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Carbide-Graphite Composites
Second Progress Report

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K. V. Davidson
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CARBIDE-GRAPHITE COMPOSITES

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K. V. Davidson, R. E. Riley, and J. M. Taub

ABSTRACT

This report is a continuation and extension of IA-3569-MS. Additional data on the effect of composition, binder content, and contaminants in the as-received powders are presented. The effect of pressure on deformation of extruded composites at high temperatures is discussed. Also presented are preliminary determinations of the effect of composition of TaC-C composites, TaC-NbC solid solution, and composites of TaC-NbC mixtures. Preliminary data on the dynamic elastic properties and electrical resistivity of NbC-C composites are given.

INTRODUCTION

The data for the previous work⁽¹⁾ were obtained primarily on hot pressed samples of a single lot (NbC-9) of NbC powder. However, subsequent work involving several lots of commercial NbC powders showed the need for further efforts in powder characterization to develop a procurement specification. The effect of composition and binder content, as well as the effect of graphite particle size, was investigated further.

Preliminary investigations were started on the effect of composition of TaC-C composites. In addition, a brief exploration was made of the TaC-NbC solid solution and the effect of composition of a solid solution-graphite composite system. The effects of pressure at elevated temperatures on the low density extruded composite were investigated.

RAW MATERIALS

The raw materials used in this investigation included several lots of NbC and a single lot of TaC powder. In addition, Varcum binder and two types of graphites were employed.

Niobium Carbide (NbC) Powder

The NbC powders were obtained from four sources. The chemical analyses of these various lots are presented in Table I. The powder characterization data are presented in Table II.

Tantalum Carbide (TaC) Powders

The TaC powder, Lot TaC-13A, was purchased from Kawecki Chemical Company. The Fisher average particle size was 2.0 μ , the bulk density was 4.70 g/cc, and the tap density was 6.91 g/cc. Chemical analysis showed it to contain 92.7 $\pm$ 0.3% Ta, 6.15% total carbon, less than 500 ppm free carbon, and 0.25% oxygen.

M-2 Graphite Flour

The standard graphite flour used in this program was grade 1008, supplied by Great Lakes Carbon Company. This material was procured to a controlled particle size distribution specification. A typical chemical analysis was 0.03% ash, 100 ppm Fe, 150 ppm Si, and all other elements less than 200 ppm total.

TABLE I

CHEMICAL ANALYSES OF SELECTIVE NbC POWDER LOTS*

Elements	Lot 9	Lot 10E	Lot 13	Lot 13**	Lot 14	Lot 15	Lot 17	Lot 17AR**	Lot 6
Nb	88.4%	88.5% ± 0.2%	88.5 ± 0.2%	88.9%	87.6%	87.3%	87.1%	88.5%	88.7%
Total C	11.08%	11.7%	10.77%	10.9%	11.4%	11.32%	11.5%	11.4%	11.2%
Free C	<0.05%	0.58%	0.077%	<0.05%	0.09%	0.14%	0.23%	0.2%	0.21%
O ₂	0.089%-0.21%	0.068%-0.11%	0.26%	0.25%	0.07%	0.12%	0.14%	0.14%	0.64%
Li	<3	<3	<3	<3	<3	<3	<3	<3	<3
Be	<1	<1	<1	<1	<1	<1	<1	<1	<1
B	<1	<1	<1	1	<1	<1	<1	<1	<1
Na	<3	<3	<3	<3	<3	<3	<3	<3	<3
Mg	<1	<1	<1	<1	<1	30	<1	<1	<1
Al	<10	30	<10	<10	<10	<10	200	<10	<10
Si	<10	<10	<10	30	20	<10	<1	30	<10
K	<30	<30	<30	<30	<30	<30	<30	<30	<30
Ca	20	<5	<5	<5	<5	300	10	<5	<5
Ti	300	<10	<10	<10	500	<10	<10	<10	<10
V	<50	<50	<50	<50	<50	<50	<50	<50	<50
Cr	200	<10	<10	<10	30	30	<10	<10	<10
Mn	3	<3	50	<3	30	<3	30	3	<3
Fe	500	1000	3000	100	300	500	3000	500	50
Co	20	<10	<10	<10	30	30	<10	<10	<10
Ni	50	<10	10	<10	30	50	<10	<10	<10
Cu	<10	<10	10	<10	<10	10	10	<10	<10
Zn	<300	<300	<300	<300	<300	<300	<300	<30	<300
Sr	<30	<30	<30	<30	<30	<30	<30	<30	<30
Zr	<100	<300	<100	100	<100	<100	500	<100	<300
Mo	<100	<100	<100	<100	<100	<100	<100	<100	<100
Ag	<1	<1	<1	<1	<1	<1	<1	<1	<1
Cd	<3	-	-	-	-	-	-	-	-
Sn	<50	<50	<50	<50	50	<50	<50	<50	<50
Ba	<10	<10	<10	<10	<10	<10	<10	<10	<10
Ta	<500	<500	<500	<500	<500	<500	<500	<500	<500
W	-	<1000	<1000	<1000	<1000	<1000	<1000	<1000	<1000
Pb	<30	<30	<30	<30	<30	<30	<30	<30	<30
Bi	<10	<10	<10	<10	<10	<10	<10	<10	<10

