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AEC RESEARCH AND DEVELOPMENT REPORT
SPECIAL

AT(30-1)-3633
DEVELOP 1800F-400F FIBROUS-TYPE INSULATION
FOR RADIOISOTOPE POWER SYSTEMS

MICROTHERM 20 Cr
Report No. ALO-3633-5

June 9, 1967

J. O. Collins

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ABSTRACT

by

J. O. Collins

As part of the effort under AEC Contract No. AT(30-1)-3633, MICROTHERM 20 Cr was evaluated as a potential thermal insulation for use in RTG's.

Due to the similarity between this material and the Johns-Manville family of MIN-K products, Johns-Manville was requested by the AEC to assemble all of the data on MICROTHERM into one report.

This report fulfills that request.

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I. INTRODUCTION

MICROTHERM 20 Cr is one of several products developed by Boucher & Company, Kidderminster, England to compete with MIN-K for application in RTG units developed by the U. K. Companion products, samples of which have neither been seen nor evaluated, are MICROTHERM 18 Si and MICROTHERM 15C. After examination of MICROTHERM 20 Cr, it became obvious that the number in the product name represented the density in pound per cubic foot, while the letters were the chemical symbols for the opacifier.

A small sample (6-in. diameter x 1-in. thick) of MICROTHERM 20 Cr was obtained through the Johns-Manville London Office. Microscopic and chemical analyses were conducted on this sample. Subsequently, three pieces, 12 x 12 x 1-in., were received for further examination such as strength, stability, conductivity, etc.

All references to MICROTHERM in this report refer to MICROTHERM 20 Cr.

II. PHYSICAL AND CHEMICAL NATURE

A. MICROSCOPIC ANALYSIS

Petrographic examination of the 6-in. diameter by 1-in. thick specimens showed the following:

Bulky Silica	- Major
Chromic Oxide	- Minor
Amosite Fiber	- Approximately 5%
Glassy Fiber and Shot	- Approximately 5%

(Shot = unfiberized glassy material)

The amosite fiber showed a brown coloration, and an elevated refractive index typical of amosite which had been heated to 760C.

The ultimate particle size of the silica was approximately 0.03 microns as shown by the electron micrograph in Figure 1.

The chromic oxide had an average particle size of 1 micron with an approximate minimum of <0.3 microns and an approximate maximum of 6 microns. This oxide shows as black specks in Figures 2 and 3.

The amosite fiber ranged in diameter from <0.3 microns to 37 microns with an average of 2 microns. The length was from <10 microns to 1430 microns with an average of 200 microns. Figure 2 shows a typical example of an amosite fiber.

The glassy fibers ranged in diameter from 5.6 to 31 microns with an average of about 14 microns. A glassy fiber may be seen in Figure 3.

