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GRAPHITE - METAL COMPOSITES
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I. INTRODUCTION AND SUMMARY

This program was conceived to investigate liquid phase sintering in graphite-metal composites during hot pressing at elevated (2800° - 3100°C) temperatures. The first year's studies [1] revealed that a strong bond is obtained by formation, diffusion, and subsequent recrystallization of a carbide-carbon eutectic phase in selected composites. Flexural strengths of greater than 14,000 psi were exhibited by graphite systems containing niobium carbide or hafnium carbide at both room temperature and 2000°C.

The purpose of this year's work is to extend the compositional studies into higher metal contents and to fully characterize the properties of composites which exhibit the greatest potential for high temperature applications. The metal phases with which we are most concerned are niobium, tantalum, hafnium, zirconium, titanium, and molybdenum. Parameters which are being investigated in these systems include type and particle size of raw materials, and relationship be-

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tween processing and carbide-carbon eutectic temperatures.

During this period compositional studies have included a broader study of tantalum and niobium composites, having metal contents of 50-90 wt%. These compositions are of prime interest to LASL for near term applications. In addition, systems containing molybdenum, vanadium and titanium have been examined in the continuing general study of metal carbide-graphite composites and their properties. The use of metal oxides as the metal source has also been examined. Experiments have been conducted to determine actual metal content in fabricated billets. Physical property measurements were made at elevated temperatures (2500°-2800°C) and thermal shock tests have been initiated.

II. DISCUSSION

A. Compositional Studies

Materials which are of prime interest at this point are niobium and tantalum systems. These are the most refractory in terms of carbide-carbon eutectic temperatures, the NbC-C being 3250°C and the TaC-C, 3450°C. Thus these systems are at present being given the most study.

Other systems such as TiC-C, ZrC-C and MoC-C are also being investigated to add to general information concerning behavior of two phased graphite-carbide composites. Molybdenum compositional studies have indicated a mechanism by which the properties of a pure graphite may be improved.

In our present studies, the relationship between carbide-carbon eutectic and processing temperatures has been investigated; complementary metal analysis has shown substantial metal loss for some systems. Thus optimum fabrication temperatures can now be defined. Preliminary experiments using the oxide instead of the metal or metal carbide as the metal source are evaluated.

1. Processing versus Carbide Eutectic Temperature

A series of metal carbide-graphite composites were hot pressed with particular attention to maintaining a processing temperature slightly below the carbide-carbon eutectic temperature. As reported in the preceding quarterly report, [2] a significant loss of metal can occur if the processing temperature is too high. Furthermore, segregation of the metal carbide phase can occur due to the mobility of a liquid eutectic, and macropores can result during crystallization of a large volume of melt. Results from these pressings are presented in Table I.

Previous work had shown that vanadium and titanium systems processed at 3000°C exhibited macropores and poor bonding. Metal analysis in the 50 wt% Mo system has shown decreasing metal content with increased processing temperatures (28000° and 3000°C). Thus the V-C and Mo-C systems were pressed at 2600°C which is lower than the 2650°C eutectic temperature listed for VC-C and slightly above that for MoC-C (2580°C). The titanium composition was processed at 2750°C; the TiC-C eutectic is reported as 2775°C.

Lowering of the processing temperature yielded excellent composites of VC-C and MoC-C but not for the TiC-C system. The present flexural strength value of 16,710 psi for the vanadium sample (Table I) reveals a vast improvement in bonding over that of the same composition fabricated at 3000°C (4710 psi). Fairly good retention of strength at higher temperatures exists for this system. The molybdenum system also showed excellent strength at room temperature with dropoffs at elevated temperatures. The Mo-C system is unique, in that exceeding the eutectic temperature by as much as 400°C in processing was not deleterious to good strength as is the case for Zr-C, Ti-C and V-C. The loss

in metal which occurs for Mo-C has little effect on bonding; in fact, an inverse relationship between carbide content and strength has been reported by other investigators[3]. As described in a later section, this may be due to a strengthening of the matrix graphite with loss of metal.

The low densities and poor bonding in the titanium system is somewhat puzzling. However, as shown in a later section on metal analysis, substantial loss of metal occurred during processing. This lowered metal content is responsible at least in part for the poor properties. The presence of some large pores in the finished billet also indicates that the eutectic temperature was exceeded. Fabrication of this composition will be conducted at an even lower temperature in an attempt to realize the potential of the TiC-C system.

For the more refractory NbC-C and TaC-C compositions higher temperatures appeared necessary for densification and good bonding. Increasing the fabrication temperature to 3100°C to more closely approach the 3250°C NbC-C eutectic resulted in high strength composites of 50 wt% Nb as seen by the data in Table I. The bonding exhibited by this sample is comparable to that shown by the same composition in earlier work (16,000 psi)[1] and considerably higher than that for a mixture processed at 3000°C (6100 psi)[2]. Thus, the hypothesis presented in the preceding report[3] i.e., that the high strengths observed in earlier experiments was due to processing at a temperature higher than the indicated 3000°C, appears to hold.

A comparison of density between the strong Nb bodies (3.38 and 3.42 g/cc) and the weak (3.52 g/cc), indicates

that an actual improvement in bonding exists and that degree of densification is not the main factor. Metal analysis has shown that the strong 3.42 g/cc material had an actual metal content of 48.2% and had attained 92.7% theoretical density; the weaker 3.52 g/cc material contained 50.4 wt% metal and was 92.6% theoretical density. From the similarity in metal content and degree of densification, it would appear that the higher processing temperature is more conducive to NbC-C eutectic diffusion and also to a higher degree of graphitization, thus yielding stronger bonds.

The 3100°C fabrication temperature produced the first bodies containing tantalum of significant strength (8570 vs 2720 psi as shown in Table I). Metal analysis shows that the stronger bodies were of lower metal content (50.4 wt% vs 56.7 wt%) but had achieved greater densification (93.7% vs 87.0% theoretical density). In addition to fabricating at a temperature closer to the TaC-C eutectic (3450°C), the use of the carbide which is somewhat finer in particle size than the metal may have enhanced good bonding. Microstructural examination (Figs 1 and 2) shows a finer carbide dispersion and less porosity for the 3100°C sample than for that processed at 3000°C.

The results for the 60 wt% Ta composition pressed at 3100°C were disappointingly poor. The increase in metal content did not produce a corresponding increase in strength as might have been expected. Nevertheless, this sample did exhibit higher strength than a previously fabricated 65 wt% Ta billet (8200 vs 3130 psi). The data for TaC-C processed at 3100°C indicates that optimum properties are yet to be obtained at these weight levels. Future pressings will consider higher pressures and temperatures and heat treatment.

2. High Metal Content

Compositions of higher metal content approaching the eutectic are of interest in that higher strengths and greater resistance to chemical attack can be realized. Arc cast compositions of the hypereutectic region which have a slight excess of free graphite have been shown to possess good thermal shock resistance[4]. Those in the hypoeutectic, on the other hand, are poor in thermal shock and are quite difficult to machine. Our pressings to date have involved compositions in the hypereutectic region for TaC-C and NbC-C.

The mold material used for these fabrications was of grade ATJ (Union Carbide). This premium grade material has a much finer grain structure and exhibits higher strengths than the standard CS mold stock. It was felt that higher fabrication pressures could be used with ATJ molds and that better retention of mold strength would prevail at the very high (greater than 3000°C) temperatures needed for TaC-C and NbC-C. The compositions which were fabricated in the niobium system were 70 and 80 wt% and in the tantalum, 82.5 and 90 wt%.

A 70 wt% Nb billet was fabricated at a pressure of 4000 psi and a temperature of 3100°C. Examination of the finished sample and mold revealed gross extrusion of material around the plungers as shown in Fig 3. At the 80 wt% Nb level, a pressure of 3000 psi was used to obviate the considerable loss of material. The results of two pressings are shown in Fig 4. Sample A showed distinct zones of recrystallized melt (area 2), graphite (area 1), and what appeared to be a dense, homogeneous area. Not included in this photograph are two zones above the graphite (area 1) layer, which fell away from the rest of the sample in removal from the mold. The recrystallized melt (area 2) was extremely fragile and showed shiny platelets of graphite which had come out of solution.

Photomicrographs of area 2 reveals a eutectic structure with long platelets of graphite (Fig 5) similar to that found in arc cast TiC-C and ZrC-C of hypereutectic composition[4]. Fig 6 shows the structure in area 3. Again, a eutectic structure is revealed with larger amounts of graphite. A much more finely structured eutectic is seen in the center. For comparative purposes, the microstructure of a 80 wt% of Nb body pressed at 3000°C is presented in Fig 7; no eutectic structure is revealed for this material.

From these results it would appear that a temperature of 3250°C (NbC-C eutectic) was attained during the processing. Such high temperatures may not be deleterious if a lower metal content such as 50 wt% is used since melting would be in isolated islands. However, in an 80 wt% mixture which is quite close to the eutectic (81.3 wt%) composition, practically the whole sample melts resulting in gross loss of material thru extrusion around the plungers and reaction with the mold. Heterogeneity also can result due to different rates of crystallization.

An example of the extent of melting which occurs for a low metal content body vs that for a eutectic composition is illustrated in Fig 8 for zirconium bodies heat treated at approximately 2800°C. Both samples were of a bar shape prior to heating.

