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Y-1716

Metals, Ceramics,
and Materials

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PALARITE THERMAL INSULATION

C. D. Reynolds
Z. L. Ardary

MASTER

UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the ATOMIC ENERGY COMMISSION under U. S. GOVERNMENT Contract W-7405 eng 26

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ABSTRACT

Requirements for a low-density, high-temperature thermal insulation material led to the development of Palarite, a substance that consists essentially of carbon-bonded silica fibers.

Palarite is generally prepared with densities in the range of 7 - 10 pounds per cubic foot (pcf), using a vacuum-molding process. The thermal conductivity of 8-pcf Palarite increases from 0.25 Btu-in/hr-° F-ft² at 70° F to 0.60 Btu-in/hr-° F-ft² at 1,000° F. In an inert atmosphere, or vacuum, the insulation can be used continuously at temperatures up to 2,000 ° F. However, in air, oxidation of the carbon binder begins at temperatures above 500° F.

CONTENTS

SUMMARY	10
INTRODUCTION	11
PREPARATION AND PROPERTIES OF PALARITE INSULATION	13
Constituent Materials	13
Fabrication Process	14
Fiber Preparation	14
Slurry Preparation	15
Vacuum-Filtration Operation	17
Curing, Drying, and Furnace Heating	18
Machining	25
Properties of Palarite Insulation	25
Chemical Composition	26
Temperature Capability	26
Density	26
Specific Heat	27
Strength	27
Thermal Conductivity	27
Hygroscopicity	28
Resistance to Vibration, Shock, and Acceleration	28
Discussion	29
Strength	29
Thermal Conductivity	36
Conclusions	43
REFERENCES	46
ACKNOWLEDGEMENTS	47
APPENDIX A	49
Compressive Stress-Strain Curves of Palarite Insulation with Various Binder Concentrations	49
APPENDIX B	51
Calculation of the Ultimate Flexural Strength and Modulus of Elasticity	51

SUMMARY

Palarite insulation consists, essentially, of carbon-bonded silica fibers. The fabrication process entails vacuum forming, from a dilute water slurry containing silica fibers and starch, a fibrous molding on a perforated mandrel of the desired shape. The molding, which is composed of starch dispersed in a fibrous mat with residual water, is then processed through a curing, drying, and furnace-heating operation to effect a rigid carbon-bonded fibrous structure.

Palarite is generally prepared with densities in the range of 7 to 10 pounds per cubic foot (pcf), using the vacuum-molding process, although densities as high as 30 pcf have been achieved by varying the process techniques. The thermal conductivity of 8-pcf Palarite increases from 0.25 Btu-in/hr-° F-ft² at a 70° F mean temperature to 0.60 Btu-in/hr-° F-ft² at a 1,000° F mean temperature. In an inert atmosphere, or vacuum, the insulation can be used continuously at temperatures up to 2,000° F. However, in air, oxidation of the carbon binder begins at temperatures above 500° F.

Palarite is especially suited for aerospace applications because of its very low density, and because the resiliency of the fibrous material enables it to withstand severe forces of vibration, shock, and acceleration. In addition, machining of relatively intricate shapes is possible because of the good structural integrity of the material.

A program was conducted to determine the density-strength-thermal conductivity interrelations in Palarite over a wide range of values. A considerable difference in these properties was established by varying the relative volume fractions of the carbon binder and silica fibers. The best method of improving the strength of Palarite insulation, in terms of maximum strength gain per unit increase in density and coefficient of thermal conductivity, is by increasing the fiber concentration by compaction.

INTRODUCTION

Thermal insulation for aerospace applications should have the following properties: (1) low density, (2) the ability to withstand severe shock, vibration, and acceleration, (3) dimensional and structural integrity over a wide range of temperatures, and (4) sufficient structural integrity to permit the fabrication of shapes to close dimensional tolerances.

In August 1967, the Oak Ridge Y-12 Plant^(a) undertook to develop a thermal-insulation material that would have the qualities just enumerated. As part of the program, procedures and equipment were devised to fabricate insulators in useful sizes and shapes.

A carbon-bonded silica fiber insulation appeared to meet the basic requirements for low-density thermal insulation. One objective of a series of parametric studies was to determine the best method for improving the strength of the insulation with a minimum sacrifice in increased density and thermal conductivity. Second, information relating to strength-density-thermal conductivity trade offs would be of great value to designers.

(a) Operated for the US Atomic Energy Commission by Union Carbide Corporation's Nuclear Division.

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PREPARATION AND PROPERTIES OF PALARITE INSULATION

CONSTITUENT MATERIALS

Materials used in the production of Palarite insulation are Grade 106 Microquartz fibers^(b) and Fisher S-516 starch. Microquartz fibers consist essentially of pure, amorphous silica (98.5% plus). They are manufactured by the chemical treatment of high-silica glass fibers with the selective removal of practically all other elements and compounds. The manufacturer lists the following chemical composition for the fibers:

	Weight Percent (maximum)
Silicon Dioxide	98.50
Calcium Oxide	0.35
Magnesium Oxide	0.35
Sodium Oxide	0.15
Aluminum	0.50
Boron	0.01
Iron	0.06

A spectrochemical analysis of Microquartz fibers is presented in Table 1.

The average diameter of Grade 106 Microquartz fibers is in the range of 0.50 to 0.75 micron. Johns-Manville determines fiber diameter by the use of a Williams freeness tester. The length of the silica fibers is predominantly in the 1/2 to 3/4-inch range.

Fisher S-516 starch is a reagent-grade starch used for iodometry. Its granule size (approximately 25 microns), relative insolubility in water of neutral pH, and ease of gelation upon heating above 60° C make it particularly well suited for the Palarite process.

The theoretical carbon, hydrogen, and oxygen contents of the starch, whose formula is $(C_6H_{10}O_5)_x$, are 44.4, 6.2, and 49.4 weight percent, respectively. A complete spectrochemical analysis of Fisher S-516 starch is given in Table 2. Pyrolysis of the starch during the Palarite fabrication process generally yields 20 to 25 weight percent carbon.

(b) A product of Johns-Manville Corporation.