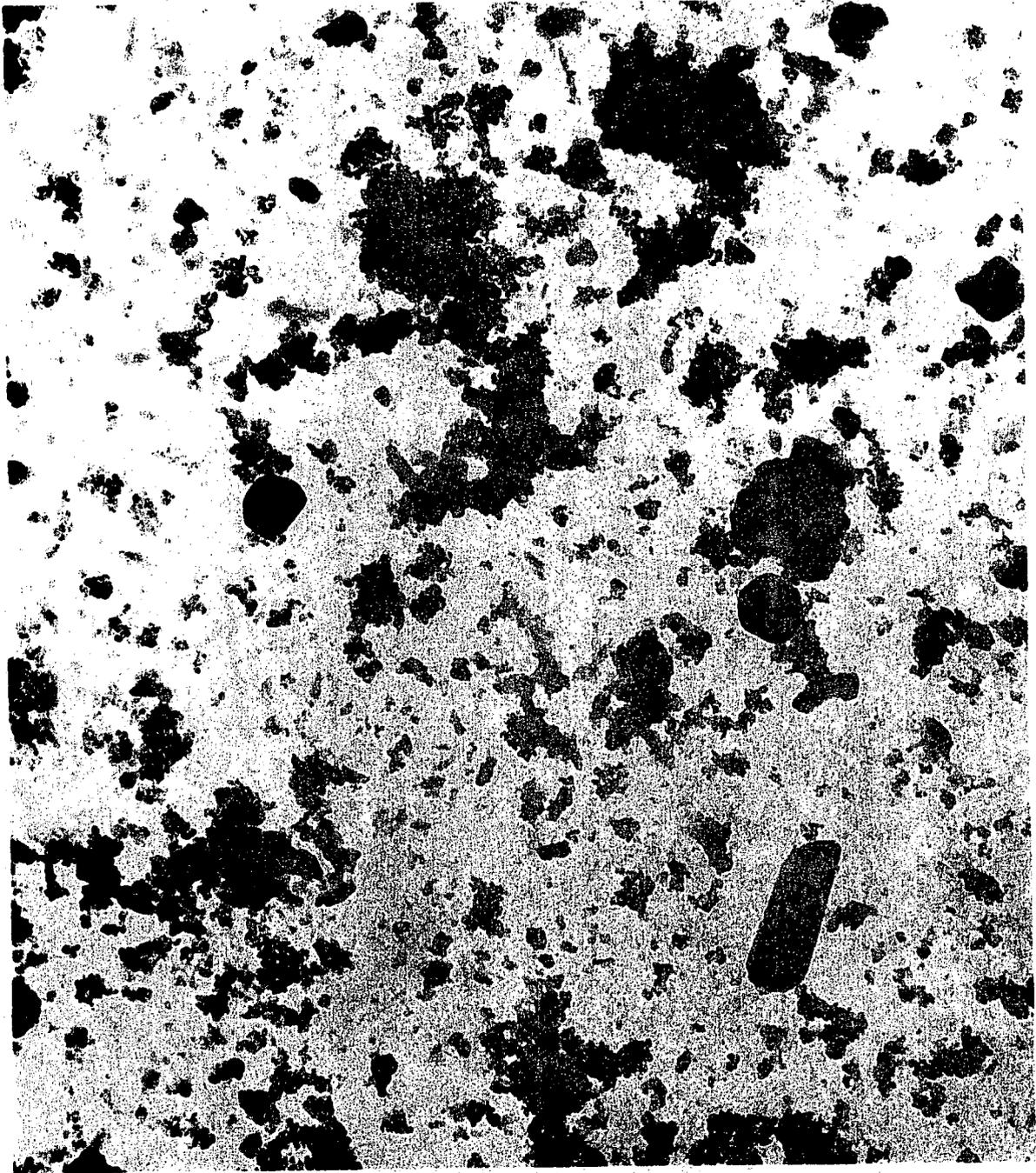
B. X-RAY DIFFRACTION

The X-ray diffractometer diagrams and the Debye-Scherrer films indicated a strong pattern of Cr_2O_3 . Additionally, the X-ray diagram of the dark brown fiber indicated it to be oxyamosite, or amosite which had been decomposed at temperatures between 500C and 800C.

C. SPECTROGRAPHIC ANALYSIS

The results of the spectrographic analysis are given in Table 1 below.

