approx. 3200°F)
4:15	1865	0.482	1985	0.86	but radiometer sig-
4:24	2326	1. 139	2485	0.95	nal became noisy.
4:38	2710	1.835	2940	0.85	White coating on
4:44	2720	1.818	2960	0.82	surface noted dur-
					ing disassembly.
Specime	en 2		100 B		
9:21	1480	0. 199	1566	0.75	· ·
9:29	1910	0.490	2047	0.79	
9:35	2280	0.954	2460	0.82	1
9:45	2840	2.336	3059	0.94	
9:53	3305	3.986	3591	0.90	
10:03	3480	4.763	3796	0.88	
Specime	en 3				
1.13	1526	0.235	1611	0.81	
1:23	1875	0.461	2007	0.79	
1:32	2333	1.030	2521	0.82	
1:38	2720	1,810	2961	0.82	
1:45	3199	3.605	3463	0.93	
Specime	l en 4				
9.38	1448	0, 195	1524	0.81	
9:44	2015	0 600	2164	0.01	
9.50	2286	1 043	2447	0.02	
10:00	2772	2.094	2991	0.91	
10:05	3330	4.339	3585	0.99	
	1_				м.
Specime	en 5	4 79 1	2000	0.07	
10:18	3480	4. 721	3802	0.87	
Specimo	en 6			1	
1:16	3605	4.954	4008	0.76	
Specime	l en 7				
9:45	3005	2.622	3286	0.87	
Specime	l en 8				
2:43	2895	2.396	3139	0.88	
2:48	3143	3.057	3451	0.80	
2:53	3262	3.441	3600	0.77	
3:06	3535	4.681	3913	0.78	
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TOTAL NORMAL EMITTANCE OF LOW-DENSITY PHENOLIC-NYLON CHAR

TABLE 11 TENSILE STRESS-STRAIN DATA FOR THE VIRGIN LOW-DENSITY PHENOLIC-NYLON

Remarks					-			<u>.</u>	Loaded to ~45 psi and unloaded before	Loaded to ~49 and unloaded before	una towung. Lateral strain did not track. Lateral strain did not track.		Loaded and unloaded before final load.		Lateral strain did not track. Heated to 400°F twice.	Lateral strain not measured. Lateral strain could not be measured due to charring of specimen.	Lateral strain not measured. Lateral strain not measured.	Specimen burned.	
Initial Poissonis	ratio	0.14	0.30	0.05		0. UB	0.05	0.06	0.11	0.14	1. 1	0.13 0.14	à	0. 11 0. 50	0.20	t 1	1.1	•	
Load time	min	9	9	9	4.2	5.3	4	ŝ	12.5	11	1 3.5	4. w	4		2.1 6.3	8.1	2.4	-	
Tensile	in percent	0.33	0.27	0.40	0.68	0.66	0.79	0.95	0.69	0.81	0.76 0.73	1.32 0.96	0.73	1.58	1.14 2.42	1.32 0.42	0.87 1.04	•	
e strength	in 10 ⁶ N/m ²	4.41	3.85	3.58	3.65	3.63	2.77	3.52	2.82	3.71	3.94	3.72 3.81	1.01	1.32	0.96 0.78	0.28	0.21 0.14	,	
Ultimat	in psi	640	559	519	530	526	402	510	409	538	572 320	539 553	147	192 234	139 113	2g 17	85	•	
l strength	in 10° N/m ²			•	,	,	2.45	3.39	.1	1	1.92	2, 55 3, 54	ı	1.13	0.46	11	1.1		e this point.
Yield	in psi		1	•	,	,	356	492	,	•	- 279	367 513	1	164	66.4	8 1	• •	,	ed before
elastic	in 10 ⁶ N/m ²	1338	1703	786	620	115	563	476	465	201	575 511	450 546	147	161 149	76.5 65.2	41 66	25	ŧ	acture occurr
Initial	in 10 [°] psi	194	247	114	0 00	83.7	81.6	69.0	67.5	73.6	83.4	65.3 79.2	21.4	23.3 21.6	11.1 9.45	8.8 8.8	3.6	,	timate and fr.
Crosshead	in./min	800	8	0.03	2	38	10	0.0	0.02	0.02	0.4 0.03	0.02 0.05	0.05	0.08 0.08	88	88 80 00	0.0 88 8	1	fined when ul
Temperature	"F/min			,			,	í.	1	*		100	100	100	100	100	100	100	. 2% offset not de temperature fron seasured.
Temperature	F i	- 200	- 200	-200	100	88	c	••	20	02	70 70	200	300	300	400 400	200	009	700	ld strength at 0 cimens held at 1 ding rates not n
SRI run		т-26	7-27	8	T00		T-23	1-24	T- 3	T- 4	T-5 T-20	4 4 8 4 7	7- 8	т- 9 Т-19	T-11 T-16	T-12 T-17	T-13 T-15	T-14	Notes: 1. Yie 3. Coo

