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Subject: IITRI G6003-Q9 (Letter Report)
"Metal Carbide-Graphite Composites"
Contract No. NASr-65(09)

Gentlemen:

I. INTRODUCTION

Investigations conducted at IITRI have resulted in the development of metal carbide-graphite composites which exhibit good high temperature properties. The early feasibility studies revealed optimum hot pressing conditions for obtaining composites of desired properties.[1] Subsequent studies were concerned with the control of properties by varying carbide content, particle size and shape, and purity of the component powders.[2,3]

Much of the work has been focused on the two most refractory systems, NbC-C and TaC-C. Fabrication of these composites has been conducted at 3150° or 3300°C (or just below the solidus temperatures) in order to obtain maximum bonding. However, these very high hot pressing temperatures are difficult to control and to reproduce. This difficulty has been periodically reflected in production of bodies which do not exhibit optimum properties. Therefore, it is evident that studies are needed for assuring the reproducibility of metal carbide-graphite composites which exhibit good high temperature behavior.

The studies for this year are concerned with the use of low melting densification aids for the following reasons:

1. Normalization of Fabrication Process - Lowering of the fabrication temperature to about 3000°C or less by using densification aids can result in greatly improved quality control in any laboratory.

2. Improved Composite Properties - Our preliminary work indicates that deformation resistance is improved by the use of additives. This may be by solid solution formation, increased graphitization, or a dispersion strengthening effect.

The NbC-C system is of primary interest; pertinent information gathered will be transferred to the TaC-C system, and to a limited extent to the ZrC-C system. Compositional studies will consider composites of 25 to 75 vol% carbide content in order to establish behavioral trends; this will provide a wider latitude of choice of properties for specific applications.

During this period, investigations have been initiated into the use of tungsten in NbC-C composites. Composites containing 25 to 75 vol% carbides have been fabricated, and evaluation has considered both room temperature and high temperature behavior.

II. DISCUSSION

The use of low melting additives as densification aids is based in part on information generated on previous contracts on this program. It has been established at IITRI[1] that amounts as small as 10 vol% of W or Mo yielded strong (~15,000 psi) composites with dense graphite matrices. This was accomplished at the relatively low fabrication temperature of 2800°C and can be attributed to exsolution of a dense graphite from the molten carbides on cooling.

Incorporation of W in the fabrication of TaC-C bodies has resulted in composites which exhibit excellent high temperature properties. Evaluation of these materials using the microprobe analyzer has shown the formation of TaC-WC solid solutions.[3]

During the initial portion of this year's program, composites containing 25, 50, and 75 vol% NbC were fabricated as the standards to which additive-containing composites could be compared. Additive composites, i.e. NbC-WC-C systems, containing 25 to 75 vol% total carbides were hot pressed for evaluation. The results of these experiments appear in the following sections.

A. Raw Materials

In the study of carbon sources during the preceding program, the use of M3 graphite flour yielded composites which exhibited good high temperature strength and deformation resistance. Furthermore, the "spherical" shape of M3 particles gave rise to composites which exhibited less anisotropy than those incorporating the platy calcined petroleum coke in past studies. This material is the standard graphite powder used by the CMB-6 Section of LASL. In view of the desirable properties of M3, it was chosen as the material to be used for our present studies. Other carbon powders such as calcined petroleum coke or carbon blacks can be substituted if different directional behavior is desired.

The detrimental effect of iron in NbC-C composites has been observed at LASL[4] and also at IITRI.[3] Rigid specifications have been set up by LASL for the morphology as well as purity of NbC powders acquired from vendors. A portion of this powder (NbC-42A) has been allotted to IITRI to reduce variations in composite properties which might result from the use of other powders not subject to these stringent specifications and to permit correlation of mechanical properties with material fabricated at LASL.

The densification aid which is being studied initially is tungsten. Molybdenum (also Group VI) performs a similar function in densification and graphitization of graphite; the choice of W over Mo was dictated by the fact that WC-NbC would be a higher melting solid solution than $MoC_{(1-x)}$-NbC, and would probably offer better high temperature properties.

A very fine tungsten powder was obtained from Fansteel Inc. The small particle size was chosen in order to realize good dispersion of the W additive throughout the NbC-W-C mixture, and thus maximize the graphitization effect of WC during fabrication.

The use of niobium carbide and graphite powders supplied through the cooperation of LASL also facilitiates comparison of property data for composites since any raw materials effects are eliminated except for the use of W. Photomicrographs of these powders are shown in Figure 1 and the powders are described in Table I.

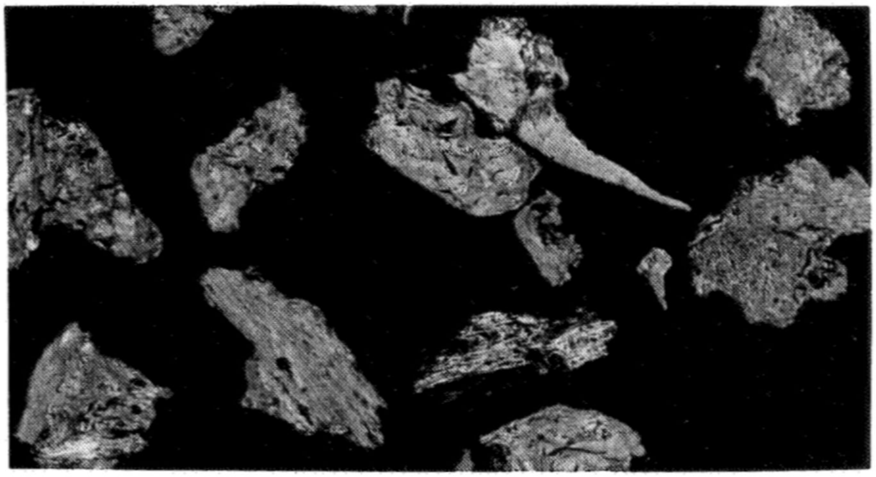
B. Processing

The various mixtures of NbC-C and NbC-W-C were blended by tumbling for 16 hr with rubber stoppers. Mixtures were then cold pressed at 1000 psi prior to hot pressing. The time-temperature-pressure schedules for the various pressings are shown in Figure 2. The standard composites containing no additive were soaked at 3150° to 3200°C for 15 min, the conditions optimum for good bonding. The tungsten containing bodies were soaked at 3000°C for 20 min. These latter conditions yield NbC-C composites (containing no WC) of high density but poor bonding.

