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AEC RESEARCH AND DEVELOPMENT REPORT
SPECIAL

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DEVELOP 1800F-400F FIBROUS-TYPE INSULATION
FOR RADIOISOTOPE POWER SYSTEMS

Fourth Quarterly Progress Report
Report No. ALO-3633-4

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J. O. Collins

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ABSTRACT

by

J. O. Collins

This is the Fourth Quarterly Report for AEC Contract AT(30-1)-3633 covering the period January 1967 through March 1967.

Some of the long term (2000 hr) evaluations of Formulations 1999 and 2002 have been completed; others will be completed during the Fifth Quarter.

Formulation 2020 has been designated for service with the SiGe RTG's at 1800F. Long term testing pertinent to this material has been initiated.

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1. INTRODUCTION

Long term testing of Formulations 1999 and 2002 for lead telluride RTG applications was completed for several of the physical properties. These included chemical compatibility with PbTe elements (final results still under analysis), load relaxation, and thermal stability. Out-gassing and thermal conductivity tests were initiated during this quarter, but have not been completed. Other physical properties such as dynamic response, moisture adsorption/desorption, and shear have been completed.

Chemical compatibility in air between SiGe elements and the fibrous candidates has completed the 2000 hr evaluation level. The same test in vacuum is approaching the 1500 hr level. Tests have also been initiated to evaluate the effect of prolonged contact between Irish REFRASIL and SiGe bars as determined by Seebeck voltage and resistivity measurements.

The effort to improve the temperature resistance of the MIN-K formulations has been concluded with the choice of Formulation 2020. Long term tests on this material were initiated.

II. FINAL SCREENING

A. CARBORUNDUM T30LR

Four specimens of Carborundum's T30LR Laminate (4 x 4 x 0.84-in.) were subjected to a constant load of 200 psi for 168 hr (1 week), at an elevated hot face to determine its load bearing characteristics. The product was sanded from its nominal 1-in. thickness to insure a flat specimen with a uniform bearing surface. The product ranged in density from 53.3 pcf to 54.9 pcf for the four specimens, and averaged 54.0 pcf.

It was originally intended that these tests be conducted at 1800F hot face. However, due to the high thermal conductance of the material (relative to standard insulations), and the power limitations of the heater unit, these tests had to be conducted at temperatures averaging approximately 1700F. The hot face indicating thermocouple of one specimen became erratic during the second and third days of test, indicating temperatures as high as 1800F and 1900F. It subsequently became useless. The control to the heaters was not changed during this period, nor for the remainder of the test, however, since the hot face couple was considered faulty. This decision was supported upon completion of the test when the measured shrinkage of the specimen was typical of the remaining three values.

Table 1 summarizes the data obtained for this product. The thickness shrinkage averaged 3.4 per cent. All changes in length or width dimensions were within the limits of measuring error.

This product did not prove itself to be as satisfactory for load bearing applications as the GLASROCK 25 product. No further work was done.

B. GLASROCK 25

Four specimens of GLASROCK 25 were tested for modulus of rupture (bend strength) at room temperature (test procedure presented in Third Quarterly Report). The specimens were 8 x 4-1/2 x 1-in. nominal size and were tested across a 7-in. span. The range of values was from 150 psi to 195 psi with an average of 172 psi. Therefore, although the material has been proven to be satisfactory in compression, it should not be subjected to excessive bending or tensile loads.

TABLE 1.

EFFECT OF 200 PSI AND 1700F (AVG) HOT FACE UPON
CARBORUNDUM'S T30LR LAMINATE AFTER 168 HR EXPOSURE

<u>Specimen</u>	<u>Density pcf</u>	<u>Hot Face °F</u>	<u>Cold Face °F</u>	<u>Thick. Shrinkage %</u>
A	54.9	1726 avg 1745 max 1700 min	398 avg 401 max 395 min	3.4
B	53.3	1695 avg 1699 max 1683 min	376 avg 382 max 369 min	2.9
C	53.6	1710 avg* 1710 max 1708 min	379 avg 385 max 375 min	3.5
D	54.2	1667 avg 1685 max 1635 min	447 avg 460 max 435 min	3.7

*Average of four readings only due to thermocouple malfunction.

GLASROCK 25 was also subjected to shear tests (see description of test procedure under III - Detailed Evaluation) and found to average 210 psi with a range of 186 psi to 225 psi for twelve specimens. This test was conducted parallel to the thickness surfaces.

A 12 x 12 x 1-in. specimen was tested for thermal conductivity in the Johns-Manville Rapid Heat Meter Apparatus at 73F and 800F mean temperatures. The values were 0.8 and 1.4 Btu in./hr sq ft °F, respectively, at the two temperatures. The manufacturer's literature indicated a thermal conductivity of 1.0 Btu in./hr sq ft °F at 1000F mean temperature. The previously verified error connected with the Heat Meter equipment is +8 per cent when testing this type of high conductivity material. Consequently, it must be assumed that the published data were incorrect. Nonetheless, the conductivity should be considered good when compared with similar type materials which have significantly less compression resistance than the GLASROCK 25.

III. DETAILED EVALUATION

A. LEAD TELLURIDE INSULATIONS

Chemical Compatibility

The chemical compatibility tests between Formulations 1999 and 2002, and PbTe 2N, 2P, 3N, 3P elements in argon at 1000 $\pm$ 20F has been completed to the 2000 exposure level. Seebeck voltage and resistivity tests have been conducted only on the 500, 1000, and 1500-hr exposure levels, however. Consequently, a detailed description of the statistical methods establishing the standard deviation of the Seebeck and resistivity tests, the effect of the Seebeck test on the resistivity test (the resistivity is measured both before and after the Seebeck test), the change of all elements as a result of exposure conditions, and the difference in Seebeck and resistivity change between the test elements and the "blanks" will be withheld until all the data are available. An investigation of the Seebeck and resistivity tests with respect to their standard deviation became necessary when it was obvious that a gross incompatibility did not exist, but that some degradation of the thermoelectric element properties might go unnoticed.

It was established that there was no significant difference between the resistivity values taken before and after the Seebeck voltage measurements, based upon the measurement of all elements before exposure to the insulations. Thus, the analysis will utilize the combined resistivity data (before and after Seebeck voltage) when evaluating the effect of exposure to the insulations. This will provide added data for the analysis.

A cursory examination of the data indicated that there was no significant change in resistivity for the 2N, 2P, and 3N PbTe elements up through the 1500-hr exposure level. The data for the 3P elements required additional study, however, as there existed a possible indication of change.

The Seebeck tests were less conclusive, primarily because of the fewer number of data points which were available for statistical analysis. Elements 3P and 2N showed a change in Seebeck at the 5 per cent significance level. This preliminary analysis grouped both the "blanks" and the "test" elements together. Further work will separate these groups to determine if the change was due to the insulation, or to the thermal exposure. Both the 3N and 2P elements showed no significant change in Seebeck voltage up to 1500-hr exposure to the insulation.

Outgassing of Formulation 2002

The preliminary stages of the outgassing test have been completed, and the specimens are currently on long term (2000 hr) exposure. The purpose of this early phase of the test was to attempt to gain some insight into the effect of various atmospheric moisture contents on the ease of removal of this moisture at low temperatures, and with numerous argon backfill cycles. The test procedure is given in Appendix A.

To summarize, four ceramic tubes were preconditioned to 1600F under vacuum, cooled to room temperature, and backfilled with air bled through magnesium perchlorate drying columns. One tube was opened, a 200-gm specimen (no specific prior conditioning) inserted, and the tube reclosed. All tubes were then heated to 1050F in the dry air atmosphere for 30 min, evacuated for an additional 30 min, and subsequently cooled to room temperature under active vacuum. This conditioning of the one specimen represented a simulation of the heat and vacuum to which the standard MIN-K 1301 specimens are subjected prior to generator assembly.

All tubes were backfilled again with dry air, and 200-gm specimens previously conditioned to 33% RH at 75F and 90% RH at 90F were inserted into two of the three empty tubes. The fourth tube remained empty throughout the test. Each tube was then individually pumped down at room temperature for 15 min, and then allowed to outgas (no active pumping) for an additional 15 min. This procedure was repeated for a total of three pumpdown and outgassing cycles.

Following the room temperature pumpdown and outgassing cycles, the system was helium leak checked. This was done by bagging each fitting and filling the bag with helium while evacuating the system through a helium detecting system. After several adjustments, all joints (including the VITON gasket joint between the ceramic tubes and the stainless steel vacuum system) showed a leak rate of less than 5×10^{-10} cc/sec. The porosity of the tubes themselves could not be measured due to the configuration of the test equipment. Consequently, these constitute the only source of leak.

The three pumpdown and outgassing cycles were then repeated on each tube individually at 400 $\pm$ 20F and at 600 $\pm$ 20F. Following the final cycle at 600F, the tubes were backfilled with argon (see Table 2 for analysis) to approximately 1030 mm Hg (20 psia) and held at these conditions for a minimum of 96 hr.

TABLE 2.
ANALYSIS OF ARGON USED IN OUTGASSING EVALUATION

<u>Constituent</u>	<u>Content, ppm</u>
N ₂	<2.0
O ₂	<1.0
CO ₂	not detected, <0.5
CO	not detected, <0.5
H ₂	not detected, <1.0
CH ₄	<0.4
H ₂ O	<8.0

A plot of pressure versus time for each of the tubes and at each of the three temperature levels is shown in Figures 1, 2, and 3. It will be noted that the pumpdown and outgassing rates followed the anticipated pattern based upon the water contents of each specimen.

Water content and residual gas analyses were run on each tube. The results of these analyses are shown in Table 3. The water analysis was run using a Consolidated Electrodynamics Corporation moisture monitor which directly measures water content by volume as it is bled from the tubes. The residual gases were determined on a mass spectrometer capable of detecting mass 2 to mass 100.

TABLE 3.
WATER CONTENT AND RESIDUAL GAS ANALYSIS AT 600F

	<u>33% RH</u>	<u>90% RH</u>	<u>Preconditioned at 1050F</u>	<u>Empty</u>
N ₂ , ppm	65	60	85	80
O ₂ , ppm	ND*	ND	ND	6
CO ₂ , ppm	47	37	32	20
H ₂ , ppm	ND	ND	ND	ND
CH ₄ , ppm	25	10	ND	ND
H ₂ O, ppm	500	900	6	20
H ₂ O, % wt (STP)	0.0010	0.0018	nil	nil

*Not detected, less than 4 ppm.

FIGURE 1.

FORMULATION 2002 PUMPDOWN AND OUTGAS CYCLES
AT ROOM TEMPERATURE

1 - 90% RH, 2 - 33% RH, 3 - Prefired to 1050F and evacuated, 4 - Empty

FIGURE 2.

FORMULATION 2002 PUMPDOWN AND OUTGAS CYCLES AT 400F

1 - 90% RH, 2 - 33% RH, 3 - Prefired to 1050F and evacuated, 4 - Empty

FIGURE 3.
FORMULATION 2002 PUMPDOWN AND OUTGAS CYCLES AT 600F

1 - 90% RH, 2 - 33% RH, 3 - Prefired to 1050F and evacuated, 4 - Empty

The water content was then converted from ppm by volume to per cent by weight of the test specimen at STP using the following equation:

$$\begin{array}{l} \% \text{ of H}_2\text{O} \\ \text{by weight} \end{array} = \frac{\left[\frac{273}{T} \times \frac{P_{\text{abs}}}{760} \times \text{Volume} \right] \left[\frac{\text{ppm}}{10^6} \right] \left[\frac{18}{22.4} \right]}{\text{Weight of Specimen (gm)}} \times 100$$

where:

- T = absolute temperature in $^{\circ}\text{K}$
- P_{abs} = absolute pressure in mm Hg
- ppm = measured parts per million of water by volume in system.

Thus, the tube containing the 33% RH specimen contained 0.0010 per cent water, and the tube containing the 90% RH specimen contained 0.0018 per cent water. No significance could be assigned to any differences between specimens in the residual gas analyses.

The three pumpdown and outgassing cycles were repeated at 600F for the two tubes showing the higher water contents. However, after each cycle, the tubes were pressurized with argon for a minimum of 5 min to investigate the effect of repetitive backfills. Following a 96 hr time period to achieve equilibrium within the tubes, the water content was rechecked. The tube containing the 33% RH specimen now contained 78 ppm water, while the tube containing the 90% RH specimen still contained 620 ppm water. At this point in the test, it appeared that the flushings had a greater effect on the specimen containing the lesser amount of water initially.

