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1 June 1967

Westinghouse Astronuclear Laboratory

HOT PRESSED NIOBIUM CARBIDE- GRAPHITE COMPOSITES

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PROGRESS REPORT

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ABSTRACT

An investigation was conducted to determine the manufacturability of 75-25 w/o hot pressed composites. Commercially available and custom ground superfine carbide and graphite powder combinations were evaluated; the hot pressing operation was studied; and machining techniques were developed. The relationship of the physical properties of the starting powders to thermal and mechanical properties of the composites was studied. Room temperature and 2400°C tensile, compressive, and flexural strengths were determined; tensile creep at 2400°C and compressive deformation (creep) at 2200°C were studied; and thermal expansion and specific heat to 5000°F were determined. In addition, the effect of a post hot-pressing heat treatment was studied.

This evaluation has shown that equally dense, high strength composites can be obtained by hot pressing large or small billets. For the composition studied, the resulting structure of the superfine carbide graphite powders was a weak carbide continuous phase in a dominant continuous graphite phase. The resulting structure from commercially available carbide/graphite powders was a dominant carbide network. Optimum mechanical properties were obtained both at room temperature and at high temperature, using normal commercially available high purity powders. Thermal properties do not appear to be dependent on the physical properties of the powders. A post hot-pressing high temperature heat treatment is important in improving the dimensional stability of the billets. Ultrasonic material removal techniques developed for rough machining and diamond tooling developed for final machining provided an economical high production method for manufacturing composite hardware.

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I. INTRODUCTION

The number of structural materials that offer satisfactory performance diminishes rapidly as temperature requirements increase. Carbide and graphite have received increasing attention since they are among the most satisfactory refractory materials known.

Graphite is one of the most widely used high temperature materials. The machinability of graphite is excellent, and grades can be selected that have excellent thermal shock resistance. On the other hand, graphite has very poor high temperature hydrogen corrosion resistance. Protective carbide base coatings have been developed for improving the corrosion resistance, but because of the difference in thermal expansions, the performance of the coatings has not been entirely satisfactory.

Refractory metal carbides possess extremely high melting points and have excellent resistance to hydrogen corrosion. However, they have very poor thermal shock resistance and are most difficult to machine into hardware.

Composites of these two materials apparently offer the most attractive means for overcoming the poor corrosion resistance of graphite and the poor thermal shock resistance and machinability of refractory metal carbides.

This program was established to investigate some of the parameters involved in hot pressing dry mixes of graphite flour and refractory metal carbides at temperatures up to 3100°C.

Recent tests at LASL indicate that the corrosion resistance and thermal shock resistance of the 75-25 w/o niobium carbide-graphite composition are satisfactory for NRX-A requirements. The initial phase of this program is concerned with evaluating some of the process variables of this composition as they relate to the thermal and mechanical properties to develop economical fabrication and machining methods.

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II. EXPERIMENTAL PROCEDURES

A number of 75-25 w/o niobium carbide-graphite composites were hot pressed at 3000°C. The raw materials, blending procedures, hot-pressing methods, and procedures used for evaluation of the product are discussed in the following sections.

A. RAW MATERIALS

The powders selected consisted of two particle sizes of graphite flour and two sizes of niobium carbide powder for blending into three combinations or mixtures for hot pressing.

For the purposes of this investigation, the carbide powders are defined as normal or fine. "Normal" is commercially available powder with an average Fisher subsieve particle size of 2-4 microns, and the "fine" powder is a custom-ground powder with a particle size of less than 1 micron. The graphite flour is also designated as normal or fine. The normal graphite flour is commercially available flour meeting the requirements of WANL-PDS-30046, screened to -150 +325 mesh particle size. This flour is manufactured from graphite with a density of not less than 1.65 gm/cc and with impurities not exceeding 3 ppm of boron, 150 ppm of silicon, and 100 ppm of iron. The particle distribution of the flour as-received is presented in Table 1.

TABLE 1
GRAPHITE FLOUR PARTICLE DISTRIBUTION SIZE

Mesh Size	Percent by Weight
+65	2 max.
-65 +100	2-6
-100 +140	13-17
-140 +200	19-23
-200 +270	11-15
-270 +325	8-12
-325	33-37

The fine graphite flour was also manufactured from PDS-30046 material and reground to an average particle size of 0.129 BET. Actual impurity analysis was 260 ppm of iron, 75 ppm of silicon, and less than 1 ppm of boron.

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Four batches of the normal -325 mesh niobium carbide powder were used, one of which was reground into fine powder. The chemical and physical analyses are shown in Table 2.

