MASTER

LOS ALAMOS SCIENTIFIC LABORATORY
of the
University of California
LOS ALAMOS, NEW MEXICO

Compatibility of Refractory Metals with Solid
Solution (UC)(ZrC) at Elevated Temperatures

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

This report expresses the opinions of the author or authors and does not necessarily reflect the opinions or views of the Los Alamos Scientific Laboratory.

Printed in USA. Price \$1.00. Available from the Clearinghouse for Federal Scientific and Technical Information, National Bureau of Standards, United States Department of Commerce, Springfield, Virginia.

NOV 8 1965

LA-3307
UC-25, METALS, CERAMICS,
AND MATERIALS
TID-4500 (43rd Ed.)

LOS ALAMOS SCIENTIFIC LABORATORY of the University of California

Report written: February 1965
Report distributed: September 28, 1965

Compatibility of Refractory Metals with Solid Solution (UC)(ZrC) at Elevated Temperatures

by
K. V. Davidson
R. E. Riley
J. M. Taub

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

ABSTRACT

The results of time-temperature thermal treatments designed to investigate the compatibility of refractory metals such as tungsten, molybdenum, and niobium in the presence of solid solution (UC)(ZrC) are reported. Also given are details of the techniques used to fabricate specimens.

ACKNOWLEDGMENTS

The authors wish to acknowledge A. L. Hipp, B. L. Barthell, and J. A. Pritchard, all members of the Powder Metallurgy Section, for preparation and thermal treatment of the various samples. We also wish to acknowledge D. J. Sandstrom for preparation of the magnetically compacted samples, the Physical Metallurgy Section for the metallographic results, the Analytical Chemistry Group for the chemical and X-ray diffraction analysis, and J. A. O'Rourke for the X-ray microprobe analysis.

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

CHAPTER I

INTRODUCTION

The inherent brittleness of specimens fabricated by various techniques from solid solution (UC)(ZrC) suggested the desirability of having a more ductile binder matrix material. Molybdenum, niobium, and tungsten, because of their high melting points, were considered for this purpose. Compatibility of these refractory metals with (UC)(ZrC) was determined by time-temperature stability tests.

CHAPTER II

RAW MATERIALS

A. Molybdenum Powder (Mo)

The molybdenum metal was purchased as powder (lot Mo-32) having an average particle size of 4.3 to 4.5 microns. Quantitative spectrographic analysis of this powder showed all impurity elements to be below the limits of detection, except for Fe, which was 4.5 ppm, and Ni, 7.5 ppm. Oxygen analysis on the starting powder showed the material to contain 2000 ppm oxygen. The powder was hydrogen-reduced immediately prior to use, but no subsequent oxygen analysis was performed.

B. Niobium Powder (Nb)

The niobium metal was purchased as powder (lot Nb-6) having an average particle size of 4.3 microns. Quantitative spectrographic analysis of this material showed all impurity elements to be below the limits of detection, except for 450 ppm Zr.

The oxygen analysis for this powder was not available.

C. Tungsten Powder (W)

The tungsten metal was purchased as powder. Lot W-90 had an average particle size of 43 microns, and lots W-121 and W-123 had an average particle size of 0.7 micron. Quantitative spectrographic analysis of these powders showed all impurity elements to be below the limits of detection, except for 100 ppm Fe and 2000 ppm Ni in lot W-90, and 500 ppm Mo and 100 ppm Ni in lots W-121 and W-123. Oxygen analysis on these powders showed 350 ppm for lot W-90 and 2000 ppm for lots W-121 and W-123.

