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WANL-TME-1611

May 1967

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Westinghouse Astronuclear Laboratory

WESTINGHOUSE ASTRONUCLEAR LABORATORY
CONTRIBUTION TO THE
TWENTY-FOURTH HIGH TEMPERATURE FUEL
COMMITTEE MEETING
May 16, 17 and 18 1967

(Title Unclassified)

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Reviewer	Class.	Date
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This report was edited by L. R. Fleisher from material provided by P. H. Wilks, F. Kirkhart, and W. F. Brizes. The work reported was performed by the personnel of the NERVA Fuel Development Section at the Westinghouse Astronuclear Laboratory

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INTRODUCTION

Electrically heated laboratory corrosion tests indicated that fuel elements produced for the NRX-A5 reactor could operate satisfactorily for 40 minutes. In the NRX-A5 reactor test, element life was about 30 minutes, as previously reported in the Westinghouse Astronuclear Laboratory contribution to the Twenty-Third High Temperature Fuel Committee Meeting (WANL-TME-1539).

Evaluation of these results led to the definition of a development program to:

1) devise a better laboratory corrosion test to evaluate fuel development and quality control of fuel production; and 2) improve fuel element performance. The first goal was met by a higher power-higher flow test which better simulated reactor corrosion. Aspects of the second part of the program are discussed in the following section.

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SECTION I

NRX-A6 FUEL DEVELOPMENT

P. H. Wilks

The dispersion fueled graphite elements contain full length longitudinal coolant channels coated with a vapor deposited layer of NbC. The different thermal expansion coefficients of the graphite and the NbC leads to cracks in the protective coating at temperatures below the NbC deposition temperature. The major attack by hydrogen coolant occurs at intermediate temperatures at the cracks in the NbC coating. Because of its longitudinally central position, this region is called the "midband" and the attack on the region is called "midband corrosion".

The cracks in the coating cannot be eliminated without a significant change in the thermal expansion characteristics of the graphite matrix or the coating. This approach was ruled out except as a long range solution to the corrosion problem. Therefore, the development program sought controllable variations in the nature of the cracks by which the crack distribution could be optimized with respect to lower corrosion rates. It was found that the crack width varied with coating temperature and thickness; fine cracks were characteristic of thin coatings, wide cracks of thicker coatings and low coating temperature led to reduced crack width. A comparison of fine and coarse cracks is given in Figure 1 - 1. The fine crack widths are of the order of 0.1 mil, the wide crack widths about 1 mil. Further, it was found that elements with fine coating cracks tended to corrode at lower rates than elements with wide cracks although the area of unprotected graphite was essentially the same. Thus, better behavior was achieved simply by coating to produce narrow cracks.

Similar goals were sought in the optimization of the LASL concept of Mo coatings deposited over the NbC coatings. It was found that Mo deposited over NbC coatings with

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Figure 1-1. Comparison of Narrow and Wide Cracks in NbC

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narrow cracks heightened the effectiveness of the overcoating technique. To illustrate, the curves in Figure 1 - 2 show the weight loss/inch of typical fuel elements versus position along the length of the element. Both elements were Mo overcoated; one was characterized by narrow cracks in the NbC coating, the other by wide cracks. It is obvious from the data that the combination of fine cracks and Mo overcoating gives superior results.

In summary, the deleterious effects of coating defects built in to the fuel materials system was minimized by altering the geometry of the defect and supporting the change with secondary protection. The production of fuel for the next reactor in the test series, NRX-A6, includes these principles to improve fuel performance: NbC protective coatings deposited to produce narrow cracks and overcoated with Mo.

Parallel development efforts were undertaken to improve the graphite matrix of the fuel element and the fuel particles. The matrix studies are, for the most part, in the area of additives, varying process parameters, and impregnation, the object being to reduce the corrosion susceptibility and permeability of the body. Experimental elements incorporating these techniques are programmed for inclusion in the NRX-A6 test.