C. Evaluation

The compositions being investigated during this program involve ternary as well as binary systems. In order to simplify

50μ



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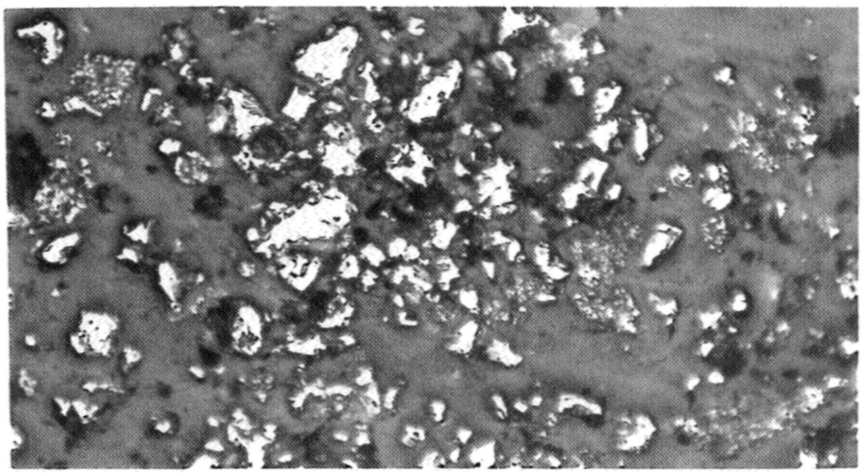
(a) M3 Graphite Flour
44-105μ



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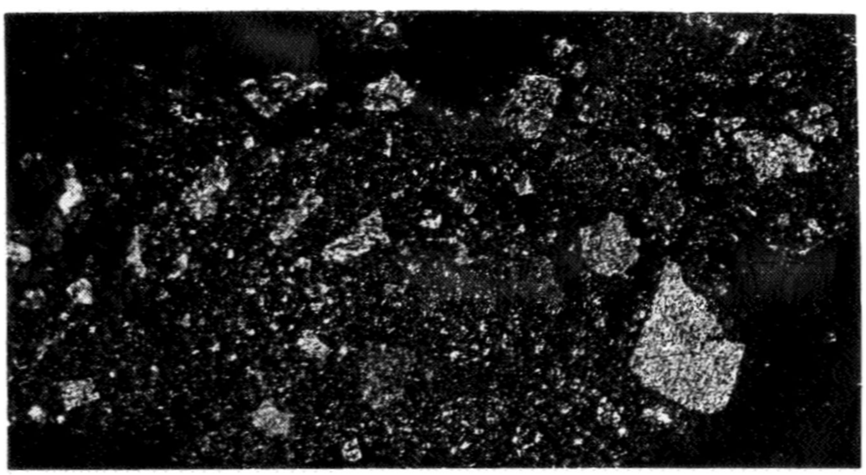
(b) NbC-42A
3.55μ (Fisher)



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(c) W-427A
1.10μ (Fisher)

Figure 1. PARTICLE SIZE AND SHAPE OF RAW MATERIALS

Table I

RAW MATERIALS USED FOR NbC-C AND NbC-WC-C COMPOSITES

Material/Designation		Supplier	Particle Size	Chemical Analysis
Graphite	M3	Great Lakes Carbon (CMB-6:LASL)	44-105µ	Fe: 40 ppm O: 700 ppm
NbC	42A	Wah Chang (CMB-6:LASL)	3.55*	Fe: 300 ppm O: 0.30%
W	427-A	Fansteel	1.10*	Fe: 15 ppm O: 1450 ppm

