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DEVELOPMENT OF 400F-2200F FIBROUS-TYPE
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Eighth Quarterly Progress Report
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ABSTRACT

by

J. O. Collins

The thermal conductivity study of the MIN-K Formulations developed under Phase I uncovered what may have been the cause of the earlier erratic data. If further testing confirms these findings, the Guarded Hot Plate test procedure for evaluating MIN-K at temperatures above 800F mean may require significant alteration.

Attempts to manufacture an opacified Glasrock product were concluded unsuccessfully. Additional load-bearing data were subsequently obtained for the unopacified product.

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1. INTRODUCTION

The Eighth Quarterly Progress Report under Contract No. AT(29-2)-2661, Mod. #3 covers the effort conducted during October, November, and December 1968. This contract previously carried the Contract No. AT(30-1)-3633. The chronological numbering of Quarterly and Final Reports will be continued, however.

The Eighth Quarter effort fell into two areas:

1. The thermal conductivity evaluation of the MIN-K Formulations developed under Phase I of this contract, and
2. The evaluation of opacified and unopacified GLASROCK 25 foam silica for load-bearing applications up to 1800F hot face.

Work under Item 1 above was continued during this quarter. Thermal conductivity tests conducted by Dynatech on MIN-K Formulation 2002 uncovered an apparent cause for the erratic conductivity data previously obtained by Johns-Manville.

Previous attempts to obtain quality slabs of GLASROCK 25 containing titania opacifier had been unsuccessful. Further attempts were made. This was followed by a more detailed study of the load-bearing characteristics of the unopacified product. With the exception of a thermal conductivity evaluation of unopacified GLASROCK in vacuum at various mean temperatures, work on the GLASROCK product was completed.

II. PHASE I, TASK 5 - DETAILED EVALUATION

Introduction

The Seventh Quarterly Progress Report (ALO-3633-10) dated October 1, 1968, summarized the thermal conductivity data obtained to date for MIN-K Formulations 1999, 2002, and 2020. The data fell into two different groups, separated in time by the complete rewinding of the test apparatus. The conductivity values in air and argon were then either "accepted" or "rejected" based upon other tests conducted at the Johns-Manville Research & Engineering Center, tests conducted by Dynatech Corporation on MIN-K 2000 (for another AEC Contract), and the known relationship between thermal conductivity in air and thermal conductivity in argon.

The two different sets of vacuum data were neither accepted nor rejected because, although one set compared favorably with Dynatech data on MIN-K 2000, the other set was closer to the anticipated values based upon theoretical considerations. In addition, the curve which confirmed the Dynatech data contained several points which were higher than the air values between 1200F and 1300F mean temperature. (The plotted curve of Figure 1, Seventh Quarterly Progress Report rejected this cross-over, although the rest of the curve was retained as one of the two possible true regressions of conductivity vs mean temperature in vacuum.)

During the Eighth Quarter, the Johns-Manville equipment was carefully dismantled, the wiring checked, the thermocouples retested, and the instruments checked. Nothing was found which could be related in any way to the erratic data points.

The thermal conductivity evaluation of the MIN-K products was subsequently turned over to Dynatech Corporation, Cambridge, Massachusetts, midway in the Eighth Quarter. This report covers the re-evaluation of MIN-K Formulation 2002 by Dynatech following its 2000-hr exposure to 1200F and argon at 1 atm. The tests officially included two points in air, and four points in argon and vacuum, although additional data were obtained as will be noted subsequently. Additionally, the technique for measuring low thermal conductivity materials in the Guarded Hot Plate apparatus is also discussed as it relates to the apparent discrepancy of data obtained by Johns-Manville.

Dynatech Thermal Conductivity Data

The Dynatech thermal conductivity data points for MIN-K Formulation 2002 after simulated service exposure are given in Table 5-1. Similarly, the "best-fit" curve for the argon and vacuum data is presented in Figure 5-1. Figure 5-2 superimposes the Johns-Manville "accepted" regression for air and argon, and the two possible vacuum relationships (previously presented in Figure 1 of the

TABLE 5-1

THERMAL CONDUCTIVITY OF MIN-K 2002*
 (AFTER SIMULATED SERVICE EXPOSURE)
 AS DETERMINED BY DYNATECH CORPORATION

<u>Atmosphere</u>	<u>Mean Temperature, °F</u>	<u>Thermal Conductivity Btu in/hr ft² °F</u>
Air (1 atm)	500	0.212
"	1200	0.274
Argon (1 atm)	500	0.156
"	780	0.193
"	1020	0.229
"	1220	0.253
Vacuum (10 ⁻⁵ torr)	500	0.112
"	810	0.153
"	1020	0.188
" (5 × 10 ⁻⁴ torr)	1200	0.297