Table 1
SPECTROCHEMICAL ANALYSIS OF MICROQUARTZ FIBERS

Element	Concentration (ppm)	Element	Concentration (ppm)	Element	Concentration (ppm)
Al	210	Hg	< 1.7	Re	< 0.8
As	< 0.3	Ho	< 1.3	Rh	< 0.8
Au	< 0.5	I	< 3.3	Ru	< 2.5
B	10	In	≤ 40	Sb	17
Ba	49	Ir	< 0.8	Sc	10
Bi	< 0.6	K	160	Se	< 1.2
Ca	2,400	La	< 1.1	Sm	< 4.4
Cd	< 1.0	Lu	< 1.4	Sn	< 8.4
Ce	< 1.2	Mg	560	Sr	80
Cl	630	Mn	6.5	Ta	≤ 65
Co	4.6	Mo	< 6.2	Tb	< 1.2
Cr	16.0	Na	8.8	Te	< 1.0
Cs	< 1.0	Nb	< 0.7	Ti	250
Cu	25	Nd	< 4.7	Tl	< 0.8
Dy	< 4.4	Ni	6.7	Tm	< 1.3
Er	< 3.9	Os	< 1.2	V	4.0
Eu	< 2.2	P	2.4	W	< 1.6
Fe	160	Pb	100	Y	6.8
Ga	< 0.9	Pd	< 3.0	Yb	< 4.2
Gd	< 5.6	Pr	< 1.1	Zn	10
Ge	< 1.5	Pt	< 1.5	Zr	15
Hf	< 5.2	Rb	< 0.9		

FABRICATION PROCESS

A vacuum-filtration process is used in the manufacture of Palarite insulation. From a dilute water slurry containing fibers and starch, a fibrous mat is vacuum formed on a perforated mandrel of the desired shape. The molding, which consists of starch dispersed in the fibrous mat with residual water, is then processed through the curing, drying, and furnace-heating operations to effect a rigid carbon-bonded fibrous structure. A process flow diagram for the production of Palarite insulation is given in Figure 1.

Fiber Preparation

As-received Microquartz fibers are intertwined tightly in small aggregates. The length of the fibers is variable, with most fibers in the 1/2 to 3/4-inch range. Vacuum conveying as-received fibers through a Wiley mill^(c) has been

(c) Product of the Arthur H. Thomas Company, Philadelphia, Pennsylvania.

Table 2
SPECTROCHEMICAL ANALYSIS OF FISHER S-516 STARCH

Element	Concentration (ppm)	Element	Concentration (ppm)
Ag	< 0.2	Li	3
Al	4	Mg	36
As	< 2	Mn	2
Au	< 0.2	Mo	< 0.2
B	0.2	Na	144
Ba	< 0.2	Nb	< 0.2
Be	< 0.2	Ni	< 0.7
Bi	< 2	P	> 180
Ca	180	Pb	< 0.2
Cd	< 0.2	Rh	< 4
Co	< 4	Sb	< 0.2
Cr	< 0.2	Si	20
Cs	< 11	Sn	0.7
Cu	4	Th	< 0.7
Fe	4	Ti	< 1
Ga	< 0.2	U	< 18
Ge	< 0.2	V	< 0.2
Hf	2	W	< 2
In	< 0.2	Zn	< 11
K	117	Zr	< 0.4

employed to aid in separating the fibers. Subsequent high-speed slurry agitation gives a good dispersion of fibers, which is necessary for homogeneous part moldings.

The amount of reduction in fiber length due to milling is not known, although it can be controlled to some extent by a sizing plate within the mill. The sizing plate used for fiber separation in the Palarite production process contains 1/4-inch-diameter holes.

A photograph of the interior of a Wiley mill is presented in Figure 2. Fibers are vacuum drawn into the mill opening and through the revolving cutter wheel and sizing plate. A partially removed sizing plate, with 1/4-inch-diameter holes, can be seen in the center of the photograph. The Microquartz fibers leave the mill through the vacuum hose at the bottom center of the picture.

Slurry Preparation

The fiber-water-starch slurry is agitated in a large tank to obtain a homogeneous dispersion of solids prior to filtration. With high-speed agitation

Figure 1. PROCESS FLOW CHART FOR THE PRODUCTION OF PALARITE THERMAL INSULATION.

(1,700 rpm), a mixing time of approximately 30 minutes provides adequate slurry preparation. A schematic diagram of the process equipment is seen in Figure 3.

Good fiber separation and dispersion is obtained with dilute slurries (a typical slurry would consist of 0.26 wt % fibers and 0.19 wt % starch). Starch concentrations in the slurry, however, have been varied from 0.06 to 0.52 weight percent in making experimental Palarite moldings.

It is very important to have the starch solution at its minimum concentration during the slurry operation. The surface activity of silica in water is sensitive to very slight changes in the amount of adsorbed polymers. A more concentrated solution of starch retards the flocculation of silica and thereby decreases the rate of filtration. Vacuum forming of parts becomes increasingly difficult with low filtration rates. For this reason, demineralized water at 15 - 17° C is used to inhibit starch dissolution. It has also become standard procedure to add the starch to the slurry during the final minutes of agitation.

Another process variable which can affect the properties of Palarite is the pH of the slurry. Significant increases in density in the fibrous molding occur

131385

Figure 2. INTERIOR OF THE WILEY MILL.

as the slurry pH becomes more acidic. Variation in slurry pH can be attributed primarily to differences in the pH of the demineralized water and the amount of acid remaining on the acid-leached fibers. As a process control procedure, the pH of the slurry is adjusted to 7.0 ± 0.5 during the mixing operation.

Vacuum-Filtration Operation

In the molding process, the slurry is fed by gravity or pumps to a smaller container in which the filter cake is formed by applying a vacuum (20 to 26 inches Hg) on the inside of the perforated mandrel. A wide variety of shapes can be molded, depending on the particular mandrel design employed. Flat slabs, hollow cylinders, and hollow conical frustums are some of the more conventional geometries which have been formed with this process.

The use of a small molding vessel minimizes the settling of solids during filtration. Fibers and starch are quickly drawn to the mandrel, and the fibrous

Figure 3. PALARITE PROCESS EQUIPMENT.

mat is formed with a uniform mixture of the two solids. A porous filter of cotton cloth allows rapid filtration with only a slight loss of solids.

After all of the solids are deposited on the mandrel, vacuum dewatering of the filter cake is continued until a water/solids weight ratio of approximately 1.25/1.00 is achieved. Control of the dewatering operation is important, as wide variations in the residual water content will affect the material properties. During drying, a high water content increases the tendency toward delamination and promotes migration of the binder to the surface of the insulator. After dewatering, the fibrous molding has enough structural integrity to be removed from the mandrel. A perforated mandrel, with the cloth filter removed, and a vacuum-cast Palarite component is shown in Figure 4.