*Fisher Sub-sieve

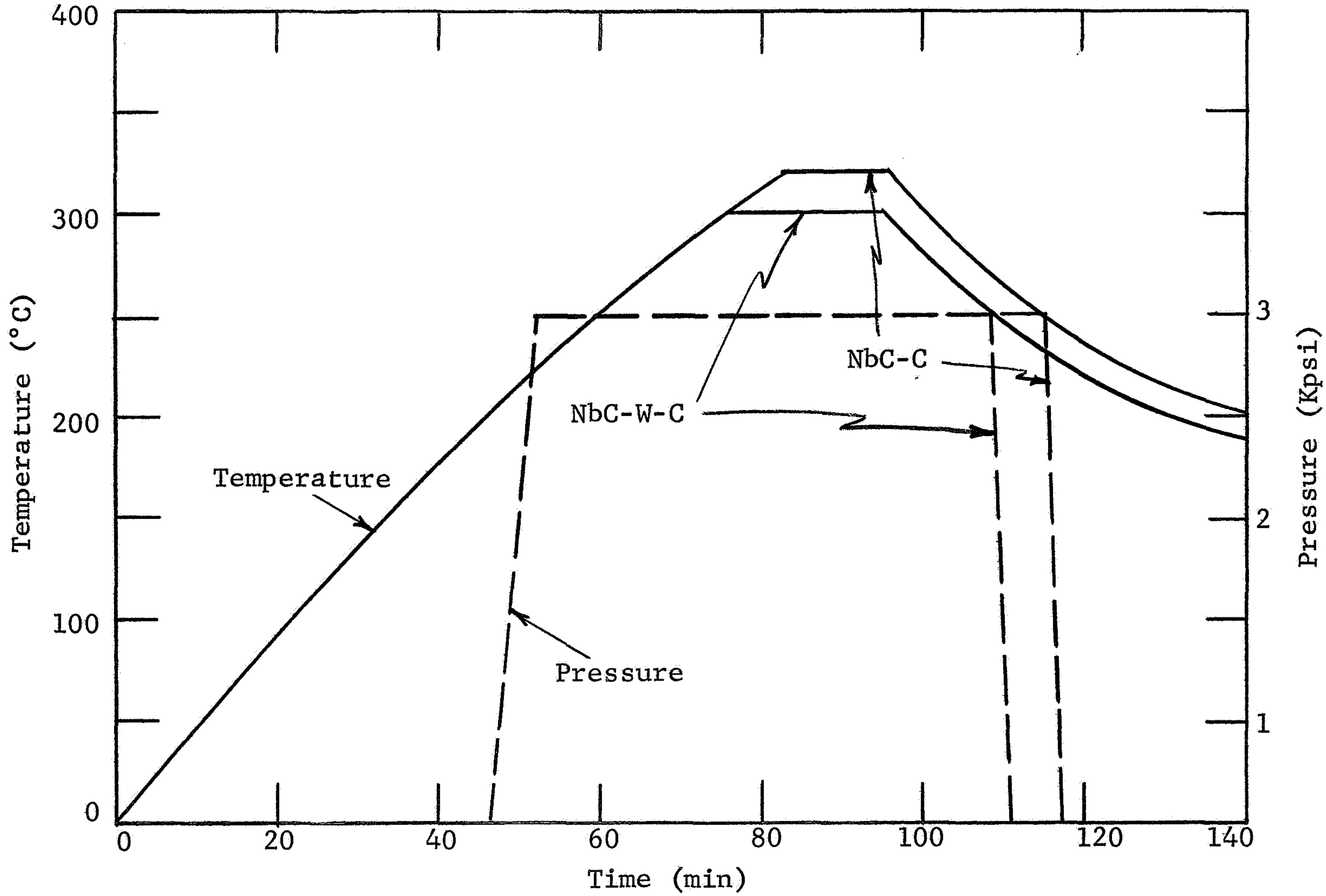

Figure 2. TIME-TEMPERATURE-PRESSURE SCHEDULES FOR HOT PRESSING OF NbC-C AND NbC-W-C COMPOSITES

compositional designations while retaining descriptiveness, the following scheme is being used. For binary system such as NbC-C, the designation consists of two numbers separated by a slash, the first number being vol% carbide and the second, vol% C. For example, 25NbC/75M3 refers to a 25 vol% NbC-75 vol% C composite prepared from NbC-42A and M3 graphite. Ternary systems have three numbers; 20NbC/5WC/75M3 contains 20 vol% NbC-42A, 5 vol% WC (added to the powder mixture as W metal), and 75 vol% M3 graphite flour. Of course it is realized that the NbC and WC form solid solutions, but this designation serves to describe what has gone into the composite. It is anticipated that these basic designations will be modified as different materials are investigated.

Composition - Compositions which have been fabricated are listed in Table II. In addition to the standard NbC-C composites incorporating 25, 50, and 75 vol% NbC, a series of composites containing WC were hot pressed. The amounts of WC added were 10 or 20 vol% (10.7 or 21.2 mol%) in relation to the total carbide content. This was done in order to determine trends which could be used for directing additional experiments.

Densification - The densities for the various composites along with extent of densification are tabulated in Table II. The non-additive composites showed increasing values for % theoretical density with increasing carbide content, a trend observed in earlier studies.[2] This can be attributed to the fact that it is more difficult to approach theoretical density in a graphite structure than in a carbide structure; hence, the low carbide (25 vol%) composite exhibited the lower extent of densification.

Theoretical densities of the NbC-WC-C composites were calculated on the basis of total weight of components divided by total volume of components, using the theoretical densities

Table II

FABRICATION SUMMARY FOR NbC-C AND NbC-WC-C COMPOSITES

Compositional Designation	Density, g/cc	% Theoretical Density
25NbC/75M3	3.37	92.5
50NbC/50M3	4.73	94.0
75NbC/25M3	6.25	97.3
22.5NbC/2.5WC/75M3	3.53	91.5
20NbC/5WC/75M3	3.79	92.9
45NbC/5WC/50M3	5.29	96.2
40NbC/10WC/50M3	5.17	--
67.5NbC/7.5WC/25M3	6.90	96.2
60NbC/15WC/25M3	7.62	95.5

of the three phases. This calculation assumed that the two carbides existed as discrete phases.

A second theoretical density was calculated based on x-ray densities of NbC-WC solid solutions using estimated lattice parameters. These estimates were based on lattice parameters which have been reported for the TaC-WC solid solutions.[5] Since NbC and TaC are of the same structure type (fcc) and exhibit similar lattice parameters, it was felt that a reasonable estimate could be made as shown by the dotted curve in Figure 3. Using this curve, the lattice parameters of NbC-WC solid solutions would be 4.452Å at 10.7 mol% WC and 4.433Å at 21.2 mol% WC. X-ray densities were calculated with the formula:

$$\rho = \frac{1.66020\ \Sigma A}{V} \qquad (1)$$

where ΣA = sum of the atomic weights of the atoms in the unit cell, and V = volume of unit cell (Å^3).

Comparison of calculated theoretical densities from method 1, i.e., regarding the carbides as separate phases, with those from method 2, i.e., regarding the carbides as having formed a solid solution, show fairly close agreement.

		Theoretical Density, g/cc	
Method	Mol% WC	25 vol% NbC-WC	50 vol% NbC-WC
1	10.7	3.845	5.429
2	10.7	3.854	5.447
1	21.2	4.045	5.829
2	21.2	4.064	5.867

The results to date are too preliminary to indicate how much solid solution has formed and if either of the carbides still exists in its free state. Therefore, the values obtained by assuming the NbC and WC exist as discrete phases appear to be a reasonable first estimate.

The various composites incorporating WC all showed high densification. The 40NbC/10WC/50M3 composite suffered

