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WESTINGHOUSE ASTRONUCLEAR LABORATORY **MASTER**
CONTRIBUTION TO THE
TWENTY-SECOND HIGH TEMPERATURE FUEL
COMMITTEE MEETING
24,25,26 May 1966

(Title Unclassified)

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Classification cancelled or changed to
by authority of DOC
by H7.C TFC, date SEP 10 1973

Edited by:

J.M.Tobin

SPECIAL REVIEW FINAL DETERMINATION	Reviewer	Class.	Date
	TR	U	5-4-82
Class: <u>U</u>

INFORMATION CATEGORY	
<u>C.R.D.</u>	
<u>B. Reich</u>	<u>5-19-66</u>
AUTHORIZED CLASSIFIER	DATE

(The preliminary information presented in this report should not be published without prior written approval of the Westinghouse Astronuclear Laboratory. The work performed was supported by the Space Nuclear Propulsion Office).

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INTRODUCTION

The third reactor in the present NERVA series to be tested at full power conditions was NRX-A4. This test was especially significant because it was the first to operate with engine system components. That is, NRX-A4 with the associated engine system components supplied its own auxiliary power and pumped its own coolant; it was functionally independent of the test stand except for mechanical support and instrumentation.

The reactor was tested in a series of four experimental plans over a four and one-half month period. It operated for time-power level combinations equivalent to 53.9 minutes at full power. Preliminary evaluation of the data indicates that actual operation time at design temperatures and power levels above 10000 MW was approximately 30.1 minutes, about twice that of NRX-A3.

NERVA fuel elements are dispersions of pyrocarbon coated UC_2 fuel particles in extruded graphite shapes. They are 52 inches long, are of hexagonal cross section, and have 19 coolant channels running the length of the element. To reduce the corrosion effect of the hydrogen coolant on the graphite fuel matrix, the coolant channel surfaces (bores) and the external surfaces of the element at the downstream end are coated with NbC.

The NRX-A4 core contained 1584 NERVA fuel elements of which 693 were produced at the Union Carbide Y-12 facility. The Y-12 elements were manufactured with 1-inch unfueled tips at the nozzle end and were back-coated 2 1/2 inches with NbC. These elements were located in the central region of the core. The remaining 891 fuel elements were produced at the Westinghouse Astrofuel Facility (WAFF). All of the WAFF elements were manufactured with 1/4-inch unfueled tips and had standard 3/4-inch NbC back-coatings on their external surfaces at the downstream end with the following exceptions;

- a) 130 elements with 2 1/2 inch NbC back-coats located adjacent to the Y-12 fuel elements.
- b) 12 elements with tapered tips and 2 1/2 inch NbC back-coats located in Cluster 4G4 and 4G5.

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- c) Eleven elements, located in Sector 6, surface coated with 1 to 1.5 mils thick pyrocarbon.
- d) Peripheral fuel elements surface coated with approximately 0.5 mil thick NbC from station 17 to the hot end.

Following the tests, the reactor was moved to the R-MAD building for disassembly and post-operative examination. Examination is still in progress; preliminary observations on the post-test condition of the fuel elements and fuel element weight loss can be reported now. A more complete evaluation of NRX-A4 fuel element behavior will be made after detailed studies of the visual data and hot cell examinations at R-MAD and LASL are finished.

PRELIMINARY POST OPERATIONAL RESULTS

The post test appearance of WAFF and Y-12 fuel elements was different. The WAFF elements displayed, in general: high weight losses; high pinhole density; a high incidence of large, localized, internal voids (corrosion pockets); moderate to severe surface corrosion; and moderate corrosion at the coated end. The locations of WAFF and Y-12 elements across the core and their corrosion behavior are shown in figure 1. The Y-12 elements, on the other hand, showed variable weight losses and pinhole formation which were highly dependent on core radius, moderate surface corrosion effects, and more severe corrosion damage at the coated end. In both types of elements, patterns of element fracture were observed which appeared to coincide with high element weight loss and high pinhole densities.