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Figure 1-2. Corrosion Rate of Elements with Thick, High Temperature Coatings vs Thin, Low Temperature Coatings, Both with Mo Overcoats

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SECTION 2

MIXED CARBIDE FUEL STUDY

F. Kirkhart

The present fuel system for NERVA elements (UC_2 coated with pyrolytic carbon) exhibits severe migration behavior at temperatures in excess of about $2400^\circ C$. It is desirable to extend the useful temperature range of the fuel for future reactors.

Theoretically, one of the most promising fuel systems is the pseudobinary solid-solution carbide system UC-ZrC. At concentrations of UC < 10 mole percent, the thermal stability has been established at between $2700^\circ C$ and $2900^\circ C$. Figure 2-1 shows a pseudo-vertical section through the U-Zr-C ternary system between terminal compositions (ZrC + C) and (UC_2 + C). According to the phase diagram, compositions of about 10 percent UC in ZrC should be thermally stable to about $2700^\circ C$. This represents an increase of about $300^\circ C$ in the useful maximum temperature of the fuel in elements.

In late 1965, Westinghouse purchased 5 kg quantities of mixed carbide beads from each of two vendors. Purchase specifications are shown in Table 2 - 1.

Table 2 - 1

PURCHASE SPECIFICATIONS FOR THE INITIAL QUANTITIES OF MIXED CARBIDE FUEL BEADS

Composition	10 mole percent UC -90 mole percent ZrC
Uranium Form	Depleted or normal
Particle Size	44-200 microns
Particle Shape	Spherical
Carbon Content	9.0 - 10.8 weight percent
Density	95 percent theoretical
Chemical Impurities	Oxygen - 2000 ppm; Nitrogen - 400 ppm; All other impurities as specified in WANL-PDS-30050-3

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Figure 2-1. Pseudo-Vertical Section through the (U-Zr-C) System between Terminal Compositions (ZrC + C) and (UC₂ + C)

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Table 2 - 2 shows the physical and chemical properties of each material. Both materials are near the purchase specification values. Material from Vendor No. 2 is slightly more pure and more dense than the Vendor No. 1 material.

Metallographs of each material show a two-phase structure. Vendor 1 material appears to be more inhomogeneous, and contains more voids than Vendor 2 material.

The lattice spacing of the solid solution in each material was checked by X-ray diffraction powder techniques. Each material showed a lattice spacing (a_0) or $4.71\text{\AA}$ which corresponds to 6 mole percent UC. The powder pattern also shows a UC_2 line at a "d" spacing of $3.04\text{\AA}$. Obviously, much of the uranium is in the form of UC_2 and appears in the metallographs to be located principally along grain boundaries in the beads.

The beads were included at a 20 volume percent level in a normal NERVA fuel body mix and extruded into 3/8 inch pieces. The stock was then given the normal 250°C cure and 850°C baking cycles. Figure 2 - 2 is a photomicrograph of the beads and matrix after an 850°C bake. As can be seen, the beads have fractured into multiple pieces and the graphite matrix has been fractured into more-or-less radial fractures radiating from beads. The cured matrix which contained Vendor 1 material, shows similar behavior. The cured matrix, which contained Vendor 2 material, did not fracture in the cured state.

The fractures in the beads are thought to be caused by the oxidation of UC_2 , which is presumably situated along grain boundaries. The resultant volume increase fractures the beads. Of course, since this occurs after the graphite matrix is physically stable, the matrix is fractured also.

Providing the previous analysis is correct, it seems obvious that the beads must be of single phase structure to be useful in elements.