COMPRESSIVE STRESS-STRAIN DATA FOR THE VIRGIN, LOW-DENSITY, PHENOLIC-NYLON

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Initial Defeature matio		0. 12	0.22	0.21 0.22	0.32 0.23	0.22 0.26	0.20 0.27	0.20 0.21	0.22	0.16 0.16 0.16	0.13 0.11	0.13 0.08	- 0.11 0.16 0.17 0.18
Load time to	nin	9	5.5	44	4 10	1 1	ৰা ৰা	4.5.	4 6 8	14 5.5 5.5	3 6.5	ର ସ୍ ସ୍	4.5 10 7.8 4.8 4.8
Compressive	96	4.9	3.5	4.0 0.3	4.0 8.4	5.0 4.9	3.0 5.8	7.2 7.6	8.8 6.1	21.7 12.5 14.7	9.0 14.6	13.1 5.4 2.2	4, 8, 8, 8, 8, 8, 8, 8, 8, 8, 8, 8, 8, 8,
ate strength	in 10 ⁶ N/m ²	18.03	18.20	16.75 14.62	13.10 14.62	9.69 9.50	9.51 9.17	9.51 9.41	8.45 6.41	7.31 4.52 5.48	2.63 2.88	2.00 2.11 2.11	12.89 14.41 14.18 13.07 11.76
Ultim	in psi	2615	2640	2430 2120	1900 2120	1405 1392	1380	1380 1365	1225 930	1060 655 795	381 418	290 306 306	1870 2090 2057 1896 1705
strength offeet	in 10 ⁶ N/m ²	15.93	12.74	11.14 8.98	7.03 9.86	7.24 7.00	6.04 3.64	4.85	4. 07 2. 55	1.56 1.63 1.91	1.41 0.95	1.28 	7.32 9.14 9.14 3.61 3.99
Yield	in psi	2310	1848	1615 1302	1020 1430	1050	876 528	703 628	591 370	226 237 277	20 4 138	186 	1062 1325 1325 523 579
stic modulus	in 10 ⁸ N/m ²	700	937	717 645	610 559	344 407	576 590	334 348	232 255	143 117 158	97 61	98 ^{°°} ' 88	430 501 433 418 418
Initial elas	in 10 ³ psi	102	136	104 94	88 81	20 20	84 86	48 50	34 37	8178	14 9	13 13 - 3	62.3 72.7 60.6 60.6
Crosshead	in./min	0,009	0.009	0.012 0.012	0.013 0.013	0.015 0.015	0.020 0.020	0. 020 0. 020	0. 030 0. 025	0, 015 0, 030 0, 030	0.030 0.024	0.030 0.020 0.025	0.015
Temperature	F/min ¹	-2372	-190²	-125^{2} -115^{2}	- 70 ² - 40 ²	1.3	100	100	100	100	100	00011000	000
Temperature	4	-200	-200	-100	00	70 70	200	300	400	200	600	750 750	2000 2000 2000 2000
SRI run	number	C-17	C-18	C-19 C-20	0 - 23 - 25	78 200	ဗ 4 ဗ 4	9 9 1 1 1 1	* * ن	с- 9 с-10 с-11	C-12 C-13	C-14 C-15 C-16	C - 25 C - 25 C - 26 C - 28 C

Notes: 1. Soaked at temperature for 5 minutes. 2. This is an approximate cooling rate due to nonlinearity of the cooling. 3. Initial modulus and strength too low to accurately determine the modulus and yield strength on SRI Run No. C-15. 4. This table supercedes Table 2 of the October 1965 progress report.