The tubes were then heated to 1000 $\pm$ 20F (argon pressure held at approximately 1030 mm Hg) and allowed to come to equilibrium for a minimum of 96 hr. The water content and residual gas analyses determined at 1000F are given in Table 4.

Based upon thermogravimetric analyses conducted on the raw materials which comprise Formulation 2002, and typical formulations (1), it was apparent that the water which was detected at 1000F constituted chemically combined water rather than adsorbed moisture.

TABLE 4.

WATER CONTENT AND RESIDUAL GAS ANALYSIS AT 1000F

	<u>33% RH</u>	<u>90% RH</u>	<u>Preconditioned at 1050F</u>	<u>Empty</u>
N ₂ , ppm	120	160	330	250
O ₂ , ppm	ND	ND	ND	ND
CO ₂ , ppm	610	750	590	320
CO, ppm	60	22	ND	ND
H ₂ , ppm	ND	330	ND	ND
CH ₄ , ppm	ND	6	ND	ND
C ₂ H ₄ , ppm	ND	15	ND	ND
H ₂ O, ppm	>1000	>2000	150	225
H ₂ O, % wt (STP)	>0.0015	>0.0030	0.00023	0.00034

Each tube was again pumped down, allowed to outgas, and backfilled with argon for a total of three cycles at 1050F. The times were changed, however, to 30 min for the first pumpdown, and 5 min for each subsequent outgas and pumpdown to coincide with current generator procedures at 1050F hot face. The tubes were then heated to 1400 $\pm$ 20F at 1030 mm Hg argon and held for 96 hr. The water content and residual gas analyses at 1400F are given in Table 5.

TABLE 5.

WATER CONTENT AND RESIDUAL GAS ANALYSIS AT 1400F

	<u>33% RH</u>	<u>90% RH</u>	<u>Preconditioned at 1050F</u>	<u>Empty</u>
N ₂ , ppm	530	260	400	260
O ₂ , ppm	ND	ND	6	ND
CO ₂ , ppm	1100	1750	500	ND
CO, ppm	480	550	820	ND
H ₂ , ppm	3600	10,000	10,000	560
CH ₄ , ppm	33	120	140	ND
C ₂ H ₆ , ppm	7	22	7	ND
H ₂ O, ppm	>2000	>2000	>2000	< 1
H ₂ O, % wt (STP)	>0.002	>0.002	>0.002	nil

It was apparent that the effect of preconditioning of the specimens no longer was significant. All three specimens showed similar levels of outgassing as compared with the empty tube.

The tubes could not be placed on extended test at 1400F since the water contents were already above the limit of the measuring device. Consequently, the three tubes containing the specimens were again pumped, outgassed, and backfilled three times. The pumpdown and outgassing times were 5 min each. Water and residual gas analysis results after 96 hr at 1030 mm Hg are shown in Table 6.

TABLE 6.

SECOND WATER CONTENT AND RESIDUAL GAS ANALYSIS AT 1400F

	<u>33% RH</u>	<u>90% RH</u>	<u>Preconditioned at 1050F</u>	<u>Empty</u>
N ₂ , ppm	150	260	450	450
O ₂ , ppm	ND	ND	ND	ND
CO ₂ , ppm	120	ND	ND	ND
CO, ppm	75	75	ND	ND
H ₂ , ppm	1000	12,000	11,400	790
CH ₄ , ppm	15	80	71	ND
C ₂ H ₆ , ppm	ND	ND	ND	ND
H ₂ O, ppm	>1000, <2000	270	4	<1
H ₂ O, % wt (STP)	>0.001, <0.002	0.0005	nil	nil

Although one specimen continued to show a relatively high level of water content, the system was placed on long term status. Water contents and residual gas analyses will be run at 500-hr intervals.

The reason for the high hydrogen content in the last two analyses at 1400F cannot be verified. It would appear that it was coming from the Formulation 2002 specimens since the empty tube registered the lowest amount in each case. It has been speculated, however, the hydrogen could come from three sources: diffusion through the ceramic tube, the INCONEL radiation shields within the tubes, and disassociation of hydrogen from silanol groups in the silica.

During the purchase of ceramic tubes, some were glazed on the outer surface, and some were not glazed. An evaluation of these tubes at elevated temperatures indicated no relationship between the glazing and the tube's ability to hold a vacuum within the required limits (5 μ /hr pressure rise at 1.44 initial pressure). Consequently, both types of tubes were used as the need arose. By coincidence, the empty tube in this series of four tubes was glazed, whereas the other three were not glazed. This may retard the diffusion of hydrogen into the tube. However, it is not likely that the concentration within the tube would exceed that of the air in the room solely by diffusion.

INCONEL is known to give off hydrogen when heated. This may account for some of the hydrogen in the empty tube, but would not account for the differing amounts in the various tubes.

There remains then the possibility that hydrogen is disassociating from the silanol or SiOH groups and combining to form the hydrogen gas. A search of the literature would be required to verify that this phenomenon can take place.

The data on the water content points out a basic, well-known characteristic of silica; the surface of silica particles tend to hydrate, or chemically combine with OH radicals whenever possible. Since the MIN-K products have silica surface areas approaching 200 m^2 /gm, a large amount of hydration must be anticipated (as measured in terms of parts per million in the residual gas). It is apparent that even the present preconditioning of the MIN-K products leaves some hydrated water behind, which eventually outgasses within that portion of the insulation that exceeds the preconditioning temperature of 1050F. Since the tests described above are conducted isothermally, the outgassing condition has been magnified.

The results to date suggest an interesting follow-on effort which would determine not only the amount of water required to obtain a measurable degree of degradation within a thermoelectric module, but which also would establish the assembly, and low temperature outgassing and backfill conditions necessary to stay below the critical water content within the generator. For example, it may be possible to change the current assembly conditions (glove boxes containing dry argon) to humidity controlled benchtop conditions, isothermal heating of the generator to 400F under vacuum, and a subsequent evacuation and argon backfill under ΔT heating conditions. Benchtop assembly conditions would undoubtedly reduce the time and cost of a generator assembly.

The presence of a small amount of getter material within the generator still appears desirable.

Dynamic Properties of Formulation 2002

The dynamic modulus of elasticity (E), the structural damping (G), and the first natural (resonant) frequency of Formulation 2002 was determined. A complete description of the test procedure, data, calculations, and results may be found in Martin Marietta Corporation Reports MND 3900-11 and MND 3900-12 presented in Appendix B. To summarize, six specimens, 2.5-in. in diameter and 2-in. thick were subjected to ten static preloads ranging from approximately 13 psi to approximately 114 psi. Data were obtained by striking the top lead weight (used to impart the static preload) with a small mallet in the direction parallel to the thickness dimension (normal to the layering plane) of the specimen, and recording the accelerometer output on an oscillograph. This procedure was followed for each increasing increment of preload. Data for one specimen were also obtained under decreasing load conditions.

The effect of static preload on the frequency of first resonance is plotted in Figure 4. Figure 5 represents the change in calculated dynamic modulus of elasticity with static preload. The method of least squares for fitting a curve of the form $y = a + bx$ was used to arrive at the equation:

$$E = 1431 + (9.79) (\text{preload, psi})$$

The first three preload conditions were not used in the calculation of the above equation since this region appeared to be non-linear. A similarity appears to exist between this region and the one encountered in the static stress-strain data⁽²⁾ below 35 psi. This non-linearity may be caused by the expansion of the product normal to the laminar plane immediately after molding. It is believed that this expansion is concentrated near the top and bottom surfaces of the product, resulting in a low apparent compression resistance at these low stress levels. It should be noted, however, that the average dynamic modulus differed only slightly from the static modulus as shown in Table 7.

TABLE 7.

AVERAGE DYNAMIC PROPERTIES

Dynamic modulus, psi	-	2100
Static modulus, psi ⁽²⁾	-	2000
Structural damping coefficient, %	-	0.5

FIGURE 4.

EFFECT OF STATIC PRELOAD ON FREQUENCY OF FIRST RESPONSE
OF FORMULATION 2002

FIGURE 5.

CHANGE IN DYNAMIC MODULUS OF ELASTICITY
WITH STATIC PRELOAD FOR FORMULATION 2002

Also shown in Table 7 is the average structural damping coefficient of 0.5 per cent. Figure 6 shows the slight, but consistent, change in damping coefficient with static preload. The slope is small enough, however, to be considered zero for all practical applications.

It is interesting to note that the damping coefficient is approximately half that measured for MIN-K 1301; Martin Report⁽³⁾. This was probably caused by the 1400F, 2 hr firing cycle to which Formulation 2002 is subjected during manufacture. Had MIN-K 1301 been subjected to 1400F prior to, or during the test, it is believed that its damping coefficient would also have been lower due to sintering or similar phenomenon which occurs within these insulations at temperatures above 1000F.

Thermal Stability of Formulation 2002

The per cent shrinkage of Formulation 2002 after 2000 hr of 1400F isothermal exposure was presented previously⁽²⁾. Table 8 shows the effect of exposing the same specimens for an additional 100 hr at 1500F and 48 hr at 1600F. Four of the original twelve specimens were then exposed to 1600F for a total of 1000 hr. These shrinkage data are also shown in Table 8.

TABLE 8.

THERMAL STABILITY OF FORMULATION 2002*

	2000 hr @ 1400F	+	100 hr @ 1500F	+	48 hr @ 1600F	+	475 hr @ 1600F	+	1000 hr @ 1600F
Length shrinkage, %	0.4		0.4		0.5		0.6		0.6
Width shrinkage, %	0.8		0.9		0.9		0.6		0.7
Thickness shrinkage, %	0.8		0.8		0.9		1.2		1.4

*All values based on dimensions prior to any thermal exposure. Values are not cumulative.

The apparent "growth" of the specimens in the width direction during the 475 hr exposure to 1600F can probably be attributed to the accuracy of measurement, coupled with the effect of frequent thermal shock, upon the specimens as they were periodically heated and cooled. Nonetheless, it would appear that Formulation 2002 has a useful service temperature above the 1400F limit for which it was designed.

FIGURE 6.

VARIATION OF DAMPING COEFFICIENT WITH
STATIC PRELOAD OF FORMULATION 2002

To establish more meaningful shrinkage data under conditions of thermal gradient, two 4 x 4 x 1-inch specimens of Formulation 2002 were inserted into the load relaxation test units, but with less than 1 psi load applied. The specimens were heated to 1400F hot face for 500 hr, removed, measured, re-inserted, and heated to 1600F hot face for an additional 425 hr. Table 9 below summarizes the data. Note that the "shrinkage" is presented in terms of dimensional change, rather than per cent shrinkage as has been done previously. The estimated precision of measurement is ± 0.002 in.

TABLE 9.

DIMENSIONAL CHANGE OF FORMULATION 2002
AFTER HOT FACE EXPOSURE

Specimen No.	Average Hot Face OF	Average Cold Face OF	Exposure Time hr	Total Dimensional Change, in.		
				Length	Width	Thickness
1	1398	283	500	nc*	-0.001	+0.002
2	1403	274	500	-0.002	-0.002	-0.004
1	1598	671**	425	-0.004	-0.005	-0.002
2	1597	666**	425	-0.008	-0.005	-0.005

* No change

** 1/2-in. MICRO-QUARTZ placed over cold face of specimen to increase mean temperature.

It can be seen that at 1400F hot face, no length and width shrinkage could be detected within the limits of measuring accuracy. Similarly, the measured thickness shrinkage was considerably below that for the isothermal exposure (0.001 in. represents approximately 0.1 per cent of the original thickness). At 1400F hot face, only one of the specimens showed what might be construed as "significant shrinkage," although even then, it was well below the unofficial goal of 1 per cent.

The 1600F hot face tests were conducted such that both the hot face and the cold face (and hence the mean temperature) exceeded normal operating limits. Despite these exaggerated conditions, the shrinkage remained below 1 per cent. The result of isothermal versus hot face stability testing (comparing Table 8 with Table 9) was most evident in the thickness dimension as anticipated, with the hot face tests representing the more realistic stability values. All dimensions represent changes from the original dimensions prior to any hot face exposure.