High tantalum composites did not exhibit such gross loss of material or reaction with the mold. The surfaces of the billets were not as hard as for niobium composites, indicating poorer sintering. These experiments suggest that future pressings in the tantalum system can tolerate higher temperatures whereas a lowering of temperature is indicated for fabricating NbC-C composites.

Samples of these various compositions are now being sectioned from the billets for evaluation. An important consideration for the Nb composites will be metal analysis to determine actual metal content since substantial loss of metal may have occurred.

3. Metal Analysis

Gross extrusion of metal to the outside walls of molds had been noted in past pressings, especially those involving the lower melting materials. It has also been observed that in systems such as zirconium and titanium, a "plateauing" was observed in densities at the 30-50 wt% metal levels. A number of samples involving various metals were subjected to an analysis for metal content. The procedure consists of oxidizing the metal carbide-graphite under a stream of oxygen at 900°C and gravimetric analysis of the metal oxide product.

Tabulated in Table II are the results of these analyses. For the titanium system considerable loss of metal occurred at both processing temperatures. This was reflected in the aforementioned low strengths for these bodies. Zirconium bodies processed at 3000°C exhibited strong loss of metal; however, a 2800°C fabricated temperature which is below the ZrC-C eutectic yielded bodies which had lost very little material. The exception was the zirconia metal source sample which probably suffered metal loss due to strong evolution of gasses in the oxide-carbon reaction.

Some of the billets prepared in the early part of the program had shown strong heterogeneity in density. The present analysis shows the major reason to be disproportionation in metal content as shown for the 50 wt% Hf and 50 wt% Nb samples (Table II). The 65 wt% Hf body had virtually the same metal

content in the processed sample as went into the raw mixture. This appears logical in view of the reported 3180°C for the HfC-C eutectic.

The niobium and tantalum samples also exhibited little, if any metal loss. Again, this is to be expected from the high eutectic temperatures in these systems.

The molybdenum system was of particular interest since fabrication was conducted at temperatures well above the reported eutectic of 2580°C. Analyses showed that at the 30 wt% level, little if any metal loss occurred. However, at the 50 wt% level, metal loss varied depending on the heat treatment. As shown, increase in pressing temperature and soaking time resulted in increased losses of metal. An interesting observation is that based on the theoretical density as calculated from actual metal content, the degree of densification of all the 50 wt% samples fall in the 95.2 to 95.5% region.

The significant loss of metal which can and does occur when the processing temperature exceeds the carbide-carbon eutectic has been verified by these experiments. Similar analyses will be conducted on all future compositions.

4. Use of Metal Oxide as Metal Source

Attempts were made to fabricate bodies substituting ZrO_2 and TiO_2 for the respective carbides as the metal source. It was felt that the oxide could be used in a much finer form than the carbide and that in forming the carbide, a fine dispersion of the carbide in the graphite matrix could be obtained. This is particularly important in lower metal content samples in which such a fine dispersion has been found to aid in densification and good bonding.

The fabrication procedure consisted of a 30 minute hold at 1800°C to permit the oxide-carbon reaction and a final processing temperature of 2800°C. Initiation of the MO-C reaction is listed at 1700°- 1800°C[5] and the eutectic temperatures are reported as 2780°C for TiC-C and 2910°C for ZrC-C[6].

During the processing, substantial evolution of gasses from the oxide-carbon reaction was evident. Only the zirconium carbide system could be evaluated; significant extrusion of material around the plunger occurred in the titanium system precluding a sample large enough for evaluation. As shown in Table I, both density and flexural strength were relatively low as compared to those for a similar composition utilizing carbide (3.29 g/cc, 13,400 psi). Microstructural examination revealed a fine but somewhat heterogeneous dispersion of the carbide phase and also the presence of large pores.

Future experiments employing oxides will consider moderation of the oxide-carbon reaction. This can be accomplished by lowering the reaction temperature from 1800° to about 1500°C, soaking at the initial plateau temperature for longer times, or pre-reacting the raw materials prior to actual hot pressing. This should prevent excessive loss of material and segregation which are detrimental to physical properties.

5. Mo-C System

During mechanical property evaluations at 2500°C, it was seen that some of the molybdenum composites formed droplets on the surfaces of the test specimens. Experiments were conducted to investigate this phenomenon. The 50 wt% Mo samples listed in Table II processed at 2800°C, 2800°C/1 hr, 3000°C, and 3000°C/1 hr were subjected to a temperature of 2600°C for two hrs. "Sweating" occurred most strongly for the 50 Mo-2800°C, or the highest metal content samples, and did not occur in the 50 Mo-3000°C/1 hr samples. The weight losses

were: 50 Mo-2800°C, 3.22%; 50 Mo-2800°C/1 hr, 1.91%; 50 Mo-3000°C, 1.80%; and 50 Mo-3000°C/1 hr, 0.11%.

Shown in Fig 9, is a "sweated" sample and microstructural examination of the cross section of a bubble. The area directly under the exuded carbide bubble shows dense graphite and large carbide particles. X-ray examination revealed the bubble to be primarily Mo_2C with small amounts of MoC and C. It would appear that the Mo_2C phase coalesces and finally escapes the parent body, leaving behind a highly oriented graphite phase as indicated by the same area under polarized light (Fig 10).

The high degree of ordering and strong bond obtained in brazing of graphite as observed at Los Alamos and Westinghouse may be due to this type of mechanism. X-ray examination of this area will be conducted to determine the degree of graphite ordering. In addition, electron microprobe analysis will be used to determine if any molybdenum may be dissolved in the graphite lattice. It may be possible to exploit this behavior in producing graphites of high density and strength.

B. Physical Properties

The property of composites which has received the most study has been flexural strength. This has been predicated on the ease of fabricating a sample specimen shape and on the speed and simplicity of flexural evaluations. A good screening of potential systems in terms of high temperature behavior is accomplished. Furthermore, other properties such as tensile and compressive strength can be predicted reasonably well using flexural data, e.g., the former is approximately 0.67 times, and the latter, 5 times the flexural value. As the optimum compositions emerge from these fabrication and flexural studies, physical properties measurements will be expanded to include compression, tension, elastic moduli, compressive

creep, thermal expansion, thermal conductivity and thermal shock. Apparatus for both tension and compression at high temperatures is now being readied.

Composites of 80, 81.3 and 84 wt% niobium prepared at IITRI have been submitted to LASL for their evaluation. Preliminary data generated at Los Alamos show that these materials exhibit less than 2% creep in compression (2700°C/2000 psi/30 min) and flexural strengths in excess of 20,000 psi.

1. Flexural Strength

During this period, flexural strength tests have been conducted at 2500°and 2800°C, employing four point loading with a load rate of 300 psi/sec. At these temperatures substantial plastic deformation was exhibited by many of the materials; a tantalum system was the only composite which showed very little deformation at 2500°C. The results of these tests are graphically presented in Figs 11-13. In Fig 14, photographs of test specimens in the various metal systems are presented. Brittle failure reflecting the lameller structure occurred for all samples. The extent of plastic deformation is also illustrated.

Composites incorporating molybdenum (Fig 11) exhibited a loss in strength at 2000°C, and at 2500°C, only the samples of lower metal content could be stressed to failure. The others revealed considerable plastic deformation; a load of greater than 25,000 psi was attained without failure. Considerable deformation occurred for the samples which did fail as shown in Fig 14. In the A/G direction, all samples showed strong deformation at 2000°C.

In Fig 12, the data obtained for a variety of compositions are presented. The zirconium containing bodies

displayed a significant increase in strength at 2000°C but a strong dropoff at 2500°C. The higher metal content composition (80.7 wt%) displayed a stronger loss and was actually weaker (11,300 psi vs 12,600) than the 50 wt% composite at 2500°C. At 2600°C, failure could not be induced as illustrated in Fig 13.

Vanadium samples exhibited behavior similar to that for molybdenum systems, i.e., lower strengths at elevated temperatures. The hafnium and tantalum composites displayed increased strength at 2000°C. Altho high plastic deformation was observed for the hafnium body at 2500°C, good retention of strength was evident. As illustrated in Fig 13, the tantalum composite showed little deformation at 2500°C where the strength was higher than that at room temperature. At 2800°C, however, plastic deformation was evident; the samples which did fail revealed a strength of 6860 psi, and those which did not fail were carried to a load of greater than 25,000 psi.

Among the niobium composites, the best high temperature properties were displayed by the 50 wt% composition (Fig 13). As was the case for zirconium composites, the higher metal content composition revealed a large decrease in strength at 2500°C. The 50 wt% composition displayed strengths at 2500°C which were about 30% higher than the room temperature value. In the A/G direction, the 30 wt% composition as well as the 50 wt% showed little change with temperatures, although plastic deformation was apparent. At a test temperature of 2800°C, failure occurred at 14,100 psi for some samples whereas others of the same composition were stressed to a load of 25,000 psi without failure. Test bars of 50 wt% Nb are shown in Fig 13.

Tests in the A/G direction have been limited due to some difficulties in obtaining billets of the proper size. Future pressings will incorporate enough materials so that such evaluations will be possible.