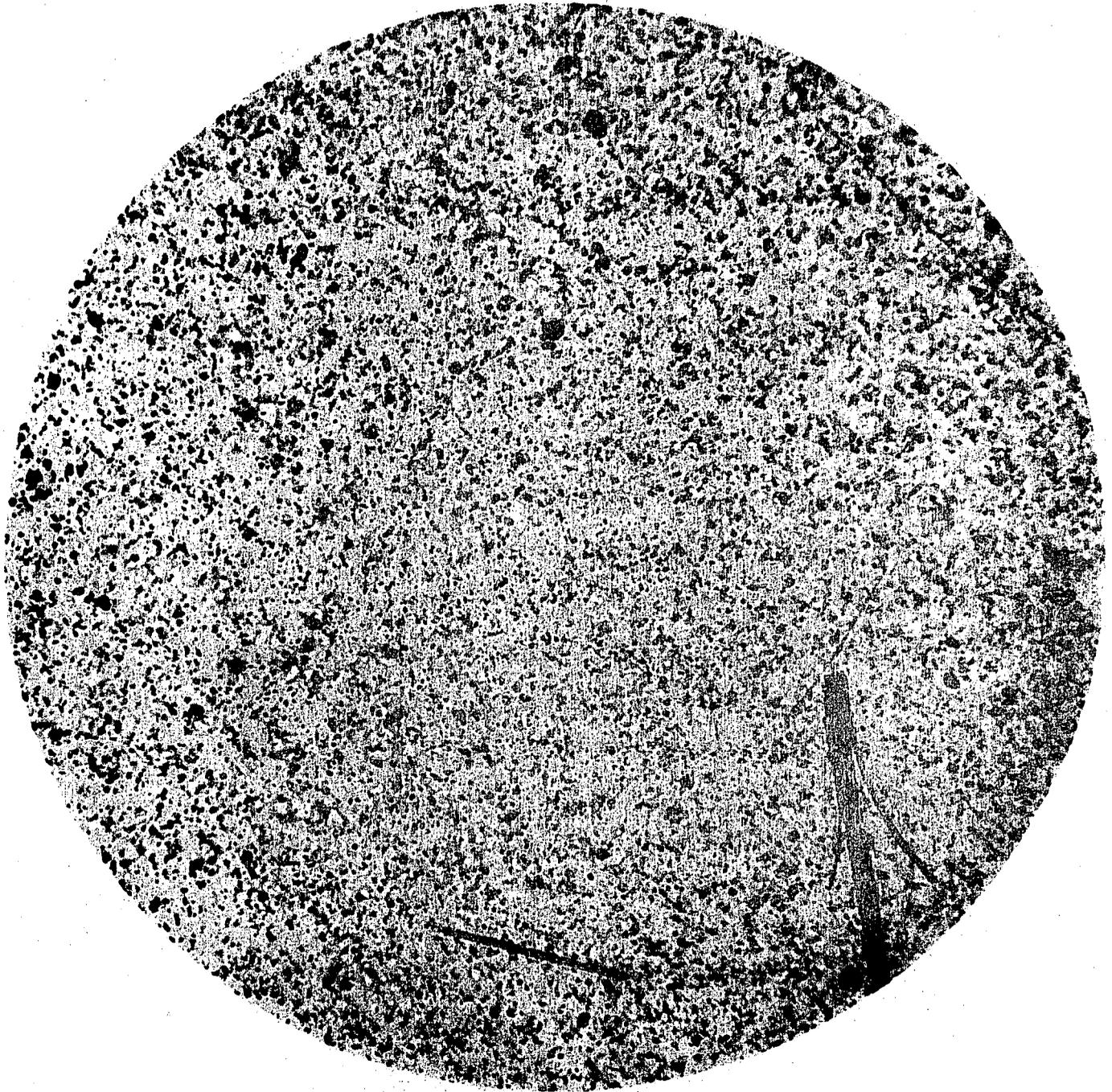
FIGURE 1.
ELECTRON MICROGRAPH OF THE SILICA PARTICLES
IN MICROTHERM (36,000X MAG.)



No. 463C

FIGURE 2.