Moisture Absorption/Desorption

The initial moisture adsorption test results for Formulation 2002 at 22% RH were reported previously⁽²⁾. (The % RH was erroneously reported as 27.) These data are reported again together with the balance of the test results.

The test specimens were 4 x 4 x 1/4-in., 1/2-in., and 1-in. thick blocks of Formulation 2002. However, due to the high degree of similarity between Formulation 2002, MIN-K 2000, and other Formulations using CAB-O-SIL M5 (not including MIN-K 1301), these data are considered applicable to these materials as well. MIN-K 1301, having a different silica base material, may differ slightly from Formulation 2002.

The moisture absorption tests were conducted in a sealed chamber into which the desired humidities were achieved through the use of the following super-saturated salt solutions:

22% RH - CH_3COOK

51% RH - $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$

91% RH - $(\text{CH}_3\text{COO})_2\text{Ca}$

These solutions, contained in a small dish were placed in the chamber, and a blower was provided to circulate the air within the chamber. The humidity was measured by a humidistat using the proper sensing elements for each humidity range, and a recording humidistat for record keeping purposes. A chainomatic balance was mounted directly above the humidity chamber. The sample holder within the chamber was suspended by a fine diameter wire from the balance arm through a small hole in the chamber. Access to the chamber for the insertion of the samples was provided by a hole fitted with a plastic sleeve, each end of which (one end inside the chamber, and one end outside the chamber) could be sealed. To insert the dry (at 230F to constant weight) sample into the chamber, the operator's arm was placed into the sleeve and sealed around the upper portion of the arm. The seal inside the chamber was then opened, permitting the operator to place the dry specimen on the balance pan and to initiate weighings.

The desorption tests were run by placing the same balance above a drying oven (operating at 230F), and suspending the balance pan into the oven. There was

no air circulation within the oven, thus providing not only reproducible conditions, but also the slowest drying conditions. The specimens were preconditioned in their respective relative humidity conditions until an equilibrium weight was attained.

The results of the moisture adsorption tests are plotted in Figures 7, 8, and 9 for 22% RH, 51% RH, and 91% RH, respectively. The curves do not necessarily include the equilibrium points, since the major significance of the curves occurs in the early stages of adsorption. They clearly show the rapidity with which dry MIN-K materials adsorb moisture from the air.

Similarly, Figures 10, 11, and 12 represent the moisture desorption or drying rates. In all cases, the per cent weight change was based upon the initial weight of the sample. Thus, on a per cent basis, the moisture pickup during adsorption may appear to be greater than the amount of moisture driven off at 230F during desorption.

Prior to the desorption tests, all specimens were placed in a desiccator containing the proper salt solution until equilibrium was attained. In two instances, 91% RH equilibrium appeared difficult to achieve in the 1/4-in. and 1-in. thick specimens. Consequently, these two specimens were placed in 100% RH conditions for 3 and 5 days (1/4-in. and 1-in., respectively), and subsequently returned to the 91% RH condition. Equilibrium was subsequently achieved in 5 and 6 days, respectively. Table 10 summarizes the equilibrium moisture contents for each thickness and at each humidity level.

Load Relaxation Characteristics of Formulation 2002

The load relaxation evaluation of Formulation 2002 at 1400F hot face has been completed at the 50 psi, 100 psi, and 150 psi initial stress levels. As predicted in Figure 7 of the Third Quarterly Report, the stress on the specimens after 2000 hr was 45 to 55 per cent of the initial stress. No apparent relationship between initial stress, and fraction of initial stress after 2000 hr could be detected, possibly because of the slight compressibility factor of the furnaces themselves. The spread of all the data points is encompassed by the crosshatched band in Figure 13.

Also shown in Figure 13 are the load relaxation characteristics of Formulation 2002 at room temperature and at 100 and 150 psi initial stress levels. For these tests, the furnace was removed from the test fixture, thus eliminating the possible error due to furnace compression. Although

FIGURE 7.

MOISTURE ADSORPTION OF FORMULATION 2002 IN 22% RH

FIGURE 8.

MOISTURE ADSORPTION OF FORMULATION 2002 IN 51% RH

FIGURE 9.

MOISTURE ADSORPTION OF FORMULATION 2002 IN 91% RH

FIGURE 10.

MOISTURE DESORPTION OF FORMULATION 2002
AFTER CONDITIONING AT 22% RH

FIGURE 11.

MOISTURE DESORPTION OF FORMULATION 2002
AFTER CONDITIONING AT 51% RH

FIGURE 12.

MOISTURE DESORPTION OF FORMULATION 2002
AFTER CONDITIONING AT 91% RH

TABLE 10.
EQUILIBRIUM MOISTURE CONTENT OF FORMULATION 2002 AT
VARIOUS HUMIDITY LEVELS

Sample Thickness, in.	Relative Humidity, %	Time to Reach Equilibrium, days	Equilibrium Moisture Content, %
1/4	22	4.0	0.60
1/2	22	7.5	0.64
1	22	1.0	0.69
1/4	51	5.0	1.53
1/2	51	7.0	1.52
1	51	7.5	1.48
1/4	91	---	7.1*
1/2	91	19.0	7.3
1	91	---	6.8*

* These values were obtained by subjecting the specimens to 100% RH and subsequent exposure to 91% RH for 5 and 6 days for the 1/4-in. and 1-in. specimens, respectively. Values obtained in the conventional manner were:

1/4-in. - 5.8 per cent after 11.0 days

1-in. - 6.0 per cent after 13.0 days.

FIGURE 13.

LOAD RELAXATION OF FORMULATION 2002

only two specimens were tested in this manner at each stress level, one for 2000 hr and one for a minimum of 1000 hr, the data points were very reproducible and permitted a distinction between the two stress levels.

The effect of loading on the thickness shrinkage under temperature gradient conditions is shown in Table 11. This effect is one which must be anticipated for this type of product.

TABLE 11.

EFFECT OF LOAD ON THICKNESS SHRINKAGE OF
FORMULATION 2002 AT 1400F HOT FACE

<u>Initial Load, psi</u>	<u>Thickness Shrinkage, %</u>
<1	0.4*
50	1.9
100	2.9
150	4.6

* Per Table 9.

Thermal Conductivity of Formulations 1999 and 2002

Figure 14 is a plot of thermal conductivity versus mean temperature for Formulations 1999 and 2002 as tested in air. Formulation 1999 was tested on the ASTM Guarded Hot Plate Apparatus, whereas Formulation 2002 was tested on the Dyna Tech Guarded Hot Plate and Consolidated Vacuum Apparatus purchased by Johns-Manville specifically for use under this contract. The latter equipment will also evaluate these materials under vacuum and inert gas conditions.

It was not anticipated that the two materials would have different conductivity values. Past experience has shown differences of up to 5 per cent between different apparatus using the same test specimen. Therefore, no conclusions can be drawn as yet between these formulations until each is tested in the other unit.

For comparison purposes, published values for MIN-K 1301 in air have also been plotted in Figure 14.

FIGURE 14.

THERMAL CONDUCTIVITY IN AIR OF FORMULATIONS 1999 AND 2002

Comparing Formulation 2002 and MIN-K 1301 at 850F mean (typical mean temperature for bulk insulation - 1400 hot face, 300 cold face), Formulation 2002 represents a 10 per cent improvement. Similarly, if the measured difference between the two formulations is significant, Formulation 1999 represents a 14 per cent improvement at 650F mean (typical module mean temperature - 1000F hot face, 300F cold face). It is questionable at this point, however, if an increase in efficiency of more than 10 per cent could be anticipated.

Shear Strength of Formulation 2002

To confirm the relative weakness of Formulation 2002 in shear parallel to its laminar plane, 12 specimens were subjected to shear at room temperature. The test utilized 1 x 2 x 3/4-in. specimens which were placed on their 2 x 3/4-in. edge within a confining fixture. The fixture was so constructed as to prevent any movement of half of the projected area, or 2 x 3/8-in., while the remaining half was subjected to a guillotine action over its entire area at a loading rate of 50 lb/min (or approximately 37.5 psi/min).

An average value of 22.1 psi was obtained for the 12 specimens, with an approximate spread of +25 per cent for the individual values. Previous work on MIN-K 1301 containing binder resulted in an average of 44 psi, with a higher spread of values. Although no data exist for a direct comparison, it would be anticipated that MIN-K 1301 with the binder removed would have a shear strength approximating that of Formulation 2002. Thus, as expected, Formulation 2002 does not represent a significant change in shear strength from the current MIN-K 1301 material, and hence, should not be required to sustain excessive shear forces during service.

B. SILICON-GERMANIUM INSULATIONS

Chemical Compatibility

The chemical compatibility between N and P silicon-germanium elements and the four fibrous insulation candidates (Irish REFRASIL, MICRO-QUARTZ, ASTROQUARTZ, and DYNAQUARTZ) was determined by embedding a series of cylindrical elements (0.444 in. diameter by 0.271 in. long) into each insulation and subjecting them to 1600F isothermal conditions in air and vacuum (less than 2 microns). Periodically, several of the elements were removed from the insulation, and approximately 5 mg removed uniformly from the cylindrical surface of each element. The powder thus obtained was then analyzed spectrographically for impurities. Since the procedure specifically evaluated the surface contamination, the analysis was qualitative only.

Of the 55 pairs of elements used in the test, 20 were analyzed prior to test by the same technique to determine not only what impurities existed in the elements as a result of manufacture, but also to determine if the sampling procedure introduced impurities. No impurities were found in any of these elements.

The results shown in Tables 12 and 13 are presented in qualitative terms since the tested material was not representative of the entire mass of the specimens. These terms were:

ND	- not detected	- $<0.001\%$
t.	- trace	- $<0.01\%$
v.s.	- very small	- $<0.1\%$
s.	- small	- $<1.0\%$

One N and one P element were analyzed following the 500, 1000, and 1500 hr exposure levels, while three N and three P elements were analyzed after the 2000 hr level. Although the 1000 hr level for Irish REFRASIL and MICRO-QUARTZ, and the 1500 hr level for ASTROQUARTZ and DYNAQUARTZ in vacuum have been completed, the spectrographic results were not available for this report.

None of the results indicated what might be considered significant contamination at the surface of the elements, and thus it would appear that none of the fibrous candidates would adversely affect the performance of the SiGe elements due to migration of chemical elements from the insulation into the SiGe elements. This tentative conclusion may be altered if subsequent results of the vacuum tests do not follow the current trend.

Attempts were made to determine the source of copper as a contaminant in the above analyses, and if the fibrous insulations were picking up chemical elements from the SiGe specimens after the 2000 hr exposure level in air. With as much care as possible, fiber samples were taken from that portion of the mass which was in contact with the 2000 hr N and P elements, as well as from a section which was not in contact with the elements. Copper from the boring tool used to cut the holes in the fiber mass was the prime suspect. Table 14 summarizes the qualitative results.

The results in Table 14 showed that the boring tool could be suspect. However, the small amounts found in the SiGe elements, together with the

TABLE 12.

SiGe COMPATIBILITY IN AIR AT 1600F

Fibrous Product	Impurity in SiGe Elements									
	Chromium		Copper		Iron		Molybdenum		Nickel	
	N	P	N	P	N	P	N	P	N	P
Irish REFRASIL										
500 hr	v.s.	v.s.	t.	t.	t.	t.	ND	ND	ND	ND
1000 hr	t.	t.	t.	t.	t.	t.	ND	ND	ND	ND
1500 hr	t.	t.	t.	t.	t.	t.	ND	ND	ND	ND
2000 hr	t.	t.	t.	ND	t.	ND	ND	ND	ND	ND
	t.	t.	v.s.	t.	s.	s.	ND	ND	ND	ND
	t.	t.	v.s.	ND	t.	ND	ND	ND	ND	ND
MICRO-QUARTZ										
500 hr	ND	ND	t.	t.	ND	ND	ND	ND	ND	ND
1000 hr	ND	ND	t.	t.	ND	ND	ND	t.	ND	ND
1500 hr	ND	ND	t.	t.	v.s.	ND	ND	ND	t.	ND
2000 hr	ND	ND	v.s.	s.	ND	ND	ND	ND	ND	ND
	ND	ND	v.s.	t.	ND	ND	ND	ND	ND	ND
	ND	ND	v.s.	v.s.	ND	ND	ND	ND	ND	ND
ASTROQUARTZ										
500 hr	ND	ND	t.	s.	ND	ND	ND	ND	ND	ND
1000 hr	ND	ND	t.	v.s.	t.	t.	ND	ND	ND	ND
1500 hr	ND	ND	t.	v.s.	t.	ND	ND	ND	ND	t.
2000 hr	ND	ND	v.s.	s.	ND	ND	ND	ND	ND	ND
	ND	ND	v.s.	s.	t.	ND	ND	ND	ND	ND
	ND	ND	v.s.	s.	ND	ND	ND	ND	ND	ND
DYNAQUARTZ										
500 hr	ND	ND	t.	s.	ND	ND	ND	ND	ND	ND
1000 hr	ND	ND	t.	v.s.	t.	ND	ND	ND	ND	ND
1500 hr	ND	ND	v.s.	t.	t.	t.	ND	ND	ND	ND
2000 hr	ND	ND	v.s.	v.s.	t.	ND	ND	ND	ND	ND
	ND	ND	v.s.	v.s.	ND	t.	ND	ND	ND	ND
	ND	ND	v.s.	v.s.	v.s.	t.	ND	t.	ND	ND

ND - not detected - <0.001%
 t. - trace - <0.01%
 v.s. - very small - <0.1%
 s. - small - <1.0%

TABLE 13.