* All values ppm unless otherwise indicated.

** Acid leached to remove Fe.

TABLE II

POWDER CHARACTERIZATION DATA OF SELECTIVE NbC POWDER LOTS

Powder Lot No.	Density, g/cc		Fisher Average Particle Size, μ	Specific Surface, m^2/g	
	Bulk	Tap		BET	Fisher
NbC-6	2.09	3.27	1.30	2.58	0.59
NbC-9	2.21	3.98	3.90	1.28	0.20
NbC-10E	2.36	3.72	3.48	1.43	0.22
NbC-13	2.90	4.24	3.60	0.92	0.21
NbC-13R	3.02	4.25	4.02	0.885	0.191
NbC-14	3.22	4.14	12.70	0.28	0.06
NbC-15	2.57	3.93	3.50	0.97	0.22
NbC-17	2.89	4.18	3.50	1.03	0.38
NbC-17AR	3.13	4.26	4.11	0.788	0.187

Many of the pressings were made from a -150 +325 mesh screened fraction of this graphite flour, designated M-2 flour.

Varcum Binder

Varcum 8251 resin obtained from Varcum Chemical Division of Reichold Chemicals is a partially polymerized furfuryl alcohol. The resin was catalyzed with 4 g. maleic anhydride per 100 cc of resin.

Varcum Flour

Some of the Varcum binder material was cured at 250°C for 63 h in air, baked for 48 h at 850°C in vacuum, and graphitized at 2500°C for 1 h. The glassy carbon made at IASL by this technique was crushed, ground, and screened to provide the -150 +325 mesh fraction.

SPECIMEN FABRICATION

The details of the fabrication of test specimens are described in LA-3569-MS;⁽¹⁾ however, briefly, the graphite dies are loaded and positioned in the induction coil, the furnace is closed, and the tank is flushed with argon. After flushing, the die is heated to 3050°C while a load of 3200 psi is gradually applied. The pressure is held for 10 min at temperature and pressure after which the power is turned off, the pressure is released, and the assembly is allowed to cool.

The hot pressed piece is broken from the die and machined to a right cylinder 1.0-in.-diam by 1.0-in.-long for deformation testing.

TESTING

Two types of data were obtained. The single-point deformation test consisted of heating a 1.0-in.-diam by 1.0-in.-long specimen to 2700°C. At this point a load of 2000 psi was applied and held for 30 min after which the load was removed and the sample cooled to room temperature. The specimen was removed from the die, the change in length measured, and the amount (%) of deformation recorded.

Data on cumulative axial deformation were obtained in a like fashion; however, the specimen was first heated to 2000°C. At this point a load of 2000 psi was applied for 30 min after which the load was removed and the specimen was cooled and measured. The specimen was then returned to the die, and the procedure was repeated increasing the temperature 200°C each time until 2600°C was reached. Above 2600°C the temperature was increased in 100°C increments until the cumulative deformation became excessive (greater than 5%). The data were then plotted as a function of cumulative deformation versus temperature.

Effect of Contaminants

Considerable scatter of the data occurred in specimens involving several powder lots. Figure 1 presents variations in properties of the seven powder lots involved as shown by specimens of 85 wt % NbC-15 wt % C composition. Earlier work⁽²⁾ involved specimens from powder lot NbC-9 which is used as the basis for all comparisons in this report.

Fig. 1. Cumulative deformation curves for various powder lots at the 85 wt % NbC-15 wt % C composition.

Since the fabrication procedures were essentially identical, we believed that this scatter was due to one or more contaminants in the as-received NbC powder. Analysis revealed that Lots NbC-13 and NbC-17, which showed the greatest deformation, were sub-stoichiometric, higher in oxygen and higher in iron content than the other powder lots shown.