*Specimens 100 & 101

FIGURE 5-1

DYNATECH THERMAL CONDUCTIVITY REGRESSION FOR MIN-K FORMULATION 2002*
IN ARGON AND VACUUM

*Specimens 100 & 101 previously exposed to 2000 hrs in argon at 1200F

FIGURE 5-2

COMPOSITE OF JOHNS-MANVILLE AND DYNATECH
THERMAL CONDUCTIVITY REGRESSION FOR MIN-K FORMULATION 2002

Seventh Quarterly Progress Report) onto the Dynatech data. It can be seen that reasonably good agreement exists between the two sets of data for air and argon, and that the Dynatech data conforms quite closely to the higher Johns-Manville vacuum curve.

It will be noted from the data in Table 5-1 that Dynatech also arrived at a conductivity value in vacuum at 1200F which was higher than that for air at the same mean temperature, a situation which plagued Johns-Manville for part of its test program. Since the cross-over of the air and vacuum curves has no logical basis for fact, these "high" points have been discarded, and the vacuum regression allowed to approach but not to cross the air regression.

Discussion of Test Procedure

Although it was not the purpose of this contract to study the test techniques for high temperature (800F-1800F mean temperatures) Guarded Hot Plate thermal conductivity measurements, the varying sets of data generated by these measurements under "similar" test conditions have dictated that some investigation be initiated to determine their cause.

C. M. Pelanne of the Johns-Manville Research & Engineering Center, and R. P. Tye of Dynatech, both of whom are directly involved with the conductivity measurement of MIN-K under this contract, are also active members of ASTM Subcommittee T-VI of Committee C-16 to study and revise the testing procedure for high temperature Guarded Hot Plate conductivity evaluations. Consequently, their concern in testing the insulations under this contract included not only a desire for accuracy, but also an interest in establishing the effect of test technique upon the measured results. The following summarizes their preliminary investigations as they apply to the MIN-K insulations.

Some years ago it was proposed, from theoretical considerations, that while testing at low to moderate mean temperatures in the Guarded Hot Plate, the temperature at the edge of the test specimen midway between its hot and cold face should not deviate greatly from the arithmetic mean temperature of the specimen. Specifically, it was concluded that:

$$\frac{T_M - T_E}{\Delta T} \leq 0.2$$

where T_M = mean temperature
 T_E = edge temperature of specimen midway between its hot and cold faces
 ΔT = temperature drop across the specimen, approximately 200F

This function has always been considered a reasonable criterion for test accuracy, although neither its experimental determination nor the monitoring of the edge temperature was ever required by the ASTM test procedures.

Following the high vacuum values at 1200F, Dynatech initiated a short test series to determine the effect of the edge temperature of the test specimen on the measured conductivity in air at 1200F mean temperature. This was accomplished by adjusting the temperature of the heated guard ring which surrounds, but is entirely separate from the test and guard sections of the main stack. (In the past, both Dynatech and Johns-Manville have operated this ring at about the mean temperature of the specimen. The ring is approximately 2 in. from the main stack.) The results of this investigation are shown in Table 5-2.

From these preliminary results, one must conclude that the edge temperature function does in fact effect the measured results, and that a factor of about 0.05, rather than 0.2, must be achieved for accurate results with MIN-K under these specific test conditions. It is also reasonable to speculate that the factor becomes more critical as the mean temperature increases, and as the thermal conductivity of the test specimen decreases. Thus, one should expect that the vacuum data at elevated temperatures would be even more prone to error.

If the edge temperature function does have such a critical bearing on the measured values for MIN-K materials, one might speculate that if the function were negative, then the measured conductivity would be less than actual. This would explain the unusually low data obtained recently by Johns-Manville under all atmospheric conditions. Similarly, this function would explain why the MIN-K data obtained in the ASTM C-177 Guarded Hot Plate (without the heated guard ring) tends to increase faster above 1000F mean temperature than the data obtained in the similar apparatus with the guard ring. It could also explain why a "correction factor" must be applied to the MIN-K data obtained by the Rapid Heat Meter (which also does not have a ring guard) at 800F and 1000F mean temperature, but not at temperatures of about 350F, so as to correlate with the ASTM C-177 test procedure. The "correction factor" lowers the measured value, thus indicating a positive edge temperature function as one might expect based upon the design of the apparatus.