SiGe COMPATIBILITY IN VACUUM AT 1600F

Fibrous Product	Impurity in SiGe Elements							
	Chromium		Copper		Iron		Molybdenum	
	N	P	N	P	N	P	N	P
Irish REFRA-SIL 500 hr	v.s.	v.s.	t.	t.	t.	t.	ND	ND
MICRO-QUARTZ 300 hr	ND	ND	v.s.	t.	ND	t.	ND	ND
ASTROQUARTZ 500 hr	ND	ND	v.s.	t.	ND	ND	ND	ND
1000 hr	ND	ND	v.s.	t.	t.	t.	ND	t.
DYNAQUARTZ 500 hr	ND	ND	v.s.	v.s.	ND	ND	ND	ND
1000 hr	ND	ND	v.s.	t.	t.	ND	ND	ND

ND - not detected - <0.001%
 t. - trace - <0.01%
 v.s. - very small - <0.1%
 s. - small - <1.0%

fact that copper is a common contaminant in spectrographic analyses indicated that no further investigation was warranted. The analyses of the fibers also indicated the presence of no unusual impurities which could be ascribed to the SiGe elements.

Similar chemical compatibility tests will be initiated on the formulation developed for 1600F service (see Material and Process Development).

TABLE 14.

COPPER CONTENT BY SPECTROGRAPHIC ANALYSIS OF THE
FIBROUS INSULATIONS - AFTER 2000 HR AT 1600F IN AIR

	<u>Adjacent to N</u>	<u>Adjacent to P</u>	<u>No Element Contact</u>
Irish REFRASIL	†.**	†.	ND*
MICRO-QUARTZ	†.	†.	†.
ASTROQUARTZ	†.	†.	ND
DYNAQUARTZ	ND	†.	ND

* Not detected, <0.001%

** Trace, <0.01%

Seebeck Voltage and Resistivity Compatibility

Tests were initiated this quarter to evaluate the compatibility between the SiGe elements and Irish REFRASIL as measured by the change in Seebeck voltage and the room temperature resistivity. The test utilized matching pairs of SiGe materials fabricated into bars 0.2 x 0.2 x 1.75-in. long. One bar of each pair was placed in intimate contact with the Irish REFRASIL, while the matching bar was embedded in DYNAQUARTZ as a standard. This procedure not only yielded a value against which the test bar (in the Irish REFRASIL) could be compared, but also eliminated the variable resulting from time-temperature history of the bar as it passed through its critical temperature during heatup and cooldown.

DYNAQUARTZ was chosen as the insulation for the standard bar because of its history of long term compatibility, while the test insulations were limited to Irish REFRASIL since it was the only fibrous insulation which differed significantly from the pure silica fibers currently used in SiGe modules.

Seebeck voltage and room temperature resistivity measurements were made by RIA, Harrison, New Jersey, prior to exposure. These measurements will be repeated after 1000 and 2000 hr exposure to Irish REFRA-SIL at 1600F in air and in vacuum. The test will be repeated using the MIN-K type formulation developed for 1800F service.

Thermal Conductivity

The thermal conductivity of the fibrous candidates (in air) are currently in progress using the ASTM C-177 Guarded Hot Plate Apparatus. Although data for these products are generally available from their manufacturers, the fibers are used at considerably higher-than-normal densities within the SiGe thermoelectric modules. Consequently, tests on Irish REFRA-SIL, MICRO-QUARTZ, and ASTROQUARTZ at 10 pcf have been scheduled. Data for DYNAQUARTZ at 10 pcf in air has been developed by Johns-Manville as part of their continuing product performance program.

Figure 15 shows the thermal conductivity versus mean temperature relationship for Irish REFRA-SIL at 10.3 pcf, and DYNAQUARTZ at 10.0 pcf. The ASTM C-177 equipment cannot exceed 1100F mean temperature, although additional data at higher mean temperatures will be obtained when the new high temperature, vacuum-air-inert gas unit completes the MIN-K formulation evaluations. The DYNAQUARTZ data were obtained using a high temperature calorimeter unit. Although this unit is not standard with the industry or ASTM with respect to this type of insulation (specified for use with insulating firebrick and refractories per ASTM C-182 and ASTM C-201, respectively), experience has proven it to be reliable for materials having lower thermal conductivities such as DYNAQUARTZ.

FIGURE 15.

THERMAL CONDUCTIVITY IN AIR OF IRISH REFRASIL AT 10.3 PCF
AND DYNAQUARTZ AT 10.0 PCF

IV. MATERIAL AND PROCESS DEVELOPMENT

Table 15 lists the formulations investigated during the fourth quarter leading to the development of an 1800F insulation. The Third Quarterly Progress Report stated that the prime area of concentration was in the upgrading of the silica material either through the use of a more stable product, or by prefiring the present silica (CAB-O-SIL N3) to some elevated temperature such as 1850F. Since no new material was found which had improved stability at 1800F, efforts were then switched to the latter technique.

As previously reported⁽²⁾, prefiring of the CAB-O-SIL N3 definitely damaged the binder-free molding characteristics of the silica in Formulations 2007 through 2010. Formulation 2012 containing AEROSIL uncompresssed also had poorer molding characteristics than the unfired silica formulations, although not as poor as Formulations 2007 through 2010. This was probably due to the lower firing temperature (1820F vs 1850F).

In order to properly assess the effect of prefiring the silicas on the thermal stability of the molded products, binder was added to Formulations 2013 through 2019. Additionally, Formulations 2017 and 2018 incorporated prefired MICRO-QUARTZ, which in one instance was opened through an aeration chamber called an Entolator prior to its prefiring cycle.

During the isothermal shrinkage evaluations, problems arose with the temperature distribution within the test furnace, as well as the relationship between the specimen temperature and the recorded control temperature. Consequently, subsequent efforts included the instrumentation of some, if not all, of the specimens with exposed thermocouples. In those cases where the specimen was not instrumented, it was placed between two specimens that were instrumented.

Table 16 summarizes the shrinkage data obtained on 4 x 2 x 1-in. specimens at 1600F and 1800F. Formulations 1999 and 2002 were included in this series for comparison purposes. The indicated temperatures represent the maximum temperature achieved as a result of the cyclic fluctuations of the furnace. This temperature never varied more than 5 degrees from cycle to cycle. All values are based upon the original dimensions of the specimen.

One of the most obvious effects on shrinkage was the MICRO-QUARTZ content. Thus, Formulations 2010, 2011, and 2012 which contained 8 per cent MICRO-QUARTZ,

TABLE 15.
FOURTH QUARTER EXPERIMENTAL MIN-K FORMULATIONS⁽¹⁾

No. (2)	Silica		Fiber				TAM - 5 TiO ₂	BRP 5095 Binder	Comments
	CAB-O-SIL M5	AEROSIL Uncompr.	MICRO- QUARTZ	ASTRO- QUARTZ- 1/2"	ASTRO- QUARTZ 1/4"	DYNA- QUARTZ			
2007	75 ⁽³⁾	---	4	5	---	---	16	0	---
2008	75 ⁽³⁾	---	4	5	---	---	16	0	---
2009	75 ⁽⁴⁾	---	4	5	---	---	16	0	---
2010	75 ⁽⁴⁾	---	8	---	1	---	16	0	CAB-O-SIL M5 premilled in Henschel 5 min.
2011	73 ⁽³⁾	---	8	---	1	---	16	2	---
2012	---	75 ⁽⁵⁾	8	---	1	---	16	0	AEROSIL premilled in Henschel 5 min.
2013	---	73 ⁽⁵⁾	8	---	1	---	16	2	AEROSIL premilled in Henschel 5 min.
2014	---	73 ⁽⁵⁾	4	5	---	---	16	2	AEROSIL premilled in Henschel 5 min.
2015	---	73 ⁽⁴⁾	4	5	---	---	16	2	AEROSIL premilled in Henschel 5 min.
2016	73 ⁽⁴⁾	---	4	5	---	---	16	2	CAB-O-SIL premilled in Henschel 5 min.
2017	73 ⁽⁴⁾	---	4 ⁽⁶⁾	5	---	---	16	2	CAB-O-SIL premilled in Henschel 5 min.
2018	73 ⁽⁴⁾	---	4 ⁽⁶⁾	5	---	---	16	2	CAB-O-SIL premilled in Henschel 5 min; MICRO-QUARTZ passed through Entolator be- fore prefiring.
2019	80	---	---	---	---	---	17	3	---
2020	75	---	4 ⁽⁶⁾	5	---	---	16	0	---
2021	79	---	---	5	---	---	16	0	---
2022	75	---	---	5	4	---	16	0	---
2023	75	---	4 ⁽⁷⁾	5	---	---	16	0	---
2024	75	---	4 ⁽⁸⁾	5	---	---	16	0	---
2025	75	---	---	5	---	4	16	0	---

(1) All numbers represent per cent by weight.

(2) Formulations 2007 through 2013 published in Third Quarterly Report - repeated here for continuity.

(3) Prefired 2 hr at 1850F.

(4) Prefired to 1850F.

(5) Prefired to 1820F.

(6) Prefired 2 hr at 1800F.

(7) Prefired 2 hr at 1850F.

(8) Prefired 2 hr at 1900F.

TABLE 16.

THERMAL STABILITY OF EXPERIMENTAL FORMULATIONS 2010 - 2018

No.	Maximum Temp, °F	Shrinkage after 24 hr @ 1600F			Maximum Temp, °F	Shrinkage after 24 hr @ 1600F + 24 hr @ 1800F		
		Length	Width	Thickness		Length	Width	Thickness
1999	1596	1.5	1.5	1.1	1804	2.0	2.0	2.6
2002	--	0.5	0.3	0.4	--	0.7	0.6	2.3
2010	1605	1.2	1.2	0.0	1794	1.6	1.6	1.6
2011	--	1.1	1.1	0.0	1813	1.5	1.5	1.3
2012	1618	1.1	1.1	0.7	--	1.6	1.6	3.5
2014	--	0.4	0.3	0.6	--	0.6	0.5	2.4
2015	1591	0.4	0.4	0.3	1799	0.7	0.6	2.2
2016	1578	0.4	0.4	0.5	1804	0.5	0.6	1.7
2017	1605	0.0	0.0	0.3	1818	0.1	0.2	1.6
2018	--	0.0	0.0	0.4	--	0.2	0.2	1.7

and which represented 1999 type formulations, had the most severe length and width shrinkage. This relationship also held for the Formulation 1999 and 2002 specimens. It also appeared that the prefiring of the MICRO-QUARTZ was a significant factor when the length and width shrinkages of Formulations 2017 and 2018 were compared with those of Formulations 2014, 2015, and 2016.

The above results led to the next series of Formulations (2019 through 2025) which investigated the effect of the fibrous reinforcement on the shrinkage characteristics. Formulation 2019 contained no fiber, but was formulated such that the amount (by weight) of CAB-O-SIL M5 and opacifier in a 9 x 9 x 1-in. block was the same as that in a block containing fiber reinforcement. This formulation was to provide a basis for comparison. However, the unreinforced block was exceptionally brittle, which precluded handling. It was eliminated from further evaluations.