D. (UC)_{.18}(ZrC)_{.82} Powder

The (UC)(ZrC) powder was prepared by arc melting elemental constituents.¹ Essentially, this procedure consists of solution treatment of the arc-melted buttons, crushing through 20 mesh, followed by solution treatment of the powder, and final crushing through 200 mesh. A typical analysis for carbides prepared by this technique follows:

U (%)	Zr (%)	Total C (%)	Free C (ppm)	N ₂ (ppm)	O ₂ (ppm)
33.3±0.3	57.5±0.3	9.20	<500	<50	700±200

CHAPTER III

EXPERIMENTAL PROCEDURE

A. Fabrication

1. (UC)_{.18}(ZrC)_{.82} + 10 w/o Mo

The pyrophoric nature of (UC)(ZrC) and the susceptibility to oxidation of the high surface area refractory metal powders used required that comminution, weighing, blending, etc., be performed in an inert atmosphere dry box. Figure 1 shows a typical dry box train with the associated auxiliary equipment such as the vacuum pump and oxygen analyzer. Some of the items used inside the dry box are shown in Fig. 2. An argon atmosphere was used, and high purity argon was used to standardize the analyzer and provide the basis for comparison of the atmosphere in the box. Entry into the dry box was accomplished through the locks at the ends and the center of the train. The material was loaded into the lock, and the lock was evacuated and back purged with argon twice and sampled for oxygen before the material was transferred to the working area of the dry box. Utilization of this lock obviated the necessity of purging the dry box each time entry was made.

A Beckman Model E-2 oxygen analyzer was used to determine the oxygen content of the argon atmosphere. The argon inlet and gas sampling tube were located at the top of the dry box and vacuum locks. In addition to the precautions described above, uranium metal powder was exposed to the dry box atmosphere to provide gettering for the traces of oxygen which may remain. Transfer of the materials or specimens from one dry box train to another or to the sintering furnaces was accomplished by use of a vacuum desiccator which had been back-filled with purified argon. Carbide powders were continuously stored in the dry boxes.

A powder charge consisting of a blend of -200 mesh (UC)_{.18}(ZrC)_{.82} powder plus 10 w/o 4 to 5 micron Mo powder (lot Mo-32) was loaded into a die made of A-331 Stackpole graphite. The blending and die loading was carried out in an inert atmosphere dry box. The die was transferred to the vacuum hot press (Fig. 3), which was evacuated, and a load of 1000 psi was applied. The die was then heated to 1200°C in 23 min while the pressure was maintained at 1000 psi. On reaching 1200°C, the pressure was raised 100 psi/min to 4400 psi, while at the same time a moderate heating rate was maintained. The temperature was allowed to increase to 1965°C and was held there until the desired die punch movement was achieved (approximately 5 min). The pressure was then reduced rapidly to 1750 psi, at which time the power was cut back and the die allowed to cool to 1000°C. On reaching 1000°C, the power was cut off and the load reduced to 0 psi. The pressed shape was removed from the die, measured, and determined to be 95.03% of theoretical density.

Specimens 0.250 inch in diameter were electrically discharge-machined from the pressed shape. The specimens were cleaned alternately in hexane and ethanol, using a standard ultrasonic cleaning procedure. A specimen was submitted for chemical analysis. The results follow:

U (%)	Zr (%)	Mo (%)	Total C (%)	Free C (ppm)
30.3±0.3	51.3±0.2	10.8±0.1	7.75	500

2. (UC)_{.18}(ZrC)_{.82} + 10 w/o Nb

Specimens containing 10 w/o Nb were prepared similarly to the Mo bearing samples except that the maximum temperature achieved during hot pressing was 1800°C. The calculated density of the as-pressed shape was 94.21% of theoretical. A specimen was submitted for chemical analysis. The results follow:

U (%)	Zr (%)	Nb (%)	Total C (%)	Free C (ppm)
29.6±0.3	50.9±0.5	11.0±0.1	8.32	500

3. (UC)_{.18}(ZrC)_{.82} + 10 w/o W

The 10 w/o W samples were prepared by four different techniques: 1) hydrostatic pressing, 2) cold die pressing, 3) magnetic compaction, and 4) loose powder sintering.

The hydrostatically pressed samples consisted of a blend of -200 mesh (UC)_{.18}(ZrC)_{.82} plus 10 w/o W (0.7 micron, lot W-121) which was carefully weighed and blended in an inert atmosphere dry box of the type shown in Fig. 2. The blend was split into two equal charges. To one charge, 0.75 w/o cetyl alcohol dissolved in ethyl alcohol was added to aid consolidation of the pressing. The other charge was similarly treated using 1.00 w/o cetyl alcohol dissolved in ethyl alcohol. The cetyl alcohol was added to the powder charges in an evaporating dish and was dried with a heat lamp with constant stirring. Stirring was continued until the blends were cool.