APPENDIX A

DETAILED MATERIAL DESCRIPTION

The following detailed description of each constituent within the low-density phenolic-nylon was supplied by the NASA Langley Research Center.

Union Carbide BRP-5549 Phenolic Resin

The properties of the resin as published by the manufacturer are shown in the table below

Property

Property value

Density

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16.8 to 19.4 lb/ft^3 (270 to 310 kg/m³)

Sieve Analysis (U.S. Std. Mesh) on 40 mesh 0.0% on 100 mesh 0.6% on 200 mesh 2.0% through 200 mesh 98.0% Hexamethylene 8.7 - 9.5% tetramine content

Measurements with a Coulter Counter (Coulter Electronics Industrial Division) at the Langley Research Center indicate that the range of diameters of the phenolic resin powder particles is 0.0001 to 0.0048 in. (2 to 120 μ m) with about 80% by volume of the powder having diameters ranging from 0.0003 to 0.0023 in. (6 to 58 μ m).

Phenolic Microballoons

The properties of the Microballoons as published by the manufacturer are given in the following table:

Property	Property value
Density (liquid displacement Density (air displacement)	15.6 lb/ft^3 (250 kg/m ³) 18.7 lb/ft^3 (300 kg/m ³)
Flotation in toluene dupanol solution	Not less than 90% must float
Average particle size (dia)	0.0017 in. $(43 \ \mu m)$
Size range (dia)	0.002 to 0.0005 in. $(13 \text{ to } 51 \mu\text{m})$

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Measurements with the Coulter Counter indicate that the range of diameters of the phenolic Microballoons is 0.0004 to 0.0100 in. (10 to 250 μ m), with approximately 85% by volume of the Microballoons having diameters ranging from 0.0024 to 0.0080 in. (60 to 200 μ m).

Nylon

Properties of the nylon powder as given by the manufacturer are listed in the table below:

Drononter

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Froperty	Froperty value
Specific Gravity	1.3 - 1.15
Melting Point	482 - 500°F (523 - 533°K)
Tensile Strength	11,800 psi (81.4 MN/m^2)
Coefficient of Linear	-
Thermal Expansion	$45.0 \ge 10^{-6}$ in./in °F
	$(81.1 \times 10^{-6} \text{ m/m} - ^{\circ}\text{K})$
Thermal Conductivity	1.31 x 10 ⁻⁴ Btu-in. /ft ² -sec-°F
	0.068 W/m - °K
Specific Heat	0.3 - 0.5 Btu/lb - °F
	1.25 to 2.09 kJ/kg - °K

Preliminary studies using a Coulter Counter indicate that about 85% by volume of the nylon powder lies in the range from about 0.0012 to 0.0157 in. (30 to 400 μ m) in diameter, with about 5% below 30 μ m and 10% above 400 μ m.

APPENDIX B

APPARATUS FOR THE DETERMINATION OF PERMEABILITY

The flow of gas through a material can be of two different types, Knudsen (molecular) flow which is governed by the relation¹

$$q = \frac{K_{m} (\Delta P) A \sqrt{RT}}{PL}$$

(1)

and Poiseuille (viscous) flow, which is described by

$$q = \frac{K_{\nu} (\Delta P) \frac{1}{2} (P_2 + P_1) A}{PL n}$$

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where

q	= volume flow rate measured at absolute pressure P
$\Delta \mathbf{P}$	= pressure drop across the specimen
Α	= area of the specimen normal to the flow of gas
L	= length of the specimen parallel to the flow of gas
R	gas constant of the gas
Т	= absolute temperature
ή	 viscosity of the gas
$\frac{1}{2}(P_2 + P_1)$	average absolute pressure on the specimen
K _m	 molecular permeability coefficient
κ _ν	 viscous permeability coefficient

For those cases where neither equation fully describes the gas flow, the flow is both molecular and viscous and the expressions for both types of flow are combined which yields:

$$q = (K_0 - \sqrt{RT} + K_1 - \frac{P_m}{\eta}) - \frac{A \Delta P}{PL}$$
(3)

¹Barrer, R. M., Diffusion In and Through Solids, Cambridge University Press, Cambridge, pp 60-61, 1951.

where K_0 and K_1 are permeability coefficients similar to K_m and K_{ν} , respectively, and $P_m = \frac{1}{2} (P_2 + P_1)$.