TABLE 2

CARBIDE POWDER BATCH PHYSICAL AND CHEMICAL PROPERTIES

Powder Batch No.	Lot No. 1 (Normal)	Lot No. 2 (Normal)	Lot No. 3 (Normal)	Lot No. 3 (Fine)
Total Carbon (%)	11.19	11.22	11.03	12.26
Free Carbon (%)	0.06	0.07	0.15	1.3
Tungsten (ppm)	1400		3000	100
Tantalum (ppm)	3000	1500	3000	5000
Cobalt (ppm)	115		200	3
Titanium (ppm)	200	100	50	400
Iron (ppm)	300	400	300	150
Chromium (ppm)	15		300	10
Average Particle Size (Microns)	4.9(Fisher)	3.2(Fisher)	1.55(BET)	0.128(BET)
Density (gm/cc)	7.44	7.91	7.91	8.04
Surface Area (M ² /G)			0.49(BET)	5.85(BET)

B. BLENDING

Two methods of blending were employed. The mixtures of the commercial powders, designated as normal blends, were blended for 1/2 hour in a Patterson-Kelly twin-shell V blender equipped with an intensifier bar. The mixtures of the fine graphite and normal carbide powders, designated as fine, and fine graphite and fine carbide, designated as superfine, were blended in a Hobart blender. In order to prevent segregation of the heavy carbide particles during handling of the mixtures prior to hot pressing, the following procedure was used.

Acetone was added to the carefully weighed mixtures of powders prior to loading in the blender to obtain a slurry with the consistency of paint. The mix was then blended for about 2 hours. At the end of this time the mixture began to ball up, and there was little or no smell of acetone. It was then die pressed into 2-inch O.D. x 2-inch long billets and

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stored in plastic bags until hot pressing time. These cold pressed billets were then crumpled into fragments and loaded into the hot pressing die. Chemical analysis of two of the cold pressed blends and the dry normal blend is shown in Table 3.

TABLE 3
CHEMICAL ANALYSIS OF BLENDED POWDERS

Blender Type	Method	Powder Mix	% NbC
Hobart	Wet	Fine	75.46
Hobart	Wet	Superfine	75.34
Patterson-Kelly	Dry	Normal	75.14

C. HOT PRESSING

The fine and superfine blends of powder were hot pressed at the Y-12 research facility at Union Carbide Company's Nuclear Division in induction heating equipment shown in Figure 1. The dies were heated by radiation from an induction heated susceptor. Heatup time was 1-1/2 hours, dwell time was 45 minutes, and cool-down time was 6 hours. A grafoil washer was used on the pressing surface of each plunger to permit easy parting of the plunger from the hot pressed billet at the completion of the pressing cycle. One-half inch thick graphite cylindrical inserts extended die life to about six billets.

The blends of normal powders were hot pressed at the New Products Division of the Carborundum Company in induction-heated furnaces large enough to accommodate 6-inch round billets. The dies were also heated by radiation from an induction heated susceptor. The heating cycle was 2-1/2 hours to 3000°C, 45 minutes at temperature, and 24 hours of cooldown to room temperature.

Temperature measurement utilized the optical pyrometer method at both installations. Sighting was on the ATJ dies with 4200°C pyrometers through an argon-purged tube. Temperature calibration at carborundum established a correction factor of 140°C, an observed temperature of 2860°C indicating a true temperature of 3000°C. The correction factor at Y-12 was

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Figure 1. Typical Furnace Assembly at Y-12 Research Facility

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50°C, an observed temperature of 2950°C indicating a true temperature of 3000°C. Correction at Carborundum was 20°C to account for the transmission through the sight tube, and 120°C to compensate for the reflection loss of light through a quartz sight glass. At Y-12 no quartz sight glass was used, and a total correction factor of 50°C was used to account for the loss through the argon-purged sight tube.

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III. RESULTS AND DISCUSSION

A. HOT PRESSING EXPERIMENTS

1. As-Pressed Through-Holes

Initially, the composition used was 85 w/o niobium carbide - 15 w/o graphite. This composition was extremely difficult to machine by conventional machining methods. Since a major portion of the machining for the intended application consists of drilling through-holes, an experiment was conducted for molding undersize through-holes in the hot pressed billets.

A proprietary process was utilized in which graphite pins were carefully located in drilled holes in the top and bottom plungers; these pins were removed mechanically from the billet after hot pressing. A multi-hole and single-center-hole billet are illustrated in Figure 2. The true location of the holes was held within ± 0.004 inch, and the outside diameter was held to within ± 0.005 inch. This work was discontinued when the decision was made to use the 75 w/o NbC - 25 w/o graphite composition which is easier to machine.

2. Multi-Billet Hot Pressing

A multi-pressing technique was successfully developed for economically hot pressing at least six billets per hot pressing cycle.

A large die was manufactured with six equally spaced cavities. Six upper and six lower plungers were made. Six quantities of the blended normal 75 - 25 mixture were weighed to within 227 ± 0.5 grams, carefully loaded into the six die cavities, and hot pressed at 3000°C , all in a single pressing. The six billets were machined into 1.075-inch diameter x 2-inch long cylinders and weighed. The theoretical density varied from 94.4 to 95.0 percent. The experiment was repeated, and the theoretical density varied from 96.1 to 96.9 percent. Four additional billets, 2-1/4 inches in diameter and 2 inches long, were hot pressed in the same manner. The theoretical density varied from 95.0 to 95.9 percent.

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Figure 2. Single- and Multi-Hole Hot-Pressed Composite Billets

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3. Effect of Billet Size on Density

Another experiment was conducted to determine the effect of the size of individually hot pressed billets on the percent theoretical density. Since many small composite cylinders, approximately 1 inch in diameter and 1 inch long, are required for each reactor for cups and element tips, large hot pressed billets are desirable. A 3-1/4 inch diameter billet 3-1/4 inches long can supply up to twenty-one cylinders for cups or tips.