Curing, Drying, and Furnace Heating

Process operations following vacuum molding are performed to effect a rigid carbon-bonded fibrous structure. The basic operations are curing (gelation) of the starch, drying, and furnace heating (pyrolysis of the starch).

131386

Figure 4. FORMING MANDREL AND VACUUM-CAST PALARITE INSULATOR.

Starch will undergo the following reactions in the presence of water and heat:

In the molded filter cake, the starch is present as crystalline granules. The granules consist of an amylopectin membrane surrounding an amylose center. Gelation of the starch occurs when the membrane surrounding the granule ruptures, releasing the amylose core which forms a colloidal dispersion in water. Disruption of the membrane begins in most starches between 64 and 71°C.

In order to achieve complete starch gelation, the fibrous molding is heated at 130° C for six hours in an air atmosphere saturated with water vapor. After gelation, the residual water is removed by low-humidity drying at 130° C. Drying is completed after 18 hours at this temperature. Pressure can be applied during the gelation process if an insulator with higher density and strength is desired. The material must be completely dried while under pressure to prevent relaxation.

Pyrolysis of the starch to effect a carbon bond is conducted by heating the insulation in an inert atmosphere at 1,000° C. Shrinkage during carbonization is generally between 5 and 10 percent. Figure 5 shows an insulator unit which has been completed through the pyrolysis operation.

131387

Figure 5. PALARITE INSULATOR AFTER PYROLYSIS OF THE STARCH BINDER.

The degree of bonding achieved in Palarite is dependent upon the extent to which the starch is gelled prior to drying. In order to obtain good bonding it is essential that the water-starch reaction be carried to at least gelation. This need becomes apparent upon examination of the microstructure using scanning and transmission electron microscopy (Figures 6 through 9). Figure 6 presents two scanning electron micrographs of Microquartz fibers bonded together with gelatinized starch. Note that the starch appears to be more concentrated at the fiber junctions, although wetting of individual fibers does occur.

(a) At 940X

C-331

(b) At 18,700X

C-331

Figure 6. MICROQUARTZ FIBERS BONDED WITH GELATINIZED STARCH.

(a) At 37,050X

PEM-200-9304

(b) At 25,800X

PEM-200-9314

Figure 7. MICROQUARTZ FIBERS AFTER PYROLYSIS OF THE GELATINIZED STARCH.

Mandrel Surface
↕

(a) At 100X

(b) At 300X

(c) At 1,000X

(d) At 3,000X

Figure 8. CARBON-COATED MICROQUARTZ FIBERS.

PEM-200-9311

Figure 9. MICROQUARTZ FIBERS AFTER PYROLYSIS OF NONGELATINIZED STARCH. (63,000X)

This relatively small amount, by weight, of gelatinized starch results in an excellent carbon bond between fibers. Two transmission electron micrographs of the microstructure after pyrolysis of the gelatinized starch are seen in Figure 7. The bond between Microquartz fibers is formed by thin and multi-layered laminar sheets and webs of carbon. The thickness of a single sheet is about 100 Å. Bonding occurs at the fiber-to-fiber junctures, rod edge to rod edge, and through complete encapsulation of fibers by thick laminar sheets. A series of scanning micrographs of the carbon-bonded structure is seen in Figure 8. The thin, translucent carbon webbing can be seen in Figure 8 (d). The internal appearance of the fibrous material in which the starch was not gelled prior to pyrolysis is clearly indicated in Figure 9. Here, the carbon is present along the edges of fibers as small granules and has a very weak bonding effect.

These photographs also show to some extent the orientation of fibers within the Palarite insulation. The tendency, during the vacuum-forming process, is for the individual fibers or fiber aggregates to align themselves in a direction parallel to the mandrel surface (or perpendicular to the applied force of vacuum). The result is a laminated or layered fibrous structure. This layering effect is well illustrated in Figure 8(a). Fibers within aggregates appear to have a random orientation.

Machining

Single-point tooling or metal-bonded diamond grinding wheels can be used in machining Palarite. Parts can be finished to close tolerances, and the surface quality and edge definition are good. Very thin slitting wheels can be used for the slicing and cutting-off operations. Figure 10 is a photograph of a machined insulation component.

PROPERTIES OF PALARITE INSULATION

A program was conducted to thoroughly characterize the thermal and physical properties of Palarite insulation. Emphasis was placed on determining the strength-density-thermal conductivity interrelations over a relatively wide range of values. Wide differences in properties were achieved by varying the relative volume fractions of binder, fibers, and porosity.

The wide range of properties available with Palarite insulation make it particularly attractive in designing thermal protection systems for specific applications. A summary of these properties is presented in the subsections that follow.

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Figure 10. MACHINED PALARITE INSULATION COMPONENT.

Chemical Composition

The principal constituents of Palarite insulation are carbon and silica. The material has been prepared with compositions ranging from 2 weight percent carbon, 98 weight percent silica to 35 weight percent carbon, 65 weight percent silica.

Temperature Capability

In a vacuum or in an inert atmosphere, Palarite can be used to temperatures above 2,000° F. Temperature limitations are dependent upon the stability of the silica fibers. Microquartz fibers can be used in continuous applications at 2,000° F, but they are dimensionally stable at temperatures up to 2,750° F for short periods of time.

In air or an oxidizing environment, degradation of the structural integrity of Palarite will occur above 550° F due to the loss of the carbon binder.

Density

Palarite has been prepared with bulk densities ranging from 7 to 32 pounds per cubic foot (pcf). This wide variation in density can be obtained by controlling porosity and the relative volume fractions of carbon and silica. For this range of densities, the insulation has pore volumes between 0.95 and 0.75 of total volume.

Specific Heat

Table 3 lists the specific heats of two Palarite insulators at four different temperatures.

Table 3
SPECIFIC HEAT OF PALARITE

Composition	Temperature (° C)	Specific Heat (cal/gm-° C)
2.0 wt % Carbon, 98.0 wt % Silica	50	0.183
	100	0.200
	150	0.209
	250	0.220
19.2 wt % Carbon, 80.8 wt % Silica	50	0.163
	100	0.200
	150	0.222
	250	0.233

Strength

The compressive and ultimate flexural strengths of Palarite are listed in Table 4.