At first glance, this function appears to be the reason for many of the problems which knowingly or unknowingly have plagued the thermal conductivity evaluation of MIN-K for many years. Actually, the Dynatech investigation represents only the first step in what could be a relatively long and involved investigation covering all aspects of the test procedure by the laboratories involved with the T-VI Subcommittee. However, in the interest of obtaining the best possible data within the present scope of knowledge for the new MIN-K products, the edge temperature will be monitored by Dynatech for subsequent tests on MIN-K Formulation 2020 (following simulated service exposure to vacuum at 1600F), and MIN-K Formulation 2002 at 35 pcf density (load-bearing material).

TABLE 5-2

EFFECT OF SPECIMEN EDGE TEMPERATURE
ON THE MEASURED THERMAL CONDUCTIVITY OF
MIN-K FORMULATION 2002

Mean Temperature = 1200F
Atmosphere = Air at 760mm Hg.

$\frac{T_M - T_E}{\Delta T}$	Measured Thermal Conductivity Btu in/hr ft ² °F
+ 0.464	0.373
+ 0.315	0.353
+ 0.047	0.265
+ 0.032	0.275

ASTM C-177 Guarded Hot Plate Testing

MIN-K Formulation 1999. In addition to the conductivity efforts in the special atmosphere test equipment, MIN-K Formulation 1999 was placed on test in the standard ASTM C-177 Guarded Hot Plate test apparatus. This equipment is designed to test only in air at 1 atm pressure. The samples had been exposed to the 2000-hr simulated service conditions of 1000F in argon. Only one point was obtained before a combination of water pump, heater cutoff, and emergency cooling water failures resulted in a water line leak and subsequent soaking of the samples. The samples were dried to constant weight at 800F and retested, although a significant loss in thermal efficiency was anticipated. Table 5-3 shows the results of this test.

The single point at 635F prior to soaking was only about 5 per cent above the average air regression shown in Figure 5-2 previously established by Johns-Manville. Past experience has shown that whenever a MIN-K product tests satisfactorily at one mean temperature, its regression over a temperature range would also match the established average regression. Consequently, it can be concluded with reasonable confidence that such would have been the case with these specimens. Additionally, it was concluded that the 2000-hr thermal exposure had no effect upon the conductivity of MIN-K Formulation 1999.

The results after water exposure were considerably lower than anticipated. A smooth relationship through the test points indicated an average increase of only 0.03 Btu in./hr ft² °F over the average regression shown in Figure 5-2. The amount of water adsorbed by the specimens (2 required for the Guarded Hot Plate test) was not determined, although it was estimated that they weighed about twice as much wet than they did dry. Similarly, the weight to which they were dried coincided with their original weight. Although soaking with water is not recommended under any circumstances, the relatively small change in conductivity lends further strength to the conclusion that no change occurred solely as a result of the thermal exposure.

MIN-K Formulation 2020. These specimens which had undergone a simulated service exposure of 2000 hr in air (1 atm) at 1600F were also placed in the Guarded Hot Plate apparatus for conductivity evaluation in air. (It will be noted that two sets of MIN-K Formulation 2020 specimens are under evaluation; one set, being tested by Dynatech Corporation, was exposed to vacuum for 2000 hr, while the other set, presently under evaluation by Johns-Manville, was exposed to air for 2000 hr.) The data from both evaluations will be reported as Ninth Quarter effort.

TABLE 5-3

THERMAL CONDUCTIVITY IN AIR OF MIN-K FORMULATION 1999*
AFTER 2000 HRS IN ARGON AT 1000F

Mean Temperature °F	Thermal Conductivity Btu in/hr ft ² °F	
	<u>Before Water Soak</u>	<u>After Water Soak</u>
653	0.220	
489		0.233
748		0.266
865		0.264
1064		0.283

*Specimens 12 & 14

III. PHASE II, TASK 3 - LOAD-BEARING INSULATION FOR 400-1800F SERVICE

Subtask 3.2 - Load-Bearing Insulation for 1800F Service

Introduction. The Seventh Quarterly Progress Report (ALO-3633-10) described the evaluation of the 5 and 10 per cent zirconia, and the 5 per cent titania opacified Glasrock specimens. It was concluded then that several additional trials to fabricate the 10 per cent titania specimens would be attempted, but that the thermal conductivity data had indicated that opacification was not reducing the conductivity as expected.

This report covers the manufacture of the 10 per cent titania product, its evaluation for thermal conductivity, the conductivity evaluation of low density (<20 pcf) unopacified Glasrock to augment the conductivity vs silica density relationship previously reported, the determination of the permanent set of 25 and 30 pcf density (nominal) unopacified Glasrock following up to 500 hr under various constant loads, and the study of the stress-strain characteristics of unopacified Glasrock. With the exception of the further evaluation of the thermal conductivity of unopacified Glasrock in vacuum over a range of mean temperatures, the work on this Subtask was completed during this quarter.