We decided to plot single-point test data for these various lots as a function of the various contaminants. The only trend as a function of increased contaminants was shown for iron (see Fig. 2).

Analyses of the hot pressed pieces showed them to be very low in iron content, indicating that iron was lost (probably by vaporization) during hot pressing but not before damaging the structure and properties.

This effect of iron, of course, was very undesirable. Our immediate concern was whether the iron

was combined in solution in the NbC or deposited as a film on the particles by the crushing and milling operation. A 500-g sample from each of the two lots highest in iron was acid leached in hot $\text{HCl}:\text{H}_2\text{O}-1:1$, washed, and dried. Specimens of the 85 wt % NbC-15 wt % C composition were hot pressed and single-point deformation tested.

It is apparent from the test results, also shown in Fig. 2, that the iron resulted from the crushing and milling operations. The chemical analysis of the leached powder (Table I) also confirms this observation since the iron content of Lots NbC-13 and NbC-17 is reduced from 3000 ppm to 100 and 500 ppm, respectively.

Metallographic examination of these samples also indicated the effect of iron content on the structure of the carbide matrix. The higher the iron content, the greater the network within the carbide structure as is shown in Fig. 3. Metallography of the as-received NbC powders showed no detectable second phases.

The presence of iron has been observed to promote the formation of large "optical domains" of graphite in various types of carbonaceous bodies upon thermal treatment to 2700°C . Such "domains" appear optically to consist of relatively perfect graphite. Since the presence of iron in carbide-graphite composites causes a grain-boundary network of relatively perfect graphite, it seems reasonable that a relatively soft grain-boundary phase (highly oriented pure graphite) might facilitate deformation by grain-boundary sliding. Just how the iron may do this is problematical.

Since these composites are hot pressed at temperatures well above 2700°C , it is conceivable that incipient grain-boundary melting occurs during the hot pressing operation. It seems logical that carbon is taken into solution as a result of this melting and that, on cooling, perfect graphite particles are precipitated out. The exact mechanism is not known, but work is underway to better understand the mechanism associated with decreased resistance to deformation with increasing iron content.

Effect of Binder Content

The earlier work on NbC-C composites showed a marked increase in deformation at elevated temperatures with increasing binder content. To better understand the influence of the binder, we cured, baked, hot pressed, and deformation tested NbC

Fig. 2. Effect of iron content of the NbC powders on the single-point deformation (Composition 85 wt % NbC-15 wt % C).

powder containing various amounts of binder. The results of cumulative deformation tests shown in Fig. 4 confirm that deformation increases with increasing binder content. The network of the microstructure as a function of binder content is shown in Fig. 5.

While the exact mechanism is not known, it is postulated that the Varcum (furfuryl alcohol) binder releases its oxygen on polymerization during the curing and baking cycles which results in an incipient liquid phase reaction at the temperatures necessary to press these materials to high density. The higher the binder content, the greater the reaction; consequently, the greater the network. X-ray microprobe analysis across this network shows it to be carbon. We are investigating this reaction with the electron microscope and x-ray diffraction as the prime tools.

Until this work is completed, it is postulated that the mechanism creating the network is similar to that proposed previously for the effect of iron.

Effect of Composition

1. Niobium Carbide-Graphite (NbC-C) Composites

Earlier work⁽¹⁾ on a limited number of specimens indicated an increase in deformation with increased NbC content. However, we determined that this effect was not real but was due to the variations in purity of the raw materials used. Therefore, the work was repeated using a known high purity powder lot (NbC-9) and M-2 flour. The results of cumulative deformation tests are shown in Fig. 6. Composition appears to have little effect on the resistance to deformation over the range investigated.

Figure 7 presents the results of single-point tests in triplicate as a function of composition using NbC-9 powder and shows only a very slight effect of composition on resistance to deformation.

2. Tantalum Carbide-Graphite (TaC-C) Composites

One specimen each of selective compositions of TaC-C composites was single-point tested at 2700°C

500 ppm Fe

500 ppm Fe

1000 ppm Fe

Fig. 3. Effect of iron content on the microstructure of 85 wt % NbC-15 wt % C composites. 500X

Fig. 4. Effect of binder content on cumulative deformation of NbC.

and 2000 psi for 30 min. A comparison of the effects of M-2 and the LASL Varcum flour in these composites was also made. The data are presented in Fig. 8 and show that composition has little effect on resistance to deformation over the range investigated. The specimens containing Varcum flour showed slightly more resistance to deformation.