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Table 2 - 2

WESTINGHOUSE ANALYSES OF 10 UC -90 ZrC FUEL PARTICLES

	<u>Vendor 1</u>	<u>Vendor 2</u>
Particle Shape	Spherical	Spherical
Carbon	9.8 w/o	10.02 w/o
Density	6.74 g/cc	7.63 g/cc
Oxygen	1280 ppm	1980 ppm
Nitrogen	40 ppm	40 ppm
Zirconium	71.72 w/o	67.26 w/o
Uranium	18.72 w/o	22.85 w/o
Aluminum	11 ppm	15 ppm
Boron	1.8 ppm	0.6 ppm
Barium	5 ppm	5 ppm
Beryllium	0.2 ppm	0.2 ppm
Bismuth	1 ppm	1 ppm
Calcium	10 ppm	59 ppm
Cadmium	10 ppm	10 ppm
Cobalt	3.6 ppm	1.9 ppm
Chromium	120 ppm	12 ppm
Cesium	20 ppm	20 ppm
Copper	18 ppm	1.2 ppm
Iron	1190 ppm	37 ppm
Potassium	1 ppm	1 ppm
Magnesium	3 ppm	18 ppm
Manganese	3 ppm	9.4 ppm
Molybdenum	780 ppm	120 ppm
Nickel	420 ppm	12 ppm
Phosphorous	20 ppm	20 ppm
Lead	1 ppm	1 ppm
Rubidium	5 ppm	5 ppm
Antimony	2 ppm	2 ppm
Silicon	40 ppm	20 ppm
Tin	4.8 ppm	2 ppm
Strontium	20 ppm	20 ppm
Titanium	2.4 ppm	120 ppm
Thallium	20 ppm	20 ppm
Vanadium	5 ppm	5 ppm
Tungsten	1000 ppm	1000 ppm
Zinc	50 ppm	50 ppm
Hafnium	46 ppm	39 ppm

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VENDOR 2 FUEL
850°C HEAT TREAT
150X MAG.

VENDOR 1 FUEL
850°C HEAT TREAT
150X MAG.

Figure 2-2. Photomicrographs of Heat-Treated Graphite Matrix Containing Inhomogeneous (U-Zr)C Fuel

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A heat-treatment program was set up to determine a suitable time at temperature to form a single phase structure in Vendor 2 beads. Gram-quantities of beads were intimately mixed with graphite powder, heated in a graphite crucible to a temperature, and held at the temperature for a period of time. The temperature-time schedule is shown in Table 2 - 3. The table also indicates whether or not UC_2 was present in the heat-treated beads.

Table 2 - 3
HEAT TREATMENT CONDITIONS FOR MIXED CARBIDE BEADS

<u>Temperature - °C</u>	<u>Time at Temperature - hours</u>	<u>Presence of UC_2</u>
2400	1	No
2400	3	No
2400	8	No
2500	1	No
2500	3	Yes
2600	1	Yes
2600	2	Yes

Figures 2 - 3, 2 - 4, and 2 - 5 are photomicrographs of the heat-treated beads. The material seems to have been converted to the single-phase structure in every heat treatment. X-ray analysis indicates that the solid solution has a lattice parameter of about $4.73 \text{ \AA}$, which corresponds to a structure containing about 13 percent UC. Apparently, zirconium has been lost in the heat treatment or the chemical analysis, which indicates ~11 mole percent UC is in error. No explanation for a loss of zirconium has been proposed. At any rate, the high uranium content can explain the presence of UC_2 in the high-temperature heat treatments. Apparently, the temperature has fluctuated high enough to form liquid in the high-uranium material. This material would partially precipitate out as UC_2 . The UC_2 could be located around the surface of the larger-grained beads or could have migrated into small pockets by itself. In either case, little evidence of it would be found in the photomicrographs.

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2400°C, 8 HR. HEAT TREAT
500X MAG.

2400°C, 1 HR. HEAT TREAT
500X MAG.

Figure 2-3. Photomicrographs of Heat-Treated Vendor 2 (U - Zr)C Fuel

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2500°C, 3 HR.
500X MAG.

2500°C, 1 HR.
500X MAG.

Figure 2-4. Photomicrographs of Heat-Treated Vendor 2 (U - Zr)C Fuel

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2600°C, 2 HR.
500X MAG.

2600°C, 1 HR.
500X MAG.

Figure 2-5. Photomicrographs of Heat-Treated Vendor 2 (U - Zr)C Fuel

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Small quantities of the material from each heat-treatment were introduced into a normal NERVA matrix mix, the mix molded into 1/2-inch diameter by 1/2-inch long slugs, and the slugs subjected to curing and baking temperature conditions. Although the resultant carbon matrix was relatively porous, enough structure was present to determine matrix cracks had any been present. In fact, the fuel did not appear to be different from that shown in previous photomicrographs (Figures 2 - 3, 2 - 4, and 2 - 5), and no matrix disruptions were evident.