Ten Per Cent Titania Glasrock. The initial attempt to manufacture Glasrock containing 10 per cent titania opacifier resulted in failure due to excessive cracking and crazing. Subsequently, three more 10 per cent batches, plus one each at the 5 and 15 per cent levels were tried. In two cases, 60 per cent of the fired slab contained cracks, while the remaining three batches were discarded due to excessive slumping during the drying stage.

The manufacturing process was then radically changed such that in place of the 18-hr mixing of the titania with the silica slurry (in a large ball mill without the grinding balls), a 30-min mixing cycle was substituted using the grinding balls. This resulted in an excellent slab, free from internal cracks and voids.

The slab was then slit into 12 x 12 x 1-in. specimens for thermal conductivity evaluation in the Rapid Heat Meter apparatus. The results of two specimens were 1.09 and 1.03 Btu in./hr ft² °F at 800F (nominal) mean temperature. This represented a 15 per cent reduction in average conductivity based upon the regression for conductivity vs silica density shown in Figure 21 of the Seventh Quarterly Progress Report. (The actual TiO₂ content was not determined, but was assumed to be 10 per cent. The silica density was then calculated to be 24.9 pcf for both specimens.)

Since it appeared that a breakthrough had been achieved, the run was repeated using the identical manufacturing procedure. Although the subsequent slabs did not appear to be quite as good as those made just previously, they were free from major cracks and voids. Breakage during shipment from the manufacturer yielded only one good 12 x 12 x 1 section for conductivity testing. This specimen had a thermal conductivity of 1.36 Btu in./hr ft² °F at 800F mean temperature. Based on an estimated 24.4 pcf silica density, this point was 12 per cent above the average conductivity vs silica relationship previously referenced. As a check to establish the validity of the relative conductivity values obtained by the Rapid Heat Meter, one of the good 10 per cent TiO₂ specimens, and one of the earlier 15 per cent TiO₂ specimens were retested. In each case, the conductivity varied only 0.01 Btu in./hr ft² °F from the initial data point, establishing that the apparatus was reporting accurate data.

It appeared, therefore, that considerably more effort would be required to establish the cause of the wide variation in product integrity and conductivity, as well as the manufacturing and quality control procedures necessary to insure a uniform product, if such were possible. It was concluded that the improved properties which might be realized did not justify what could be a relatively involved program. Consequently, further efforts to opacify Glasrock foam silica were halted.

Low Density Opacified Glasrock. Concurrently with the evaluation of the 10 per cent TiO₂ opacified Glasrock product, a special low density slab of unopacified Glasrock was made at approximately 18 pcf density in order to complete the conductivity vs silica density relationship initiated during the Seventh Quarter. Two 12 x 12 x 1-in. samples of unopacified Glasrock, 18.2 and 18.3 pcf density, were evaluated in the Rapid Heat Meter apparatus at 800F mean temperature. The values were 0.95 and 1.04 Btu in./hr ft² °F, respectively.

Summary of All Thermal Conductivity Data. All of the Rapid Heat Meter thermal conductivity data for both the opacified and unopacified Glasrock are plotted in Figure 3.2-1. Previously (Seventh Quarterly Progress Report, Figure 21), two separate regressions for the opacified and unopacified data were indicated, differing only slightly in slope, and crossing at about 25 pcf silica density. With the scatter of the most recent 10 per cent titania data, it was concluded that two significantly different regressions should not be drawn. Consequently, Figure 3.2-1 depicts a single conductivity vs silica density relationship for all data. Tables 3.2-1 and 3.2-2 show the specific data and specimen description from which Figure 3.2-1 was plotted. Specimens 18 and 19 of Table 3.2-1 represent re-runs of Specimens 10 and 16, respectively.

The cause for the apparent ineffectiveness of the opacifiers will not be investigated under this program. However, it can be speculated that it is related to the relative indices of refraction of opacifier and silica, and the