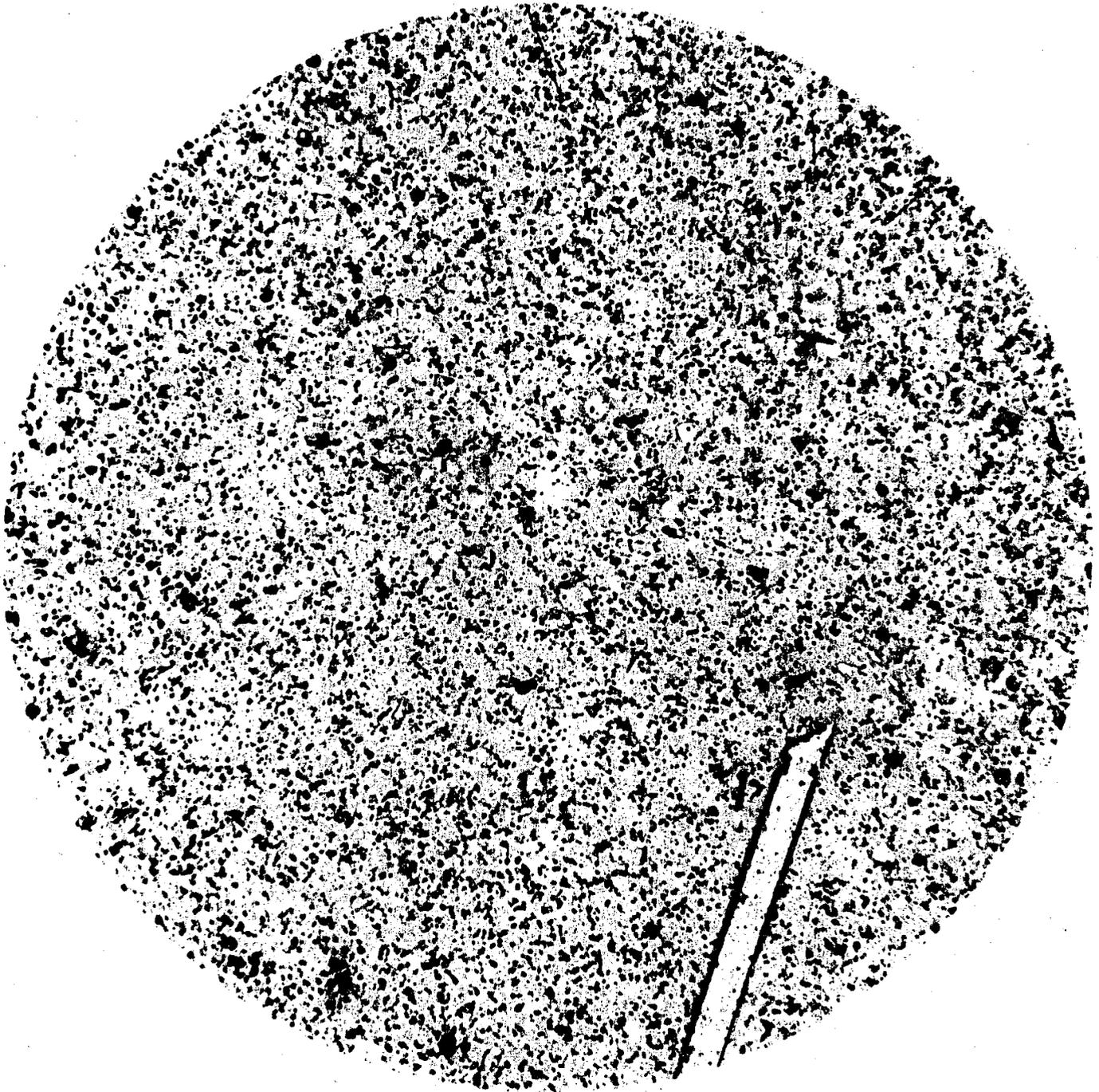
MICROTHERM AT 500X MAGNIFICATION SHOWING CHROMIC OXIDE
(BLACK SPECKS) AND AMOSITE FIBER



No. E-3497B

FIGURE 3.

MICROTHERM AT 500X MAGNIFICATION SHOWING CHROMIC OXIDE
(BLACK SPECKS) AND GLASSY FIBER



No. E-3497A

TABLE 1.
SPECTROGRAPHIC ANALYSIS OF MICROTHERM*

SiO ₂ (by difference)	62 <u>+2</u> per cent
Cr ₂ O ₃	32 <u>+2</u> per cent
Al ₂ O ₃	3.7
Fe ₂ O ₃	1.8
TiO ₂	0.26
MgO	0.24
Na ₂ O	0.1
Total ignition loss at 1800F - 1.9 per cent	

* All elements expressed as oxides on the ignited basis.

A spectrographic analysis of the glassy fiber and shot extracted from the MICROTHERM gave the qualitative analysis shown in Table 2.