Table 4
COMPRESSIVE AND ULTIMATE FLEXURAL STRENGTHS OF PALARITE

Bulk Density (pcf)	Ultimate Flexural Strength (psi)	Compressive Strength	
		at 10% deflection (psi)	at 20% deflection (psi)
8	120	35	50
10	200	60	80
14	430	125	170
18	730	190	260
22	1,030	270	375
26	1,340	410	560

Thermal Conductivity

Table 5 summarizes the thermal conductivity of Palarite insulation with densities between 8 and 30 pcf.

Table 5
THERMAL CONDUCTIVITY OF PALARITE

Bulk Density (pcf)	Thermal Conductivity(1) (in argon) (Btu-in/hr-°F-ft ²)		
	at 400° F	at 800° F	at 1,000° F
8	0.30	0.38	0.45
10	0.31	0.39	0.46
14	0.34	0.44	0.52
18	0.41	0.52	0.62
22	0.49	0.63	0.73
26	0.57	0.75	0.86
30	0.66	0.88	0.99

(1) Data obtained from Northrup comparative apparatus.

Hygroscopicity

The amount of moisture absorbed in Palarite at several levels of relative humidity and temperature is given in Table 6.

Table 6
HYGROSCOPICITY OF PALARITE

Relative Humidity (%)	Temperature (°F)	Moisture Gain (wt %)
100	120	2.5
100	70	2.0
50	120	0.4
50	70	1.4

These data were obtained on insulation which had been furnace heated to 1,000° C in the fabrication process. Pyrolysis of the starch at higher temperatures (ie, 1,300 - 1,500° C) can result in a lower hygroscopicity due to a decrease in the surface activity of the carbon.

Resistance to Vibration, Shock, and Acceleration

Palarite is quite resilient and can withstand severe forces of vibration, shock, and acceleration without undergoing permanent dimensional change. Some of the conditions under which the insulation has been tested are as follows:

Vibration - 10 percent repeated cyclic compressive strain at 220 cps for five seconds; 1.6 percent repeated cyclic compressive strain at 500 cps for five seconds.

Shock - 50 g's, 11-millisecond half-sine pulse; 70 g's, 1-millisecond half-sine pulse.

Acceleration - 200 g's along longitudinal and lateral axes.

DISCUSSION

A development program was conducted to characterize the thermal and physical properties of Palarite. Emphasis was directed toward the determination of density-strength-thermal conductivity interrelations over a fairly wide range of values. A primary objective of the study was to determine the best method of improving the strength of the insulation in terms of maximum strength gain per unit increase in density and thermal conductivity.

By varying the relative volume fractions of carbon and silica, a broad range in properties was realized. Material and process conditions used to control the concentrations of the solid phases were: slurry composition (ie, the starch/fiber weight ratio), fiber length, and mechanical compaction.

Strength

The effect of carbon binder concentration, fiber length, and fiber packing density on the strength of Palarite was determined. Compressive and flexural testing was performed on insulation with binder contents between 4 and 30 weight percent carbon, and compacted insulation with densities ranging from 10 to 27 pcf. The physical properties were determined for Palarite which had been prepared with silica fibers with lengths from 0.080 to 0.500 inch.

Binder Concentration - The binder concentration in Palarite is dependent upon the amount of starch present in the fiber-water-starch slurry, as practically all of the starch is trapped in the fibers during the vacuum molding process. A series of insulators was prepared with binder contents ranging from 4 to 30 weight percent carbon by varying the starch/fiber weight ratio from 0.25/1.0 to 2.0/1.0 in the slurry preparation.

Several discs of insulation, approximately 12 inches in diameter and 1 1/2 inches thick, were vacuum molded from each slurry composition. The materials were cured and dried at 90° C. Pyrolysis of the starch binder was conducted at 1,000° C in an inert atmosphere. Compressive test specimens three inches in

diameter by one inch thick, and flexural test specimens 3 inches by 1/2 inch by 1/4 inch were machined from the larger discs. The Microquartz fibers used in making these samples were initially passed through a Wiley mill which contained a sizing plate with 1/4-inch-diameter holes.

Results of the compressive testing are summarized in Table 7. Testing was performed on an Instron universal testing machine at a crosshead speed of 0.05 in/min. Load-strain curves were obtained up to 10 percent deflection; specimen strain was measured with a Tinius-Olsen deflectometer. The weight percents of carbon present as binder were determined by carbon burnout after completing the physical testing.

Table 7
EFFECT OF BINDER CONCENTRATION ON THE COMPRESSIVE STRENGTH OF PALARITE

Sample Number ⁽¹⁾	Slurry Composition (starch/fiber weight ratio)	Bulk Density (pcf)	Binder Content ⁽²⁾ (wt %)	Compressive Strength (psi) ⁽³⁾					Modulus of Elasticity (x 10 ³ psi)
				2	4	6	8	10	
A-4-1	0.25/1.00	7.49	2.1	11	15	16	17	18	0.8
A-4-2	0.25/1.00	7.43	5.1	3	10	14	15	16	0.4
A-4-3	0.25/1.00	7.61	4.2	3	9	14	16	17	0.4
B-6-1	0.50/1.00	7.99	8.0	7	26	32	34	35	1.1
B-6-2	0.50/1.00	8.30	8.4	8	25	31	33	34	1.0
B-6-3	0.50/1.00	7.80	8.3	15	29	32	33	35	1.2
C-2-1	0.75/1.00	8.74	12.9	5	43	50	53	55	2.4
C-2-2	0.75/1.00	8.36	15.4	23	49	53	55	56	2.6
C-2-3	0.75/1.00	8.99	16.7	19	49	54	56	58	2.3
C-2-4	0.75/1.00	8.99	12.6	23	54	59	61	63	2.6
D-1-1	1.50/1.00	10.73	34.0	14	73	76	79	81	2.2
D-1-2	1.50/1.00	10.67	28.0	20	52	58	62	65	2.0
D-1-3	1.50/1.00	10.73	28.0	19	50	64	70	73	1.7

(1) Each specimen was 3 inches in diameter by 1 inch thick.

(2) Carbon.

(3) Numbers represent the compressive strength at that percent deflection.

An increase in the binder content from about 4 to 30 weight percent carbon resulted in an increase in bulk density from 7.5 to 11.0 pcf and a compressive strength (at 10 percent deflection) from 15 to 70 psi. The relation between concentration of carbon binder and compressive strength is indicated by the graph of Figure 11. In Figure 12, the variation in compressive strength with density is presented. Changes in density were due to the increased binder content.

Figure 11. COMPRESSIVE STRENGTH OF PALARITE AS A FUNCTION OF THE BINDER CONCENTRATION.