The anisotropy ratio for most of the 50 wt% material has been about 4:1 in flexure. It is anticipated that as the carbide becomes the matrix phase, this ratio will drop. Work at LASL has shown this ratio to be about 1.5 near the eutectic.

These data show that both tantalum and niobium compositions have good potential as materials for use at 3000°C. The other compositions display either drastic losses in strength or high deformation at 2500°C. An additional limitation, of course, is that except for hafnium, eutectic temperatures of the metal carbide-carbon are all lower than 3000°C. Tests at 3000°C will be conducted upon modification of the flexural test apparatus using high strength premium grade graphite fixtures.

2. Elastic Modulus

Room temperature measurements of elastic modulus were conducted on a series of compositions. Deflection as a function of load using four point loading was measured with an extensometer. The data are tabulated in Table III.

Samples of lower metal content, i.e., 50 wt%, had relatively low moduli which are comparable to those of premium grade graphites such as the ZT grades. Similar to the hot pressed Zt materials, considerable anisotropy was shown as seen for 60 wt% Ta and 50 wt% W composites.

With increasing metal content in the niobium system into the eutectic composition region, the carbide becomes the dominant phase and a sharp increase in elastic modulus

exists. A stress strain diagram of a 84 wt% Nb exhibiting non-plastic behavior is shown in Fig 15. It can be seen that deformation is slightly retarded at the higher loads. The lower metal content materials all exhibited slightly increasing deformation with greater loads.

Although the 80-84 wt% Nb bodies have high room temperature elastic moduli, flexural strength evaluations have shown that high niobium composites exhibit as much deformation at high temperature as the lower (50 wt%) material. Thus a large decrease in elastic modulus is indicated. Tests will be conducted in the near future at the higher temperatures.

3. Compression Tests

Four of the most refractory systems were examined for compressive creep behavior under a 4000 psi load at 2500°C. The compositions were tested in the form of 1/4 in. diameter x 3/4 in. high right cylinders. Sample deformation was monitored visually with a cathetometer. Listed in Table IV are the systems tested and creep exhibited.

Table IV

Compressive Creep
of
Metal Carbide-Graphite Composites
at 2500°C/4000 psi

Composition - Pressing Temperature	Creep %	Load Time Period
50 wt% Ta - 3100°C	2.2	One hour
50 wt% Nb - 3100°C	6.2	One hour
50 wt% Zr - 2800°C	12.5	One hour
50 wt% Hf - 3000°C	15.0	Thirty minutes

As indicated in the Table, the tantalum system exhibited good resistance to creep. A slightly larger deformation was shown for the niobium sample, and the hafnium and zirconium bodies both exhibited significant creep. A column action occurred in the Zr and Hf bodies and bowing of the cylinders indicated that loading was not perfectly uniaxial. This may have been caused by creep occurring in the test fixtures. Modifications will be made in the furnace and test apparatus so that higher temperature experiments can be conducted. This will consist of installing a support rod directly under the loading rod; the present system employs support rods under the two points of load in a flexural apparatus. In addition, higher strength graphite fixtures will be employed.

4. Thermal Shock

Preliminary experiments were conducted to examine resistance to thermal shock of various composites. The test consisted of rapid heating of one end of a 1/4 in. cross section test bar with a plasma jet at a 1/2 in. distance under an inert (argon) atmosphere. Three cycles of 30 sec on, followed by a cooling gas stream of argon, were used. The equipment used was a Plasmadyne SG-3, 25 KW unit mounted within a dry box arrangement, capable of a horizontal travel to obtain the on-off cycles on stationary samples.

During the test, temperatures of greater than 3000°C was achieved at the test tip of the sample within 20 sec. On subsequent cycles, this temperature was reached in 15 sec. On the off cycles, cooling to below 800°C was accomplished within 10 sec.

These early tests indicate that the metal carbide-graphite composites have good resistance to thermal shock. No failure due to cracking occurred in any of the samples. Compositions which were subjected to this test include tantalum, niobium and zirconium bodies; in addition, ATJ graphite test bars were subjected to this test. The attainment of very high temperatures (> 3250°C) as monitored by an L & N pyrometer was verified by the fact that both niobium and zirconium composites underwent melting; tantalum composites did not show such melting, indicating that the highest temperature was in the range of 3250° - 3450°C.

Improvements in testing technique will be incorporated prior to any further testing. Some difficulty was encountered in torch alignment, and some slight oxidation prevented accurate data gathering as to dimensional and weight changes. Pure carbide materials which are known to have poor thermal shock resistance will be obtained for comparative evaluation.

Efforts will also be made to use different types of thermal shock tests. The present test involves small samples and altho thermal gradients are induced in the longitudinal axis, radial gradients are quite limited. Experiments in which discs are thermally stressed in the center resulting in strong radial gradients to the periphery will be used.

III. CONCLUSIONS AND FUTURE WORK

During this period, the relationship between processing temperature and carbide-carbon eutectic temperature has been examined. This has been supplemented by metal analysis which indicates substantial metal loss in some of the systems. Physical property measurements have been extended to 2500° - 2800°C. In addition, thermal shock evaluations have been initiated. The most significant results of the present work can be considered as follows:

(1) Fabrication at temperatures which greatly exceed the metal carbide-carbon eutectic can result not only in significant loss of metal and a concomitant degradation of properties, but also in gross segregation of the two phases and formation of large pores. This has been shown for the vanadium system which exhibits much higher flexural strength when prepared at 2600°C as opposed to 3000°C,

(2) For the lower (50 wt%) metal content tantalum and niobium composites, increasing processing temperature to 3100°C yields composites of higher flexural strength. However, at the higher niobium metal levels near the eutectic compositions, melting will occur in nearly the entire billet when a temperature of 3250°C is approached unlike the 50 wt% material in which the major constituent, graphite, will remain as a solid. Thus a moderation in processing temperature is necessary in the niobium system with increasing metal content.

(3) Examination of a molybdenum-graphite composite heat treated at 2600°C shows "sweating" of the sample. The exuded material, which is primarily Mo_2C with small amounts of MoC, leaves behind a highly oriented graphite phase.

(4) Flexural tests at 2500° and 2800°C show that plastic deformation occurs for all samples to varying degrees. The tantalum and niobium systems have exhibited the best properties, both in flexure and compression.

(5) Preliminary tests have indicated good thermal shock resistance for hot pressed metal carbide-graphite composites.

Future work will involve complete evaluation of the physical properties of niobium and tantalum composites with special emphasis on compositions near the eutectic. These evaluations will be conducted at temperatures up to 3000°C.

In addition, a limited study will be conducted on the effect on molybdenum carbide on the degree of ordering and improved properties of graphite. Zirconium and molybdenum composites prepared at LASL by vacuum hot pressing will be evaluated.

IV. CONTRIBUTING PERSONNEL AND LOGBOOK RECORDS

Contributing personnel include M. A. Schwartz, G. A. Rubin, S. A. Bortz, R. Baker and D. Berent. Data are contained in Logbook Nos. C16487, C16500, C16802 and C16806.

Respectfully submitted,

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Table I

PROPERTIES OF METAL CARBIDE-GRAPHITE COMPOSITES

Metal Additive		Vol % Carbide	Pressing Temp., °C	Density g/cc	Flexural Strength, psi	
Form	Wt%				Room Temp.	2000°C
ZrO_2	50	32.7	2,800	2.40	7,540	
VC	50	37.8	2,600	2.46	16,710	16,980
VC	50	37.8	3,000	2.29	4,710	7,200
Mo	50	23.7	2,600	3.07	19,040	15,670
TiC	50	43.5	2,750	2.03	5,570	
Nb	50	27.1	3,100	3.42	14,530	21,080
Nb*	50	27.1	3,000	3.38	16,080	18,660
Nb	50	27.1	3,000	3.51	6,100	9,100
TaC	50	15.2	3,100	3.86	8,570	11,980
Ta	50	15.2	3,000	4.01	2,750	3,810
TaC	60	22.0	3,100	4.64	8,200	10,540 (W/G)
					3,200	3,340 (A/G)

*First year's program. See Reference (1).