TABLE 2.
SPECTROGRAPHIC ANALYSIS OF THE GLASSY FIBER
AND SHOT IN MICROTHERM

Al and Si	Major
Zr	Small
Ti	Trace
Fe	Not Detected
Na	Not Detected
B	Not Detected

III. PHYSICAL PROPERTIES

A. THERMOGRAVIMETRIC ANALYSIS

The thermogravimetric curve for MICROTHERM is given in Figure 4. The sample was preconditioned for 20 hr at 54 per cent RH and room temperature. An initial weight loss of 1.3 per cent at 120C represented adsorbed moisture, while the balance of the loss (up to 1.8 per cent) probably represented water chemically combined with silica.

A further qualitative examination indicated that an organic binder was not present in quantities greater than a few tenths of one per cent.

B. THERMAL STABILITY

Thermal stability, and all other physical properties, were determined on the three 12 x 12 x 1-in. samples.

Specimens nominally 2 x 4 x 1-in. were subjected to isothermal conditions of 1600F, 1800F and 2000F for 24 hr each. Three specimens were subjected to 1600F for 24 hr and measured for shrinkage. The same three, plus an additional three (no previous thermal history) were subjected to 1800F for 24 hr. Likewise, the six which had been exposed to 1800F, plus an additional three were subjected to 2000F for 24 hr. In all instances, the previous thermal history had no effect upon the per cent shrinkage, based upon the original dimensions. Table 3 below summarizes the results.

TABLE 3.
MICROTHERM SHRINKAGE AFTER 24 HOUR ISOTHERMAL EXPOSURE

<u>Temperature, °F</u>	<u>Number of Specimens</u>	<u>Average Shrinkage, %</u>		
		<u>Length</u>	<u>Width</u>	<u>Thickness</u>
1600	3	2	2	2
1800	6	4	5	14
2000	9	35	35	45

C. MODULUS OF RUPTURE

The strength of MICROTHERM was evaluated in accordance with the test procedure described in the Appendix. This evaluation was done on both the "as received" material, and on material which had been exposed to 1400F for 2 hr. Table 4 below summarizes the results.

FIGURE 4.