Two large 75 - 25 w/o billets, 3-1/4 inch O.D. x 3-1/4 inches long and consisting of normal powders, were individually hot pressed at 3000°C true temperature. Each billet was sectioned into eight rectangles, 0.5 inch x 0.5 inch x 3 inches long, four from each billet across the grain (in the direction of pressing) and four with the grain (90 degrees to the direction of pressing). Density determinations tabulated in Table 4 were equivalent to those of the multi-pressed or individually pressed small billets.

TABLE 4

DENSITY VARIATIONS IN 3 X 3 INCH HOT PRESSED BILLETS

Billet No.	Orientation	Density (gm/cc)	Percent TD
1	With grain	4.556	94.2
1	With grain	4.572	94.5
1	With grain	4.573	94.5
1	With grain	4.573	94.5
2	With grain	4.558	94.2
2	With grain	4.572	94.5
2	With grain	4.586	94.8
2	With grain	4.577	94.6
1	Against grain	4.595	95.0
1	Against grain	4.618	95.5
1	Against grain	4.567	94.4
1	Against grain	4.579	94.5
2	Against grain	4.560	94.3
2	Against grain	4.572	94.5
2	Against grain	4.551	94.1
2	Against grain	4.572	94.5
Average Percent Theoretical Density			94.5
Percent Variation			+1.0 -0.3

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4. Pressing Temperature Versus Percent Theoretical Density

A series of 75 - 25 w/o billets, 1-3/4 x 1-1/2 inches in size, were hot pressed from normal powders at 2950, 3000, 3050, and 3100°C. Results of density determinations as they relate to the temperature of hot pressing are shown in Table 5.

TABLE 5
HOT PRESSING TEMPERATURE VERSUS DENSITY

Temperature (°C)	Percent Theoretical Density
2950	91.3
3000	94.2
3050	95.1
3100	94.4

At 3100°C pressing temperature, radiographic examination disclosed the presence of a banded structure. At 3050°C and below, the structure was homogeneous and no banding was apparent. On the basis of density, it appears that the minimum hot pressing temperature is 3000°C.

5. Powder Blend Type Versus Density and Billet Size

Thirty billets of the 75 - 25 w/o composition were fabricated from the normal, fine, and superfine blends of powders. Billet sizes varied from 1-3/4 x 1-1/2 to 3 x 3 inches. The hot pressing temperature of the thirty billets was maintained at 3000°C; pressure was from 2500 psi to 3000 psi, and dwell time was 45 to 60 minutes. Table 6 shows the processing parameters and results of the density determinations for each of the billets.

Figure 3 shows a plot of the three powder blends versus the percent theoretical density and illustrates that the normal blend resulted in the highest density. The graphite powder size is probably not a density-controlling factor, but varying the carbide powder from a commercial size of 2 to 4 microns to an ultra-fine size reduced the density by 3 percent. For each billet size, densification of the fine carbide powder was approximately 3 percent lower than that achieved with the commercial carbide, regardless of the graphite powder size.

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Figure 3. Powder Type Versus Percent Theoretical Density of Hot-Pressed Material

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TABLE 6

DATA SUMMARY OF THIRTY BILLETS HOT PRESSED AT 3000°C

S/N	Pressure (psi)	Powder Type	Billet Size Dia. x Length (Inches)	Percent Theoretical Density
1	3000	Normal	3 x 3	93.7
2	3000	Normal	3 x 3	94.5
3	3000	Normal	3 x 3	95.0
4	2800	Fine	3 x 3	92.5
5	2800	Fine	3 x 3	93.8
6	2800	Superfine	3 x 3	89.3
7	2800	Superfine	3 x 3	89.6
8	3000	Normal	2-1/4 x 2-1/4	95.2
9	3000	Normal	2-1/2 x 2-1/2	93.5
10	3000	Normal	2-1/4 x 2-1/4	94.2
11	3000	Normal	2-1/4 x 2-1/4	94.7
12	3000	Normal	2-1/4 x 2-1/4	93.0
13	3000	Normal	2-1/4 x 2-1/4	94.5
14	3000	Normal	2-1/4 x 2-1/4	94.2
15	3000	Normal	2-1/4 x 2-1/4	94.8
16	2800	Fine	2-1/2 x 2	94.3
17	2800	Superfine	2-1/2 x 2	91.1
18	2800	Superfine	2-1/2 x 2	91.4
19	2500	Superfine	2-1/2 x 2	90.4
20	2500	Superfine	2-1/2 x 2	90.5
21	3000	Normal	1-3/4 x 1-1/2	95.3
22	3000	Normal	1-3/4 x 1-1/2	95.5
23	3000	Normal	1-3/4 x 1-1/2	94.0
24	3000	Normal	1-3/4 x 1-1/2	95.5
25	2800	Fine	1-3/4 x 1-1/2	93.2
26	2800	Fine	1-3/4 x 1-1/2	93.9
27	3000	Normal	1 x 1-1/2	94.4
28	3000	Normal	1 x 1-1/2	95.2
29	3000	Normal	1 x 1-1/2	95.0
30	3000	Normal	1 x 1-1/2	95.4