Formulations 2020, 2023, 2024, and 2025 evaluated the effect of the MICRO-QUARTZ prefiring temperature on the thermal stability. The extreme of the prefiring temperature was represented by the use of DYNAQUARTZ fiber in Formulation 2025. Formulations 2021 and 2022, employing only the relatively stable ASTROQUARTZ fibers, was to be used as a baseline comparison in place of Formulation 2019 above. However, both Formulations 2021 and 2022 resulted in poor moldings, having many hairline cracks at the edges, and a poor ceramic "ring."

It was apparent that the presence of the short, fine diameter MICRO-QUARTZ fibers was important to a sound molding. Because of their delamination tendencies, these formulations were not evaluated for thermal shrinkage since the thickness measurement would have been suspect.

Table 17 shows the isothermal shrinkage test results of Formulations 2020, 2023, 2024, and 2025, together with 1999 and 2002 specimens. Each specimen was instrumented as before to insure a valid comparison between results.

Prefiring the MICRO-QUARTZ had a significant effect on the length and width shrinkage as seen by comparing the Formulation 2002 shrinkage (0.8 avg) with that of the newer formulations (0.4 avg). The thickness shrinkage, however, remained in the relatively high range of 4 per cent.

TABLE 17.

ISOTHERMAL SHRINKAGE AT 1800F OF FORMULATIONS
2020, 2023, 2024, AND 2025

No.	Max. Temp Of	Shrinkage after 24 hr, %			Shrinkage after 70 hr, %		
		Length	Width	Thickness	Length	Width	Thickness
1999	1786	2.1	2.1	2.3	2.2	2.3	2.9
2002	1813	0.6	0.8	3.6	0.7	0.9	4.1
2020	1799	0.2	0.3	3.9	0.3	0.4	4.4
2023	1790	0.2	0.3	3.9	0.3	0.4	4.6
2024	1794	0.2	0.5	3.6	0.3	0.5	4.1
2025	1762	0.3	0.4	2.7	0.4	0.5	3.1

To evaluate further the effect of the prefiring temperature of the fiber, four modulus of rupture tests were run on each of the four experimental formulations. These results are given in Table 18 below.

TABLE 18.

MODULUS OF RUPTURE OF FORMULATIONS 2020, 2023, 2024, AND 2025

<u>Formulation No.</u>	<u>Modulus of Rupture, psi</u>
2020	82.9
2023	87.4
2024	61.6
2025	41.9

Thus, although Formulation 2025 may be the best from a thickness shrinkage standpoint, a very large sacrifice in strength would be necessary.

The choice of which formulation had the more desirable characteristics consisted of a series of trade-offs. Formulations 2017 and 2018 had the lowest shrinkage characteristics. This was achieved by prefiring both the CAB-O-SIL M5 (to 1850F) and the MICRO-QUARTZ (1800F, 2 hr). These formulations, however, required binder in order to make a satisfactory molding. Although the binder was, for all practical purposes, removed during the 2 hr, 1400F firing after molding (same procedure as followed for Formulations 1999 and 2002), traces of organic gases would probably desorb from the insulation under elevated temperatures and

high vacuum. From a practical standpoint, the cost of prefiring the CAB-O-SIL M5 would be quite high. It would be difficult to justify the much higher product cost in terms of only a few per cent improvement in thickness shrinkage under isothermal test conditions.

In contrast, Formulation 2020 (or 2023) could be molded without binder, and did not involve the prefiring of the CAB-O-SIL M5. The sacrifice, of course, was thickness shrinkage. However, a true picture of the thickness shrinkage requires a A/T exposure such as that conducted for Formulation 2002 (see Detailed Evaluation, Thermal Stability). Since the length and width shrinkage was satisfactory, indicating that the hot surface of the insulation would not have a tendency to "neck down," either of these formulations (2020 or 2023) would probably prove satisfactory under service conditions. Formulation 2020 was therefore chosen as the Formulation XXXX material for 1800F hot face service.

Based upon the research done to date with respect to the increased thermal stability of the MIN-K products, it appears that the service temperature limit will not be pushed above 1800F with the currently available materials, and the known processing techniques. However, potential areas of investigation into more stable materials appear to offer promise. These areas, which are beyond the scope of the current program plan, will probably yield a material with a trade-off of thermal conductivity for thermal stability. However, a 200 to 500F increase in temperature limit would probably warrant such a trade-off. Such a program will be recommended for Phase II.

V. SUMMARY OF FOURTH QUARTER RESULTS AND CONCLUSIONS

A. FINAL SCREENING

1. Carborundum T30LR laminate shrank 3.4 per cent in thickness after 168 hr at 1700F hot face and 200 psi compressive loading. No further work is scheduled for this product.
2. GLASROCK 25 had a modulus of rupture (bend strength) of 172 psi (average), and a shear strength of 210 psi.
3. The thermal conductivity of GLASROCK 25 was 0.8 and 1.4 Btu in./hr ft² at 73F and 800F mean temperatures, respectively, as measured by the Rapid Heat Meter Apparatus. This was higher than that published by the manufacturer, but relatively good when compared with products of similar compression characteristics.
4. GLASROCK 25 remains the most favorable candidate for load bearing applications.

B. DETAILED EVALUATION

1. A cursory examination of the PbTe Seebeck voltage and resistivity data indicated no significant change in resistivity for the 2N, 2P, and 3N elements, and no significant change in Seebeck voltage for the 3N, and 2P elements after 1500 hr of intimate exposure to Formulations 1999 and 2002 at 1000F. Further analysis of these data and the data following 2000 hr exposure is necessary before final conclusions can be drawn.
2. The amount of water outgassed by Formulation 2002 was relative to its initial humidity preconditioning up to 1000F. At 1400F, all specimens outgassed to the same degree. Conditions will now be held constant for extended periods to determine if additional outgassing occurs with time.
3. The average dynamic modulus of elasticity of Formulation 2002 was within 5 per cent of the static modulus (2100 psi vs 2000 psi). The calculated dynamic modulus increased, however, with increasing preload.
4. The structural damping of Formulation 2002 was 0.5 per cent.
5. The thermal shrinkage of Formulation 2002 after 1000 hr at 1600F isothermal conditions was 0.6, 0.7, and 1.4 per cent for length, width, and thickness, respectively.
6. Under ΔT conditions of 1600F hot face and 670F cold face, the dimensional change of 4 x 4 x 1-in. specimens of Formulation 2002 after 425 hr

averaged -0.005 in. This indicated that under A T heating, Formulation 2002 would be satisfactory above its design limit of 1400F hot face.

7. The average equilibrium moisture content of Formulation 2002 was 0.65, 1.51, and 7.1% for 22, 51, and 91% RH, respectively.
8. Formulation 2002 specimens supported 45 to 55 per cent of their initial loading after 2000 hr at 1400F hot face. This relationship was independent of the initial load (50, 100, or 150 psi).
9. Formulations 1999 and 2002 had thermal conductivities approximately 10 per cent lower than those published for MIN-K 1301 as determined by two Guarded Hot Plate Apparatuses.
10. The shear strength of Formulation 2002 parallel to the laminar direction averaged 22.1 psi.
11. No chemical incompatibility was evident between SiGe elements and Irish REFRASIL, MICRO-QUARTZ, ASTROQUARTZ, and DYNAQUARTZ after 2000 hr in air at 1600F. Similarly, no incompatibility was evident after 500 hr in vacuum at 1600F with Irish REFRASIL and MICRO-QUARTZ, and after 1000 hr in vacuum at 1600F with ASTROQUARTZ and DYNAQUARTZ.
12. Irish REFRASIL (10 pcf) and DYNAQUARTZ (10 pcf) had similar thermal conductivities of 0.65 Btu in./hr ft² OF at 950F mean.

C. MATERIAL AND PROCESS DEVELOPMENT

1. AEROSIL uncompressed showed no greater thermal stability than CAB-O-SIL M5.
2. Formulations containing prefired CAB-O-SIL M5 required binder to obtain a sound molding, although the resultant product was more stable than those using unfired CAB-O-SIL M5.
3. Prefiring the MICRO-QUARTZ reduced the shrinkage of the products.
4. A significant loss in strength was noted in Formulations containing MICRO-QUARTZ which was prefired above 1850F.
5. Formulation 2020 was chosen as the material for 1800F service after consideration of several trade-off factors.

VI. FIFTH QUARTER EFFORT

1. Complete Formulation 2002 outgassing evaluation.
2. Continue thermal conductivity tests of Formulations 1999 and 2002.
3. Conduct dynamic verification tests (vibration) on Formulation 2002.
4. Complete chemical compatibility tests between SiGe elements and the candidate fibrous insulations in vacuum.
5. Continue Seebeck voltage and resistivity compatibility tests between SiGe bars and Irish REFRASIL.
6. Initiate long term testing (thermal stability, chemical, and Seebeck voltage compatibility, and load relaxation) of Formulation 2020.
7. Conduct machining and dynamic response evaluations of Formulation 2020.
8. Continue preparation of Phase II follow-on program.

VII. REFERENCES

1. Develop 1800F - 400F Fibrous Type Insulation for Radioisotope Power Systems, First Quarterly Progress Report, Report No. ALO-3633-1, July 1, 1966, under AEC Contract AT(30-1)-3633.
2. Develop 1800F - 400F Fibrous Type Insulation for Radioisotope Power Systems, Third Quarterly Progress Report, Report No. ALO-3633-3, January 3, 1967, under AEC Contract AT(30-1)-3633.
3. Structural Evaluation of MIN-K 1301, Report No. MND-3169-45, January 1965, under AEC Contract AT(30-1)-3169.

VIII. APPENDIX A.

**OUTGASSING OF MIN-K FORMULATIONS 1999 AND
2002 IN ARGON AT 1 ATMOSPHERE**
Revision D dated April 11, 1967

1.0 Purpose

The purpose of this test is to determine the amount and composition of outgassing products of the experimental MIN-K formulations when exposed to 1400F in an argon atmosphere at a pressure of approximately 1030 mm Hg. The test is designed to evaluate the effectiveness of low temperature outgassing with regard to adsorbed oxygen and moisture which is common to all types of MIN-K materials, and to remove these residuals prior to the prolonged testing if necessary.

2.0 Equipment

The equipment will consist of four ceramic tubes nominally 4-3/8-in. diameter by 30-in. long, each having the following attachments:

1. VITON or similar gasket and flange assembly for sealing tube.
2. Valved gas inlet port (for introduction of argon) with 0 to 2 atmosphere gauge attached for reading positive argon pressure within tube.
3. One and one-half inch diameter vacuum valve with flange for connecting to vacuum pump system.
4. Blank flange with 1/4-in. valve attached. (This flange will be connected to the vacuum flange (Item 3) upon completion of all evacuation and backfill cycles for periodic gas sampling.)
5. Argon bottle and stainless steel-copper manifold system leading to gas inlet port (Item 2). The argon line will remain connected throughout the prolonged portion of the test. The four tubes will be placed into a single box furnace per the original equipment designs.

3.0 Samples

Six samples of Formulation 1999 or 2002 (to be run separately) approximately 3 x 6 x 1-in. (to be measured accurately prior to test) will be placed in three of the four tubes, two per tube. (Two samples per tube are used to increase the MIN-K volume with respect to the tube furnace volume.) These samples will have been subjected to the standard 1400F, 2-hr isothermal firing (in air) which is considered part of the manufacturing procedure. The fourth tube will be run concurrently as a blank.

4.0 Test Procedure

4.1 Preliminary Conditioning

4.1.1 Condition one pair of specimens at 90% RH and 90F for a minimum of 48 hr. Continue conditioning until Step 4.1.7 can be conducted.

4.1.2 Condition one pair of specimens at 30% RH and 75F for a minimum of 48 hr. Continue conditioning until Step 4.1.7 can be conducted.

4.1.3 Heat the four tubes to 1600F under active vacuum, and hold these conditions for 24 hr.

* * * * *

4.1.4 Leak check each tube. Maximum permissible leak rate is 5 microns per hour. (This corresponds to a rate of approximately 5×10^{-2} cc/hr.)

4.1.5 Cool to <250F under active vacuum.

* * * * * Indicates system may be held overnight before proceeding to the next step.

- 4.1.6 Backfill each tube with dry air (by connecting a drying column containing magnesium perchlorate to the backfill inlet with flexible tubing).