Figure 12. COMPRESSIVE STRENGTH OF PALARITE AS A FUNCTION OF THE BULK DENSITY.

There was a substantial improvement in strength with additional binder between 4 and 15 weight percent carbon. Above 15 weight percent carbon (~ 9 -pcf density), a lower rate of strength enhancement was observed with increasing binder content.

The compressive stress-strain curves for the individual samples are reproduced in Appendix A. The curves for Palarite insulation generally contained three distinct phases: a "tail" that usually occurs during initial loading (ie, 0 - 3 percent deflection) due to surface irregularities or nonparallelism of surfaces; the linear, or elastic, portion of the stress-strain diagram extending over a fairly short range of strain (ie, 2 or 3 percent); a final segment of the curve beyond the yield point (here the insulation yielded inelastically up to very large deflections).

The elastic portion of the stress-strain curves was likely experienced during the bending of silica fibers. Breaking of carbon-silica bonds is believed to occur at the yield point. If the compressive load is relaxed prior to reaching the yield stress, the insulation will return to its original dimensions. Loading above the yield point will result in a permanent set, as can be seen by the curves of Figure 13. Here, a Palarite insulator of 10.5-pcf density was compressively loaded to 45 percent deflection. The permanent set after relaxation of the load was 10 percent of the original thickness.

Figure 13. PERMANENT DEFORMATION IN PALARITE AFTER LOADING TO LARGE STRAINS.

The ultimate flexural strengths of Palarite insulation containing from 4 to 30 weight percent carbon binder were determined. The results are reported in Table 8. Testing was performed on an Instron universal testing machine at a crosshead speed of 0.05 in/min using a thirds-point loading scheme. Specimen strain was measured with a Tinius-Olsen deflectometer. The ultimate flexural strength increased from approximately 120 to 280 psi, with an increase in binder concentration from 4 to 30 weight percent carbon (Figure 14). Increase in the modulus of elasticity was from near 1.5×10^4 psi to 4.0×10^4 psi. A typical flexural stress-strain curve is reproduced in Appendix B, along with a sample calculation of flexural strength and modulus of elasticity.

Table 8
EFFECT OF BINDER CONCENTRATION ON THE ULTIMATE FLEXURAL STRENGTH OF PALARITE

Sample Series(1)	Slurry Composition (starch/fiber weight ratio)	Number of Specimens	Average Bulk Density (pcf)	Average Binder Content(2) (wt %)	Average Ultimate Flexural Strength (psi)	Average Modulus of Elasticity ($\times 10^4$ psi)
P-1-4	0.25/100	12	9.9	3.79	130	1.69
P-2-1	0.50/1.00	16	7.8	7.62	123	1.51
P-3-5	1.00/1.00	12	12.4	22.18	228	3.39
P-4-6	1.50/1.00	16	9.4	25.30	267	3.94
P-5-3	2.00/1.00	14	10.2	28.70	253	3.92

(1) Bar samples were 1/2 by 1/4 x 3 inches.

(2) Carbon.

The moduli of elasticity of Palarite determined from flexural testing were more than an order of magnitude greater than the moduli observed from compressive testing ($\sim 4 \times 10^4$ psi vs 2×10^3 psi). This difference may be largely due to differences in applied stresses with respect to the anisotropy of the insulation. Stresses arising in compressive testing are perpendicular to the laminated fibrous structure; flexural testing generates internal stresses which are parallel to the preferred orientation of the fibrous laminae.

Fiber Length - The fiber concentration in Palarite is influenced by the length of the Microquartz fibers. It would be logical to expect a higher concentration of fibers with decreasing fiber length. Shorter fibers should give a higher insulator density and, therefore, an increased thermal conductivity and strength.

As-received Microquartz fibers are intertwined tightly in small aggregates. The length of the fibers is variable, with most fibers in the 1/2 to 3/4-inch range. Vacuum conveying as-received fibers through a Wiley mill is an effective method of separating fibers. Subsequent high-speed slurry agitation gives a good fiber

Figure 14. VARIATION OF FLEXURAL STRENGTH AS A FUNCTION OF THE BINDER CONCENTRATION.

dispersion and homogeneous part castings. The amount of reduction in fiber length due to milling is not known, although it can be varied to some extent by a sizing plate within the mill. The sizing plate used for fiber separation in the Palarite production process contains 1/4-inch-diameter holes.

A study was conducted to determine the relation between the fiber length and the density and strength of Palarite. Wiley mill sizing plates containing holes with diameters of 0.500, 0.250, 0.125, and 0.079 inch were used to obtain fibers of different lengths. Identical slurry compositions, with a starch/fiber weight ratio of 0.75/1.00, were used in the sample preparation.

Insulation prepared from fibers which had been passed through a sizing plate with 0.500-inch-diameter holes had a higher strength and density than that made from fibers which had been treated through smaller holes (Table 9). The density, compressive strength, and ultimate flexural strength of this material were 10 - 11 pcf, 107 psi at 10 percent deflection, and 348 psi, respectively. In going from 0.250 to 0.079-inch-diameter holes in milling the fibers, the resultant insulator properties were affected as follows: (1) the density increased from about 8 to 10 pcf, (2) the compressive strength at 10 percent deflection remained essentially the same at 35 - 40 psi, and (3) the ultimate flexural strength was relatively unchanged at 135 - 190 psi.

It was probable that fiber separation was incomplete in using the sizing plate with 0.500-inch-diameter holes. As a result, the high-density fiber aggregates were carried through the process, giving the insulation a higher density and strength. A higher strength might be expected because of the entanglement of

Table 9
EFFECT OF FIBER PRETREATMENT ON THE PHYSICAL PROPERTIES OF PALARITE INSULATION

Series(1)	Sizing Plate Hole Diameter (inch)	Number of Samples	Average Bulk Density (pcf)	Average Binder Content ⁽²⁾ (wt %)	Average Compressive Strength (psi) ⁽³⁾					Average Ultimate Flexural Strength (psi)
					2	4	6	10	20	
M-1(4)	0.500	5	10.9	17.75	66	92	98	107	126	-
M-2(4)	0.250	5	8.2	12.20	12	30	34	37	44	-
M-3(4)	0.125	5	8.5	10.85	14	32	36	40	48	-
M-4(4)	0.079	5	10.1	14.63	21	31	33	36	44	-
M-1(5)	0.500	14	10.2	19.51	-	-	-	-	-	348
M-2(5)	0.250	14	8.6	13.60	-	-	-	-	-	168
M-3(5)	0.125	12	8.9	12.40	-	-	-	-	-	191
M-4(5)	0.079	16	10.0	24.20	-	-	-	-	-	136

(1) Pretreatment consisted of passing as-received Microquartz fibers through a Wiley mill containing sizing plates with different hole diameters.