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Figure 4 shows the billet size versus the hot pressed density for the normal blend of powders. There is evidently little difference in the percent theoretical density for billet sizes varying from $1 \times 1\frac{1}{2}$ to 3×3 inches. It appears that for a diameter/length ratio of about one, there is no significant billet size effect on the hot pressed density. Although no small $1\frac{1}{2} \times 1\frac{1}{2}$ inch billets were fabricated from the superfine blend, this observation was also true for the 3×3 and $2\frac{1}{4} \times 2\frac{1}{4}$ inch hot pressed fine and superfine blends. As noted in the preceding paragraph, the density of the superfine powder billets was consistently about 3 percent below that of the normal and fine blends.

B. MICROSTRUCTURE

Hot pressed 75 - 25 w/o blends of the normal graphite resulted in a dominant continuous carbide phase as illustrated in Figure 5. A dominant graphite network with the grain was formed when fine graphite was used, regardless of carbide powder size, as illustrated. The fine graphite also resulted in a more anisotropic billet. This difference was confirmed by room temperature flexural tests with the grain and across the grain, as discussed later in Section D.

The superfine carbide powder agglomerated extensively during hot pressing, and actually resulted in carbide grains larger than those obtained with the large-grained normal carbide powders. It initially appeared that this effect was the result of not properly blending the carbide and graphite powders, but increased blending times did not result in any change in the microstructure.

Using graphite flour and carbide powder from a normal blend, eight billets were hot pressed under identical conditions, the composition varying from 71 - 29 to 85 - 15 w/o NbC-graphite in 2 percent increments. The microstructure for each billet with the grain is shown in Figure 6. Composition by weight percent and volume percent of the carbide graphite mixture is included.

The photomicrographs illustrate that with the grain a more dominant continuous phase of carbide begins to exist at a composition of 47 - 53 v/o (75 - 25 w/o). At the 50 - 50 v/o composition and above, the structure from a corrosion point of view appears to be the highly

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Figure 4. The Effect of Billet Size on the Density of Hot-Pressed Normal Powder

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Normal Blend - Transverse

Normal Blend Longitudinal

Fine Blend - Transverse

Fine Blend Longitudinal

Superfine Blend - Transverse

Superfine Blend - Longitudinal

Figure 5. Transverse and Longitudinal Microstructure of Three Combinations of Hot-Pressed Powders, 100X

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71-29 W/O
41-59 V/O

79-21 W/O
53-47 V/O

73-27 W/O
44-56 V/O

81-91 W/O
56-44 V/O

75-25 W/O
47-53 V/O

83-17 W/O
59-17 V/O

77-23 W/O
50-50 V/O

85-15 W/O
62-38 V/O

Figure 6. Microstructure of With-Grain Compositions of Hot-Pressed Normal Blends

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desirable weak graphite phase in a dominant carbide phase. The 41 - 59 v/o (71 - 29 w/o) composition is a weak carbide phase in a graphite matrix.

C. MACHINABILITY

A feasibility study of material removal methods for the 75 - 25 composites was conducted. Diamond tooling, carbide tooling, EDM, and ultrasonics were investigated.

One-inch rounds were trepanned from large 3-1/4 inch OD x 3-1/4 inch long billets by the EDM at a rate of feed of 1-3/10 inch per hour with electrode rotation and at a rate of 1 inch per hour without electrode rotation. Electrode wear in both instances was considered excessive, and surface finish was rated as poor. Diamond trepanning was at a rate of feed of 1 inch per minute, tool wear was insignificant, and a good surface appearance was obtained. Ultrasonics is a method by which an extremely high frequency reciprocating tool used in conjunction with a slurry of fine carbide suspended in water produces material cavitation. This method was used to trepan 1-inch cylinders at a rate of feed of 1/4 inch per minute. Surface finish was rated excellent although a small amount of tool wear was noted. Evidently, diamond tooling is preferable for trepanning rounds, while ultrasonics must be utilized for trepanning hexagonals or rectangles.

With carbide tooling, 0.100-inch diameter holes were drilled at a rate of feed of 1 inch in 3 minutes. The carbide drill dulled rapidly and had to be resharpened twice per inch of feed during this experiment. By the ultrasonic method, 0.100-inch diameter holes were trepanned at the same rate of feed.

From the above feasibility studies, it was concluded that specialized machining, utilizing diamond tooling, and ultrasonics are the prime material-removal methods for manufacturing hardware from the hot-pressed composite material.

However, many problems were encountered with the ultrasonic method while machining the first group of protection cups for reactor application. The most disturbing problem was maintaining concentricity. This problem, which appears to stem from fixturing of the part, has not been resolved.

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Tool wear caused undersized conditions after the fifth or sixth cup was machined. This may or may not be corrected by using harder materials for the tools. Material has been purchased to correct this situation. Another problem is tool breakage. Sufficient strength in the brazed area of the tool has been a problem in the past. Because of tool wear, it becomes necessary or more economical to braze tools on the toolholders rather than machine tool configurations on the toolholders. Induction brazing should permit controlled uniform heat and a stronger braze joint.