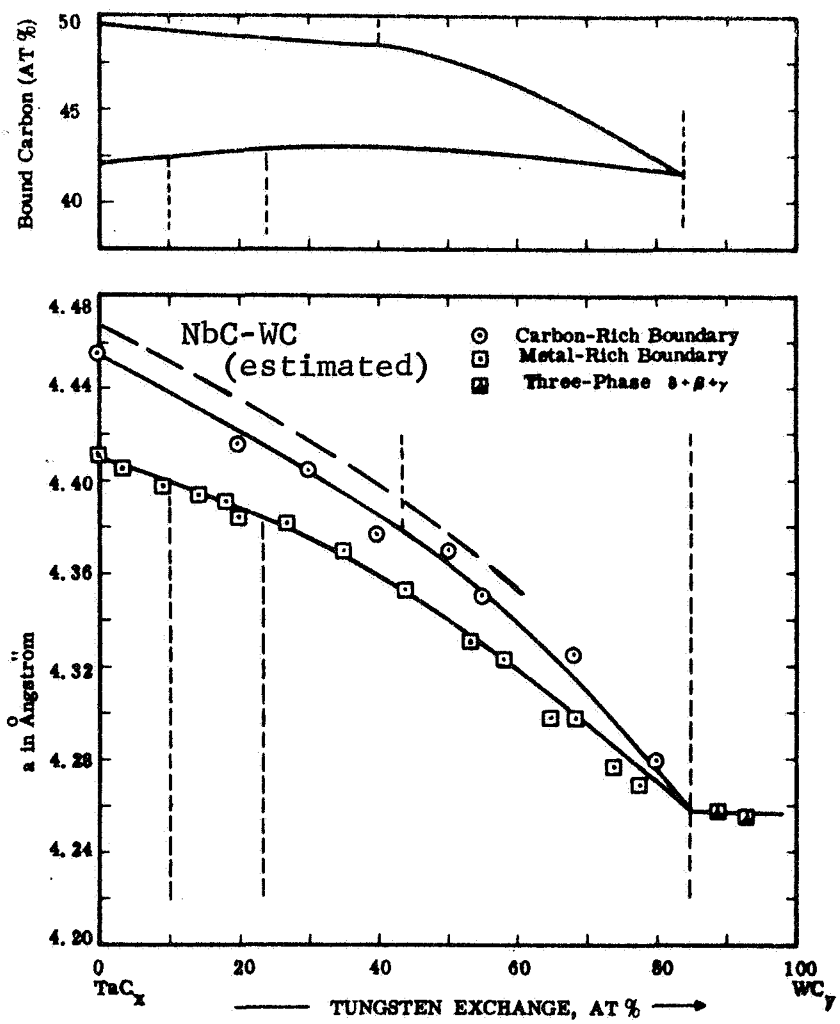

Figure 3. Ta-W-C: LATTICE PARAMETERS OF THE CUBIC MONOCARBIDE SOLID SOLUTION AT THE METAL- AND CARBON-RICH BOUNDARIES AT 2200°C (Reference 5)

– – – – Location of vertices of adjacent three-phase equilibria some loss of material during hot pressing, and thus a theoretical density (based on starting materials) could not be assigned to this system. However, the excellent properties exhibited by this composite (as detailed later in this report) suggests that this body probably had exceeded 95% theoretical density.

X-Ray - The various composites were evaluated using x-ray techniques. Debye-Scherrer powder pictures were made, and lattice parameters of the NbC-WC solid solutions were calculated from the films using the Nelson-Riley Function. In all of the patterns, only the solid solution and graphite were evident.

The lattice parameter data are listed in Table III along with mol% estimates based on the values in Figure 3. It would appear that with one exception, most of the WC had been tied up in solid solution for the various composites. The 40NbC/10WC/50M3 composition showed a significant departure (13 mol% WC in solid solution as compared to the 21 mol% WC as mixed) in WC content. As pointed out before, this billet had exhibited material loss due to hot pressing; from this data, it appears that a substantial amount of WC had been extruded out of the sample in fabrication. This composite had the largest amount of WC, and thus, the most liquid in any of the composites.

Microstructure - The microstructures of composites incorporating 25 vol% carbides and 50 vol% carbides are presented in Figures 4 and 5. The graphite-rich composites incorporating WC exhibited a similar structure to that for the binary NbC-C (Figure 4). As has been observed before, the difference in particle size between the carbide (3.55μ) and graphite (>44μ) results in clustering of the finer carbide particles. There is a fairly high porosity, and a lack of orientation for the carbide grains. These structures suggest that holding times in fabricating be increased to form a tighter structure.

Somewhat less porosity is observed for the composites containing 50 vol% carbides (Figure 5). Again, a clustering of

Table III

LATTICE PARAMETERS OF NbC-WC SOLID SOLUTIONS IN NbC-WC-C COMPOSITES

Compositional Designation	Lattice Parameter, Angstroms	Mol% WC	As-Mixed Mol% WC
22.5NbC/2.5WC/75M3	4.454	9	10.7
20NbC/5WC/75M3	4.435	20	21.2
45NbC/5WC/50M3	4.457	8	10.7
40NbC/10WC/50M3	4.447	13	21.2
67.5NbC/7.5WC/25M3	4.457	8	10.7
60NbC/15WC/25M3	4.437	19	21.2
NbC	4.470		

50μ

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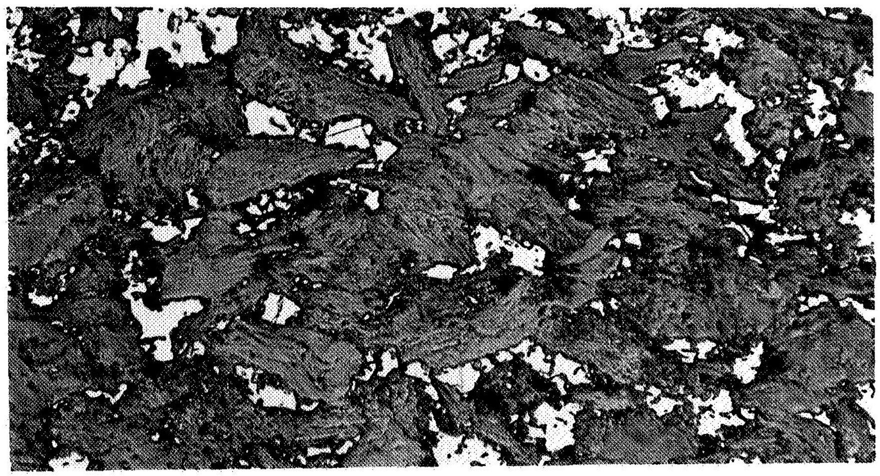
(a) 25NbC/75M3 (W/G)



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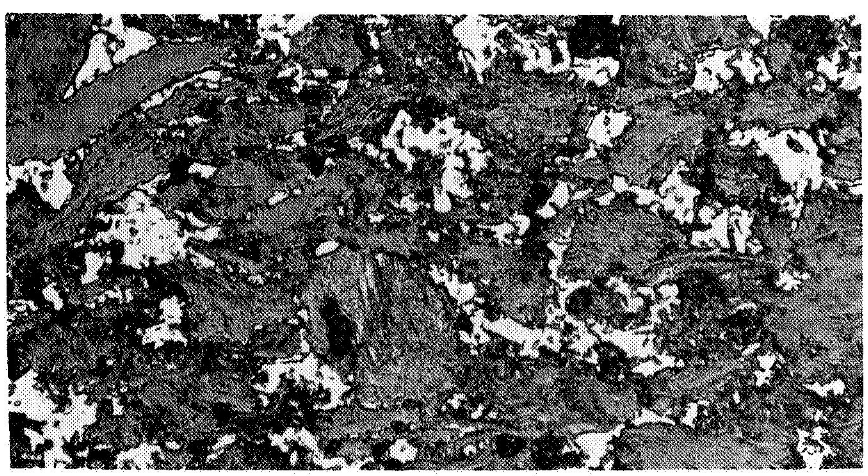
(b) 22.5NbC/2.5WC/75M3 (W/G)