Table II

METAL ANALYSIS OF METAL CARBIDE-GRAPHITE COMPOSITES

Composition	Pressing Temp., °C	Sample	Density g/cc	Actual Metal Content	Actual Theoretical Density	% Theoretical Density	
50 Ti	3,000	T1	2.08	13.3	2.48	83.9	
50 Ti	2,750	B3	2.04	17.3	2.56	79.7	
50 Zr	3,000	T2	2.22	16.1	2.57	86.4	
50 Zr	2,800	1B	3.32	48.3	3.53	94.1	
50 Zr*	2,800	T5	2.41	21.0	2.68	89.9	
65 Zr	3,000	T3	2.12	17.9	2.61	81.2	
80.7 Zr	2,800	T4	5.55	79.5	5.59	99.3	
50 Hf	2,800	T8	3.71	51.7	4.14	89.6	same billet
50 Hf	2,800	T25	2.33	23.5	2.85	81.8	
65 Hf	3,000	4B	4.99	65.1	5.27	94.7	
50 Nb	3,000	T1	3.18	42.4	3.43	92.7	same billet
50 Nb	3,000	T6	3.51	49.0	3.73	94.1	
50 Nb	3,000	3T	3.52	50.4	3.80	92.6	
50 Nb	3,100	C5	3.42	48.2	3.69	92.7	
81.3 Nb	3,000	3B	6.30	81.3	6.53	96.5	
50 Ta	3,000	2B	4.01	56.7	4.61	87.0	
50 Ta	3,100	Tc3	3.88	50.4	4.14	93.7	

Table II (Cont'd)

Composition	Pressing Temp., °C	Sample	Density g/cc	Actual Metal Content	Actual Theoretical Density	% Theoretical Density
30 Mo	2,800	T3	2.50	30.4	2.97	84.2
30 Mo	3,000	T1	2.60	29.9	2.96	87.8
50 Mo	2,600	B4	3.10	38.5	3.25	95.4
50 Mo	2,800	5B	3.12	39.0	3.27	95.4
50 Mo	2,800/1 hr	5B	2.97	34.8	3.12	95.2
50 Mo	3,000	5B	2.97	34.7	3.11	95.5
50 Mo	3,000/1 hr	5B	2.75	27.5	2.89	95.2
50 W	3,000	3A	3.70	48.9	4.08	90.7

*ZrO_2 as source of metal

Table III

ELASTIC MODULUS OF METAL CARBIDE-GRAPHITE COMPOSITES

Composition	Pressing Temp.,°C	Secant Elastic Modulus $X10^6$	Flexural Strength psi	Volume Percent Carbide
50 Zr	2,800	2.17	13,400	32.7
65 Hf	3,000	4.28	7,305	27.2
50 V	2,600	4.44	16,750	37.8
50 Nb	3,100	2.54	14,530	27.1
80 Nb	3,000	24.0	19,520	72.9
81.3 Nb	3,000	25.60	26,720	76.4
84 Nb	3,000	29.52	23,200	84.2
50 Ta	3,100	1.70	8,570	15.2
60 Ta	3,100	3.04 (W/G)	7,230	22.0
		0.75 (A/G)	2,350	22.0
50 Mo	2,800	2.70	18,930	10.9
50 Mo	3,000/1 hr	3.39	17,020	16.8
50 W	3,000	3.88 (W/G)	14,200	14.2
		1.20 (A/G)	4,660	14.2

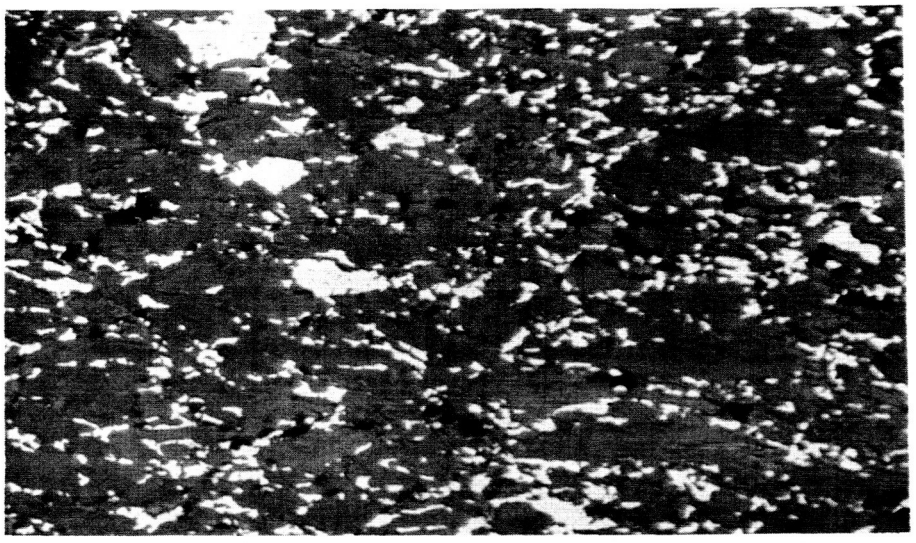


FIG 1 - MICROSTRUCTURE OF 50 WT% Ta-GRAPHITE
PRESSED AT 3100°C (320x)

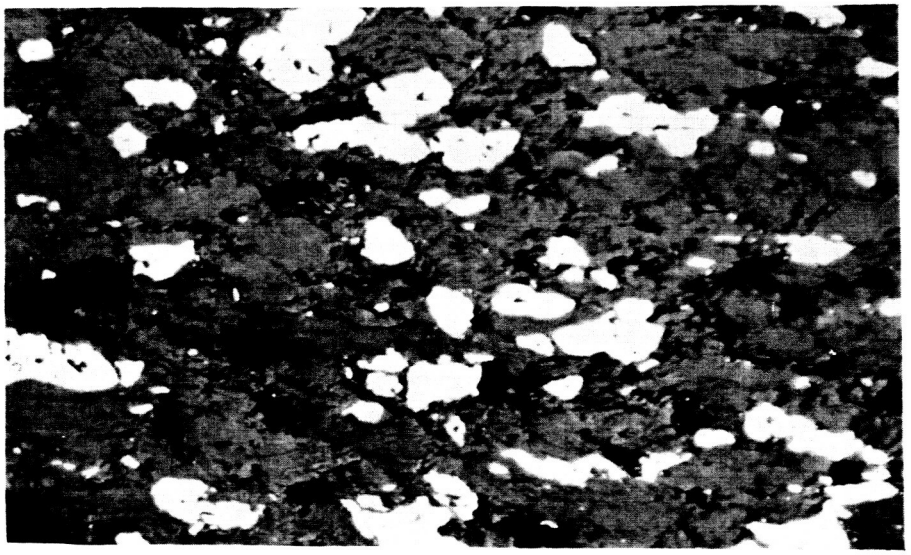


FIG 2 - MICROSTRUCTURE OF 50 WT% Ta-GRAPHITE
PRESSED AT 3000°C (320x)

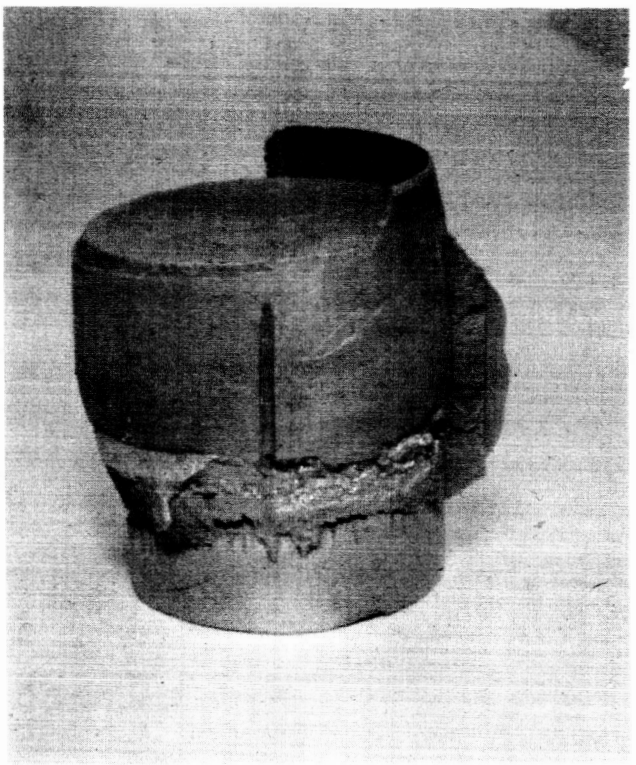
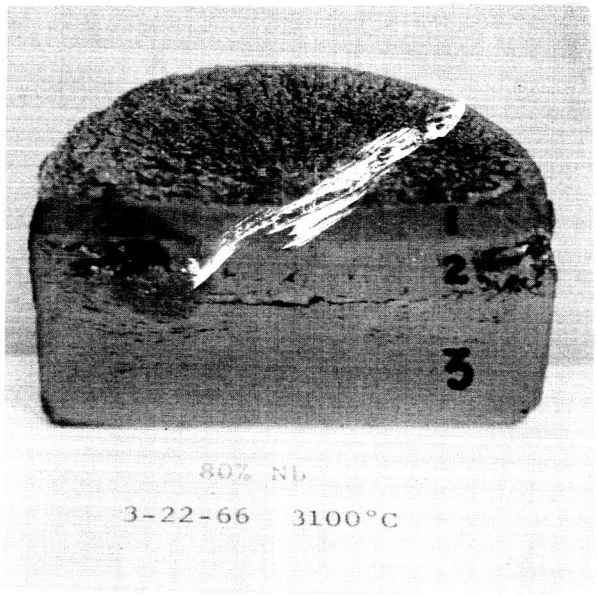
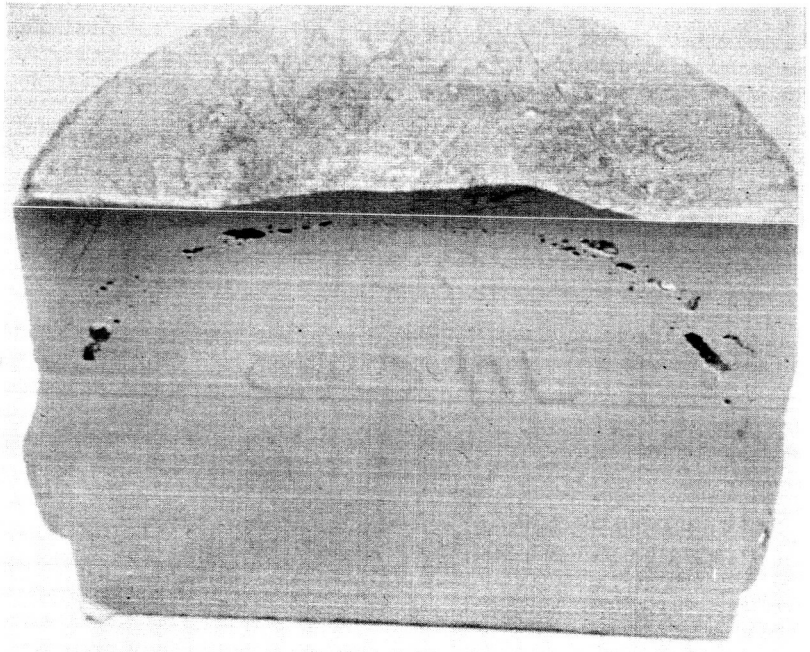