Broken Elements in NRX-A4

Five hundred twenty-eight elements were observed to be broken during core disassembly. Of these 387 were broken in several places while the remainder displayed only single breaks. Of the WAFF elements, 45.7 percent were broken as compared to only 9.5 percent of the Y-12 elements.

A major cause of element fracture seemed to be localized pinholes and corrosion pocket formation which caused a general weakening of the elements. Additional contributions

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Figure 1. Cross Section of Core Showing Positions of WAFF and Y-12 Elements

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to element weakening were made by surface corrosion effects particularly when these effects were superimposed in axial areas of general bore corrosion and corrosion pocket formation as shown in figure 2. While the evaluation of surface corrosion patterns in the core awaits more detailed visual and depth studies, there is evidence, illustrated in figure 3, of moderate-to-severe tile pattern corrosion having occurred on peripheral elements and of fairly heavy surface corrosion caused by gas exiting from adjacent face pinholes. The longitudinal streak corrosion patterns which were observed in NRX-A3¹ fuel elements were also observed on the A4 elements although preliminary observations indicate they varied considerably in axial location.

Pinhole Formation in A4 Fuel Elements

A preliminary evaluation of pinhole data was made on 836 NRX-A4 fuel elements. The variation of pinhole frequency with core radius obtained from this study is shown in figure 4. No attempt has been made in this plot to differentiate between pinholing in WAFF and Y-12 elements. These data indicate a very low pinhole frequency in Y-12's and a very high frequency in the WAFF elements at comparable radii. The peak WAFF frequency appears to be at about 35 - 40 centimeters with a reduction in pinhole frequency on the core periphery. Peak frequency in the Y-12 elements occurs in the center region of the core.

Pinhole data from 149 of the A4 fuel elements from Sectors 1, 2, and 6 were used to obtain preliminary axial pinhole distributions. Figures 5 to 7 show the distribution obtained for peripheral fuel elements and for WAFF and Y-12 elements near their junction in the core. For all three groupings, maximum pinhole densities occurred in the axial range 20 - 30 inches with a secondary concentration occurring at 35 - 45 inches. There are considerable differences however in the pinhole frequencies for Y-12 and WAFF elements; the Y-12's averaged 3 pinholes per element while the WAFF's averaged 19 pinholes per element.

WEIGHT LOSS DISTRIBUTION IN NRX-A4 ELEMENTS

The preliminary radial weight loss distribution curve for unbroken NRX-A4 elements is shown in figure 8. The average weight loss data varies considerably with core radius, with

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Figure 2. End View of an Element Break Showing Extensive Surface Corrosion on One Flat and Internal Corrosion Around the Coolant Channels

Figure 3. Tile Corrosion Pattern on a Peripheral Element

Figure 4. Average Pinhole Frequency as a Function of Core Radius (Preliminary Examination of 836 NRX-A4 Fuel Elements)

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Figure 5. Axial Distribution of Pinholes in WAFF Peripheral Fuel Elements from NRX-A4 (Preliminary Data Based on 15 Elements)

Figure 6. Axial Distribution of Pinholes in WAFF Code 99 Fuel Elements from NRX-A4 (Preliminary Data Based on 16 Elements)

Figure 7. Axial Distribution of Pinholes in Y-12 Code 99 Fuel Elements from NRX-A4 (Preliminary Data Based on 121 Elements)

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the Y-12 fuel elements showing the greatest range, i.e., 10 grams for elements in mid-core locations and 40 grams for elements in the center of the core. The overall average weight loss for Y-12 elements was 13.7g. The WAFF fuel element weight losses showed less variation ranging from approximately 23g to 33g with an average of 29.7 g/element. At comparable radii (27 to 33 cm) the Y-12 fuel elements showed lower average weight losses than the WAFF elements. As noted, this data was taken from unbroken elements and may not represent the absolute weight loss distribution particularly in the region of the core periphery where a relatively high fraction of elements were broken. Further evaluation of weight loss data for broken elements is being undertaken.