At this point, it seemed desirable to heat-treat enough material to include in an extruded carbon matrix for study at graphitization temperatures. About 500 grams of Vendor 2 material was heated to 2400°C for four hours. The resultant material was only slightly altered in particle shape. Metallography showed the expected single phase structure and X-ray analysis indicated that no UC₂ was present. However, the lattice spacing (a_0) of about 4.73 Å indicated 12-15 mole percent UC. This concentration of UC can be a cause of a phenomenon which will be discussed later.

A NERVA matrix mix was prepared which contained about 20 volume percent of the heat-treated beads. This mix was extruded into 3/8-inch diameter rods. Subsequent curing, baking, and normal graphitization to 2200°C was carried out with some rods in vacuum and some in helium atmospheres. No disruption in the matrix or the beads could be detected with either atmosphere or at 250, 850, or 2200°C temperatures.

Small samples of the vacuum heat-treated stock were further heat treated to final temperatures of 2400, 2450, 2500, 2550, 2600, 2650, and 2700°C. In each case, the final temperatures were maintained for 1/2 hour except for the 2700°C. Some samples were held at 2700°C for 10 minutes and others held 1/2 hour. Photomicrographs of the matrix structure and of bead structure are shown in Figures 2 - 6 through 2 - 14.

Figures 2 - 6, 2 - 7, 2 - 8, and 2 - 9 are all essentially identical. The fuel is of single phase structure and the matrix is intact.

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FUEL
500X MAG.

MATRIX
150X MAG.

Figure 2-7. Photomicrographs of Graphite Matrix Containing Homogenized (U - Zr)C Fuel - Heat Treated 2400°C, 1/2 Hr.

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FUEL
500X MAG.

MATRIX
150X MAG.

Figure 2-9. Photomicrographs of Graphite Matrix Containing Homogenized (U - Zr)C Fuel - Heat Treated 2500°C, 1/2 Hr.

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Figure 2 - 10 shows distinct densification of fuel particles and very slight diffusion through the matrix. Presumably, the matrix would be weakened only very slightly since the strength of the matrix depends upon the bond to the bead only to a very slight degree.

Figures 2 - 11 and 2 - 12 show densification in the fuel and the beginning of some diffusion of the solid solution throughout the graphite matrix. Apparently, 2600 to 2650°C is the temperature limit of this particular composition of fuel.

Figure 2 - 13 indicates severe melting of the bead with almost complete diffusion throughout the matrix.

Figure 2 - 14 is a micrograph of a piece of the matrix which has been heat-treated at 2700°C for one half hour. It simply shows complete dispersion of the liquid carbide throughout the matrix. Obviously, severe melting has occurred.

The mixed carbide beads, as received, apparently contained some quantity of UC_2 dispersed around grain boundaries of the solid solution. The UC_2 can readily oxidize at relatively low temperatures to form a larger bulk volume than the original volume. The bead then becomes larger in volume and splits the carbon matrix.

Apparently, the mixed carbide fuel from Vendor 2 can be homogenized by heating it to a temperature of 2400°C with a short soaking period. The homogenized material, when included in a matrix, performed in a satisfactory manner (physically) when heated, until a temperature of about 2600°C was reached.

One would expect the homogenized material of the composition, shown analytically, (11 mole percent UC) to be stable to about 2700°C. However, an increase in UC composition to about 13 percent would result in a drop in stable temperature to about 2600°C. X-ray analysis indicates that, in fact, the material is of about this composition. One possible explanation for the discrepancy in the X-ray and analytical data, showing the quantity of UC in the material, is that the material may have been inhomogeneous in bead-to-bead chemistry. No particular effort was made to completely blend the material before samples were taken. Therefore, the chemistry sample may not have been representative of the lot of material.

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FUEL
500X MAG.

MATRIX
150X MAG.

Figure 2-10. Photomicrographs of Graphite Matrix Containing Homogenized (U - Zr)C Fuel - Heat Treated 2550°C, 1/2 Hr.

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FUEL
500X MAG.

MATRIX
150X Mag.