FIG 3 - BILLET OF 70 WT% Nb-GRAPHITE
PRESSED AT 4000 PSI AND 3100°C



A



B

FIG 4 - BILLETS OF 80 WT% Nb-GRAPHITE
PRESSED AT 3000 PSI AND 3100 °C

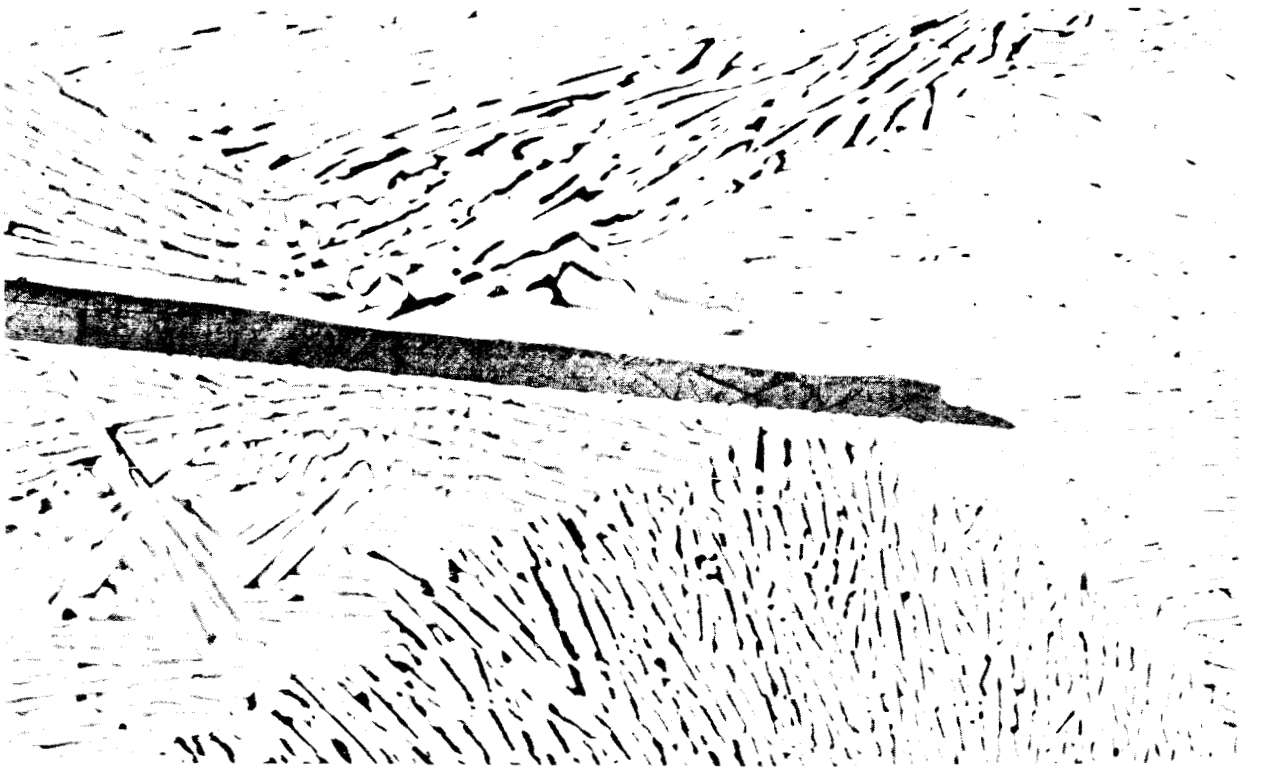


FIG 5 - MICROSTRUCTURE OF ZONE OF RECRYSTALLIZED MELT IN 80 WT% Nb PRESSED AT 3100°C SHOWING EUTECTIC STRUCTURE (x200)

FIG 6 - MICROSTRUCTURE OF 80 WT% Nb-GRAPHITE
PRESSED AT 3100°C (x200)

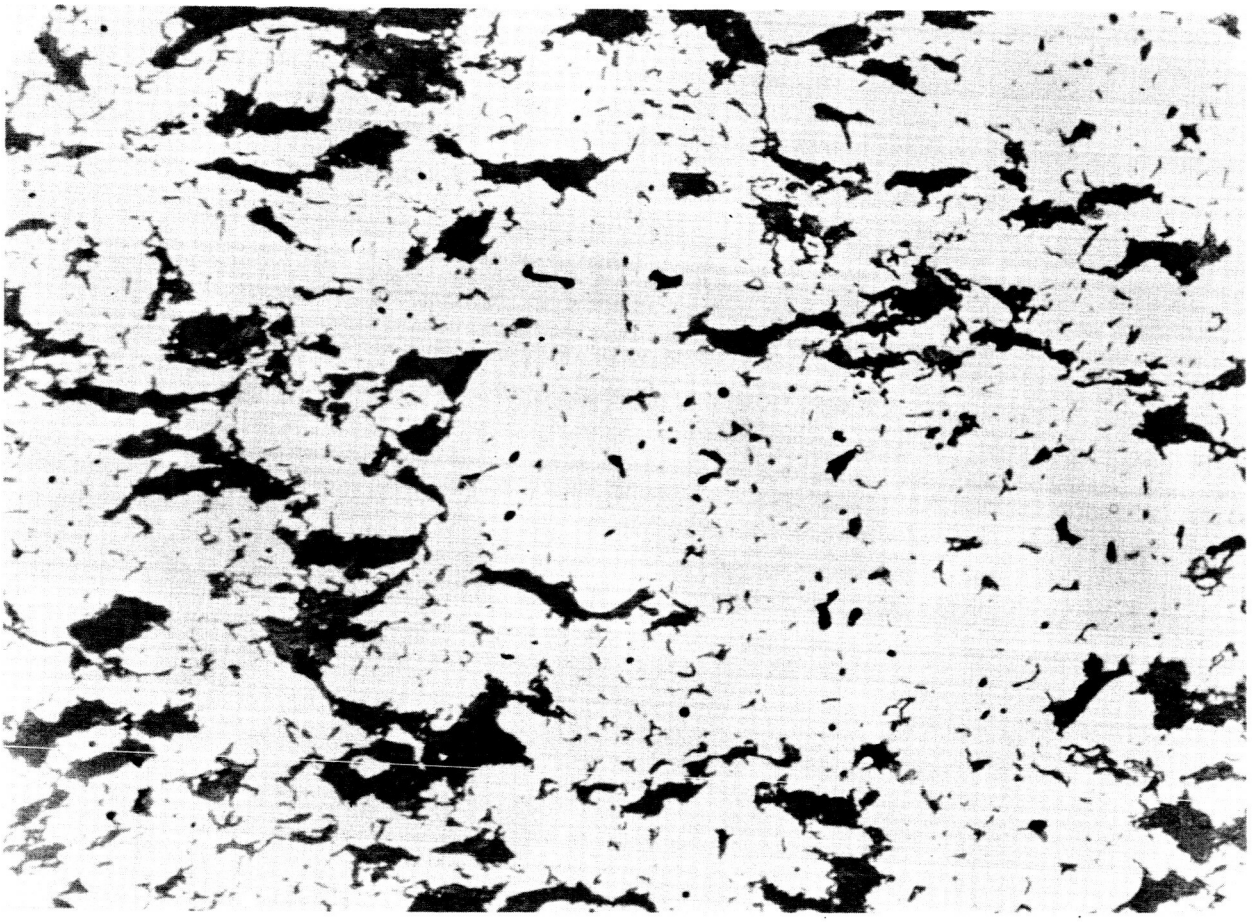
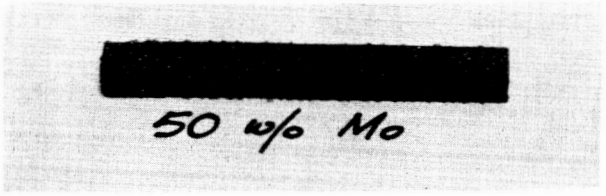


FIG 7 - MICROSTRUCTURE OF 80 WT% Nb-GRAPHITE
PRESSED AT 3000°C (x320)