The two charges were then separately screened through a 35 mesh screen and jar-blended for 30 min. The charges were then placed with moderate tamping in an expanded-metal supported, flat bottom plastisol sac. After loading, a 1/4 inch thick cap was placed on top of the charge, after which a plastisol cap was inserted and sealed with a MEK-acetone mix.

The sacs were then evacuated by inserting a hypodermic needle into the closure cap and resealing at needle location upon removal. The sacs were then hydrostatically pressed at 30,000 psig, and the pressure was released slowly. The pressing oil was cleaned from the sacs which were then transferred to the atmosphere dry box for removal of the pressed compacts.

The compacts were transferred to prefired Al₂O₃ boats which were placed in a quartz furnace tube to facilitate binder removal. The quartz tube was transferred from the dry box to a clam shell type furnace, where the tube was evacuated to 2x10⁻⁵ torr before heat was applied. The temperature was regulated by a program controller and was raised to 1160°C over a 24 hr period, at which time the power was cut off and the furnace allowed to cool to room temperature under vacuum. Upon cooling, the tube was back-filled with argon, sealed off, and transferred to the dry box for removal of the specimens and subsequent processing.

Difficulty was encountered on attempts to die press -200 mesh (UC)_{.18}(ZrC)_{.82} with W; therefore, a blend was similarly prepared using -400 mesh (UC)_{.18}(ZrC)_{.82} with 10 w/o W (0.7 micron, lot W-121) added. The blend was again split into two equal charges to which were added 0.75 w/o cetyl alcohol dissolved in ethyl alcohol.

After drying, screening, and reblending, the individual charges were placed in a 0.960 inch I.D. steel die. The die was transferred through the locks to a 500 ton Rodgers dry box contained press (Fig. 4), where the material was pressed at 130,000 psi. The ejected pressed compacts were then transferred back through the locks and placed in Al_2O_3 boats for binder removal in the manner previously discussed. They were then held for subsequent processing.

The magnetically compacted specimens, which contained no binder, consisted of two blends of -325 mesh $(\text{UC})_{.18}(\text{ZrC})_{.82}$ plus 10 w/o W; one contained 0.7 micron W (lot W-123) powder and the second contained 43 micron W (lot W-90) powder. The two charges were weighed, blended, and loaded in the dry box system previously described.

The pressing container consisted of copper tubing, approximately 12 inches long, with a 0.508 inch I.D. and a 0.35 inch wall. Inside the copper tube was a cellulose acetate tube which reduced the inner diameter to 0.408 inch. A wood spacer plug was inserted into one end of the tube and the powder was loaded. A second wood spacer plug was inserted into the open end of the tube. This gave an uncompacted cylinder of $(\text{UC})_{.18}(\text{ZrC})_{.82} + 10$ w/o W that was 0.408 inch in diameter and 3 inches long.

Compaction was accomplished in a standard setup² with 9.45 kilojoules of energy which required approximately 40 microseconds to attain peak power. This energy is equivalent to 30,000 to 40,000 psi.

A section of each compact was submitted for chemical analysis. The results for samples containing coarse W (lot W-90) and fine W (lot W-123) respectively follow:

U (%)	Zr (%)	W (%)	Total C (%)	Free C (ppm)
29.0 ± 0.3	51.1 ± 0.5	12.0 ± 0.2	7.7	500
29.8 ± 0.3	51.5 ± 0.5	10.5 ± 0.2	8.0	500

The loose powder sintered samples utilized the same powder blends that were used for the magnetically compacted samples. Four slip-cast W crucibles of each blend were used as starting samples for the time-temperature thermal treatments.