Figure 2-11. Photomicrographs of Graphite Matrix Containing Homogenized (U - Zr)C Fuel - Heat Treated 2600°C, 1/2 Hr.

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FUEL
500X MAG.

MATRIX
150X MAG.

Figure 2-12. Photomicrographs of Graphite Matrix Containing Homogenized (U - Zr)C Fuel - Heat Treated 2650°C, 1/2 Hr.

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FUEL
500X Mag.

MATRIX
150X Mag.

Figure 2-13. Photomicrographs of Graphite Matrix Containing Homogenized (U - Zr)C Fuel - Heat Treated 2700°C, 10 Min.

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FUEL
500X Mag.

MATRIX
150X MAG.

Figure 2-14. Photomicrographs of Graphite Matrix Containing Homogenized (U - Zr)C Fuel -Heat
Treated 2700°C, 1/2 Hr.

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A homogeneous material of the system (U - Zr) has been demonstrated to be physically stable in a NERVA type carbon matrix to a temperature of at least 2600°C . Results indicate that the material must be single phase and free of UC_2 , at least in the grain boundaries. The maximum working temperature is strongly dependent upon close control of the amount of uranium in the solid solution U-Zr-C system.

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SECTION 3

COMPRESSIVE CREEP BEHAVIOR OF THE GROUPS IV B AND V B MONOCARBIDES

W. F. Brizes

The creep behavior of the transition metal carbides is presently being studied:

1) to establish a base for the development of high carbide content composites; and 2) as a foundation for alloy carbide development. At present, of the six monocarbides of groups IV B and V B transition metals, TiC, ZrC, HfC, and NbC have been studied. Specimens of theoretically dense carbides, of the order of 3/4 inch in length by 3/8 inch in diameter have been prepared by carburizing the liquid metals into carbide rods. This method produces carbides with carbon to metal ratios near the maximum attainable.

Samples were tested in compressive creep over the 1600° to 2700°C temperature range and 1000 to 4000 psi stress range. The total strains for a series of one hour creep tests, and the minimum creep rates observed in these tests are plotted against test temperature in Figure 3 - 1. Note that HfC is the most creep resistant of the carbides studied. The high creep resistance is thought to be directly related to the high yield stress of HfC compared to the other monocarbides, in contrast to TiC which has the lowest yield point and highest creep rate. The yield stresses of the carbides studied are plotted against temperature in Figure 3 - 2. It was also noted that the creep resistance varied with the melting temperature of the monocarbides, HfC having the highest and TiC the lowest.

The stress dependence of the minimum creep rate was also studied. The expression $\dot{E} = A \sigma^k$ was found to represent all the monocarbides studied, where A is an experimental constant, $\dot{E}$ is the minimum compressive creep rate in a one hour test, σ is the stress, and k is an empirical constant approximately equal to 2.5 as shown in Table 3 - 1. Values of k in the indicated range suggest a Peierl's stress controlled deformation mechanism.

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Figure 3-1. Total Strain and Minimum Compressive Creep Rate after One-Hour Test at 4000 psi Stress vs. Temperature for Group IVB and Group VB Monocarbides

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Figure 3-2. Yield Stress for 0.2% Offset vs. Temperature for Refractory Metal Monocarbides

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Table 3 - 1

KNOOP HARDNESS AND k VALUES OF THE TRANSITION METAL CARBIDES

<u>Carbide</u>	<u>k</u>	<u>Knoop Hardness (room temperature, 100g load)</u>
TiC	2.39	3430
VC		2483
ZrC	2.47	2378
NbC	2.56	2144
HfC	2.46	2075
TaC		1297

Knoop hardness values for the carbides were determined at room temperature. These hardnesses, also listed in Table 3 - 1 were found to be proportional to the yield points extrapolated to room temperature. A special set of specimens were prepared with varying carbon to metal ratios ($\frac{C}{Me}$) less than the maximum previously used. The room temperature hardness of these samples varied with $\frac{C}{Me}$, decreasing with decreasing $\frac{C}{Me}$ in the group IV B monocarbides and increasing with decreasing $\frac{C}{Me}$ in the group V B monocarbides. High temperature creep deformation as a function of $\frac{C}{Me}$ has not been studied.

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