(2) Carbon.

(3) Numbers represent the compressive strength at that percent deflection.

(4) Discs, 3 inches in diameter by 1 inch thick.

(5) Bars, 1/2 by 1/4 by 3 inches.

fibers within aggregates, as well as the increased anisotropy resulting from a fibrous mat of nonoriented aggregates.

It appears that the sizing plate with 0.250-inch-diameter holes gave good fiber separation with little reduction in fiber length. This result would perhaps explain the observed minimum in insulator density resulting from the various degrees of fiber sizing. On decreasing the hole size from 0.250 to 0.125 and 0.079 inch in diameter, fiber length was being reduced and the insulator density increased.

Fiber Compaction - The effect of fiber concentration, or the volume fraction of silica fibers, on the compressive and flexural strength of Palarite was determined. In preparing the samples, vacuum-molded discs of the material were pressed between metal plates at pressures ranging from 100 to 300 psi. The compacted insulation was held at the pressed thickness during the subsequent gelation and drying operations. The metal plates were removed prior to pyrolysis of the starch binder. Fibers used in making these insulators were initially passed through a Wiley mill which contained a sizing plate with 1/4-inch-diameter holes. To maintain similar binder concentrations, identical slurry formulations were used (a starch/fiber weight ratio of 0.75/1.00) in sample preparation.

Using this technique, insulator densities from 10 to 27 pcf were obtained. Compressive tests were conducted on 12 specimens of the compacted insulation.

Binder contents were determined by carbon burnout after physical testing; the volume fractions of silica were calculated for individual specimens.

Increasing the density of Palarite by compaction resulted in significantly higher compressive strengths. The data are presented in Table 10; variation in compressive strength with density is shown in Figure 15. An increase in density from 10 to 27 pcf resulted in an increase in compressive strength, at 10 percent deflection, from 60 to 460 psi. For this range of densities, the increase in the volume fraction of silica was from 0.071 to 0.186.

Table 10
COMPRESSIVE STRENGTH OF COMPACTED PALARITE INSULATION

Sample Number ⁽¹⁾	Bulk Density (pcf)	Volume Fraction of Silica, VF _s	Binder Content ⁽²⁾ (wt %)	Compressive Strength (psi) ⁽³⁾				
				2	5	10	15	20
S-1-4-1	10.3	0.071	7.74	8	40	64	74	84
S-1-4-2	10.4	0.071	7.72	20	55	68	78	86
S-1-4-3	10.5	0.074	7.40	59	69	79	89	98
S-1-4-4	10.9	0.083	6.09	54	66	78	88	98
S-2-3-2	17.2	0.106	18.58	64	131	163	191	223
S-2-3-3	20.0	0.134	11.83	180	212	251	294	340
S-4-1	20.8	0.131	13.18	42	164	221	266	318
S-2-3-4	21.1	0.142	10.85	71	191	250	300	347
S-4-2	22.5	0.156	14.25	149	232	332	410	494
S-4-5	23.4	0.152	13.20	50	208	290	347	410
S-4-4	24.2	0.156	12.97	70	255	329	389	453
S-4-3	27.5	0.186	8.72	113	396	488	577	679

(1). Three-inch-diameter by 1/2-inch-thick discs.

(2). Carbon.

(3). Numbers represent the compressive strength at that percent deflection.

The ultimate flexural strength of the insulation also increased substantially with increasing fiber concentration. Flexural tests were performed on 16 specimens with densities between 15 and 26 pcf; the results are given in Table 11.

Compacted Palarite of 15-pcf density had a flexural strength of 500 psi; increasing the density to 26 pcf increased the flexural strength to about 1,340 psi. Variation in flexural strength with density is shown in Figure 16.

Thermal Conductivity

In fibrous insulating materials such as Palarite, the total heat transfer is generally viewed as a summation of the contributions of gaseous conduction, solid

Figure 15. COMPRESSIVE STRENGTH AS A FUNCTION OF THE DENSITY IN COMPACTED PALARITE INSULATION.

conduction, and thermal radiation. Convection heat transfer is usually neglected with the assumption that convection currents are not established within the small voids of the material. At steady state, the effective conductivity is given by:

Table 11

ULTIMATE FLEXURAL STRENGTH OF COMPACTED PALARITE INSULATION

Sample Number	Bulk Density (pcf)	Ultimate Flexural Strength (psi)	Modulus of Elasticity ($\times 10^5$ psi)
S-1-1-1	15.1	565	0.96
S-1-1-4	15.9	514	1.06
S-1-1-3	16.8	700	1.31
S-1-1-5	17.4	765	1.37
S-1-1-6	17.6	722	1.35
S-1-1-2	17.8	806	1.46
S-3-5-2	20.1	718	1.16
S-3-5-5	21.2	874	1.50
S-2-1-3	21.8	914	1.66
S-3-5-1	22.8	1,202	1.61
S-2-1-2	23.5	955	1.91
S-3-5-4	24.0	1,360	1.92
S-2-1-1	24.8	1,042	2.29
S-3-5-3	25.1	1,411	2.06
S-2-1-4	25.8	1,545	2.40
S-2-1-6	26.3	1,328	2.44

Figure 16. ULTIMATE FLEXURAL STRENGTH AS A FUNCTION OF THE DENSITY IN COMPACTED PALARITE INSULATION.

$$k_e = k_s + \delta k_g + k_r,$$

where:

k_e represents the effective thermal conductivity,

k_s the solid conduction contribution,

k_r the radiant contribution,

δ the volume fraction of pores, and

δk_g the gaseous conduction contribution.

Analyses of heat transfer in fibrous insulations indicate that the solid mode of conduction is dependent upon the fiber radius, the number of fiber contact points, the thermal conductivity, K , of the fiber material, and the orientation of fibers with respect to the direction of heat flow.⁽¹⁻³⁾

For a particular fiber material, such as silica, an increase in the fiber radius or the number of fiber contact points would enhance the solid contribution to

conductivity; alignment of the fibers in a direction parallel to the heat flow would enhance the solid conductivity due to the longer conduction paths.