Another problem has been failure to get the abrasive slurry or the cutting agent down into a cavity or at the face of the tool, which caused the cutting speeds of the machine to be reduced. However, feeding the slurry up through the bottom of the workpiece by drilling a feed hole through each workpiece prior to the ultrasonic operation provided satisfactory cutting speeds.

Conventional single-point diamond tooling for machining composite material will probably always be necessary for certain machine operations. However, 90 percent of the material removal can be most expeditiously accomplished with ultrasonics. Figure 7 shows four stages of protective cup manufacture, i. e., the trepanned billet, the ultrasonically hogged out shape, the part finished with diamond tooling, and the microstructure of the coated part. Cutting this material has been greatly improved by improvising holding fixtures so that the ultrasonic cutting agent can be fed from the bottom of the fixture. This eliminates starving the tool of its cutting agent when sinking cavities such as those required in manufacturing the protective cup. Reverse feeding the cutting agent has improved cutting speeds. Substitution of low cost silicon carbide for the boron carbide slurry should result in a further cost improvement. Hard tooling is being designed and built for further improvement of efficiency. In addition, the development of ultrasonic multi-hole drilling techniques is being evaluated.

D. MECHANICAL PROPERTIES OF THE 75 - 25 W/O COMPOSITION

1. Flexural Strength

Flexural strength determinations were conducted at Southern Research Institute were made with the grain (90 degrees to the direction of hot pressing) and across the grain (parallel

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As-Pressed Hot Pressed Billet

Ultrasonically Rough Machined

Finished To Drawing With
Diamond Tooling

Coated Cup - 250X

Figure 7. The Four Manufacturing Phases for Protective Cups

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to the direction of hot pressing) at room temperature, at 2400°C, and at room temperature after a 2400°C stabilization heat treatment of the specimens. Four-point loading at 1/3 points was employed. The flexural, compressive, and tensile specimens are shown in Figure 8. Continuous load deflection curves were obtained on each run. The high temperature tests were made by heating the specimen in a graphite resistor furnace in an inert atmosphere. All of the tests were conducted at a loading rate of 10,000 psi per minute.

At room temperature the average flexural strength of the hot pressed normal powders was 10,440 psi with the grain and 6,075 psi across the grain. After a 2400°C heat treatment, the flexural strength with the grain showed no significant change, the average values being 10,521 psi with the grain and 5,825 psi across the grain. At 2400°C, the strength with the grain dropped to an average value of 8,800 psi, about 15 percent lower than that determined at room temperature. Across the grain at 2400°C, the strength increased to 7,760 psi, or 28 percent over that determined at room temperature without the heat treatment.

The average elastic modulus in units of 10^6 psi was 8.48 with the grain and 4.08 across the grain at room temperature. After a 2400°C heat treatment, the elastic modulus was 6.17 with the grain and 3.17 across the grain at room temperature. At 2400°C, the average elastic modulus was 3.37 with the grain and 2.05 across the grain.

Typical stress-deflection curves at room temperature, at 2400°C, and at room temperature after a 2400°C heat treatment are presented in Figures 9 through 12. Results of each test are shown in Table 7.

The hot pressed fine powders had an average flexural strength at room temperature of 7,540 psi with the grain, which is about 70 percent of that obtained with normal powders. Across the grain at room temperature, the average strength was 4,495 psi, again about 70 percent of the average strength of the hot pressed normal powders. At 2400°C the strength with the grain was 6,200 psi and the strength across the grain was 3,390 psi, these values being 70 percent and 50 percent, respectively, of the 2400°C values for the hot pressed normal powders.

The average elastic modulus for the fine powder billets at room temperature was 6.90×10^6 psi with the grain and 3.50×10^6 psi across the grain. At 2400°C, the values were 1.31×10^6 psi with the grain and 0.73×10^6 psi across the grain.

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Top and bottom surfaces flat and parallel to within $\pm 0.0005''$

Flexural Specimen

Tensile Specimen

Compressive Specimen

Figure 8. Tensile, Compressive, and Flexural Specimens

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Figure 9. With-Grain and Across-Grain Flexural Strength Versus Midpoint Deflection of Hot-Pressed Normal Powder at Room Temperature

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Figure 10. Flexural Stress With-Grain and Across-Grain Versus Midpoint Deflection for Hot-Pressed Normal Powders at Room Temperature after 2400°C Heat Treatment

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Figure 11. Flexural Stress With-Grain Versus Midpoint Deflection for Hot-Pressed Normal Powders at 2400°C

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Figure 12. Flexural Stress Across-Grain Versus Midpoint Deflection for Hot-Pressed Normal Powders at 2400°C