FIG 8 - ZIRCONIUM CARBIDE - GRAPHITE SPECIMENS
HEAT TREATED AT 2800°C/10 MINUTES



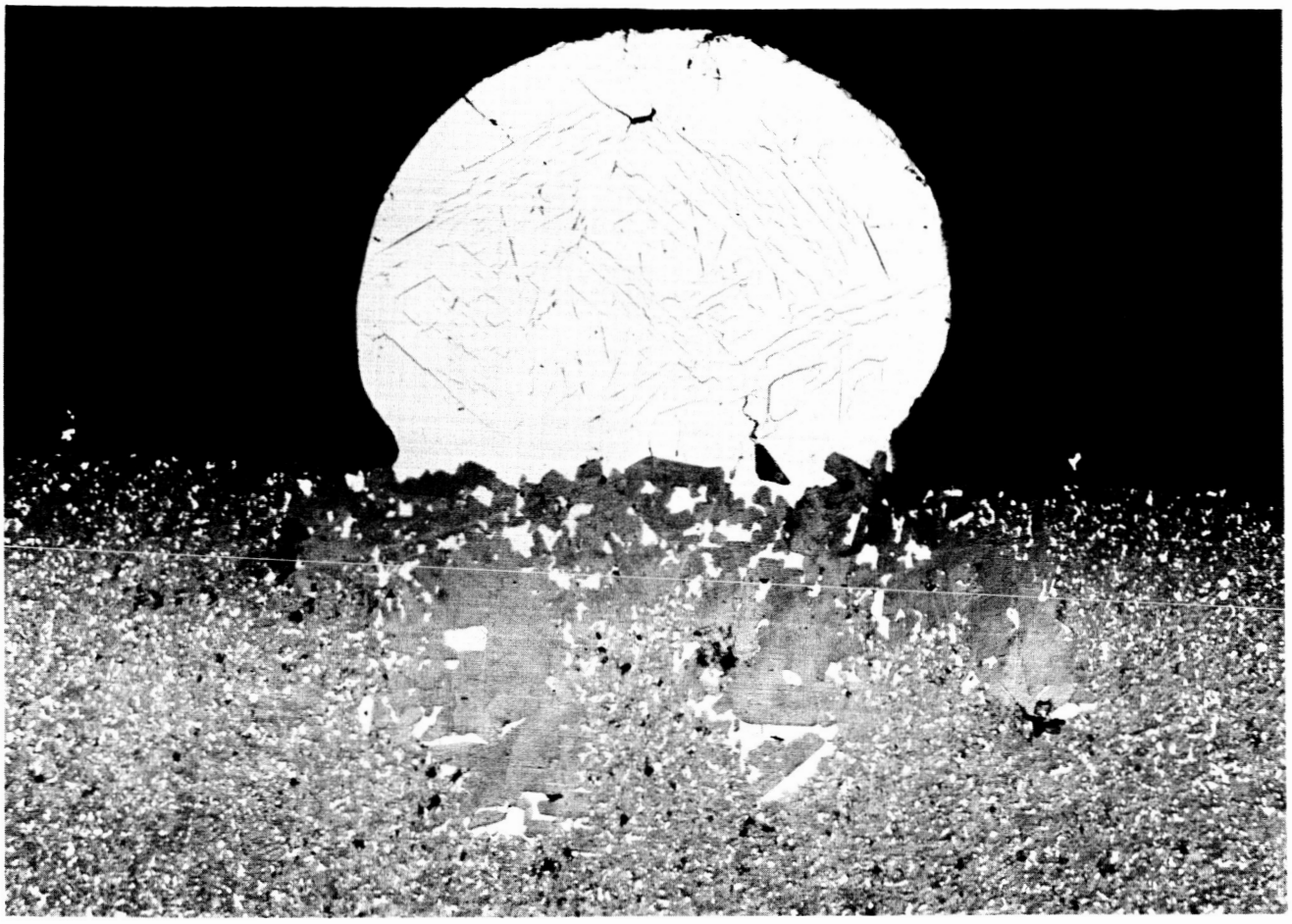


FIG 9 - MICROSTRUCTURE OF "SWEATED" AREA IN
50 WT% Mo-GRAPHITE SAMPLE HEAT
TREATED AT 2600°C/2 HOURS (x100)

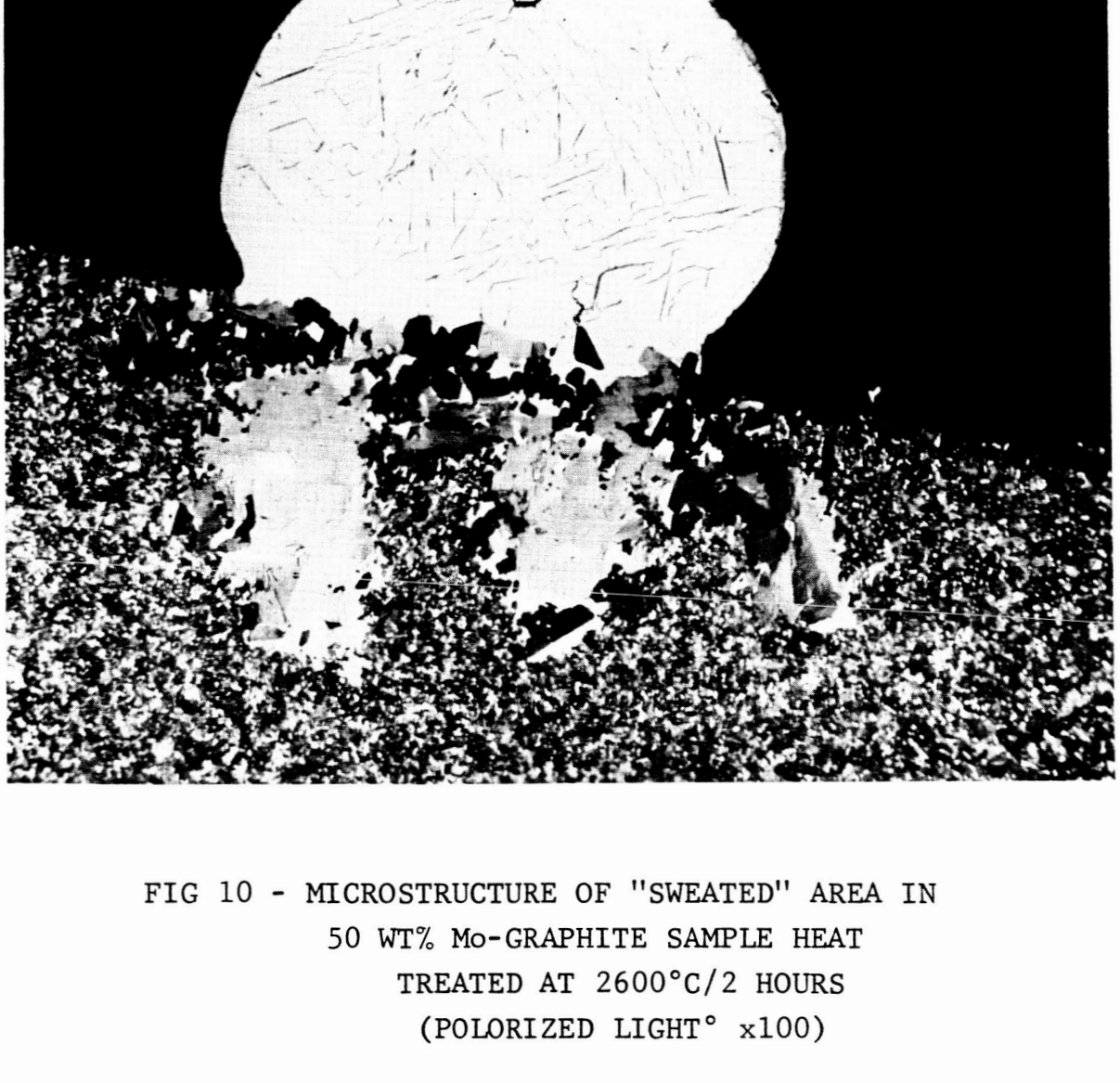


FIG 10 - MICROSTRUCTURE OF "SWEATED" AREA IN
50 WT% Mo-GRAPHITE SAMPLE HEAT
TREATED AT 2600°C/2 HOURS
(POLORIZED LIGHT° x100)