B. Time-Temperature Thermal Treatment

1. $(\text{UC})_{.18}(\text{ZrC})_{.82} + 10$ w/o Mo or Nb

One specimen each containing Nb or Mo was fired at 1800°C for 24, 48, and 96 hr and at 2000°C for 24 and 48 hr. Portions of these samples were submitted for metallographic and X-ray diffraction examination.

Designated specimens were placed in an indexed W fixture similar to that shown in Fig. 5. The fixture was then positioned in a LASL designed resistance furnace (Fig. 6) which employed a 200 KVA Crown type saturable reactor as a power source. Figure 7 is a photograph of the furnace interior showing the W resistance heating element and the Ta radiation shields. The furnace was evacuated to 1×10^{-5} torr, using a Kinney Model KD-110 mechanical pump and a 10 inch diameter diffusion pump. The vacuum in the furnace was measured with a Phillips dual range vacuum gauge. As power was increased, the fixture and specimens were heated by radiation from the W heating element. Optical temperature readings were taken by sighting on the center of the top fixture support plate through a hole in the top Ta radiation shields. The observed optical readings were corrected for sight glass and for emissivity. The run was interrupted after each 24 hr run for removal of specimens.

2. $(\text{UC})_{.18}(\text{ZrC})_{.82} + 10$ w/o W

The hydrostatic and die-compacted specimens described previously were sintered in the resistance furnace shown in Fig. 6, in accordance with the following sintering schedule.

Sample No.	Consolidation Technique	Cetyl Alcohol Binder (%)	Sintering Treatment
3030-R1	Hydrostatically pressed	0.75	13 hr at 2000°C in vac, plus 2 hr at 2200°C in argon
3030-R2	Hydrostatically pressed	1.00	13 hr at 2000°C in vac
3030-R3	Die pressed	0.75	15 hr at 2000°C in vac
3030-R4	Die pressed	0.75	15 hr at 2000°C in vac

For reasons which will be described in Chapter IV, additional specimens containing no binder were prepared by magnetic compaction and free sintering of loose powders. The sintering treatments imposed on these materials are tabulated as follows:

Magnetically Compacted Specimens		
Sample No.	W Particle Size (micron)	Sintering Treatment
9	43	24 hr at 2000°C in vac
11	43	48 hr at 2000°C in vac
13	43	96 hr at 2000°C in vac
10	0.7	24 hr at 2000°C in vac
12	0.7	48 hr at 2000°C in vac
14	0.7	96 hr at 2000°C in vac
Loose Powder Samples		
1	43	2 hr at 2000°C in vac
2	0.7	2 hr at 2000°C in vac
3	43	24 hr at 2000°C in vac
4	0.7	24 hr at 2000°C in vac
5*	43	2 hr at 2000°C in vac
6*	0.7	2 hr at 2000°C in vac
7*	43	24 hr at 2000°C in vac
8*	0.7	24 hr at 2000°C in vac

*Sintered with a W weight on top of powders.

CHAPTER IV

DISCUSSION OF RESULTS

A. $(UC)_{.18}(ZrC)_{.82} + 10 \text{ w/o Mo}$

The as-pressed microstructure is shown in Fig. 8a. Figures 8d, 8e, and 8f show the structure after 24, 48, and 96 hr respectively at 1800°C in vacuum. While there was no metallographic evidence of a liquid phase in the as-hot-pressed condition, an incipient liquid phase reaction may be noted after 24 hr at 1800°C, showing some rounding and consolidation of the carbide particles. This liquid phase reaction had progressed even further after 48 hr, but little change was observed between 48 and 96 hr at 1800°C.

At 2000°C, an extensive liquid phase reaction had taken place after 24 hr (Fig. 8b), showing almost complete spheroidization of the carbide particles. After 48 hr (Fig. 8c) the carbide matrix, now assumed to be a U-Zr-Mo-C complex, has resintered into a very dense system and appears to be forming a new solid solution with a recrystallized equiaxed structure.

X-ray diffraction results of $(UC)_{.18}(ZrC)_{.82}$ with 10 w/o Mo as pressed and after selective thermal treatments are presented in Table I.