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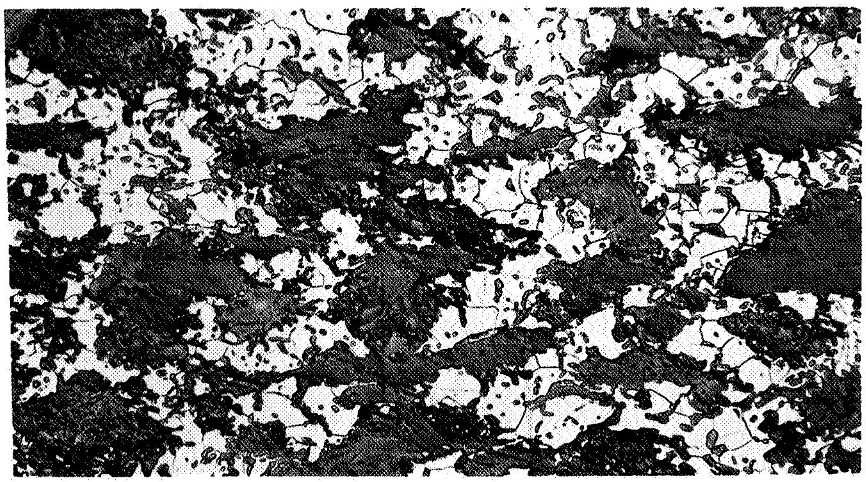
(c) 20NbC/5WC/75M3 (W/G)

Figure 4. MICROSTRUCTURES OF COMPOSITES CONTAINING 25 VOL% CARBIDES

50μ

Neg. 36060 Mag. X200

(a) 50NbC/50M3 (W/G)



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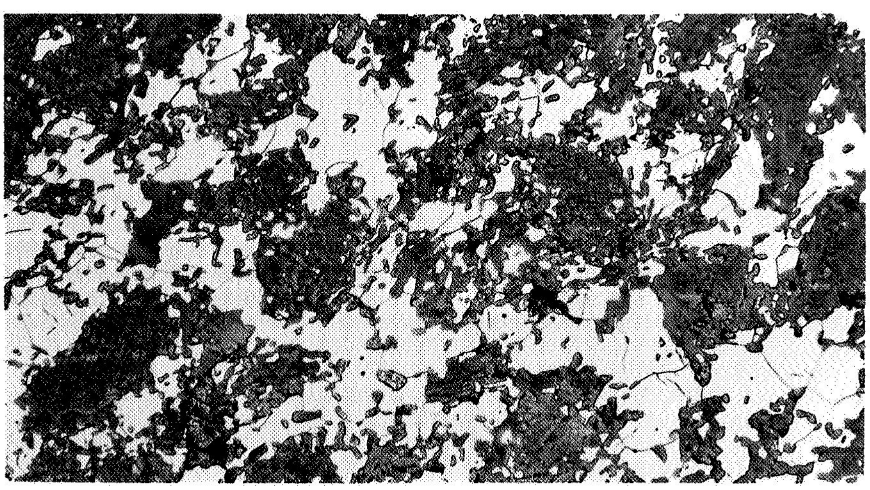
(b) 45NbC/5WC/50M3 (W/G)



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(c) 40NbC/10WC/50M3 (W/G)

Etchant: $K_3Fe_2(CN)_6$-NaOH

Figure 5. MICROSTRUCTURES OF COMPOSITES CONTAINING 50 VOL% CARBIDES

carbide particles is seen. The graphite phase appears to be denser and less "grainy" than in the lower carbide content structures. This may be due to the WC acting as a graphitization aid more effectively in the 50 vol% graphite than in the 75 vol% graphite composites. The volume ratios of C to WC are 5:1 and 10:1 in the former, and 15:1 and 30:1 in the latter. These results suggest increasing the WC content in graphite-rich composites so that the graphitization effect might be more fully realized, producing a stronger body.

Room Temperature Properties - Room temperature flexural strength, moduli and electrical conductivity data are tabulated in Table IV. The flexural strengths of the standard non-additive materials can be compared to those for comparable composites incorporating calcined petroleum coke (CPC).

	Flexural Strength, psi	
	W/G	A/G
25NbC/75M3	9,800	4,700
25NbC/75CPC	11,000	3,500
50NbC/50M3	19,200	10,300
50NbC/50CPC	16,500	6,500

The present composites incorporating the spherical M3 show a lower anisotropy than those containing the needle-like CPC, which is highly directional. This is especially evident at the 50 vol% NbC level in the A/G direction. These data indicate that good bonding exist for the standards.

Composites incorporating WC at the 25 vol% carbide level showed strengths which were considerably lower than that for the standard, i.e., 5,900 and 7,100 psi vs 9,800 psi. It would appear that conditions for realizing good graphitization and bonding at the 3000°C fabrication temperature require longer holding times and/or higher amounts of the WC densification aid. The composite incorporating 5 vol% WC showed a higher strength (7100 psi) than the composite containing 2.5 vol% (5900 psi).

Table IV

PROPERTIES SUMMARY FOR NbC-C AND NbC-WC-C COMPOSITES

Compositional Designation	Grain Direction	Flexural Strength, psi			Elastic Modulus, x 10^6 psi	Electrical Conductivity, x 10^4 mho-cm
		RT	2000°C	2500°C		
25NbC/75M3	W/G	9,830	10,980	11,080	2.7	0.62
	A/G	4,650	5,300	5,740	1.1	0.31
50NbC/50M3	W/G	19,180	18,640	13,070*	7.6	1.51
	A/G	10,270	11,750	9,470*	4.1	1.09
22.5NbC/2.5WC/75M3	W/G	5,860	5,770	6,340	3.3	0.48
20NbC/5WC/75M3	W/G	7,070	7,320	6,620	3.5	0.48
45NbC/5WC/50M3	W/G	14,170	14,550	12,540	8.1	1.07
40NbC/10WC/50M3	W/G	17,280	26,340	18,290	7.0	0.99

*Highest stresses which could be applied before deformation rate exceeded load rate (crosshead speed = .04 in./min).