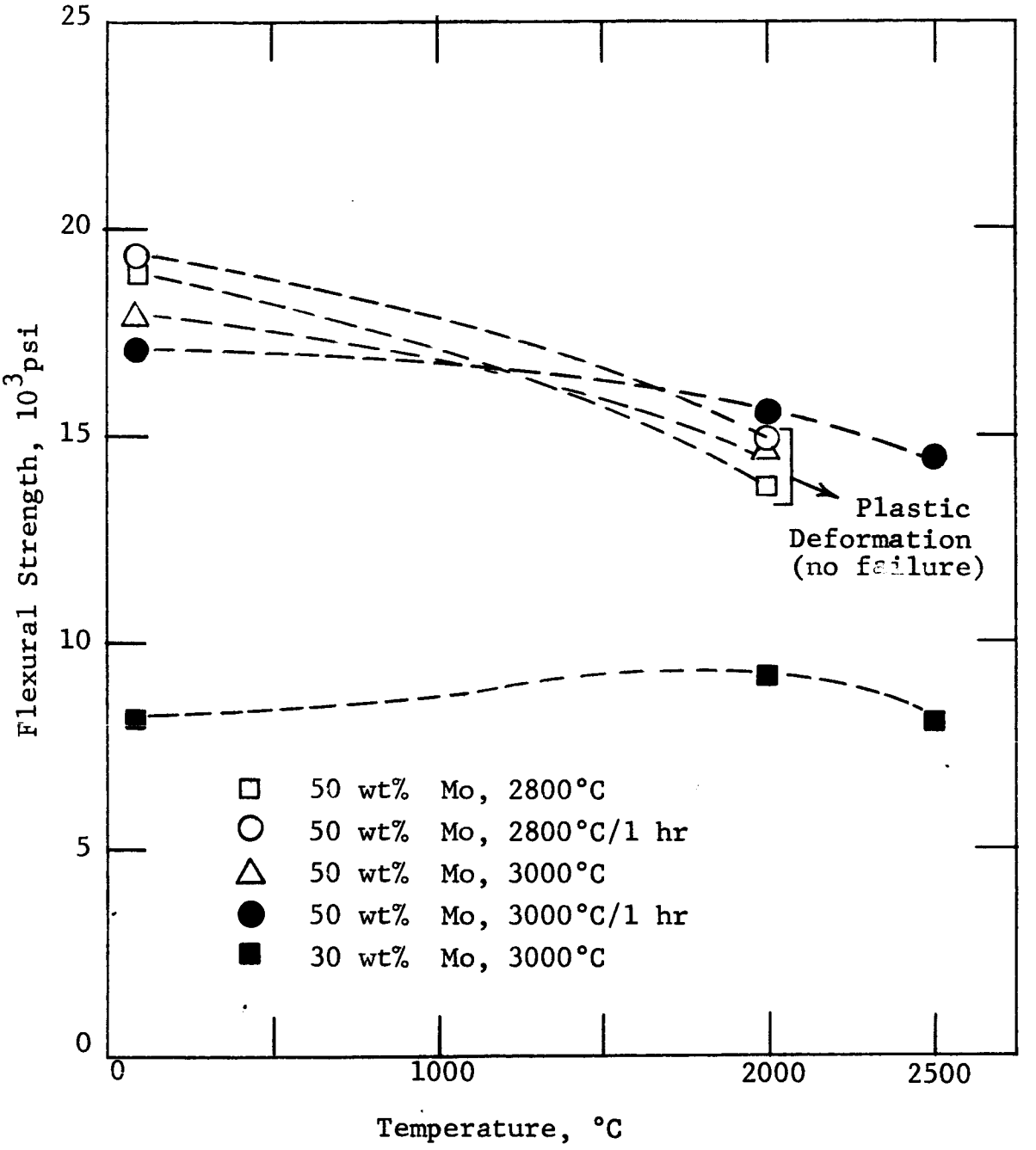


Fig 11 HIGH TEMPERATURE FLEXURAL STRENGTH OF Mo-C COMPOSITES

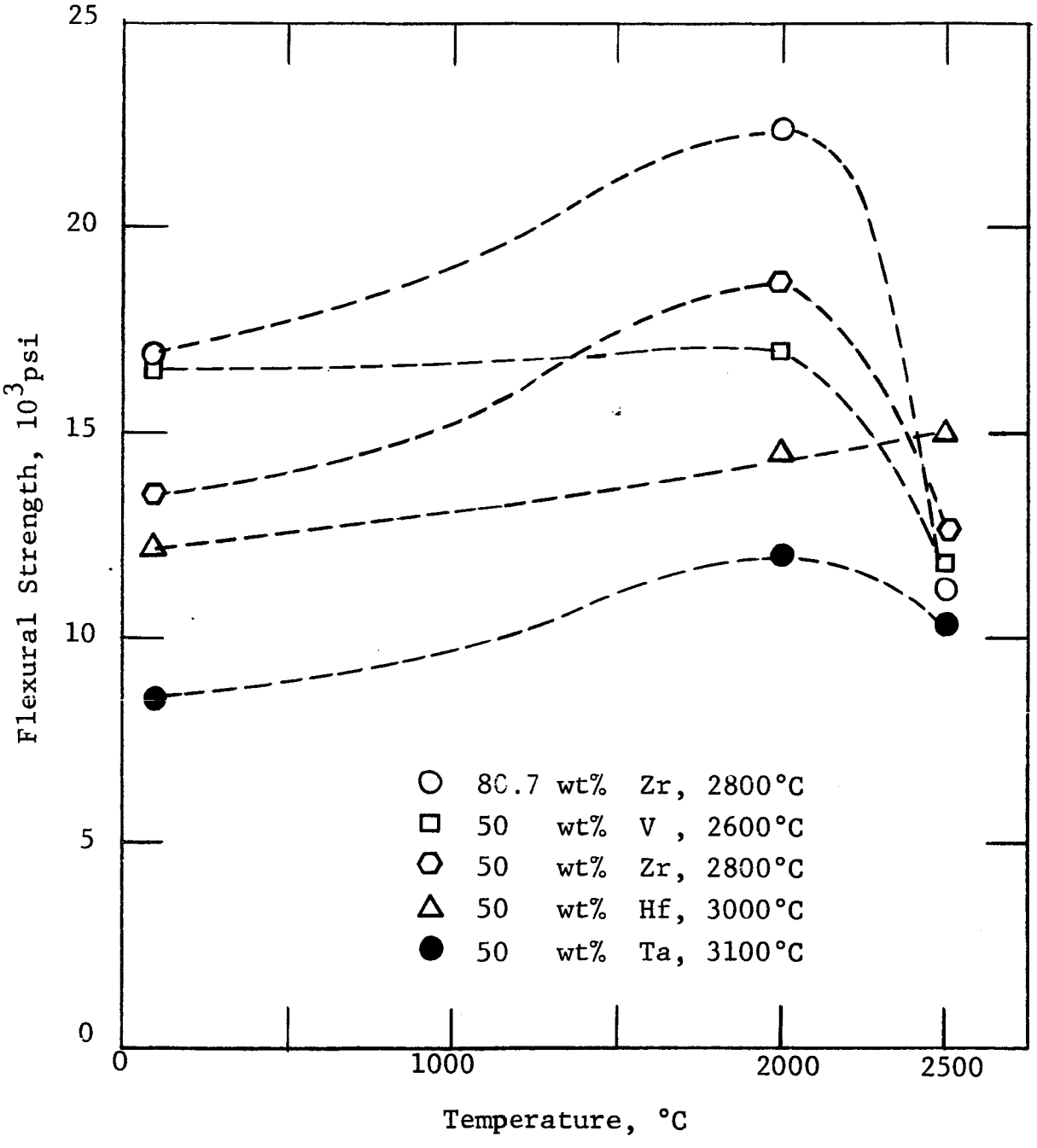


Fig 12 HIGH TEMPERATURE FLEXURAL STRENGTH OF METAL CARBIDE-GRAPHITE COMPOSITES

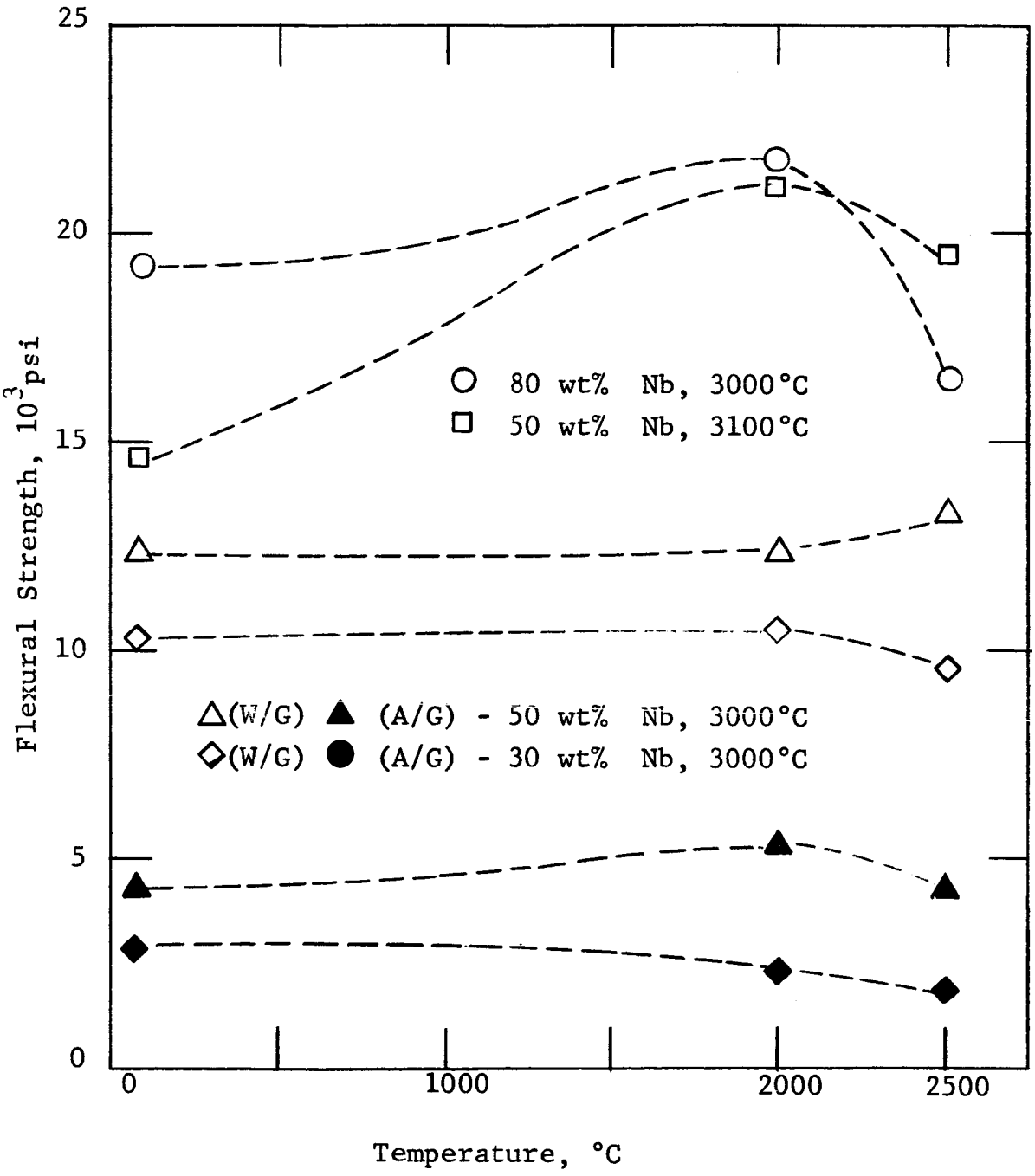