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TABLE 7

MECHANICAL PROPERTIES OF HOT-PRESSED NORMAL POWDERS

Property	Loading Direction	Temp. (°C)	Ultimate Strength (psi)	Elastic Modulus (10 ⁶ psi)	Total Strain (10 ⁻³ in./in.)	Poisson's Ratio
Tension	W. G.	2400	6,940	2.58	10.0	
			7,000	2.30	10.0	
	A. G.	2400	3,860	1.26	6.0	
		3,640	1.36	5.0		
W. G.	30	5,880	15.0			
		6,000	13.2			
Compression	W. G.	2400	14,000	4.0		0.29
			14,100	4.5		0.24
Flexure	A. G.	2400	19,100	2.2		0.38
			18,200	2.4		0.38
	W. G.	30	34,200	8.15		0.26
			W. G.	2400		
					9,000	3.25
	Tensile Creep	W. G.	2400	10,200	8.35	
Compressive Creep	A. G.	2200	11,480	8.62		
Tensile Creep	W. G.	2400	9,640	9.70		
Compressive Creep	A. G.	2200	10,700	6.63		
Tensile Creep	W. G.	2400	10,300	5.71		
Compressive Creep	A. G.	2200	8,070	1.9		
Tensile Creep	W. G.	2400	7,450	2.2		
Compressive Creep	A. G.	2200	5,230	4.32		
Tensile Creep	W. G.	2400	6,920	3.84		
Compressive Creep	A. G.	2200	5,410	3.83		
Tensile Creep	W. G.	2400	5,740	3.60		
Compressive Creep	A. G.	2200	Stress Level (psi)		Creep	
					Strain (10 ⁻³ in/in)	Time (Minutes)
			2,000	2.18	23	46
			2,000	2.78	24	52
Compressive Creep	A. G.	2200	Stress Level (psi)		Percent Creep	
Compressive Creep	A. G.	2200	2,000		0.43	
			4,000		1.3	

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Typical stress-strain curves at room temperature and 2400°C are shown in Figures 13, 14, 15, and 16. Results of each test are shown in Table 8.

For the hot pressed low density superfine powders, the average flexural strength at room temperature was only 5,200 psi with the grain, which is 50 percent of the value for the normal powders; at 2400°C the average strength increased slightly to a value of 5,680 psi, which is about 70 percent of the value for the normal blend. The average elastic modulus was about the same as for the hot pressed fine blend.

Figure 17 shows the room temperature and 2400°C flexural properties of these blends of powder. The normal blend was anisotropic at room temperature with a ratio of with-grain to across-grain values of 1.7; however, at 2400°C the material was almost isotropic with a ratio of 1.1. The fine material was anisotropic at both room temperature and 2400°C with a ratio of 1.65 at room temperature and 1.80 at 2400°C.

2. Tensile Strength

Room temperature and 2400°C tensile tests were conducted at SRI. The tensile specimen is shown in Figure 8.

The ultimate strength, elastic modulus, and total strain were determined. The tensile testing apparatus included a tensile frame with two spherical gas bearings, two optical strain analysers for monitoring strain in both the axial and lateral directions, and a graphite resistance furnace for the high temperature tests. The furnace and specimen were purged with helium to provide an inert atmosphere. Ports with visual openings were provided on opposite sides of the furnace to allow viewing the gage flags on the specimen and for optically tracking the motion of the gage points through the strain analysers. Specimen temperature was determined by optical pyrometer readings taken through another small sight port containing a sapphire window.

At room temperature the average tensile strength of the hot pressed normal powders with the grain was 5,940 psi. No across-grain determinations were made. At 2400°C the

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Figure 13. Flexural Stress With-Grain and Across-Grain Versus Midpoint Deflection for Hot-Pressed Fine Powders at Room Temperature

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Figure 14. Flexural Stress Across-Grain Versus Midpoint Deflection for Hot-Pressed Fine Powders at 2400°C

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Figure 15. Flexural Stress With-Grain Versus Midpoint Deflection for Hot-Pressed Fine Powders at 2400°C

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Figure 16. Flexural Stress With-Grain and Across-Grain Versus Midpoint Deflection for Hot-Pressed Superfine Powder at 2400°C

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Figure 17. Flexural Stress Versus Temperature for Normal, Fine, and Superfine Blends of Hot-Pressed Powders

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TABLE 8
MECHANICAL PROPERTIES OF HOT-PRESSED FINE AND SUPERFINE POWDERS

Powder	Property	Loading Direction	Temp. (°C)	Ultimate Strength (psi)	Elastic Modulus (10 ⁶ psi)	Total Strain (10 ⁻³ in./in.)
Fine	Tension	W. G.	2400	3,600	0.72	-
				3,380	0.79	11.0
	Flexure	W. G.	30	7,540	6.90	6.0
				6,020	1.30	
		A. G.	30	6,400	1.32	7.0 8.0
				4,300	3.27	
				4,660	3.74	
				2,990	0.67	
		A. G.	2400	3,790	0.80	
Superfine	Compressive Creep	A. G.	2200	Stress Level (psi)	Percent Creep	
				4,000	3.4	
				6,000	6.3	
	Flexure	W. G.	30	5,300	6.14	5.0
				5,100	5.45	6.0
				5,810	1.06	
				5,550	1.19	

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average tensile strength with the grain was 6,970 psi, and across the grain was 3,750 psi. The ratio of anisotropy was 1.8, which was not in good agreement with the 1.1 value obtained from the 2400°C flexural test. The average elastic modulus was 14.1×10^6 psi at room temperature. Typical 2400°C stress-strain curves are shown in Figures 18 and 19. Test data are summarized in Table 7.