By increasing the WC level to about 10 vol%, the C to WC ratio would be 7.5:1, and greater graphitization may be realized.

The composites incorporating 50 vol% total carbides reveal the same trend, i.e., higher strengths are obtained with increasing amounts of WC. As listed in Table IV, the strengths are 14,170 psi for 45NbC/5WC/50M3 and 17,280 psi for 40NbC/10WC/50M3. The strengths of the additive composites can be compared to those of the standard composites in the following manner to show this trend:

Composition	$\frac{\text{Vol WC}}{\text{Vol C}}$, %	$\frac{\sigma}{\sigma_{standard}}$, %
22.5NbC/2.5WC/75M3	3.3	60
20NbC/5WC/75M3	6.7	72
45NbC/5WC/75M3	10.0	74
40NbC/10WC/50M3	20.0	90

The elastic moduli of the additive composites are comparable to those of the standard composites (Table IV). Electrical conducrivity values, on the other hand, are considerably lower for the composites incorporating WC; this behavior has been observed in earlier work.[3] A recent value for the resistivity of WC is 25μΩ-cm[6], which is lower than that for NbC (32μΩ-cm). The high resistivity of the additive composites suggest that the solid solutions have higher resistivities than either of the carbides. The electrical properties of solid solutions must be characterized in order to render electrical measurements a useful evaluation tool.

High Temperature Properties - The composites incorporating 25 vol% carbides exhibited somewhat higher strengths at elevated temperatures. The data tabulated in Table IV and shown graphically in Figure 6 show that strengths at 2500°C were about the same or somewhat higher than at room temperature. This maintenance of strength at 2500°C has been noted previously for graphite-rich bodies and can be attributed to the dominance of graphite behavior.

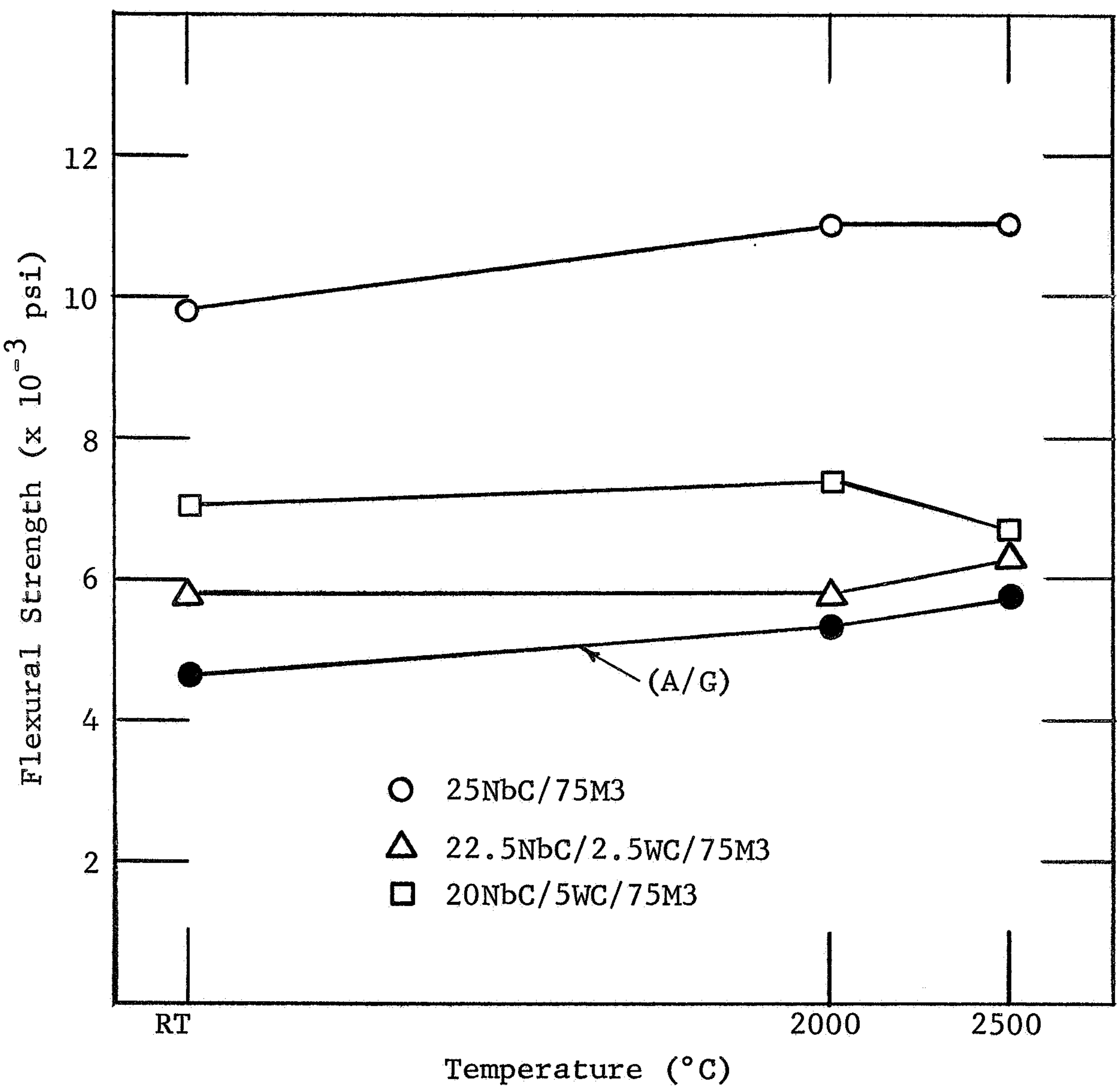

Figure 6. FLEXURAL STRENGTH VS TEMPERATURE FOR NbC-C AND NbC-WC-C COMPOSITES CONTAINING 25 VOL% CARBIDES

The lack of a noticeable peak in strength is not surprising in view of the information gathered on M3 containing bodies in recent work. It has been shown that M3 composites display a maximum in the strength-temperature curve at temperatures from 1500° to 2250°C depending on the system. Obviously tests at temperatures other than 2000° and 2500°C are necessary to determine the peak; however, the 2500°C strength is significant in indicating maintenance of strength, and determination of maximum strength vs temperature will be conducted only when deemed necessary.