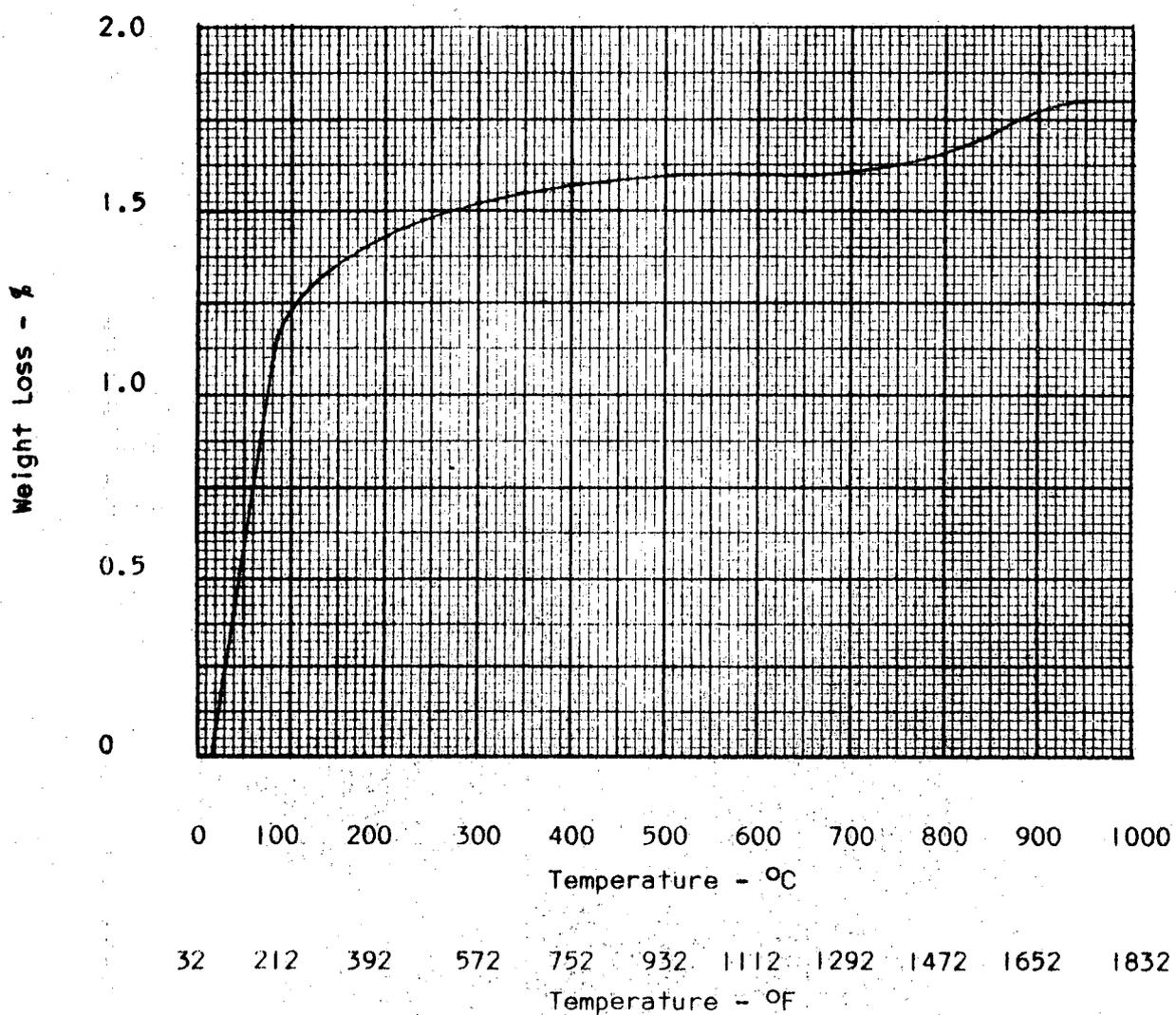
THERMOGRAVIMETRIC ANALYSIS OF MICROTHERM
(in air)

TABLE 4.
MODULUS OF RUPTURE OF MICROTHERM

<u>Thermal History</u>	<u>Number of Specimens</u>	<u>Average MR, psi</u>
None	4	16.5
1400F, 2 hr	4	20.7

Although the number of test specimens was limited, the effect of the firing cycle appeared evident. However, the low strengths verified pretest impressions that the material was relatively fragile which could create handling problems during generator assembly.

D. STRESS-STRAIN

Eight specimens, 4 x 4 x 1-in., were evaluated for stress-strain characteristics. Four were tested "as received" and four after 2 hr at 1400F. The tests were conducted on a Riehle Hydraulic Model KA-60 at a loading rate of 60 psi per minute, at room temperature, and with a 0.001-in. deflection dial gauge. The load was applied over the entire area with no lateral restraint provided the specimens during test. A static preload of 0.34 psi was applied to each specimen.

Figure 5 is a plot of the average stress-strain data obtained at 0.005 in./in. strain increments. In each group of four specimens, there was very little variation from the average, indicating good uniformity within a given 12 x 12 x 1-in. block. The curves appeared to consist of three straight-line segments. The first encompassed the 0 to 0.02 in./in. strain range, while the third went from 0.12 to 0.15 in./in. The first range was probably a result of thickness regain after molding, while the third may signify an internal fracture. The middle portion of the curve was used to calculate the modulus of elasticity values of 1000 psi and 1140 psi for unfired and fired specimens, respectively.

E. THERMAL CONDUCTIVITY

The thermal conductivity of MICROTHERM was evaluated in air using the Guarded Hot Plate apparatus (ASTM C-177). The results are given in Table 5 below.

FIGURE 5.