FIGURE 3.2-1

THERMAL CONDUCTIVITY VS SILICA DENSITY FOR
OPACIFIED AND UNOPACIFIED GLASROCK

TABLE 3.2-1

THERMAL CONDUCTIVITY AND DENSITY OF
OPACIFIED GLASROCK SPECIMENS

Specimen No.	Opacifier Amt and Type	Density, pcf			Conductivity @ 800F* Btu in/hr ft ² °F
		Product	Opacifier	Silica	
1	5% ZrO ₂	28.0	1.3	26.7	1.24
2	5% ZrO ₂	27.8	1.3	26.5	1.24
3	10% ZrO ₂	30.2	2.6	27.6	1.34
4	10% ZrO ₂	29.4	2.6	26.8	1.29
5	15% ZrO ₂	28.0	4.0	24.0	1.28
6	15% ZrO ₂	28.0	4.0	24.0	1.26
7	5% TiO ₂	29.2	1.5	27.7	1.38
8	5% TiO ₂	29.0	1.5	27.5	1.36
9	15% TiO ₂	29.6	4.2	25.4	1.28
10	15% TiO ₂	29.4	4.2	25.2	1.26
11	15% ZrSiO ₄	27.2	3.9	23.3	1.15
12	15% ZrSiO ₄	27.1	3.9	23.2	1.14
13	15% Cr ₂ O ₃	25.0	4.0	21.0	1.01
14	15% Cr ₂ O ₃	24.6	3.9	20.7	0.99
15	10% TiO ₂	27.7	2.8**	24.9	1.09
16	10% TiO ₂	27.7	2.8**	24.9	1.03
17	10% TiO ₂	27.1	2.7**	24.4	1.36
18	15% TiO ₂	29.4	4.2	25.2	1.25
19	10% TiO ₂	27.7	2.8**	24.9	1.04

*Nominal mean temperature - actual ranged between 770F and 805F

**Estimated

TABLE 3.2-2

THERMAL CONDUCTIVITY AND DENSITY OF
UNOPACIFIED GLASROCK SPECIMENS

<u>Specimen No.</u>	<u>Product Density</u> <u>pcf</u>	<u>Conductivity @ 800F*</u> <u>Btu in/hr ft² °F</u>
1	30.4	1.40
2	29.3	1.33
3	28.4	1.35
4	28.6	1.40
5	27.3	1.31
6	27.3	1.32
7	25.4	1.25
8	25.2	1.24
9	18.2	0.95
10	18.3	1.04

*Nominal mean temperature - actual ranged between 770F and 805F

physical location of the opacifier particles within the product. The opacifiers chosen for consideration are "scatterers" rather than "reflectors", and as such, their ability to opacify is a function of their high refractive index, or more specifically, the ratio of their refractive index to that of their immediate surroundings.

Glasrock is cellular in structure, and hence, it is not unreasonable to assume that most, if not all of the opacifier particles were partially or totally embedded in the silica cell wall. The advantage of their high index of refraction may therefore be negated considerably since their effectiveness must now be measured in terms of the refractive index of silica rather than of air (or space). For example, amorphous silica has a refractive index of approximately 1.5, while rutile titania has a refractive index of 2.6. In air, titania acts as an excellent opacifier; in silica, the ratio of the titania and silica indices may not be significantly different from the ratio of the silica and air indices.

This is a convenient hypothesis which would explain not only why the opacifiers in general did not improve the thermal conductivity of Glasrock, but also the reason for the greater spread of data for the opacified specimens (extent to which the particles were embedded in the cell wall). To the extent that this hypothesis has validity, however, it is undoubtedly over-simplified.

A second and more apparent difference between the 10 per cent titania specimens which differed significantly in thermal conductivity, although similar in composition, was the cell and wall size thickness of the specimens. A thin section from each specimen was prepared (in accordance with accepted techniques developed specifically for microscopic examinations) and magnified 50X. Photomicrographs of these sections are presented in Figures 3.2-2 and 3.2-3.

The difference in the pore size is immediately evident. Random measurements of the pores (which appear as bubbles) indicated that the specimen having the lower conductivity (Figure 3.2-2) had an average pore size of about 0.2 mm with a maximum size of about 0.5 mm. The other specimen (Figure 3.2-3) had an average pore size of about 0.3 mm with a maximum size of about 0.7 mm. Similar measurements of the wall thickness indicated 0.07 mm and 0.1 mm for the low and high conductivity material, respectively. The wall thickness measurements are obviously less reliable, however, since wall thickness was considerably more difficult to define.

These thin sections were also studied under a polarizing microscope at 500X magnification using transmitted light. This permitted the observation of the titania particles. This study indicated that the particles were well distributed throughout the walls of both specimens. Assuming an average titania particle size of 2 microns, essentially all of the particles would be entirely embedded in the 70 and 100 micron average wall thicknesses.

FIGURE 3.2-2

17.

THIN SECTION OF 10 PER CENT TITANIA OPACIFIED GLASROCK
HAVING A LOW THERMAL CONDUCTIVITY

MAG: 50X

THIN SECTION OF 10 PER CENT TITANIA OPACIFIED GLASROCK
HAVING A HIGH THERMAL CONDUCTIVITY

MAG: 50X

Thus, it appears that cell size, and possibly cell wall thickness, exert a major influence on the thermal conductivity of the Glasrock. The larger the cells, the fewer the number of interruptions to radiation afforded by the cell walls. Similarly, the thicker the walls, the less devious would be the path of heat flow by solid conduction. The latter factor, however, is probably considerably less important. This cell size theory does not invalidate the original speculation that the effectiveness of the titania as an opacifier is diluted due to its presence in a silica matrix. However, cell size probably represents the major cause of the spread in conductivity values between "similar" specimens.