As indicated in the section on x-ray evaluation, NbC-WC solid solutions exist in both of the additive composites (22.5NbC/2.5WC/75M3 and 20NbC/5WC/75M3). The lack of any significant increase in bonding at either room or elevated temperatures suggest that the WC was tied up in solid solution with NbC before any graphitization effects had been realized. Additional amounts of WC appear necessary for improving properties at this carbide level.

The high temperature flexural strengths of 50 vol% carbides composites are illustrated in Figure 7 and tabulated in Table IV. The standard composite, 50NbC/50M3, exhibited significant deformation at 2500°C and could not be stressed to failure; however, the stress levels which could be maintained, i.e., 13,000 psi W/G and 9,500 psi A/G, were quite high.

The 45NbC/5WC/50M3 composite displayed fairly good strength at 2500°C. The loss in strength compared to room temperature strength was only about 10%, indicating the beneficial effect of graphitization and/or solid solution formation.

Remarkable high temperature behavior was exhibited by the 40NbC/10WC/50M3 composite. The strength increase at 2000°C was more than 50%, and the 2500°C strength was still higher than that at room temperature. In reviewing this composite, a loss

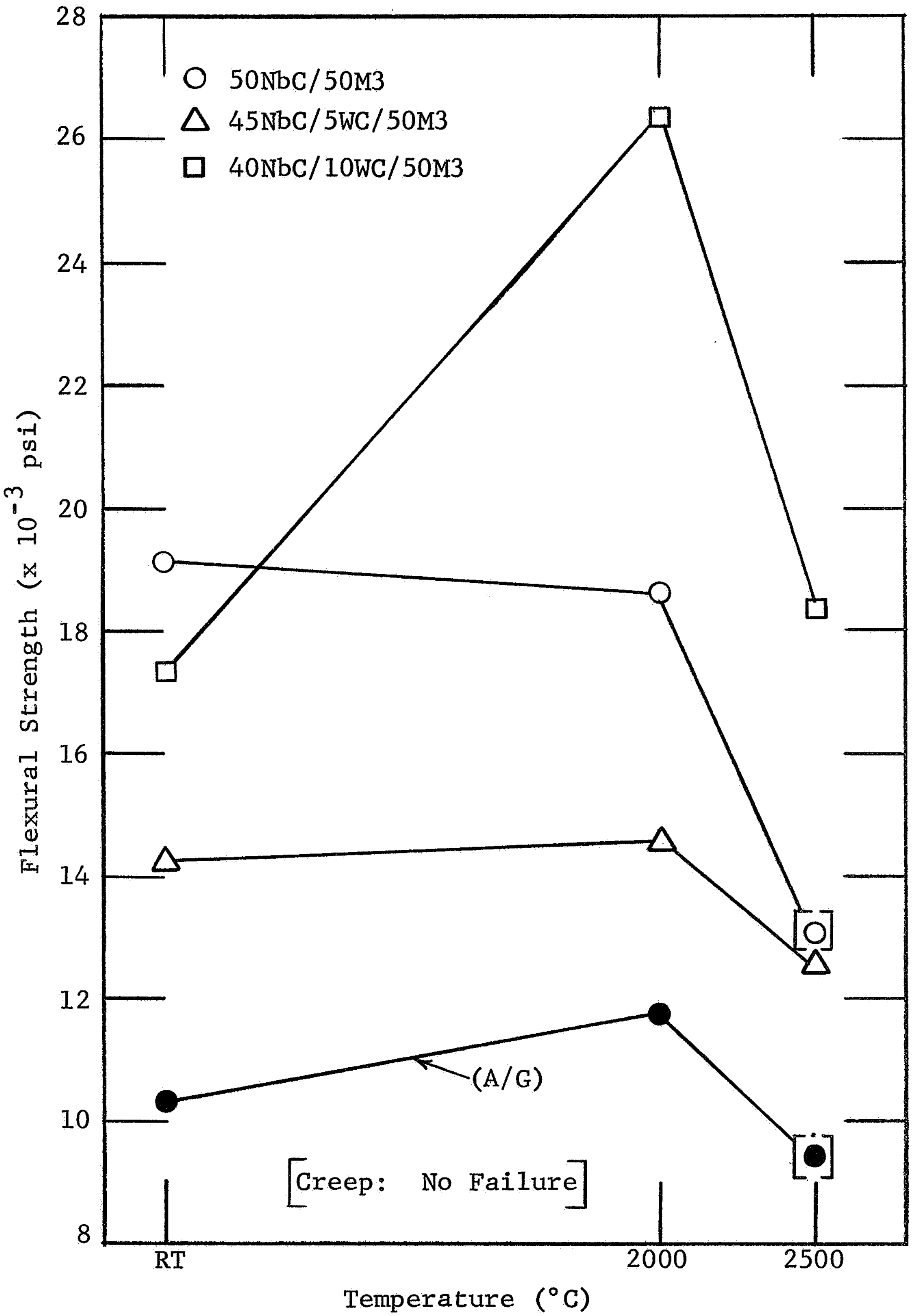

Figure 7. FLEXURAL STRENGTH VS TEMPERATURE FOR NbC-C AND NbC-WC-C COMPOSITES CONTAINING 50 VOL% CARBIDES

of material was incurred in hot pressing, and this was reflected in the composition of the solid solution as shown by lattice parameter measurements (Table III). It would appear that this excellent high temperature behavior is due more to a graphitization effect of a molten WC rather than the formation of a solid solution. It has been observed that solid solutions in the 25 vol% carbides composites did not result in such dramatic behavior.

The superior high temperature behavior of the 40NbC/10WC/50M3 composite was reflected in flexural deformation studies as shown in Figure 8. The deflections observed for the standard 50NbC/50M3 were significantly higher than that for the 40NbC/10WC/50M3. Among the 25 vol% carbides composites, the bodies containing additives displayed very rapid deformation. This may be due to the poorer graphitization at a 3000°C fabrication (as compared to the 3150° to 3200°C for the standard 25NbC/75M3). As suggested earlier, greater amounts of WC appear necessary to realize the benefit of WC as a densification aid.