STRESS-STRAIN RELATIONSHIP OF MICROTHERM

Loading Rate of 60 psi/min
Tested at Room Temperature

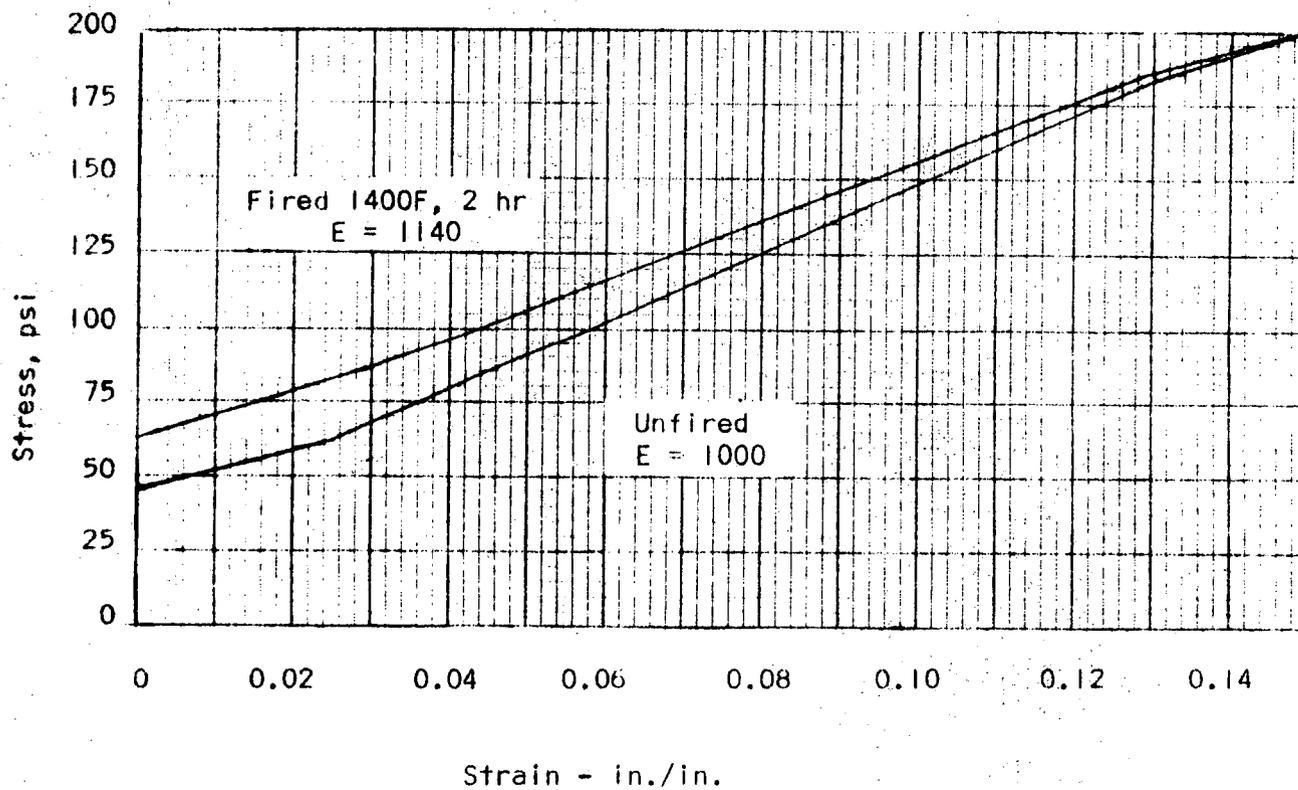


TABLE 5.
THERMAL CONDUCTIVITY OF MICROTHERM

<u>Mean Temperature, °F</u>	<u>Conductivity, Btu in./hr ft² °F</u>	
	<u>Actual</u>	<u>Published</u>
400	0.22(5)	0.20
500	0.23(0)	0.22
600	0.23(7)	0.23(5)
700	0.24(4)	0.25
800	0.25(1)	0.26(3)
900	0.26(3)	0.28
1000	0.27(7)	0.29(3)
1100	0.29(5)	

(The third decimal place is used to plot the thermal conductivity versus mean temperature curve, but is generally not considered significant for normal quoting purposes.)

It will be noted that the published values describe essentially a straight line, whereas the test values describe a curvilinear function as one would anticipate. There was also excellent agreement between MICROTHERM and MIN-K 1301 data.