TABLE I
X-RAY DIFFRACTION DATA OF
 $(UC)_{.18}(ZrC)_{.82}$ WITH 10 w/o Mo ADDED

Condition	Lattice Spacing (Angstroms)	Phases Identified
Starting $(UC)(ZrC)$ powder	4.7513	$(UC)_{.18}(ZrC)_{.82}$
$(UC)_{.18}(ZrC)_{.82} + 10 \text{ w/o Mo}$ as hot pressed	4.7345	$(UC)_{.14}(ZrC)_{.86} + Mo$
$(UC)_{.18}(ZrC)_{.82} + 10 \text{ w/o Mo}$ 1800°C for 24 hr in vac	4.733	$(UC)_{.13}(ZrC)_{.87} + Mo$
$(UC)_{.18}(ZrC)_{.82} + 10 \text{ w/o Mo}$ 1800°C for 48 hr in vac	4.733	$(UC)_{.13}(ZrC)_{.87} + Mo$
$(UC)_{.18}(ZrC)_{.82} + 10 \text{ w/o Mo}$ 2000°C for 24 hr in vac	4.715	$(UC)_{.07}(ZrC)_{.93} + Mo$

The fact that Mo is not compatible at 1800°C and above, as observed metallographically, is further corroborated by these X-ray diffraction results. The decrease in the lattice spacing with increased solution of Mo in the (UC)(ZrC) is to be expected.

B. $(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o Nb}$

The microstructure of $(\text{UC})_{.18}(\text{ZrC})_{.82}$ in the as-hot-pressed condition is shown in Fig. 9a. First examination metallographically of the Nb bearing samples showed that the reaction went nearly to completion during the first 24 hr at 1800°C in vacuum. Figures 9d, 9e, and 9f are for samples heated at 1800°C in vacuum for 24, 48, and 96 hr respectively. Figures 9b and 9c show the grain growth encountered during treatment for 24 and 48 hr at 2000°C in vacuum.

X-ray diffraction data corroborates these metallographic results and are presented in Table II.

C. $(\text{UC})_{.18}(\text{ZrC})_{.82}$ with 10 w/o W

Early attempts to consolidate $(\text{UC})_{.18}(\text{ZrC})_{.82}$ with 10 w/o W were unsuccessful. Therefore, commensurate with conventional powder metallurgy techniques, it was decided to add a lubricant to the mix. Based on earlier work, cetyl alcohol was selected. Specimens prepared by hydrostatic pressing, while very low in density, had sufficient green strength to facilitate handling for subsequent

sintering treatments. Preliminary examination under the microscope showed no evidence that W was present following the sintering treatments previously described. However, careful examination at much higher magnifications showed that W particles were in evidence. Figure 10 (sample No. 3030-R1) is a typical microstructure of samples prepared in this manner.

Similar specimens were prepared by die pressing in an effort to obtain a higher green density. Figure 11 (sample No. 3030-R3), sintered for 15 hr at 2000°C in vacuum, contained what was estimated to be approximately 8% undissolved W, whereas Fig. 12 (sample No. 3030-R4), identically treated, showed what was estimated to be less than 3% undissolved W. The only apparent difference was in the particle size of the W powder itself, as is evidenced in the photomicrographs.

This was the first evidence indicating that $(\text{UC})(\text{ZrC})$ may not be stable in the presence of W. In view of this fact, lattice parameters were obtained on the die-pressed and sintered specimens to determine the possibility of W going into solution with the $(\text{UC})(\text{ZrC})$. The results are shown in Table III.

Lowering of the lattice parameter indicates that W is indeed going into solution in the $(\text{UC})(\text{ZrC})$. Since it had been proposed by other investigators^{3,4} that $(\text{UC})(\text{ZrC})$ might be stable at 1800°C and higher for times up to 1 year or longer, it was first suspected that the instability described above might be due to the lubricant used. Therefore, specimens were prepared which contained no lubricant.