Tensile tests at 2400°C with the grain were made on the hot pressed fine powders. The average value with the grain was only 3,490 psi, or about 50 percent of that obtained with the normal powders. The data are summarized in Table 8. A typical stress-strain curve is shown in Figure 20.

3. Tensile Creep

Two 2400°C tensile creep tests on the hot pressed normal blends were conducted at SRI with the grain at a stress level of 2,000 psi, which was approximately 25 percent of the ultimate strength. The creep in both cases was at a rate of 0.0005 inch per minute, failure occurring after 46 minutes on one test and 52 minutes on the other. A typical creep strain versus time curve is shown in Figure 21. The data are summarized in Table 7.

4. Compressive Strength

Compressive strength and Poisson's ratio were determined at SRI at room temperature and 2400°C on the hot pressed normal blend billets. The specimen design is shown in Figure 7. Since the problem of specimen alignment in compression is nearly as critical as that for uniaxial tension, the gas-bearing apparatus was used.

At room temperature the average of two compressive strength determinations with the grain was 34,200 psi, and the elastic modulus was 8.15×10^6 . At 2400°C the average strength with the grain was 14,050 psi and across the grain was 18,900 psi. Typical compressive stress strain curves are shown in Figures 22 and 23, with the data tabulated in Table 7.

The average Poisson's ratio at 2400°C was 0.26 with the grain and 0.38 across the grain. Typical curves of lateral strain versus axial strain in compression at 2400°C are shown in Figures 24 and 25. The data are tabulated in Table 7.

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Figure 18. Tensile Stress Versus Axial Strain for Hot-Pressed Normal Powders Across-Grain at 2400°C

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Figure 19. Tensile Stress Versus Axial Strain for Hot-Pressed Normal Powders With - Grain at 2400°C

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Figure 20. Tensile Stress Versus Axial Strain for Hot-Pressed Fine Powders With-
Grain at 2400°C

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Figure 21. Tensile Creep Strain at 25 Percent of Ultimate Strength Versus Time at 2400°C for Hot-Pressed Normal Powders

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Figure 22. Compressive Stress Versus Axial Strain for Hot-Pressed Normal Powders Across-Grain at 2400°C

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Figure 23. Compressive Stress Versus Axial Strain for Hot-Pressed Normal Powders With-Grain at 2400°C

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Figure 24. Lateral Strain Versus Axial Strain in Compression for Hot-Pressed Normal Powders at 2400°C

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Figure 25. Lateral Strain Versus Axial Strain in Compression for Hot-Pressed Normal Powders Across-Grain at 2400°C

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E. COMPRESSIVE CREEP RESISTANCE

Right-cylindrical specimens, 1/2 inch in diameter and 1 inch long, of the hot pressed normal and fine powders were tested at WANL in compression with a sustained load between graphite plates in a graphite resistance-heated furnace under an argon atmosphere. The specimen length was carefully measured before and after the run.

Tests were conducted at 2200°C for 1 hour on across-grain specimens at 2000, 4000, and 5000 psi. At a stress level of 4000 psi, the normal powder exhibited a resistance to compressive creep almost three times that of the fine powder material. The deformation (creep) of the normal material was 1.3 percent, while the deformation of the fine powder material was 3.4 percent. The ratio appeared to exist at all loadings as illustrated in Figure 26, a log plot of the compressive creep rates of deformation.

F. THERMAL PROPERTIES OF THE 75 - 25 W/O COMPOSITION

1. Thermal Expansion of As-Hot-Pressed Billets

Determinations of the triaxial thermal expansion of the hot pressed normal and fine powders were conducted at SRI from room temperature to 5000°F in a helium atmosphere, using graphite dilatometers in a high-temperature furnace. The weights, lengths, and diameters of the specimens were measured before and after the determinations.

Two across-grain specimens (taken in the "c" direction) and four with - grain specimens, two at 90 degrees to the other two (taken in the "a" and "b" directions), were used.

The thermal expansion of both materials was similar. The values were nearly identical in the two directions with the grain (the plane perpendicular to pressing). In this direction the material expanded almost linearly to 5000°F, a total of 17.5×10^{-3} inch per inch. The coefficient of thermal expansion in this direction increased from about 3.3×10^{-6} inch/inch/°F at room temperature to approximately 4.2×10^{-6} inch/inch/°F at 5000°F. There was a permanent reduction in length of the 2.755-inch long specimens of 0.003 inch, or about 0.12 percent.

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Figure 26. Compressive Deformation of Hot-Pressed Normal and Fine Powders Across-Grain at 2200°C

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The across-grain (parallel to the direction of pressing) thermal expansion of both materials was also similar, but higher than the with-grain values. The total expansion at 5000°F was 27×10^{-3} inch per inch. The coefficient of thermal expansion in this direction increased from about 3.3×10^{-6} inch/inch/°F at room temperature to approximately 10.5×10^{-6} inch/inch/°F at 5000°F. There was a permanent increase in length of the 2.755-inch specimen after exposure at 5000°F and cooldown to room temperature of about 0.015 inch, or about 0.5 percent permanent growth.