A recheck (unaided eye) of the various unopacified specimens tested indicated that the cell size was quite uniform for samples within a given density range. The difference noted in the thin sections presented above was also evident qualitatively with the unaided eye. Thus, it appears that the presence of these "foreign" titania particles within the silica slurry resulted in an uncontrolled foaming action, and a subsequent variation in cell size. Until this can be more closely controlled, it would be difficult to determine the true effectiveness of the opacifier.

Thermal Conductivity of Unopacified Glasrock. Three 12 x 12 x 1-in. specimens of unopacified Glasrock were selected for thermal conductivity evaluation in the ASTM C-182 High Temperature Calorimeter apparatus for Insulating Firebrick. The specimens of 27.3, 27.4, and 29.0 pcf density were chosen to represent an average density about midway between the 25 to 30 pcf limits normally encountered in production. The average density was 27.9 pcf. The specimens were tested together in series and instrumented such that for given hot and cold face temperatures of the apparatus, three conductivity values at three different mean temperatures could be obtained simultaneously. The hot face temperature was controlled at three levels between 1000F and 1800F, thus giving a total of nine conductivity values ranging between 300F and 1600F mean temperature. Table 3.2-3 presents the pertinent data from this test series, while Figure 3.2-4 is a plot of the conductivity data vs the mean temperature.

The deviation of the data from a straight line regression was remarkably small. Similarly, the value of 1.38 Btu in./hr ft² °F at 800F mean temperature was in excellent agreement with the 1.36 value shown in Figure 3.2-1 at 28 pcf silica density.

Load-Bearing Capability of Unopacified Glasrock. In lieu of the evaluation of an opacified Glasrock product as originally anticipated, additional attention was given to the load-carrying ability of the unopacified material. Since the normal density range of the Glasrock 25 product is 25 to 30 pcf, unopacified specimens representing these approximate density limits were evaluated separately. Test loads ranged from 500 psi down to 200 psi in

TABLE 3.2-3
THERMAL CONDUCTIVITY OF UNOPACIFIED GLASROCK
AS DETERMINED BY ASTM C-182

Equipment Temperatures, °F		Specimen ΔT °F	Specimen Mean Temp, °F	Thermal Conductivity Btu in./hr ft ² °F
Hot Face	Cold Face			
1048	118	270	913	1.46
		291	633	1.26
		370	303	1.02
1403	138	350	1228	1.69
		393	856	1.42
		522	399	1.09
1809	168	440	1589	1.94
		498	1120	1.60
		702	519	1.17

FIGURE 3.2-4
THERMAL CONDUCTIVITY OF UNOPACIFIED GLASROCK 25 SILICA FOAM
UP TO 1600F MEAN TEMPERATURE

100 psi increments. The specimens, 2 x 2 x 1 in., were tested for up to 500 hr under constant load. The measure of performance was the permanent set imparted to the specimen as a result of the load. Since Glasrock is a relatively rigid ceramic, it was considered that any significant permanent set constituted a failure.

It was intended that the tests be conducted at 1800F hot face, thus simulating service conditions. However, frequent heater failures and long replacement delivery times necessitated that the last part of the test series be conducted at room temperature. A comparison of room temperature and 1800F hot face results at the 300 psi load level, however, indicated that the permanent set was no greater at 1800F than at room temperature, and that it may be even slightly less. Thus, the room temperature tests were considered sufficiently indicative of product performance to justify their conductance.

Table 3.2-4 is a description of all of the specimens tested. It will be noted that the permanent set was determined twice: "as tested", and "after scraping". Failure of Glasrock under compressive load resulted in a crushing of the cellular structure, and a subsequent accumulation of silica dust on the surface of the specimen. To establish just how much of the specimen was crushed as a result of the load, it was necessary to remove this layer of compacted dust.

It became apparent that significant crushing of the specimens occurred at loads greater than 200 psi, as shown by the difference in the thickness measurements after test. It was also noted that loose dust could be blown from the surface of the 30 pcf specimens after the 200 psi test, while no dust was evident on the 25 pcf specimens.

The existence of a measurable amount of silica dust initially constituted an arbitrary indication of failure to sustain the applied load. It was considerably more difficult, however, to catalogue a permanent set of up to 7 mils. Due to the ever-present loose particles of silica on the surface, repetitive measurements have shown that a plus or minus 0.002 in. thickness measurement variation might exist. However, the extent to which a 0.005 to 0.010 in. permanent set constitutes a failure can only be judged with a specific set of service conditions and requirements.