To evaluate the coefficients K_0 and K_1 , let

$$K_{m\nu} = K_0 \sqrt{RT} + K_1 \frac{P_m}{n}$$

Equation (3) may then be written

$$q = K_{m\nu} \frac{A\Delta P}{PL}$$

 \mathbf{or}

$$K_{m\nu} = \frac{PLq}{A\Delta P}$$

 $K_{m\nu}$, often called the admittance, can then be determined for the specimen at any pressure by measurement of the flow rate q at that pressure. Values of $K_{m\nu}$ for several different pressures can then be plotted versus P_m and, as seen from equation (4), the resulting line will have a slope of K_1/η and a zero intercept of $K_0 \sqrt{RT}$. Thus, K_1 and K_0 can be determined from the properties of the gas used.

If metric units are used throughout equation (3), K_0 will be in cm and K_1 in cm².

These coefficients should be dependent only on the structure of the material; that is, independent of specimen configuration, viscosity and molecular weight of the gas, temperature, pressure gradient, and the average pressure.

The data required to calculate the admittance $(K_{m\nu})$ are pressure at the flowmeter (P), indicated flow rate at the flowmeter (q), thickness of specimen (L), area (A), and pressure drop across the specimen (ΔP). For cases where the temperature of the specimen is at room temperature or the same temperature as the flowmeter, the product of Pq measured at the meter is equal to the product Pq at the specimen, since from continuity

63

(4)

(5)

(6)

$$W = \rho AV = \rho q = constant$$

$$\rho = \frac{P}{RT}$$
(8)

(7)

Therefore, from (7), equating conditions at the specimen and the flowmeter

$$\rho_{\rm s} q_{\rm is} = \rho_{\rm m} q_{\rm m} \tag{9}$$

where the subscripts s and m refer to the specimen and meter, respectively.

Substituting from (8)

$$\frac{P_s}{RT_s} q_s = \frac{P_m}{RT_m} q_m$$
(10)

Therefore, for isothermal flow

3

$$P_{s} q_{s} = P_{m} q_{m}$$
(11)

If the temperatures at the specimen and meter are different, then the term $P_m q_m$ (Pq in equation 6) must be multiplied by the ratio $\frac{T_s}{T_m}$.

A schematic of the apparatus being used for the determinations of the permeability coefficients is shown in Figure 1. The primary side of the specimen is exposed to desired gage pressures which can be measured by a laboratory gage or a U tube manometer. Since the secondary side of the specimen is exposed to atmospheric pressure or a known pressure, the pressure drop across the specimen is readily obtained. The flow of gas is measured at the exit side of the specimen with either a bubble flowmeter, for which a stopwatch is employed to measure the displacement time of a soap bubble, or a wet test flowmeter. The bubble flowmeter operates at atmospheric pressure, and the wet test meter operates at a slightly higher pressure which is monitored. For specimen materials that can be machined with good tolerance, the brass assembly containing the specimen is shown in Figure 2. The 1 inch diameter $x \frac{1}{2}$ inch thick specimen is placed on the $\frac{1}{64}$ inch shoulders and sealed by filling the annulus around the specimen with the sealer material. For porous and weaker structured materials, the specimens are mounted and sealed in the brass assembly shown in Figure 3. For this assembly, the specimens can be roughly machined in the form of a disc of approximately 1 inch diameter.

Several types of sealants were investigated for room temperature use with Dow Corning Silastic RTV 731 giving the best results. This material is viscous and can be applied easily. It wets the specimen well and provides an excellent seal after curing to a solid rubbery form. The system is thoroughly purged before securing the specimen assembly.