TABLE II
X-RAY DIFFRACTION DATA ON
 $(\text{UC})_{.18}(\text{ZrC})_{.82}$ WITH 10 w/o Nb ADDED

Condition	Lattice Spacing (Angstroms)	Phases Identified
Starting $(\text{UC})(\text{ZrC})$ powder	4.7513	$(\text{UC})_{.18}(\text{ZrC})_{.82}$
$(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o Nb}$ as hot pressed	4.736	$(\text{UC})_{.14}(\text{ZrC})_{.86} + \text{Nb}$
$(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o Nb}$ 1800°C for 96 hr in vac	4.693	No Nb or NbC detected
$(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o Nb}$ 2000°C for 48 hr in vac	4.697	No Nb or NbC detected

TABLE III
X-RAY DIFFRACTION DATA OF
(UC)_{.18}(ZrC)_{.82} WITH 10 w/o W ADDED

Condition	Lattice Spacing (Angstroms)	Phases Identified
Starting (UC) _{.18} (ZrC) _{.82} powder	4.7513	(UC) _{.18} (ZrC) _{.89}
(UC) _{.18} (ZrC) _{.82} + 10 w/o W (contained est. 8% free W in structure)	4.7435	(UC) _{.15} (ZrC) _{.85}
(UC) _{.18} (ZrC) _{.82} + 10 w/o W (contained est. 3% free W in structure)	4.7277	(UC) _{.07} (ZrC) _{.93}

Because of the high energy available, specimens were magnetically compacted to provide a more dense compact, thereby providing a more intimate contact between the (UC)(ZrC) and the dispersed W. In the case of the coarse W (43 micron lot W-90) powder addition to the (UC)(ZrC), only slight rounding of the W particles was evidenced. However, in the case of the fine W (0.7 micron, lot W-123) powder addition to the (UC)(ZrC), there was a reacted zone around agglomerated W areas, as is shown in Figs. 13 and 14 (sample No. 10). Also it may be noted that the W agglomerate is surrounded by (UC)(ZrC) particles which have been separated from the (UC)(ZrC) matrix by sintering of this dense particle. Figure 15 (sample No. 14) shows a W particle completely surrounded by (UC)(ZrC). There is an unidentified phase within the W particle.

Concurrent with this work, the loose powder samples were prepared by filling 7/16 inch I.D. x 5/8 inch O.D. x 1 inch high slip cast W crucibles. Two samples each of the blends with the fine W and coarse W powder were vacuum sintered at 2000°C. One each of these samples was sintered for 2 hr, and the second sample of each blend sintered for 24 hr. Examination of these samples showed that the system had been evacuated too rapidly and that some of the powder had blown out of the crucible. Therefore, two more samples of each blend were prepared and treated in the same manner, except that W rod plugs were fitted loosely in the crucible to hold the powder in place.

Metallographic examination of cross sections of the eight specimens so prepared showed essentially no difference in structure. Figure 16 (sample No. 4) shows a reacted area around a W agglomerate after sintering at 2000°C for 24 hr in vacuum without the W plug. The sample is very low density, but shows sintering and considerable shrinkage of the W agglomerate. Figure 17 shows a higher magnification of the same sample. It is obvious that a reaction has taken place, as is evidenced by the irregular intimate contact between the W agglomerate and the (UC)(ZrC) particle adhering to it. There is also evidence of reaction in the (UC)(ZrC) surrounding the particle. This is shown more dramatically in Fig. 18 (sample No. 7).

Figure 18 shows a W agglomerate surrounded by a reacted area in a matrix of angular (UC)(ZrC) particles. It is evident that the reaction between (UC)(ZrC) and W is real, since there was no lubricant present in these samples. Therefore, we decided to submit a representative sample for X-ray microprobe analysis. Figure 19 shows a typical microprobe traverse for this sample with the counter recording W-M α reflections. The quantity of W in solution with the (UC)(ZrC) was calculated to range from approximately 8 to 13 w/o. Figure 20 is the sample-current pattern. A large W particle is visible in the lower left hand corner of Fig. 20a and particles of (UC)(ZrC) are apparent. The W-M α reflections for the same area are shown in Fig. 20b. The dense white area is W. Significant in this photograph is that the adjacent (UC)(ZrC) particles are white, showing W in solution with the (UC)(ZrC).