* * * * *

- 4.1.7 Place one pair of specimens having no specific preconditioning into one tube and reseal tube. Heat the tubes to 1050 $\pm$ 20F without vacuum for a minimum of 30 min.

* * * * *

- 4.1.8 Actively evacuate all tubes for 30 min.

- 4.1.9 Cool tubes to room temperature under active vacuum. At room temperature check for leakage. An increase of 5 μ /hr is permitted. If this rate of pressure rise is exceeded, actively re-evacuate for extended periods of time and recheck pressure rise. The 5 μ /hr maximum rate of increase must be met before proceeding with the test. If tube is suspect, replace tube and recondition at 1600F.

* * * * *

- 4.1.10 Backfill all tubes with dry air per 4.1.6.

- 4.1.11 Open two of the three empty tubes and insert the two pairs of specimens previously conditioned per 4.1.1 and 4.1.2, one pair into each tube. Reseal tubes immediately.

* * * * *

Perform Steps 4.1.12 through 4.1.17 on each tube individually.

- 4.1.12 Open the vacuum line to the tube, and record the rate of pressure drop for 15 min starting with 100 μ pressure as the zero time. Record time and pressure every 15 sec if the pressure change is

significant. The frequency of recordings may be reduced as the rate of pressure change decreases.

4.1.13 Close vacuum valve and record pressure rise as a function of time for 15 min, or until pressure reaches 100μ , whichever occurs first.

4.1.14 Open vacuum valve and record pressure drop with time for 15 min. If starting pressure is above 100μ , evacuate to 100μ before initiating the time-pressure recordings. If pressure is below 100μ , time zero is represented by the maximum pressure reading when the vacuum valve is opened.

4.1.15 Repeat 4.1.13.

4.1.16 Repeat 4.1.14.

4.1.17 Repeat 4.1.13. Compare rate of pressure rise for the three cycles. Check for leaks if system is suspect.

4.1.18 Hook up argon bottle and manifold system. Evacuate all tubes and manifold simultaneously, and helium leak check each connection.

4.1.19 Close vacuum valves and heat furnace to $400 \pm 20^\circ\text{F}$.

* * * * *

4.1.20 Repeat 4.1.12 through 4.1.17 for each tube individually.

4.1.21 Evacuate all tubes simultaneously for 15 min (or until pressure is below 50μ , whichever is longer).

4.1.22 Close vacuum valves and heat to $600 \pm 20^\circ\text{F}$.

* * * * *

4.1.23 Repeat 4.1.12 through 4.1.17 for each tube individually.

4.1.24 Evacuate all tubes individually for 15 min (or until pressure is below 50 μ , whichever is longer). (Open argon manifold to vacuum during this evacuation.)

4.1.25 Close vacuum valves, and backfill tubes with argon to approximately 1030 mm Hg (20 psia). Maintain 600 $\pm$ 20F and approximately 1030 mm Hg for a minimum of 96 hr (4 days). During this period, disconnect the flexible vacuum hose from the tubes and connect the blank flange and 1/4-in. Hoke valve assembly.

* * * * *

4.1.26 Run a water analysis and take a gas sample through the blank flange and valve assembly of each tube.

4.1.27 Repeat 4.1.12 through 4.1.17 for each tube individually which contains 500 ppm or more of water. After each outgassing cycle, however, backfill with argon to approximately 1030 mm Hg and wait 5 min before proceeding with subsequent pumpdown cycle.

4.1.28 After final backfill, maintain 600 $\pm$ 20F and approximately 1030 mm Hg argon pressure for a minimum of 96 hr.

4.1.29 Run water content analysis on all tubes involved in Steps 4.1.27 and 4.1.28.

4.1.30 Increase temperature to 1000 $\pm$ 20F maintaining an argon pressure of approximately 1030 mm Hg. Maintain this temperature for a minimum of 96 hr (4 days).

* * * * *

4.1.31 Repeat 4.1.26.

4.1.32 Repeat 4.1.12 through 4.1.17 for each tube individually. Change the time of the first pumpdown to 30 min, and each subsequent outgas and pumpdown to 5 min.

- 4.1.33 Backfill to approximately 1030 mm Hg argon after final outgassing cycle.
- 4.1.34 Increase temperature to 1400 $\pm$ 20F maintaining an argon pressure of approximately 1030 mm Hg. Maintain this temperature for a minimum of 96 hr (4 days).
- 4.1.35 Repeat 4.1.26.
- 4.1.36 Repeat 4.1.12 through 4.1.17 for each tube individually containing >1000 ppm water. Change each pumpdown and outgas cycle time to 5 min. Backfill after each outgassing cycle with argon to approximately 1030 mm Hg.

* * * * *

- 4.1.37 Maintain 1400 $\pm$ 20F and approximately 1030 mm Hg for a minimum of 96 hr.

* * * * *

- 4.1.38 Repeat 4.1.26.

4.2 Prolonged Testing

- 4.2.1 Hold temperature at 1400F for 500 hr, repeat water and mass spectrometer analysis, and adjust pressure to 1030 mm Hg.
- 4.2.2 Repeat 4.2.1 at 500 hr intervals up to 2000 hr.
- 4.2.3 Heat to 1500F (adjusting pressure) and hold for 100 hr.
- 4.2.4 Repeat water and mass spectrometer analysis.
- 4.2.5 Heat to 1600F (adjusting pressure) and hold for 48 hr.
- 4.2.6 Repeat water and mass spectrometer analysis.
- 4.2.7 Cool to room temperature. System may be vented to atmosphere.
- 4.2.8 Disconnect flange, remove samples and measure.
- 4.2.9 Calculate per cent shrinkage of each sample.

IX. APPENDIX B.

**INSULATION DEVELOPMENT PROGRAM
DYNAMIC PROPERTIES TEST REPORT**

MND-3900-11

13 March 1967

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FOREWORD

This report was prepared for submittal as part of the work done under Contract No. AT(30-1)-3663, Subcontract 3900-1, Dynamic Tests. The work was performed by the MARTIN MARIETTA CORPORATION, Baltimore Division.

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7.

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SUMMARY

Six (6) samples were tested in an "as received" condition (unbaked). All six (6) samples were cut from the same batch of parent material, three (3) from block No. 2002-38, and three (3) from block No. 2002-39. All of the test samples completed the tests outlined in MND 3900-7 (Appendix B). From the tests conducted, the following was determined:

- 1.) Min-E 2002 has a dynamic modulus of elasticity (E) of approximately 2100 PSI (determined by averaging the results for each pressure condition for each test sample).
- 2.) The percent difference between the dynamic modulus of elasticity (E) and the static modulus of elasticity (E) is 4.4%. The static modulus of elasticity (E) was determined by John Manville.
- 3.) Min-E 2002 has a structural damping of approximately 0.5% (determined by averaging the results for each pressure condition for each test sample).

It is felt that Min-E 2002 may be used as a structural material within the limits shown in this report, since there were no failures during testing.

I. INTRODUCTION

This phase of the insulation development program was conducted to determine the dynamic modulus of elasticity (E), the structural damping (G), and the first natural (resonant) frequency of experimental K1a-X formulation 2002 for engineering data to use in the design of future nuclear power systems. The above dynamic properties were determined for six (6) test samples at various pressures, and the results for each test sample were averaged together to obtain the values for the modulus of elasticity (E) and the structural damping (G).

II. DESCRIPTION OF SPECIMENS AND TEST METHODS

The test arrangement is shown in Figure No. 2 and a list of instrumentation used during testing is included in Table No. 2. The Min-K samples (see Figures numbered 3 and 4) were placed directly on top of a flat, level steel block. The samples were positioned as close as possible to the geometric center of a circular base plate (see Figure No. 4). A loading head (see Figure No. 4) was placed directly on top of a Min-K sample and centered such that it passed through the center of the circular base plate. The samples could be observed during testing by looking between the circular base plate and the floor.

Three (3) steel wires, approximately 39 inches long, were attached to a threaded rod as shown in Figure No. 2. These steel wires were necessary to stabilize the system when more than three (3) lead weights were stacked on the loading head, and also, the wires acted as a safety device (prevented toppling) when observing the samples during testing. "Rocking" was minimized, and pure vertical loading was insured by the use of the steel stabilizing wires. Preliminary tests were run using one, two, and three lead weights, first without utilizing the steel wires, and then using the steel wires. The frequencies obtained, for both conditions, were the same. As a result of these preliminary tests, it was decided that the

steel stabilizing wires introduced no discrepancies into the system.

The test was conducted by successively adding 54 lb. lead weights on the loading head and exiting the system for each weight condition. Each lead weight was placed as close as possible to the center of the loading head, then the steel stabilizing wires were attached to a rod which protruded out of the top of the loading head. Data was obtained by striking the top lead weight by hand near the lead weights center of gravity, and recording the output of an Endevco Accelerometer (see Figure No. 4) on an oscillograph. Data was obtained for each weight condition until a total of ten (10) lead weights were stacked on the loading head. An Instrumentation Schematic is shown in Figure No. 5.

Five (5) out of six (6) of the Min-X samples were tested only under increasing load, that is, data was obtained for ten (10) weight conditions (first through tenth weight inclusive), then all of the weights were removed. For sample No. 2002-39-1 data was obtained with increasing and decreasing load. (Total of twenty (20) weight conditions.) This was done to see if loading significantly changed any characteristics of the specimen. The results are tabulated in Appendix A.

III. DISCUSSION OF DYNAMIC PARAMETERS

Determination of Experimental Spring Constant (k)

The first natural frequency (f_n) of a single-degree-of-freedom, spring mass system is defined by the following equation:

$$f_n = \frac{1}{2\pi} \sqrt{\frac{k}{m}}$$

where (k) is the spring constant in (lbs./inch), and (m) is the mass in [lbs/(inches/sec.²)]. From the relationship,

$$m = w/g,$$

where (w) is in (lbs.), and (g) is the acceleration due to gravity (386 inches/sec.²), an equation can be obtained relating f_n , k, w, and g (test parameters) is:

$$k = \frac{4\pi^2 f_n^2 w}{g}$$

which may be further reduced to: $k = (0.102)(f_n^2)(w)$. For each frequency obtained from the oscillograph records during testing, the spring constant (k) can be determined for each weight condition.

Example:

P/N 2002-58-1, first weight condition, k is found to be:

$$\begin{aligned} k &= (0.102)(f_n^2)(w) \\ &= (0.102)(24.6^2)(61.375) \\ &= (0.102)(605.16)(61.375) = 3788 \text{ lbs./inch.} \end{aligned}$$

The rest of the values of (k), for all samples, are tabulated in Table No. 1.

Determination of the Experimental Dynamic Modulus of Elasticity (E)

The modulus of elasticity (E) of an elastic material subjected to axial forces may be determined from the following equation:

$$E = \frac{FL}{kAL},$$

where (F) is the axial force in lbs., (L) is the total length of material in inches, (A) is the cross-sectional area (in²), and (ΔL) is the change in length (in). Since $k = F/AL$, we have,

$$E = \frac{FL}{\frac{F}{AL}L}.$$

$$\text{Because } k = \frac{4\pi^2 f_n^2 m}{\delta},$$

which may be further reduced to

$$E = \frac{(0.102)(f_n^2)(\omega L)}{1}.$$

For each frequency obtained from the oscillograph records during testing, the dynamic modulus of elasticity (E) can be determined for each weight condition.

Example:

P/N 2002-38-1, first weight condition, (E) is found to be:

$$\begin{aligned}
 E &= \frac{(0.102)(f_n^2)(wL)}{A} \\
 &= \frac{(0.102)(28.6)^2(122.75)}{4.74} \\
 &= 1599 \text{ lbs./in.}^2
 \end{aligned}$$

The rest of the values of (E), for all samples, are included in Data Sheet No. 4.3.6. (Appendix A).