The thermal expansion data for the hot pressed normal powder are shown in Figure 27, and for the hot pressed fine powder in Figure 28.

2. Thermal Expansion of Thermally Stabilized Hot-Pressed Billets

The thermal expansion of the hot-pressed normal powders was measured triaxially in several successive determinations at SRI. The first determination was made with a quartz dilatometer at 800°F in air. Each specimen was then evaluated in three successive determinations with graphite dilatometers to 5000°F in helium.

The thermal expansion data for the three specimen orientations are shown in Figures 29, 30, and 31. The expansions in the with-grain direction (the "a" and "c" orientations) were similar and slightly lower on the first exposure to 5000°F than on subsequent exposures. There was only a slight permanent shrinkage after exposure to 5000°F and then additional but less shrinkage on the successive runs. In the longitudinal ("b") direction the expansion was about 13 percent higher on the first exposure to 5000°F than on subsequent runs. After the initial expansion to about 29×10^{-3} inch per inch at 5000°F, the expansion was consistent on subsequent runs, or about 25×10^{-3} inch per inch. There was about 0.5 percent permanent growth on the first exposure, which checks with prior thermal expansion data, and about 0.1 percent on each subsequent exposure.

3. Heat Capacity

Heat capacities were derived from the slope of the enthalpy versus temperature curves determined with adiabatic and ice calorimeters at SRI. The adiabatic calorimeter was used to measure enthalpy at specimen temperatures below 800°F. The heated specimen was dropped into a

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Figure 27. Thermal Expansion of Hot-Pressed Normal Powders

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Figure 28. Thermal Expansion of Hot-Pressed Fine Powders

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Figure 29. Thermal Expansion of Hot-Pressed Normal Powders With-Grain (Direction "a") on Several Successive Determinations

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Figure 30. Thermal Expansion of Hot-Pressed Normal Powders With-Grain (Direction "b") on Several Successive Determinations

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Figure 31. Thermal Expansion of Hot-Pressed Normal Powders Across-Grain (Direction "c") on Several Successive Determinations

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thermally guarded, calibrated cup; and the enthalpy was measured as a function of the temperature change of the cup. The measurements were referenced to zero enthalpy at 85°F, using a linear interpolation. The values were then referenced to 32°F.

The drop-type ice calorimeter was used over the temperature range from 1000°F to 5000°F. In this calorimeter the specimen container is surrounded by a frozen ice mantle. The enthalpy was determined by measuring the volume of ice melted as the specimen cooled from the drop temperature to 32°F.

The heat capacities were determined graphically from the slopes of the enthalpy versus temperature curves, and also by differentiating an equation for enthalpy obtained using the least squares method.

Results of these determinations for both the normal and fine blends of powders showed no dependence of enthalpy on the physical characteristic of the starting powders. The enthalpies and heat capacities are shown in Figure 32. A summary of the specific heat data is shown in Table 9.

TABLE 9

HEAT CAPACITY VERSUS TEMPERATURE

Temperature (°F)	Heat Capacity (Btu/lb/°F)
70	0.110
1000	0.185
2000	0.213
3000	0.230
4000	0.235
5000	0.239

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Figure 32. Enthalpy and Heat Capacity of Normal and Fine Hot-Pressed Powders

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IV. CONCLUSIONS AND RECOMMENDATIONS

The following are the main conclusions resulting from this evaluation of the manufacturability and thermomechanical responses of hot pressed 75 - 25 w/o NbC-graphite composites:

- 1) Large billets, 3-1/4 inch OD x 3 inches long, can be hot pressed to a quality equivalent to that produced in small billets, 1-1/2 inch diameter x 2 inches long. The large billet concept is an economical fabrication method for manufacturing many small components.
- 2) The minimum hot pressing temperature appears to be 3000°C. The maximum hot pressing temperature was previously established as about 3200°C, above which eutectic melting occurs.
- 3) The normal blend of powders, -150 +325 graphite flour and -325 NbC powder, resulted in hot-pressed billets with the best mechanical properties.
- 4) Thermal expansion and heat capacity are a function of composition, the behavior being independent of the size distribution of the starting powders.
- 5) The particle sizes of both the carbide and graphite have a pronounced effect on the microstructure. The normal blend showed a more dominant continuous carbide phase structure that is highly desirable for corrosion resistance.
- 6) Procedures and methods were established for machining components. The combination of ultrasonics for rough machining and diamond tooling for the finishing operations was most effective in producing close-toleranced parts economically and at a high rate of production.
- 7) A post hot-pressing high temperature heat treatment is important in achieving dimensional stability prior to exposing hardware to elevated temperatures. No apparent degradation of the mechanical properties was observed.

Recommendations for future work on this subject are as follows:

- 1) Continued development of the ultrasonic material removal method for finish contouring and multi-hole trepanning (drilling).
- 2) Development of methods and procedures for calibration and verification of hot-pressing temperature systems.

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- 3) Development of improved temperature measurement monitoring of the hot-pressing operation.
- 4) Determination of the practical optimum hot-pressing temperature range.
- 5) Investigation of isostatic hot pressing.

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