IV. GENERAL COMMENTS

MICROTHERM is definitely similar to MIN-K and consists of very finely-divided silica opacified with about 32 per cent chromic oxide and reinforced with both amosite and alumina-silica fibers. It cannot be established definitely whether the amosite was fired prior to mixing with the other ingredients, or whether the MICROTHERM was fired after molding. The TGA curve would lead one to suspect the former due to the loss in weight above 120C.

The handleability appeared to be poor although no real test of its handleability was undertaken due to the relatively small amount of material available. Corners were easily chipped and broken even with careful handling. The low degree of integrity and handling strength may be due to any one or a combination of the following:

1. High opacifier content and hence lower silica.
2. Low fiber reinforcing efficiency per unit weight due to large fiber diameters.
3. A different silica material.

MICROTHERM has a strong dusting tendency as noted by an inability to "clean" cut edges of dust using a small fine hard brush.

Boucher appears to have excellent control over the molded thickness and the thickness regain (after molding). The nominal 1-in. thick blocks range from 0.988 in. to 1.011 in. thick. The average density of the MICROTHERM blocks, however, was slightly below 19 pcf.

Fiber distribution within the blocks also appear excellent with only a few scattered minute clumps visible to the eye. There were, however, very small pockets of silica indicating incomplete mixing of the silica with the opacifier. Neither of these small inconsistencies in distribution should affect the thermal efficiency of the product.

A cloth or fine mesh screen pattern was evident on both faces of the blocks indicating that molding was not done directly against machined die surfaces as is the case with MIN-K.

V. CONCLUSIONS

From a thermal efficiency standpoint, MICROTHERM 20 Cr appears to be as good as MIN-K 1301. Its other properties, however, appeared to be poorer than MIN-K 1301 and definitely less desirable than those of the new formulations developed under Contract AT(30-1)-3633.

On this basis MICROTHERM 20 Cr was not considered or evaluated further for potential use in radioisotope thermoelectric generators.

VI. APPENDIX

MODULUS OF RUPTURE TEST PROCEDURE

1.0 Purpose

The purpose of this test is to determine the flexural strength of preformed block-type thermal insulations such as MICROTHERM. This test does not necessarily yield an absolute value, but is useful in determining relative changes and/or the effect of formulation modifications upon:

- a. handleability
- b. fiber distribution
- c. fiber openness
- d. fiber reinforcing efficiency

2.0 Equipment

The equipment shall consist of the following:

- 2.1 Any form of testing machine capable of applying and measuring the required load. Load application shall consist of a single, self-aligning bearing edge approximately 1/2-in. diameter and 6-in. long.
- 2.2 Parallel, self-aligning cylindrical bearing edges approximately 1/2-in. diameter and 6-in. long whose distance between centers can be adjusted and accurately set to provide a given specimen span.

3.0 Specimens

The test specimens shall be nominally 4-1/2 x 4-1/2 x 1-in. in size, cut from any sized slab or block which is nominally 1-in. thick. The width and thickness of the specimen shall be measured to the nearest 0.001-in.

4.0 Test Procedure

- 4.1 Adjust the parallel bearing edges so as to provide a specimen span of 4.0 \pm 0.05-in.
- 4.2 Place the specimen on the parallel bearing edges such that the width dimension is parallel to the edges.
- 4.3 Center the loading edge such that it contacts the specimen midway (\pm 0.05-in.) between the bearing edges.

- 4.4 Counter-balance the scale to read zero load.
- 4.5 Apply the load at a rate not to exceed 250 lb/min.
- 4.6 Continue loading until a maximum load in pounds is indicated by the test equipment, indicating a failure of the specimen.
- 4.7 Record the maximum load attained in Step 4.6.
- 4.8 The modulus of rupture shall be calculated as follows:

$$MR = \frac{3Wl}{2bt^2}$$

where: MR = Modulus of Rupture, psi
W = maximum load in pounds
l = span (in this case, 4 in.)
b = width of specimen in inches
t = thickness of specimen in inches.