Figure B2. Permeability Specimen Assembly



Figure B3. Permeability Specimen Assembly

APPENDIX C

STRESS-STRAIN CURVES IN TENSION AND COMPRESSION

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Figure C2. Axial tensile stress-strain and bi-directional strains at 70°F for a virgin low-density phenolic-nylon



Figure C3. Axial tensile stress-strain and bi-directional strains at 70°F for a virgin low-density phenolic-nylon







Figure C5. Axial tensile stress-strain and bi-directional strains at 200°F for a virgin low-density phenolic nylon

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Ultimate tensile strength in psi

Figure C6. Axial tensile stress-strain at 300°F for a virgin low-density phenolic-nylon



Figure C7. Axial tensile stress-strain and bi-directional strains at 300°F for a virgin low-density phenolic-nylon



Ultimate tensile strength in psi







Ultimate tensile strength in psi

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Ultimate tensile strength in psi



Figure C12. Axial tensile stress-strain and bi-directional strains at 400°F for a virgin low-density phenolic-nylon



Figure C13. Axial tensile stress-strain at 500°F for a virgin low-density phenolic-nylon







Figure C15. Axial tensile stress-strain and bi-directional strains at 70°F for a virgin low-density phenolic-nylon

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Ultimate tensile strength in psi



Figure C17. Axial tensile stress-strain and bi-directional strains at 0°F for a virgin low-density phenolic-nylon

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Figure C18. Axial tensile stress-strain and bi-directional strains at 0°F for a virgin low-density phenolic-nylon





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Figure C21. Axial tensile stress-strain and bi-directional strains at -100°F for a virgin low-density phenolic-nylon

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Figure C23. Axial compressive stress-strain and bi-directional strains at 70°F for a virgin low-density phenolic-nylon

Ultimate compressive strength in psi

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Figure C24. Axial compressive stress-strain and bi-directional strains at 70°F for a virgin low-density phenolic-nylon

Ultimate compressive strength in psi

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Figure C27. Axial compressive stress-strain and bi-directional strains at 300°F for a virgin low-density phenolic-nylon

Ultimate compressive strength in psi

96

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Ultimate compressive strength in psi

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Figure C28. Axial compressive stress-strain and bi-directional strains at 300°F for a virgin low-density phenolic-nylon







Ultimate compressive strength in psi

j

Figure C30. Axial compressive stress-strain and bi-directional strains at 400°F for a virgin low-density phenolic-nylon



Figure C31. Axial compressive stress-strain at 500°F for a virgin low-density phenolic-nylon

Ultimate compressive strength in psi

100



Figure C32. Axial compressive stress-strain and bi-directional strains at 500°F for a virgin low-density phenolic-nylon

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Figure C33. Axial compressive stress-strain and bi-directional strains at 500°F for a virgin low-density phenolic-nylon

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Ultimate compressive strength in psi

102

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Ultimate compressive strength in psi

3

Figure C34. Axial compressive stress-strain and bi-directional strains at 600°F for a virgin low-density phenolic-nylon











Figure C37. Axial compressive stress-strain and bi-directional strains at 700°F for a virgin low-density phenolic-nylon






Figure C39. Axial compressive stress-strain and bi-directional strains at -200°F for a virgin low-density phenolic-nylon











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Figure C42. Axial compressive stress-strain and bi-directional strains at 0°F for a virgin low-density phenolic-nylon



Figure C43. Axial compressive stress-strain and bi-directional strains at 0°F for a virgin low-density phenolic-nylon



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Figure C46. Axial compressive stress-strain and bi-driectional strains at 70°F for a virgin low-density phenolic-nylon

Ultimate compressive strength in psi

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Lateral strain in 10⁻³ in./in.

Figure C47. Axial compressive stress-strain and bi-directional strains at 200°F for a virgin low-density phenolic-nylon

Ultimate compressive strength in psi

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NASA-Langley, 1967 — 18 CR-809

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"The aeronautical and space activities of the United States shall be conducted so at to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the underst practicable and appropriate distemination of information concerning its activities and the results thereof."

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