It is interesting to note that the 30 pcf (nominal) specimens consistently had a higher average permanent set than the 25 pcf (nominal) specimens at the 200 psi stress level. This may indicate that the lower density material is more "resilient", or that the cell walls tend to yield under load rather than fracture as in the case of the 30 pcf material. Intuitively, however, one might not expect such a difference in performance with a relatively small difference in density.

TABLE 3.2-4

PERMANENT SET OF 25 AND 30 PCF UNOPACIFIED GLASROCK SILICA FOAM

Specimen No.	Specimen Density pcf	Compressive Load psi	Test Time hr	Permanent Set, in.		Thermal Conditions
				As Tested	After Scraping	
25C-1	25.2	500	23-1/2	0.011	0.020	1800F hot face
25C-2	25.1	500	24	0.032	0.050	1800F hot face
30C-1	28.2	500	3/4	0.028	---	1800F hot face
30C-2	29.9	500	24	0.010	0.020	1800F hot face
25C-3	24.9	400	197	0.020	0.027	1800F hot face
25C-4	25.0	400	164	0.014	0.017	1800F hot face
30C-3	30.4	400	270	0.017	0.021	1800F hot face
30C-4	30.3	400	271	0.030	0.042	1800F hot face
25C-5	25.0	300	354	0.017	0.020	1800F hot face
25C-7	24.9	300	504	0.009	0.014	1800F hot face
30C-5	29.1	300	336	0.009	0.013	1800F hot face
30C-6	28.3	300	500	0.021	0.030	1800F hot face
30C-7	29.1	300	503	0.012	0.015	1800F hot face
25-0-1	25.6	300	504	0.009	0.013	cold
25-0-2	25.1	300	504	0.008	0.011	cold
30-0-1	27.2	300	504	0.025	0.038	cold
30-0-2	28.1	300	504	0.014	0.025	cold
25-0-3	25.2	200	504	0.002	0.002	1400F hot face
25-0-4	25.0	200	504	0.002	0.002	1400F hot face
25-0-5	25.1	200	504	0.002	0.002	cold
25-0-6	25.0	200	504	0.002	0.002	cold
25-0-7	25.0	200	502	0.002	0.002	cold
30-0-3	27.9	200	504	0.007	0.007	cold
30-0-4	28.9	200	504	0.005	0.005	cold
30-0-5	27.6	200	502	0.004	0.005	cold
30-0-6	29.1	200	502	0.002	0.005	cold
30-0-7	27.8	200	502	0.002	0.005	cold

Stress-Strain of Unpacified Glasrock. The stress-strain characteristics of unpacified Glasrock was determined on 2 x 2 x 1-in. specimens both before and after the prolonged 200 psi loading. As before, the specimens were separated into the two nominal density groups representing the normal production limits. Three fresh specimens at each density, and the ten specimens previously subjected to the 500-hr permanent set evaluation, were tested with a 1000-lb Tinius-Olsen test machine. This machine applies the load mechanically rather than hydraulically and therefore load application can be stopped instantaneously at any desired level. This feature was utilized for these tests at 12.5 psi load increments to facilitate the strain readings. Although the strain dial had 0.001 in. divisions, the readings were estimated to 0.00025 in. The hesitations at each load increment were limited to several seconds.

Figure 3.2-5 presents the stress-strain relationships for the four groups of specimens. The existence of two distinct slopes for each group is probably an indication of a surface consolidation, although one would probably expect such a phenomenon to be strain-dependent rather than stress-dependent. Each break in the regression occurred between 90 and 100 psi stress.

As expected, the fresh 30 pcf specimens (curve A) were slightly stronger than the fresh 25 pcf specimens (curve B). Similarly, the constant load of 200 psi resulted in irreversible internal damage, thus weakening slightly the 25 pcf specimens (curve C). However, the extent to which this took place for the 30 pcf specimens (curve D) was somewhat unexpected. Table 3.2-5 summarizes the modulus of elasticity for each group of specimens at the 0-100 psi and 100-200 psi stress levels.

As a further check of the effect of sustained loading on the subsequent stress-strain relationships, Table 3.2-6 shows the average permanent set of each group of specimens following the stress-strain test. This was determined two ways: immediately following the release of the load while the specimen was still in the compression fixture, and thickness measurements (external of the test fixture) before and after test. Although the two measurement techniques did not agree exactly, they both indicated the relative effect of the specimen history.