In Palarite insulation, a quantitative estimate of the solid conductivity is complicated by the addition of a second solid phase (carbon binder) and the very unordered fiber structure. Theoretical analyses are limited to qualitative predictions. Basically, it would be expected that there would be an increase in the solid contribution to the total heat transfer with increasing volume fraction contents of silica fibers and carbon binder. It can be assumed that fiber radii and orientation are quite similar from part to part and neglect the effect of these variables in comparing the heat transfer properties of Palarite insulation.

The highly open porosity of Palarite makes it possible to readily predict the gaseous conduction contribution to the effective thermal conductivity. The relatively large interfiber spacing with respect to the mean free path of gas molecules implies that the gas can diffuse as in a free gas volume. In Palarite, with a typical δ (or porosity) between 0.90 and 0.95, the gaseous contribution at 200° F would be about 0.13 Btu-in/hr-° F-ft² and increase to approximately 0.26 Btu-in/hr-° F-ft² at 1,200° F in an argon environment.

The radiant contribution to effective thermal conductivity in fibrous insulations has been analyzed by several investigators.^(4 - 9) Assuming steady-state heat flow, the radiant transmission through the material was explained in terms of absorption, scattering, and reradiation mechanisms. The theoretical equation obtained for the radiant contribution is:

$$k_r = \frac{8\sigma J^2 T^3}{P + 2N},$$

where:

J represents the index of refraction,

T the mean sample temperature,

P the absorption cross section,

N the scattering cross section, and

σ the Stefan-Boltzmann constant.

Rolinski and Purcell experimentally determined the absorption and scattering cross section of Dynaquartz (silica fiber) insulation using transmittance

measurements. They estimated the radiant contribution to be negligible at 400° F, about 0.10 Btu-in/hr-° F-ft² at 800° F, and approximately 0.23 Btu-in/hr-° F-ft² at 1,200° F.

Again, theoretical predictions of the heat transfer in Palarite are complicated by the presence of carbon. It could be qualitatively expected that there would be a decrease in the radiant contribution with increasing concentration of both silica and carbon, as the scattering and absorption cross section would be increased with additions of these materials.

An investigation was made concerning the effect of carbon binder concentration, fiber length, and fiber-packing density on the thermal conductivity of Palarite. Thermal conductivity was determined on insulation with binder contents between 2.0 and 35.0 weight percent carbon, compacted insulation with densities between 19.9 and 32.0 pcf, and material prepared from fibers with lengths between 0.079 and 0.500 inch.

Binder Concentration - The thermal conductivity of Palarite with binder contents between 2.0 and 35.0 weight percent carbon was determined in the temperature range of 300 to 1,400° F. Measurements were taken in an argon atmosphere using a Northrup comparative apparatus.

The coefficient of thermal conductivity of Palarite was found to increase with increasing binder content, and the variation of the thermal conductivity, K , with temperature for the various materials was essentially linear, as Figure 17 indicates. For the material containing 2.0 weight percent binder (7.2-pcf density), the thermal conductivity increased from 0.26 Btu-in/hr-° F-ft² at 300° F to 0.47 Btu-in/hr-° F-ft² at 1,200° F; for Palarite containing 35.0 weight percent binder (12.7-pcf density), the thermal conductivity increased from 0.48 Btu-in/hr-° F-ft² at 300° F to 0.74 Btu-in/hr-° F-ft² at 1,200° F.

In Figure 18, the thermal conductivity at several temperatures is plotted against the binder concentration. The rate of increase in K with binder content appears to become greater above about 20 weight percent binder.

An increase in the thermal conductivity of Palarite with the carbon concentration can be attributed to the enhanced solid conductivity within the composite. Encapsulation and bridging of fibers by the carbon provides additional solid conduction paths, and the thermal conductivity of carbon is relatively high. Increasing the carbon concentration would provide a greater absorption cross section for the attenuation of radiant energy. However, since there is no converging or crossing of the K -versus-temperature curves, any reduction in

Figure 17. THERMAL CONDUCTIVITY OF PALARITE INSULATION. (1/4-Inch Microquartz Fibers)

the radiant contribution to heat transfer must be offset by the increase in solid conduction.

Fiber Length - The thermal properties of Palarite are significantly influenced by the volume fraction of the silica fibers. A higher fiber concentration would, of course, increase the density of the insulation, and a higher coefficient of thermal conductivity might also be expected.

Figure 18. EFFECT OF BINDER (CARBON) CONTENT ON THE THERMAL CONDUCTIVITY OF PALARITE INSULATION.

One method of preparing Palarite with variable fiber concentrations was to use silica fibers of different lengths. A milling operation, described earlier in this report (Page 14), was used to separate and size the fibers. Sizing plates with hole diameters of 0.500, 0.250, 0.125, and 0.079 inch were employed to obtain fibers of different lengths.

The thermal conductivities of Palarite insulation prepared from fibers milled through 0.500, 0.250, and 0.125-inch-diameter holes were quite similar. The data are presented in Table 12. Ongoing from 400 to 1,000° F mean temperature, the thermal conductivity, K, of these insulators increased from about 0.31 to 0.45 Btu-in/hr-° F-ft².

Table 12
EFFECT OF FIBER PRETREATMENT ON THE THERMAL CONDUCTIVITY OF PALARITE INSULATION

Sizing Plate Hole Diameter (1) (inch)	Palarite Density (pcf)	Binder Content (2) (wt %)	Thermal Conductivity in Argon (Btu-in/hr-° F-ft ²)		
			at 400° F	at 800° F	at 1,000° F
0.500	11.20	13.05	0.33	0.41	0.45
0.250	7.60	12.15	0.31	0.40	0.45
0.125	9.40	15.75	0.31	0.41	0.45
0.079	10.60	15.90	0.50	0.57	0.61

(1) Pretreatment consisted of passing as-received Microquartz fibers through a Wiley mill containing sizing plates with variable hole diameters.

(2) Carbon.

Thermal conductivity of the insulation prepared with fibers which had been passed through a 0.079-inch-diameter holes was significantly higher. For this material, the K value increased from 0.50 Btu-in/hr-° F-ft² at 400° F to 0.61 Btu-in/hr-° F-ft² at 1,000° F. The high coefficient of thermal conductivity can be attributed to an increase in the solid conduction component of the total heat transfer. The reduction in fiber length has apparently created a significant increase in the number of fiber contact points within the insulator molding.

Fiber Compaction - Another method of varying the volume fraction of fibers is by compaction of the insulation during the fabrication process. This technique was described earlier in this report (Page 35) and was used to prepare thermal-conductivity samples of Palarite with densities up to 32 pcf.