Further evidence of this is shown in Fig. 20c, the Zr- L_{α} reflections. Here the W appears dark and the light areas show the presence of Zr. The adjacent particles which showed W in Fig. 20b are shown here with Zr present, thereby confirming that the W is indeed in solution in the (UC) (ZrC).

D. Summary

Molybdenum and niobium react with (UC) (ZrC) at temperatures as low as 1800°C, even for short periods of time. However, regarding the compatibility of W with (UC) (ZrC), the results of this program show that a reaction occurs at temperatures as low as 2000°C for sintering times as short as 2 hr. Since nothing is known regarding the compatibility and stability of the quaternary solid solution formed, the usefulness of W in the presence of (UC) (ZrC) is questionable. In fact, the data suggests that this combination of materials may have very limited application in the nuclear field.

REFERENCES

1. Davidson, K.V., Riley, R. E., and Taub, J. M., "Solid Solution Carbides of Uranium and Zirconium," Los Alamos Scientific Laboratory Report LA-3217 (1964).
2. Sandstrom, D. J., "Consolidating Metal Powders Magnetically," *Metal Prog.* **86**, 91-94 (1964).
3. Weinberg, A.F., et al., "Investigation of Carbides as Cathodes for Thermionic Space Reactors," General Atomic Quarterly Progress Report GA-2670 (1961).
4. Yang, L., et al., "Investigations of Carbides as Cathodes for Thermionic Space Reactors," General Atomic Quarterly Progress Report GA-4173 (1963).

Fig. 1 — Dry box train used for powder processing

Fig. 2 — Items used in dry box train for comminution and storage of powders

Fig. 3 — Cross section of vacuum hot press showing induction coil and die assembly

Fig. 4 — A 500 ton Rodgers dry box contained press and dry box train

Fig. 5 — The indexed W fixture used to support specimens during time-temperature thermal treatments

Fig. 6 — Exterior view of resistance furnace showing control console, saturable reactor, and vacuum system

Fig. 7 — Interior view of resistance furnace showing W element and Ta radiation shields

(a) As hot pressed

(d) 1800°C—24 hr

(b) 2000°C—24 hr

(e) 1800°C—48 hr

(c) 2000°C—48 hr

(f) 1800°C—96 hr

Fig. 8 —(UC)_{.18}(ZrC)_{.82} + 10 w/o Mo (approximately 500X)

(a) As hot pressed

(d) 1800°C—24 hr

(b) 2000°C—24 hr

(e) 1800°C—48 hr

(c) 2000°C—48 hr

(f) 1800°C—96 hr

Fig. 9 — $(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o Nb}$ (approximately 500X)

Fig. 10

100X

$(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o W}$ hydrostatically pressed and sintered at 2000° for 13 hr in vac.

Fig. 11

750X

Fig. 12

1000X

$(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o W}$ die pressed and sintered 15 hr at 2000°C in vacuum. Two different samples showing effect of W particle size on rate of reaction.

Fig. 13 **100X**

Fig. 14 **500X**

Magnetically compacted $(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o W}$ sintered at 2000°C for 24 hr in vacuum.

Fig. 15 **1000X**

Magnetically compacted $(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o W}$ sintered at 2000°C for 96 hr in vacuum. Structure shows W particle completely surrounded by $(\text{UC})(\text{ZrC})$ which contains an unidentified phase within the W particle.

Fig. 16 100X

Fig. 17 1000X

$(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o W}$ sintered as a loose powder showing a reaction zone around a W agglomerated particle.

Fig. 18 100X

$(\text{UC})_{.18}(\text{ZrC})_{.82} + 10 \text{ w/o W}$ sintered as a loose powder showing a W agglomerate, a $(\text{UC})(\text{ZrC})$ reacted zone, and an unreacted angular $(\text{UC})(\text{ZrC})$ area.

Fig. 19 — X-ray microprobe linear traverse for W-M α reflections

(a)

(b)

(c)

Fig. 20 — X-ray microprobe analysis showing (a) sample current pattern, (b) W-M α reflections, and (c) Zr-L α reflections ($\sim 400\times$)