Solid Solutions of TaC and NbC

The advantages of solid-solution strengthening similar to that for metal alloys systems were proposed for carbide systems several years ago and now seem a reality. Single-point tests were run on pressings of TaC, NbC, and a 50/50 wt % mixture of TaC and NbC. The results are presented in Fig. 9.

The data show that the solid solution formed during hot pressing was significantly more resistant to deformation than are the individual carbides.

4% Binder

8% Binder

12% Binder

Fig. 5. Metallographic results showing effect of binder content on the structure of NbC. 500X

Fig. 6. Effect of composition on cumulative deformation of 70-85 wt % NbC-C composites.

Fig. 7. Effect of composition on single-point deformation of 70-85 wt % NbC-C composites.

Fig. 8. Effect of composition on single-point deformation of 80-90 wt % TaC-C composites.

Fig. 9. Single-point deformation of TaC, NbC, and a 50/50 wt % solid solution of TaC and NbC.

The solid solution systems offer a new family of materials having mechanical and physical properties sometimes vastly different from those of the individual metallic carbide components.

The effect of composition of composites, using the 50 wt % TaC-50 wt % NbC mixture, on resistance to deformation is shown in Fig. 10. To compare the individual carbides to the solid solution, it was necessary to plot average values of deformation versus volume percent carbide. This plot is presented in Fig. 11 and shows the mixed carbide composites to be more resistant to deformation than NbC but not so resistant as the TaC composites.

Fig. 10. Effect of composition of (50 wt % TaC-50 wt % NbC) carbide-carbon composites.

Fig. 11. Deformation vs. composition of selective carbide-graphite composites.

One should remember that the TaC-NbC composites were not a true solid solution but a mechanical mixture of the individual carbides with graphite. Metallography confirms that complete alloying did not occur during the hot pressing. Therefore, it seems that the full benefit of solid solution strengthening was not evidenced. Work is continuing with mixtures of these materials, as well as prealloyed solid solution carbide mixtures.

Extruded NbC-C Composites

Two small batches of 75 wt % NbC-25 wt % C with Varcum resin binder were extruded to a nominal 1.0-in. round and given the conventional cure, bake, and graphitizing treatment described in Reference 1. One rod contained the standard M-2 flour as the

graphite addition, and the other contained Varcum flour. Specimens were cut approximately 1.0-in. long and single-point tested at 2700°C for 30 min at various loads from 500 to 2200 psi. The data are presented in Fig. 12 and show an increase in deformation with increasing load.

The cumulative deformation curves for the M-2 and Varcum flour samples were also developed and are presented in Figs. 13 and 14, respectively. Metallography showed this material to have a graphite matrix.

Effect of Graphite Particle Size

The effect of graphite particle size and distribution was determined early in the program. Therefore, a -150 +325 mesh fraction was used for most of the development work. A very limited effort was directed toward the optimization of the particle size of the graphite flour.

Figure 15 shows the data for the conventional -150 +325 mesh versus three other fractions of graphite flour. The data are based on the 85 wt % NbC-15 wt % C composition and only a single test. The trend will have to be confirmed, but if it is real, it seems that the -150 +200 mesh flour approaches optimum for this particular NbC powder. The high deformation value for the -200 +325 mesh carbon flour sample is not understood.

Dynamic Elastic Properties of NbC-C Composites

Elastic properties⁽³⁾ were determined on thin rod and cube specimens for hot pressed NbC-C composite samples of four compositions: 70, 75, 80, and 85% by weight of NbC. The thin rod and two cubes were taken from parallel locations along the pressing axis of each pressing, and the axis was identified on each sample. Sample densities were calculated from weights and dimensions. The

Fig. 12. Single-point deformation vs. load of extruded 75 wt % NbC-25 wt % C using M-2 and Varcum flour.

Fig. 13. Cumulative deformation versus compressive load of extruded 75 wt % NbC-25 wt % C composites using M-2 flour.

Fig. 14. Cumulative deformation versus compressive load of extruded 75 wt % NbC-25 wt % C composites using Varcum flour.

Fig. 15. Effect of graphite particle size on single-point deformation of 85 wt % NbC-15 wt % C composites.

difference in densities of the cube pairs indicates a probable density gradient in the pressing.