The thermal conductivity of compacted Palarite insulators with densities of 19.9, 26.2, and 32.0 pcf as a function of the mean temperature is given in Figure 19. Measurements were taken in an argon atmosphere between approximately 200 and 1,200° F. Included in this graph for comparison is the thermal

Figure 19. THERMAL CONDUCTIVITY OF COMPACTED PALARITE INSULATION.

conductivity of a conventional, noncompacted Palarite insulator with a density of 9.6 pcf.

As Figure 19 indicates, thermal conductivity of the compacted materials was found to increase with increasing density. At 1,000° F, for example, K values for the 9.6, 19.9, 26.2, and 32.0-pcf insulators were 0.51, 0.66, 0.88, and 0.99 Btu-in/hr-°F-ft², respectively.

The increase in total heat transfer with increasing fiber concentration or compacted density is most likely due to the enhanced solid conduction. As in the case of shorter fibers, the number of fiber contact points becomes greater with increased compaction. In addition, with a constant binder concentration, higher compacted densities would increase the volume fraction of carbon which would further enhance the solid conduction contribution.

CONCLUSIONS

The more important findings of the parametric studies are:

1. An increase in the binder concentration in Palarite insulation from 4 to 30 weight percent carbon increased the density from 7.5 to 11.0 pcf, the

compressive strength at 10 percent deflection from 15 to 70 psi, the flexural strength from 120 to 280 psi, and the coefficient of thermal conductivity, K , at 1,000° F from 0.43 to 0.63 Btu-in/hr-° F-ft².

2. The density, compressive strength, and ultimate flexural strength of the insulation that was prepared from fibers which had been passed through a sizing plate with 0.500-inch-diameter holes were: 10 - 11 pcf, 107 psi at 10 percent deflection, and 348 psi, respectively. In going from 0.250 to 0.079-inch-diameter holes in milling fibers, the resultant insulator properties were affected as follows: (1) the density increased from about 8 to 10 pcf, (2) the compressive strength at 10 percent deflection remained essentially the same at 35-40 psi, and (3) the flexural strength was relatively unchanged at 135 - 190 psi.

The thermal conductivity of Palarite prepared from fibers milled through 0.500, 0.250, and 0.125-inch-diameter holes was about 0.45 Btu-in/hr-° F-ft² at 1,000° F; Palarite prepared with fibers which had been passed through 0.079-inch-diameter holes had a coefficient of thermal conductivity of 0.61 Btu-in/hr-° F-ft² at 1,000° F.

3. Increasing the density of Palarite by compaction resulted in significantly higher compressive and flexural strengths. An increase in density from 10 to 27 pcf gave an increase in compressive strength, at 10 percent deflection, of from 60 to 460 psi. Noncompacted Palarite of 8.0-pcf density had an ultimate flexural strength of 120 psi; increasing the density to 26 pcf increased the ultimate flexural strength to about 1,340 psi.

Thermal conductivity of the compacted insulation increased with increasing density. For example, at 1,000° F, the coefficients of thermal conductivity for 9.6-pcf noncompacted Palarite and 32.0-pcf compacted insulation were 0.51 and 0.99 Btu-in/hr-° F-ft², respectively.

4. The best method for improving the strength of Palarite insulation, in terms of maximum strength gain per unit increase in density and coefficient of thermal conductivity, was by increasing the fiber concentration by compaction. Compressive strength-density and compressive strength-thermal conductivity trade offs are shown by the graphs of Figures 20 and 21 for variations in binder concentration and degree of compaction.

Figure 20. PROPERTY TRADE OFFS IN PALARITE INSULATION. (Density)

Figure 21. PROPERTY TRADE OFFS IN PALARITE INSULATION. (Thermal Conductivity)

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APPENDIX A

COMPRESSIVE STRESS-STRAIN CURVES OF PALARITE INSULATION WITH VARIOUS BINDER CONCENTRATIONS

In Figures A-1 through A-4, compressive stress-strain curves are given for the samples reported in Table 7. Moduli of elasticity were determined by calculation of the slopes in the most linear portion of the curves. Refer again to Table 7 (Page 30) for the density and binder concentrations of the test specimens.

Figure A-1. STRESS-STRAIN CURVES FOR SAMPLE SERIES A.

Figure A-2. STRESS-STRAIN CURVES FOR SAMPLE SERIES B.

Figure A-3. STRESS-STRAIN CURVES FOR SAMPLE SERIES C.

Figure A-4. STRESS-STRAIN CURVES FOR SAMPLE SERIES D.

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APPENDIX B

CALCULATION OF THE ULTIMATE FLEXURAL STRENGTH AND MODULUS OF ELASTICITY

A typical flexural load-deflection curve for a Palarite bar specimen (Series P-4-6, Table 8) is shown in Figure B-1. The flexural strength was calculated from the following equation for thirds-point loading:

$$S = \frac{3 Pl}{bd^2},$$

where:

- S represents the flexural stress in psi,
- P the load in pounds,
- l the distance between loading points in inches,
- b the width of the specimen in inches, and
- d the height of the specimen in inches.

For the Series P-4-6 sample:

P = 4.05 pounds (at failure),

l = 0.7 inch,

b = 0.50 inch, and

d = 0.25 inch.

Therefore, the ultimate flexural strength is:

$$S = \frac{3 \times 4.05 \times 0.7}{0.50 \times (0.25)^2} = 272 \text{ psi.}$$

The modulus of elasticity, E, in psi is determined from the formula:

$$E = \left(\frac{5 l^3}{bd^3} \right) \left(\frac{\Delta P}{\Delta Y} \right),$$

where:

l represents the distance between the loading points in inches,

b the specimen width in inches,

d the specimen height in inches, and

$\frac{\Delta P}{\Delta Y}$ the slope of the linear portion of the load-deflection curve in pounds/inch.

For the Series P-4-6 sample:

$l = 0.7$ inch,

$b = 0.50$ inch,

$d = 0.25$ inch, and

$$\frac{\Delta P}{\Delta Y} = \frac{4.25 - 0.00}{0.030 - 0.005} = 170.0 \text{ lbs/inch.}$$

$$\text{Therefore, } E = \frac{5 \times (0.7)^3}{0.50 \times (0.25)^3} \times 170.0 = 3.7 \times 10^4 \text{ psi.}$$

Figure B-1. FLEXURAL LOAD-DEFLECTION CURVE FOR PALARITE INSULATION.