Determination of Structural Damping (G)

The most widely used method for obtaining damping is by the log decrement process. With this process, the system is excited sinusoidally at its natural frequency and the means of excitation suddenly removed. As the motion of the system decays, the output of an accelerometer mounted on the structure is recorded by an oscillograph. Damping can be obtained from this trace by using the equation:

$$G = \frac{1}{\pi N} \ln (A_0/A_N),$$

where A_0 is the trace amplitude at the start of readable decay, A_N is the trace amplitude after N cycles. For the case $A_N = 0.5 A_0$, the above equation can be reduced to a simpler form:

$$G = \left(\frac{0.3184}{f} \right) \ln \left(\frac{A_0}{0.5A_0} \right)$$

$$= \left(\frac{0.3184}{f} \right) \ln(2)$$

$$= \left(\frac{0.3184}{f} \right) (0.693)(\ln(2) = 0.693)$$

$$G = 0.2206/f$$

Example: P/E 2002-38-1

Using semi-log paper, vibration amplitude was plotted on the logarithmic ordinate and frequencies of vibration on the linear abscissa. From the slope of the line, the number of cycles, N , from A_0 to $0.5A_0$ was determined. Using this data and the equation

$$G = 0.2206/N,$$

the structural damping was determined. See Figure No. 1 for a plot of amplitude vs. frequency (damping). The values of (G) for all samples are included in Data Sheet No. 4.3.6. (Appendix A).

IV. TEST RESULTS AND CONCLUSIONS

From the test conducted, the following was determined:

- 1.) Min-K 2002, approximately 20 lbs./ft.³ density, has a dynamic modulus of elasticity (E) of approximately 2100 PSI,* (Determined by averaging the values of (E) for each pressure level for each sample.)
- 2.) The percent difference between the dynamic modulus of elasticity (E) and the static modulus of elasticity (provided by Johns Manville, E_s = 2000 PSI) is 4.4%.
- 3.) Min-K 2002 has a structural damping (G) of approximately 0.5% (determined by averaging the values of (G) for each pressure level for each sample).

All of the samples completed the tests contained in Appendix B without failure. Since there were no failures, it is felt that Min-K 2002 may be used as a structural material within the limits shown in this report.

As the weight levels increased, the value of (E) also increased. At this time there is no explanation for this phenomenon, however, it is felt that upon closer evaluation of the oscillograph records, this phenomenon (increasing E) may be explained.

P/N 2002-38-1

FIRST WEIGHT CONDITION

$$A_0 = 0.5$$

$$A_N = 0.25$$

$$N = 55 - 15 = 40$$

$$G = \frac{.2206}{40}$$

$$G = .0055$$

$$G = 0.55\%$$

Figure No. 1
Sample Damping Display

FIGURE NO. 2
TEST ARRANGEMENT

FIGURE NO. 3
"MIN-K" SAMPLES PRIOR TO TEST

FIGURE NO. 4
CLOSE-UP OF TEST ARRANGEMENT

Figure No. 5
Instrumentation Schematic

Table No. 1
Values for Spring Constant (k)

Specimen No.	2002-38-1	2002-38-2	2002-38-3	2002-39-1	2002-39-2	2002-39-3
Weight Condition	k					
1	3768	4007	4564	4838	4463	4167
2	4002	4089	4634	4871	4402	4222
3	4097	4204	4703	4818	4478	4204
4	4281	4344	4929	5132	4599	4407
5	4576	4504	5094	5247	4721	4576
6	4886	4644	5301	5472	4967	4724
7	5122	4944	5487	5581	5122	4944
8	5432	5140	5732	5937	5531	5236
9	5770	5557	5879	6322	5770	5663
10	6047	6047	6164	6526	6047	5931
Average	4800	4748	5249	5474	5010	4807

LIST OF TEST EQUIPMENT AND INSTRUMENTATION

<u>Item</u>	<u>Manufacturer</u>	<u>Model</u>	<u>Calibration</u>
Accelerometer	Endevco	2217-G/8 1238	Every 6 months
Brace-Monitor (Amplifier)	Endevco	27028	" " "
Oscillograph	C.E.C.	5-124	" " "
Galvanometer	C.E.C.	7-326	Prior to test
Amplifier	NEFF	119	Every 6 months
Oscilloscope	Tektronix	561	" " "
Lead Weights - approximately 54 lbs. each, 11" x 13" x 1"			

TABLE NO. 2

APPENDIX A
DYNAMIC PARAMETERS AND RESULTS

PROPERTIES			E X P E R I M E N T						
			2002-36-1	2002-36-2	2002-36-3	2002-37-1	2002-37-2	2002-37-3	
Spec. Material Type			MIN-K	MIN-K	MIN-K	MIN-K	MIN-K	MIN-K	
Dimensions	D		2.46	2.48	2.49	2.48	2.48	2.46	
	T		2.0	2.0	2.0	2.0	2.0	2.0	
Surface Area (In ²)			4.74	4.80	4.86	4.80	4.80	4.74	
Weight (Lbs.)			.1095	.1091	.1113	.1127	.1104	.1105	
Volume (In ³)			9.48	9.60	9.72	9.60	9.60	9.48	
Density (Lbs./In ³)			19.87	19.62	19.69	20.21	19.00	20.04	
Temperature °F			74	74	74	74	74	74	
Relative Humidity %			30	30	30	30	30	30	
Weight Conditions	1	W	61.375	61.375	61.375	61.375	61.375	61.375	
		P	10.95	10.79	12.63	12.79	12.79	12.95	
	2	W	115.875	115.875	115.875	115.875	115.875	115.875	
		P	24.45	24.14	23.84	24.14	24.14	24.45	
	3	W	169.375	169.375	169.375	169.375	169.375	169.375	
		P	35.73	35.29	34.85	35.29	35.29	35.73	
	4	W	223.625	223.625	223.625	223.625	223.625	223.625	
		P	47.18	46.59	46.01	46.59	46.59	47.18	
	5	W	278.125	278.125	278.125	278.125	278.125	278.125	
		P	58.68	57.94	57.23	57.94	57.94	58.68	
	6	W	332.625	332.625	332.625	332.625	332.625	332.625	
		P	70.17	69.30	68.44	69.30	69.30	70.17	
Dynamic Results	1	E	24.6	25.3	27.0	27.8	27.0	26.7	25.8
		G	.0055	.0055	.0049	.0029	.0027	.0038	.0071
		E	1599	1669	1878	2016	1901	1859	1758
	2	E	18.4	18.6	19.8	20.3	20.0	19.3	18.9
		G	.0040	.0046	.0047	.0042	.0045	.0049	.0060
		E	1689	1703	1907	2029	1969	1834	1781
	3	E	15.4	15.6	16.5	16.7	16.6	16.1	15.6
		G	.0049	.0052	.0049	.0058	.0068	.0046	.0070
		E	1729	1752	1936	2007	1923	1866	1773
	4	E	13.7	13.8	14.7	15.0	15.1	14.2	13.9
		G	.0049	.0047	.0038	.0049	.0049	.0052	.0042
		E	1807	1810	2029	2138	2167	1916	1859
	5	E	12.7	12.6	13.4	13.6	13.8	12.9	12.7
		G	.0049	.0042	.0050	.0037	.0044	.0063	.0045
		E	1931	1876	2047	2186	2251	1967	1930
	6	E	12.0	11.7	12.5	12.7	13.0	12.1	11.8
		G	.0048	.0041	.0043	.0045	.0044	.0042	.0059
		E	2068	1935	2182	2280	2339	2069	1943

Appendix A

Page A1

Data Sheet No. 4.3.6

MWD-3900-11

D = Sample diameter (in.)

T = Sample thickness (in.)

W = Weight of loading head plus actual weight of lead weights (lbs.)

P = Pressure on sample surface (psi)

f = Frequency of 1st resonance (cps)

G = Structural damping coefficient

E = Dynamic modulus of Elasticity (psi) = $\frac{(.102) f^2 WT}{(\text{Area})}$

PROPERTIES			IDENTIFICATION NUMBER					
			00-30-2	00-30-3	00-30-4	00-30-5	00-30-6	00-30-7
Spec. Material Type			MTAL-K	MTAL-K	MTAL-K	MTAL-K	MTAL-K	MTAL-K
Dimensions	D		2.46	2.48	2.49	2.48	2.48	2.46
	T		2.0	2.0	2.0	2.0	2.0	2.0
Surface Area (in ²)			4.74	4.80	4.86	4.80	4.80	4.74
Weight (lbs)			.1095	.1091	.1113	.1127	.1104	.1105
Volume (in ³)			9.48	9.60	9.72	9.60	9.60	9.48
Density (lbs/in ³)			19.87	19.52	19.69	20.21	19.00	20.04
Temperature °F			74	74	74	74	74	74
Relative Humidity%			30	30	30	30	30	30
Weight Conditions	7	V	386.375	386.375	386.375	386.375	386.375	386.375
		P	81.51	80.49	79.50	80.49	80.49	81.51
	8	V	440.125	440.125	440.125	440.125	440.125	440.125
		P	92.85	91.69	90.56	91.69	91.69	92.85
	9	V	494.125	494.125	494.125	494.125	494.125	494.125
		P	104.24	102.94	101.67	102.94	102.94	104.24
	10	V	548.125	548.125	548.125	548.125	548.125	548.125
		P	115.64	114.19	112.78	114.19	114.19	115.64
		V						
		P						
		V						
		P						
Dynamic Results	7	E	11.4	11.2	11.2	11.9	12.5	11.4
		G	.0038	.0044	.0049	.0052	.0044	.0038
		B	2161	2059	2257	2325	2565	2134
	8	E	11.0	10.7	11.3	11.5	11.7	11.1
		G	.0048	.0045	.0038	.0051	.0048	.0049
		B	2292	2141	2359	2473	2560	2304
	9	E	10.7	10.5	10.8	11.2	11.3	10.7
		G	.0035	.0037	.0033	.0045	.0049	.0052
		B	2435	2315	2480	2634	2681	2404
	10	E	10.4	10.4	10.5	10.8	10.8	10.4
		G	.0037	.0049	.0025	.0056	.0046	.0041
		B	2552	2519	2537	2707	2707	2519
	AVE.	E	—	—	—	—	—	—
		G	.0044	.0046	.0043	.0047	.0046	.0047
		B	2026	1978	2160	2280	2317	2087
		E						
		G						
		B						

Appendix A

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Data Sheet No. 4.3.6

MND-3900-11

D = Sample diameter (in.)

T = Sample thickness (in.)

W = Weight of loading head plus actual weight of lead weights (lbs.)

P = Pressure on sample surface (psi)

f = Frequency of 1st resonance (cps)

G = Structural damping coefficient

E = Dynamic modulus of Elasticity (psi) = $\frac{(.102) f^2 WT}{(\text{Area})}$

APPENDIX B
DYNAMIC PROPERTIES TEST PROCEDURE,
INSULATION DEVELOPMENT PROGRAM

DYNAMIC PROPERTIES
TEST PROCEDURE
INSULATION DEVELOPMENT PROGRAM

MND-3900-7

30 December 1966

/s/ W. M. Griffin

W. M. Griffin

Approved: /s/ W. H. Lambert

W. H. Lambert

Approved: /s/ D. B. O'Brien

D. B. O'Brien

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1.0 OBJECTIVE

The purpose of this test is to determine the dynamic modulus of elasticity and the structural damping of insulation material. Data obtained from this test will be used to supplement results of structural properties tests performed by Johns-Manville Research and Engineering Center in which static modulus of elasticity, yield stress, failure stress, and load relaxation of the material will be performed in the Martin Company's Environmental Test Facility. Together, these data will provide the basic information necessary for analysis and design of applications in which the material is subject to the influence of external forces.

2.0 SCOPE

Each material will be subjected to transient excitation tests to determine the dynamic modulus of elasticity (E) and the structural damping coefficient (G) at room temperature. Samples will be cut from the same batch of parent material with the test axis normal to the "layering plane" of the parent material.

Effects of temperature, long-term temperature exposure, edge restraint, and consistency of formulation (batches) are assumed to have no effect on the structural properties of the material. These assumptions, based on results from previous tests of a related material (MND-3169-45), are subject to validation by the Johns-Manville static tests. If any of the above listed assumptions are shown to be incorrect, it may be necessary to perform additional tests.