III. CONCLUSIONS AND FUTURE WORK

Preliminary work has been conducted in the use of tungsten as a densification aid to obtain carbide-graphite composites with good high temperature properties at a fabrication temperature of 3000°C, or more than 200°C below the solidus. Studies in the NbC-WC-C system have involved compositions incorporating 25 and 50 vol% carbides. The results suggest great potential for not only lowering of the fabrication temperature, but also for improving composite properties. The following information is thought to be most significant:

1. Composites of NbC-WC solid solutions plus graphite are formed at the 3000°C fabrication temperature from the starting powder mixture of niobium carbide, tungsten, and graphite. X-ray studies show no other phase (such as NbC or WC) to be present.

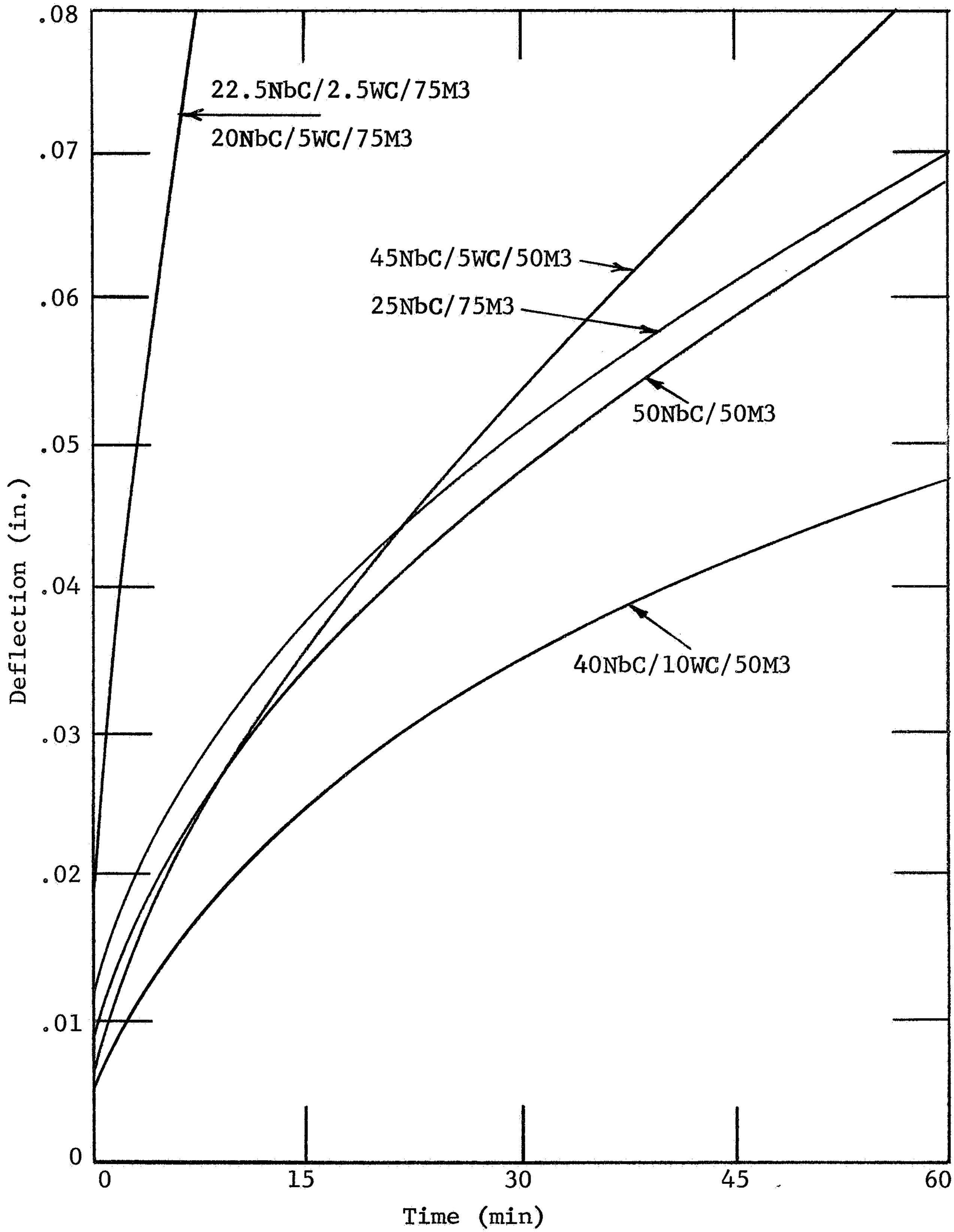

Figure 8. FLEXURAL DEFLECTION VS TIME FOR NbC-C AND NbC-WC-C COMPOSITES (2500°C/5000 psi)

2. An important compositional consideration in fabrication appears to be the WC to C ratio. A volume ratio of 1:5 yields composites which exhibit high temperature behavior superior to that for comparable NbC-C composites containing no WC. Lower ratios of from 1:10 to 1:30 decrease the effectiveness of WC as a densification aid.

   Thus, the obvious direction for future study is incorporation of greater amounts of WC in the composite. However, it is realized that an optimum NbC-WC-C composition will be reached, beyond which adverse effects will be encountered. These effects are excessive material loss during fabrication and/or formation of a solid solution which has a melting point low enough so that high temperature mechanical properties will be lowered. Based on data for TaC-WC solid solutions, a NbC-WC solid solution of 40 mol% WC may exhibit liquification at about 3000°C.

3. Graphitization appears to be the important factor in processing at 3000°C. The mere existence of a solid solution is in itself insufficient to yield good properties. The WC must promote graphitization prior to forming the solid solution in order to obtain good high temperature materials.

Future work will be based on the observed desirable trend of decreasing the C to WC ratio. It is realized that an optimum ratio will be reached, beyond which the fabrication process and/or composite properties will be affected adversely. Compositional studies will consider carbide levels from 25 to 55 vol% in order to characterize the graphite-rich bodies.

IV. CONTRIBUTING PERSONNEL AND LOGBOOK RECORDS

The following personnel have contributed to this research program: S. A. Bortz, Y. Harada, J. L. Sievert, and C. J. Levesque. The data are recorded in Logbook Nos. 18915, 18924, 19057, 19063, and 19068.

Respectfully submitted,
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