Fig 13 HIGH TEMPERATURE FLEXURAL STRENGTH OF Nb-C COMPOSITES

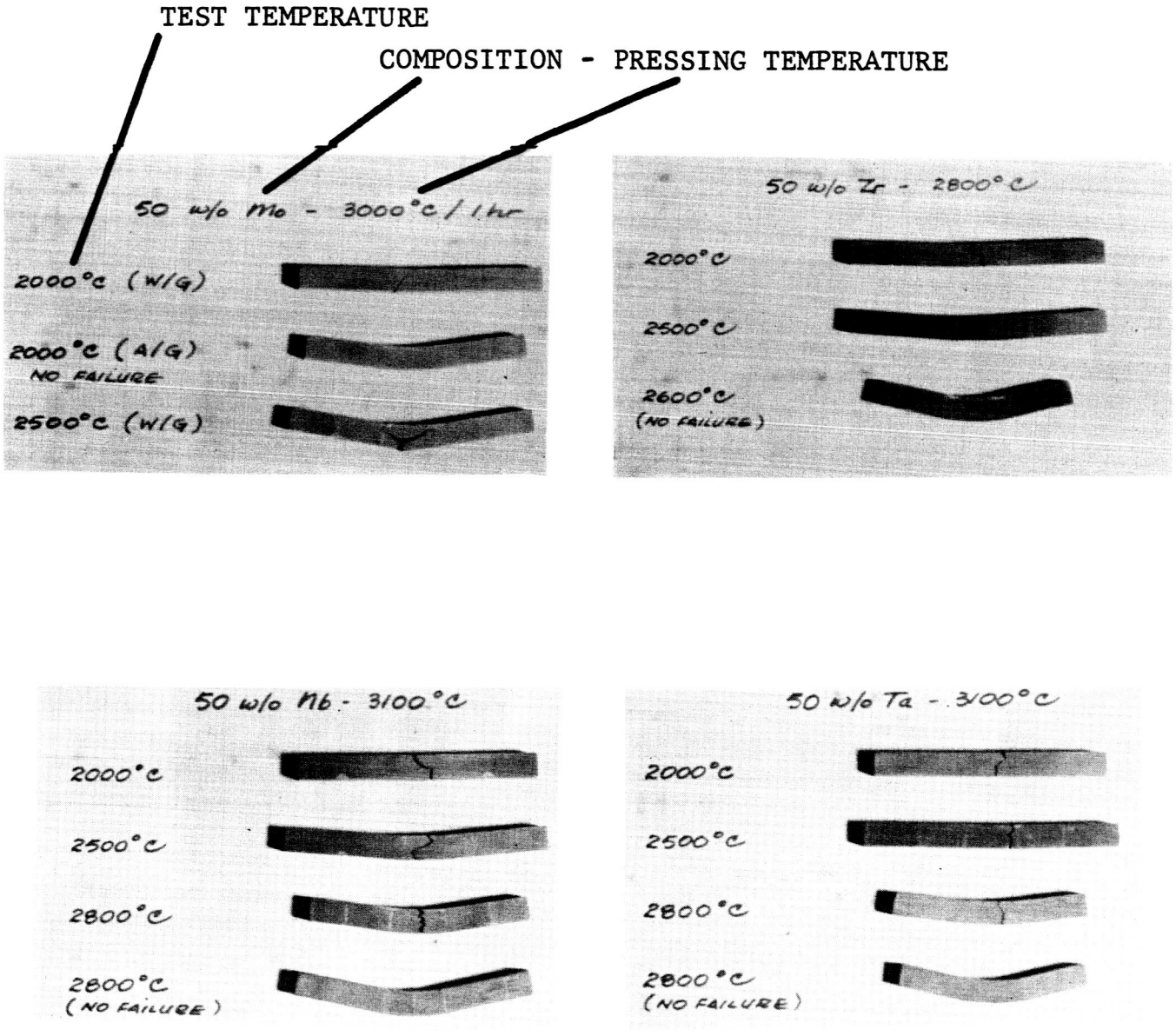


FIG 14 - HIGH TEMPERATURE FLEXURAL TEST SAMPLES OF VARIOUS METAL CARBIDE-GRAPHITE COMPOSITIONS

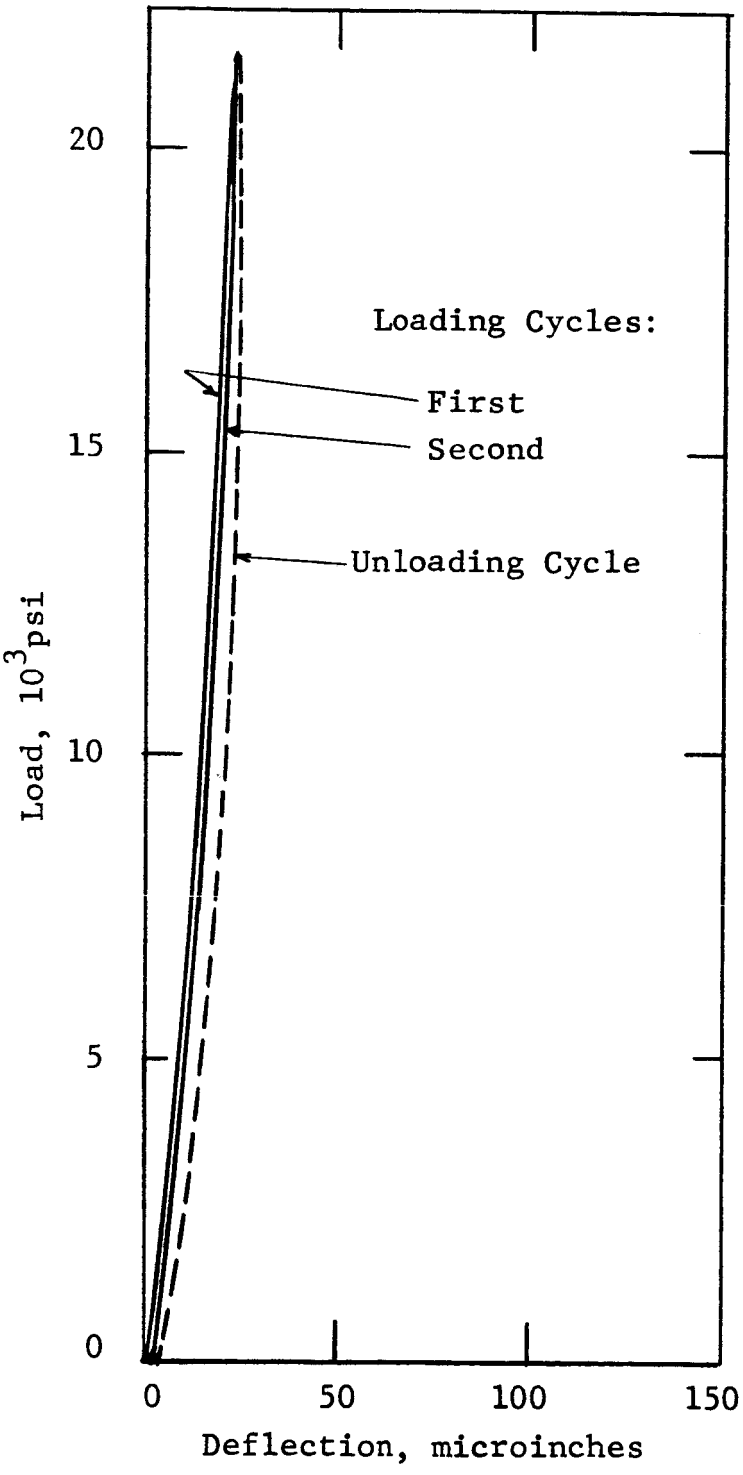


Fig 15 LOAD-DEFLECTION CURVE IN FLEXURE FOR 84 wt% Nb-GRAPHITE COMPOSITE