2.1 General Notes

2.1.1 Measurement Error

The maximum allowable instrument error shall be as follows:

Vibration Amplitude $\pm 2\%$

Vibration Frequency $\pm 2\%$

Additional tolerances as specified

2.1.2 Standard Conditions

Unless otherwise specified, the standard conditions for conducting tests shall be as follows:

Temperature $77 \pm 18^{\circ}\text{F}$

Relative Humidity 90% or less

Barometric Pressure local ambient

2.1.3 Test Equipment and Instrumentation

<u>Item</u>	<u>Manufacturer</u>	<u>Model</u>	<u>Freq. of Calib.</u>
Accelerometer	Endevco	2216	6 months
Dyna-Monitor Amplifier	Endevco	2702	6 months
Photo-Oscillograph	C.E.C.	5-119-P3-50	6 months
Galvanometer	C.E.C.	7-326	Prior to test
Test Fixture*	Martin-Baltimore	88-3000514	---
Lead Weights	---	Approx. 54 lbs, 11" x 13" x 1"	

*Existing Facilities Fabricated Under Another Program of a Similar Nature

2.2 Applicable Documents

Martin Document MND-3169-45, January 1965, Structural

Evaluation of MIN-K 1301.

3.0 DYNAMIC PROPERTIES TESTS

3.1 Description of Test Samples

All test samples shall be right circular cylinders, 2.5 inches in diameter and 2.0 inches thick. Test loading and material properties will be referenced to an axis normal to the circular faces of the samples.

Six samples of insulation material are required for testing, with the test axis normal to the "layering plane" of the parent material from which the samples are taken.

3.2 Recording System

All data will be recorded on a portable oscillograph. The equipment (or the equivalent thereof) indicated in Figure 1 will be utilized.

3.3 Test Procedure

3.3.1 Assign an identification number to the specimen and record this number on data sheet 3.3.8, and record the specimen material type. Measure the diameter, and thickness of the specimen to be tested to the nearest $1/64$ inch and its weight to the nearest 0.5 gram, and record these dimensions on data sheet 3.3.8. Calculate the surface area and density of the specimen and record on data sheet 3.3.8.

3.3.2 Install the specimen into the test fixture (refer to Figure No. 2). Read the ambient temperature and relative humidity in the environmental test laboratory, and record on data sheet 3.3.8.

- 3.3.3 Subject the specimen to a compression load by adding one lead weight to the top of the fixture (see Figure No. 2). Determine the actual weight on the specimen and record on data sheet 3.3.8. Calculate the total pressure on the specimen and record on data sheet 3.3.8.
- 3.3.4 Start the oscillograph to record the accelerometer output. Excite the specimen by striking the lead weight near the center of gravity with a rubber hammer or equivalent object. Continuously record the accelerometer output for a period of time sufficient to measure the rate of decay of the vibration amplitude.
- 3.3.5 Successively add lead weights and repeat the procedures of 3.3.3 and 3.3.4 for each weight condition. This process shall be repeated until a total of six or more weights are loaded on the assembly, as determined by the test engineer.
- 3.3.6 With the data from the oscillograph records, determine for each weight condition the fundamental response frequency, dynamic modulus of elasticity, and structural damping coefficient as follows:

On semi-log paper, plot vibration amplitude on the logarithmic ordinate versus number of cycles of vibration from a convenient starting point as the linear abscissa. Determine from the slope of the line the number of cycles, n , for the amplitude to decrease to half value. Calculate the structural damping coefficient G from the relationship $G = \frac{.2206}{n}$, and enter its value on data sheet 4.3.6.

Calculate the dynamic modulus of elasticity, E , according to the relationships shown on data sheet 4.3.6, and enter the value in the appropriate column.

3.3.7 Repeat steps 3.3.1 through 3.3.6 inclusive for each of the six (6) samples.

Figure No. 1
Recording Schematic

Figure No. 2
Basic Test Set-Up

**THERMAL INSULATION
DYNAMIC PROPERTIES TEST REPORT**

MND-3900-12

29 March 1967

Prepared by:

W. M. Griffin
W. M. Griffin

Approved by:

W. H. Lambert
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FOREWORD

This report was prepared for submittal as part of the work done under Contract No. AT(30-1)-3655, Subcontract 3900-1, Dynamic Tests. The work was performed by the Martin Marietta Corporation, Baltimore Division.

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SUMMARY

An investigation was made of apparent variations of (E) with increasing load for experimental Min-K Formulation 2002.

The oscillograph traces of all six (6) samples were analyzed by averaging the time (T) over fifty (50) cycles of vibration. Comparing the original values of (E) with the re-examined values, there is found to be approximately a 0.4% difference between the two. This percent difference is considered to be insignificant.

(E) vs. (PSI) was plotted on rectangular co-ordinate paper. The curve seems to be non-linear in the region below 35 PSI whereas in the region above 35 PSI, the curve appears to be linear. The method of least squares was used to determine the straight line which best fit the data points. There is a definite slope associated with the plot of (E) vs. (PSI).

I. PURPOSE

The purpose of this report is to investigate apparent variations of the dynamic modulus of elasticity (E) with increasing compressive load (P) of experimental Min-K Formulation 2002, as reported in MND-3900-11.

II. ANALYSIS OF THE DYNAMIC MODULUS OF ELASTICITY

The oscillograph records of all six (6) samples were re-read by averaging the time (T) over fifty (50) cycles of vibration. It was felt that this method would be more accurate than reading the number of cycles over a time span of one (1) second, as was the method used in MND-3900-11. The results of this analysis are tabulated in Table No. 1. Table No. 2 is a tabulation of the original values of (E). Upon comparing these two (2) sets of values, it is found that there is very little difference between the two (average of 0.4%). Figure No. 3 is a reproduction of a typical oscillograph trace.

III. NUMERICAL COMPUTATIONS

A. METHOD OF LEAST SQUARES

The method of least squares is an explicit mathematical procedure for fitting a curve of the form $y = a + bx$ to a given set of data. For a derivation of the method of least

squares, refer to: Unified Calculus and Analytic Geometry,
 Rainville, Earl D., The Macmillan Company, 1961, pp. 324-326.

B. SAMPLE CALCULATIONS

In the following calculations, the values of (E) and (G) are the average values of all six (6) samples for each weight condition and the values of (PSI) are the average values of all six samples at each of the ten (10) pressure (weight) conditions.

The first three (3) pressure (weight) conditions were not used in the calculation of the equation of the curve for (E) vs. (PSI) because the curve appears to be non-linear in the region less than 35 PSI. This could be caused by uneven loading on the sample (point-contacts) until significant loading occurs. Significant loading seems to occur in the region above 35 PSI in which the curve appears to be linear. Figure No. 1 is a plot of (E) vs. (PSI) using the method of least squares to "best-fit" the curve to the data points. Figure No. 1-a is also a plot of (E) vs. (PSI) connecting all of the data points by a smooth curve.

In the case of (G) vs. (PSI) as shown in Figure No. 2, it is apparent that a straight line is a very good representation of the data. There is no apparent change in slope at any loading, so all of the ten (10) pressure (weight) conditions were used in calculating the equation of the curve.

B. SAMPLE CALCULATIONS

3.

MODULUS OF ELASTICITY Vs. PSI

PSI	10.22	21.20	35.36	46.69	52.07	69.45	80.66	91.89	103.16	114.44
E	1799	1837	1848	1924	1992	2092	2196	2310	2442	2574

$$\sum(PSI) = 636.74 - 72.32 = 564.36$$

$$\sum(PSI)^2 = 51064 - 2000.32 = 49063.68$$

$$\sum(E) = 21086 - 5424 = 15542$$

$$\sum(PSI)(E) = 1420575 - 132823 = 1287712$$

$$na + b \sum(PSI) = \sum(E)$$

$$a \sum(PSI) + b \sum(PSI)^2 = \sum[(PSI)(E)]$$

By SUBSTITUTING THE APPROPRIATE VALUES INTO THE ABOVE EQUATION, THE FOLLOWING EQUATION IS OBTAINED:

$$7a + 564b = 15542 \quad (\text{MULTIPLY BY } 80.571428)$$

$$564a + 49064b = 1287712$$

$$564a + 45442b = 1252241$$

$$\therefore 564a + 49064b = 1287712$$

$$-3622b = -35471$$

$$\underline{\underline{b = 9.79}}$$

$$7a + (564)(9.79) = 15542$$

$$7a + 5522 = 15542$$

$$7a = 10020$$

$$\underline{\underline{a = 1431}}$$

From $y = a + bx$, THE EQUATION OF THE LINE IS OBTAINED,

$$\underline{\underline{y = 1431 + 9.79x}}$$

FOR $y = 0 \rightarrow x = -146$

FOR $x = 0 \rightarrow y = 1431$

B. SAMPLE CALCULATIONS

4.

STRUCTURAL DAMPING Vs. PSI

PSI	42.22	24.20	25.36	46.69	52.07	69.45	80.66	91.19	103.16	114.44
G	.0051	.0017	.0054	.0046	.0043	.0046	.0044	.0048	.0043	.0043

$$\sum(PSI) = 636.74$$

$$\sum(PSI)^2 = 51064$$

$$\sum(G) = 0.047$$

$$\sum(PSI)(G) = 2.915$$

$$na + b\sum(PSI) = \sum(G)$$

$$a\sum(PSI) + b\sum(PSI)^2 = \sum[(PSI)(G)]$$

By substituting the appropriate values into the above equation, the following equation is obtained:

$$10a + 636.74b = 0.047 \quad (\text{Multiply by } 63.674)$$

$$636.74a + 51064b = 2.915$$

$$636.74a + 40544b = 2.993$$

$$636.74a + 51064b = 2.915$$

$$-10520b = 0.078$$

$$b = -(7.4 \times 10^{-6})$$

$$10a + (636.74)(-7.4 \times 10^{-6}) = 0.047$$

$$10a - 0.0047 = 0.047$$

$$10a = 0.0517$$

$$a = (5.17 \times 10^{-2})$$

From $y = a + bx$, the equation of the line is obtained,

$$y = (5.17 \times 10^{-2}) - (7.4 \times 10^{-6})x$$

For $y=0 \rightarrow x=698$

For $x=0 \rightarrow y=0.00517$

IV. CONCLUSIONS

The following may be concluded from this investigation.

1. The original values of (E) are not significantly different from the "re-analysed" values (approximately 0.4% difference).
2. The plot of (E) vs. (PSI) appears to be approximately linear except in the region below 35 PSI.

3. The equations obtained by the method of least squares are:

$$y = 1431 + 9.79x$$

$$\text{or } E = 1431 + 9.79 (\text{PSI})$$

$$y = (5.17 \times 10^{-3}) - (7.41 \times 10^{-6})x \quad \text{or } G = (5.17 \times 10^{-3}) - (7.41 \times 10^{-6}) \text{ PSI}$$

4. The slope of (E) vs. (PSI) may be explained partly because a fixed length of the sample was used throughout the calculations for (E), whereas changing the weight condition changes the actual length of the sample. However, this accounts for only approximately 1% - 2% variation of the slope, and the remaining change cannot be accounted for.

5. The slope of (G) vs. (PSI) is so small that it may be considered to be zero.

$E_{avg.}$ vs. PSI

$E_{avg.}$ vs. PSI

Figure No. 1a

Figure No. 2

Specimen No.		E (PSI)					
		2002-38-1	2002-38-2	2002-38-3	2002-39-1	2002-39-2	2002-39-3
WEIGHT CONDITIONS	1	1612	1669	1906	2016	1831	1758
	2	1689	1722	1926	2049	1853	1781
	3	1729	1752	1936	2031	1842	1796
	4	1806	1836	1992	2138	1889	1896
	5	1931	1876	2065	2186	1967	1930
	6	2062	1968	2182	2280	2069	2026
	7	2199	2059	2259	2364	2171	2127
	8	2292	2182	2359	2517	2304	2208
	9	2481	2315	2510	2634	2404	2344
	10	2601	2471	2586	2717	2568	2502
AVERAGE		2040	1985	2172	2293	2090	2035

TABLE NO 1

"Re-Examined"(E)

SPECIMEN NO.		E (PSI)					
		2002- 38-1	2002- 38-2	2002- 38-3	2002- 39-1	2002- 39-2	2002- 39-3
WEIGHT CONDITIONS	1	1599	1669	1878	2016	1859	1758
	2	1689	1703	1907	2029	1834	1781
	3	1729	1752	1936	2007	1866	1773
	4	1807	1810	2029	2138	1916	1859
	5	1931	1876	2097	2186	1967	1930
	6	2062	1935	2182	2280	2069	1993
	7	2161	2059	2257	2325	2134	2085
	8	2292	2141	2359	2473	2304	2209
	9	2435	2315	2420	2634	2404	2389
	10	2552	2519	2537	2707	2519	2502
AVERAGE		2026	1978	2160	2280	2087	2028

TABLE NO. 2

"Original" (E)