Fundamental resonance frequencies of the thin rods were determined in air at room temperature by capacitive and eddy-current techniques. High frequency (2 to 5 MHz) pulse-echo measurements of bulk and shear sound velocities were made along all three axes of the cubes. For the shear measurements, two perpendicular polarizations of the quartz crystal transducers were used in each direction. Young's and shear moduli were then calculated with good agreement for comparable directions. Data for these samples are summarized in Table III.

The effect of increasing carbide content shows clearly in the results. Density differences, internal friction, and modulus directionality all decrease, proportionately, with increasing carbide content. The modulus values increase with increasing carbide content (Figs. 16 and 17) and at 85 wt % NbC are roughly half the modulus values for pure NbC. The modulus values shown for graphite and NbC were obtained from the literature.^(4,5)

Electrical Resistivity

Electrical resistivity measurements were obtained on the elastic property samples. The results are given in Fig. 18 and show a decrease in electrical resistivity with increasing carbide content with a sharp change occurring in the region where the structure changes from a carbide matrix.

TABLE III
SOME DYNAMIC ELASTIC PROPERTIES OF NbC-C COMPOSITES

	wt % NbC			
	70	75	80	85
Density, g/cc				
Thin rod	4.021	4.521	4.478	5.481
Cube	3.938, 4.092	4.431, 4.604	4.411, 4.539	5.439, 5.440
Shear modulus, 10^6 psi				
Thin rod*	2.4	4.5	4.2	8.4
Pulse-echo:				
Weak direction*	2.1, 2.8	4.2, 5.0	4.1, 4.4	8.2, 8.0
Strong direction	3.3, 4.3	5.2, 6.2	4.7, 5.2	9.5, 10.8
Young's modulus, 10^6 psi				
Thin rod*	4.0	9.1	8.8	17.5
Pulse-echo:				
Weak direction*	4.7, 4.8	9.9, 11.1	7.7, 11.4	18.4, 16.2
Strong direction	7.9, 9.7	12.6, 14.0	10.5, 12.3	22.3, 24.9
Log decrement of internal friction (thin rod only)	1.3×10^{-2}	7.0×10^{-3}	5.8×10^{-3}	3.5×10^{-3}

* These properties are for comparable directions in these materials.

Fig. 16. Young's modulus versus composition for NbC-C composites.

Fig. 17. Shear Modulus versus composition for NbC-C composites.

Fig. 18. Room temperature electrical resistivity of NbC-C composites as a function of composition.

SUMMARY AND CONCLUSIONS

The technology of carbide-graphite composite materials is extremely limited; however, as new data are generated certain facts become apparent.

1. The purity of the starting carbide powders profoundly effects the final properties of a fabricated shape.

2. High iron content in the starting powders, present as a result of the comminution process, drastically lowers the high temperature resistance to deformation of finished hot pressed composites. High iron content appears to cause a grain-boundary network of relatively soft graphite which seems to facilitate deformation by grain-boundary sliding, but the exact mechanism is not known. A similar effect was observed in samples containing furfuryl alcohol binder.

3. Composition appears to have little effect on resistance to deformation of both NbC and TaC composites over the range investigated.

4. Preliminary results indicate that solid solution strengthening occurs by the combinations of carbides in the TaC-NbC pseudo-binary system. However, considerable work will be necessary to demonstrate this fully.

5. Composites made from a 50 wt % TaC-50 wt % NbC mixture showed greater resistance to deformation as a function of carbide composition than NbC-C composites of comparable volume percent carbide loading. However, the composite mixtures did not perform so well as TaC-C composites. It should be remembered that these were physical mixtures of TaC and NbC and not a true solid solution mixture.

6. Data are presented on the deformation of extruded 75 wt % NbC-25 wt % C stock as a function of load. Deformation increased with increasing load, probably relative to the lower density of the extruded material.

7. A limited study of the effect of graphite particle size suggested that it may play a significant role on the properties of carbide-graphite composites at elevated temperatures. Considerable work will be necessary to verify this fact.

8. Elastic property data are presented for NbC-C composites. A linear relationship occurs for both the predominantly graphite matrix and the carbide matrix compositions with the curves changing

slope at about 46 vol % NbC where the structure changes occur. The curves also show that the specimens were nearly isotropic.

9. Room temperature electrical resistivity data show a decrease with increasing carbide content with a sharp change occurring in the region where the composition changes from a graphite to a carbide matrix.

PLANS FOR FUTURE WORK

A thorough investigation of the solid solutions of the pseudobinary system of TaC and NbC is planned. Some work is also planned on the effect of graphite particle size. Efforts will continue in obtaining physical property data of the various composites being investigated.

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