Cyclic Loading Up to 200 psi. Two 25 pcf (nominal) and two 30 pcf (nominal) specimens having no stress history, were subjected to cyclic loadings to determine if the sudden change in slope of the stress-strain curve was related to the permanent set, and if the material followed the same relationship after several cycles as it did on the first cycle (as shown in Figure 3.2-3). Each cycle was separated by a 15 sec period of zero load. All strain and permanent set measurements were taken in the test fixture. The cycles were:

1. Up to 75 psi in 12.5 psi increments
2. Up to 125 psi in 12.5 psi increments

FIGURE 3.2-5

STRESS - STRAIN OF UNOPACIFIED GLASROCK
AT 25 AND 30 PCF DENSITY

TABLE 3.2-5

MODULI OF ELASTICITY FOR UNOPACIFIED GLASROCK
BEFORE AND AFTER SUSTAINED 200 PSI LOADING

<u>Nominal Density</u> <u>psi</u>	<u>Previous History</u>	<u>Modulus of Elasticity, psi</u>	
		<u>0-100 psi Strain</u>	<u>100-200 psi Strain</u>
25	none	25,000	57,500
25	500 hr @ 200 psi	25,000	51,000
30	none	27,500	67,500
30	500 hr @ 200 psi	20,800	30,000

TABLE 3.2-6
 PERMANENT SET OF UNOPACIFIED GLASROCK SPECIMENS
 FOLLOWING THE STRESS-STRAIN EVALUATION

<u>Nominal Density</u> <u>psi</u>	<u>Previous History</u>	<u>Average Permanent Set, in.</u>	
		<u>In Fixture</u>	<u>External to Fixture*</u>
25	none	<0.0005	0.001
30	none	0.0005	0.001
25	500 hr @ 200 psi	0.001	0.002
30	500 hr @ 200 psi	0.003	0.004

* Thickness measured to nearest 0.001 in.

3. Up to 175 psi in 25 psi increments
4. Up to 200 psi in 25 psi increments

All four specimens had the same stress-strain relationship, they followed quite closely the 25 pcf relationship (curve B of Figure 3.2-5), and they appeared to deviate only slightly from this relationship on successive cycles. The permanent set also appeared to increase slightly after each cycle, although it achieved only 0.0005 in. after the final cycle. Thus, if any significant progressive changes did occur either in the relationship or the permanent set, the measuring devices were of insufficient accuracy to detect them.

IV. CONCLUSIONS

A. Phase I, Task 5 - Detailed Evaluation

1. A recheck of the Johns-Manville thermal conductivity equipment indicated it to be in complete working order, thus eliminating this factor as a possible source of the erratic data.
2. Thermal conductivity data obtained by Dynatech on MIN-K Formulation 2002 following 2000 hr at 1200F and 1 atm of argon compared well with the original Johns-Manville data in air, argon, and vacuum (prior to exposure), and the standard Guarded Hot Plate tests in air following exposure. This indicated that Formulation 2002 was not affected by the 2000-hr simulated service exposure.
3. Dynatech discovered that the relationship between the edge temperature of the test specimen and the arithmetic mean temperature may significantly effect the thermal conductivity measurement of MIN-K products. Since this relationship was not determined during the vacuum tests, the latest data in vacuum may still be somewhat in error.

B. Phase II, Task 3 - Load-Bearing Insulations for 400-1800F Service

1. Opacified Glasrock foam silica cannot be successfully made consistently without additional developmental effort. The apparently small improvement in thermal conductivity which might be achieved, however, did not warrant the further effort.
2. Unopacified Glasrock 25 (at 25 to 30 pcf) could not withstand static loads above 200 psi without resulting a significant amount of permanent set.
3. The modulus of elasticity of unopacified Glasrock 25 silica foam (with no stress history) ranged from 25,000 psi to 27,500 psi between 0 and 100 psi stress, and from 57,500 psi to 67,500 psi between 100 and 200 psi stress.
4. Prolonged exposure to 200 psi constant stress reduced the modulus of elasticity up to 55 per cent, with the greatest reduction occurring with the 30 pcf specimens between the 100 and 200 psi stress levels.

5. Cyclic loadings of unopacified Glasrock up to 200 psi showed no significant change in the stress-strain relationship within the accuracy of the measuring device used.
6. The thermal conductivity of unopacified Glasrock at 27.9 pcf density was a straight line relationship between 1.02 Btu in./hr ft² °F at 300F and 1.94 Btu in./hr ft² °F at 1600F.

V. NINTH QUARTER EFFORT

1. Complete the thermal conductivity evaluations of the MIN-K formulations developed under Phase I.
2. Evaluate the thermal conductivity of MIN-K Formulation 2002 at 35 pcf (load-bearing product) in air and argon.
3. Evaluate the thermal conductivity of unopacified Glasrock 25 silica foam in vacuum.
4. Prepare and submit the draft of the Final Report.