Electrical hydrogen corrosion tests have been used to qualify elements and to provide estimates of in-core weight losses. In figure 9, the results of electrical tests are compared with actual weight losses observed in NRX-A3 and NRX-A4. The results indicate that present electrical simulation tests cannot be used to predict the reactor performance of fuel elements.

COATED END BEHAVIOR

Visual examination of a sampling of 57 NRX-A4 fuel elements of various end coating types and from various core locations was used to obtain representative coated end behavior data. Based on these data, the behavior of coated ends of NRX-A4 fuel elements varied from poor to excellent as summarized in table 1. The back-coated ends of the WAFF elements performed considerably better than those of the Y-12 elements at comparable core positions. In addition, corrosion damage, including channel exposure and general end tapering was observed to increase in severity on Y-12 elements located in the center of the core.

The coated ends of WAFF elements with back-coated, tapered tips were in excellent condition and showed practically no external effects due to reactor testing. The typical condition of the tapered tip elements is shown in figure 10. The coated ends of A4 peripheral fuel elements were, with some exceptions, in good condition. In general, the corrosion characteristics observed at the coated end of the standard WAFF back-coated elements were similar to those observed in NRX-A3 elements, i.e., a high bearing area remaining (BAR), with

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Figure 8. Preliminary Weight Loss of NRX-A4 Unbroken Fuel Elements as a Function of Radial Position

Figure 9. Comparison of Reactor Test Weight Losses of NRX-A3 and NRX-A4 Elements with Electrical Test Weight Losses

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TABLE 1

BEHAVIOR OF COATED ENDS IN NRX-A4 FUEL ELEMENTS

Type	Examined	BAR		% of End Flats With Channel Exposure Average	% of Elements With Corrosion Local to Joint
		Average	Range		
Back Coated WAFF	10	99	98-100	10	10
Back Coated Y-12*	10	82	50-95	93	30
Back Coated Y-12**	5	35	20-50	97	***
Tapered, Back- Coated, WAFF	12	98	95-100	2.8	0
Standard WAFF Back Coats	20	93	45-100	30	15

* Y-12 elements adjacent to WAFF elements in core

** Y-12 elements in center region of the core

*** General corrosion originating at position other than joint.

Figure 10. Fuel Element Cluster During Disassembly Showing Excellent Condition of Back-Coated, Tapered Tips

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channel exposure mainly limited to the shoulder area of the undercut region.

Behavior of Pyrocarbon Coated Fuel Elements

Eleven NRX-A4 fuel elements in Sector 6 were coated externally with pyrocarbon varying from 1 to 1.5 mils thick. Seven of these elements were observed to have been broken during core disassembly and all were pinholed. The average weight loss determined on both broken and unbroken elements was 30.0 grams and the average pinhole density was 30 pinholes per element. The preliminary data do not show any performance benefit resulting from external pyrocarbon coatings.

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NERVA FUEL DEVELOPMENT

A comparative study of diffusion rates of carbon through six transition element monocarbides was completed and the results are summarized below. Uncoated alloy carbide fuel beads of a 10 UC-90 ZrC composition were found to be difficult to incorporate in a graphite matrix and the problems are discussed. Thermal analysis of liquids and solid boundaries in the U-Zr-C ternary system in the presence of excess graphite are also reported. Initial hydrogen corrosion test data for beta SiC coated graphite indicate a relatively high corrosion rate of the SiC coating itself at 1500°C.

Comparative Diffusion Study

A comparative study of diffusion of carbon in the monocarbides of the six elements in periodic groups IVB and VB has been completed in this laboratory^(1, 4, 5, 6 and 7). The particular value of a comparative study in identical crystal structures is that it provides, in addition to reaction kinetics and diffusivity data, a basis for generalization of the interrelationships among the data which can lead to both an understanding of the diffusion mechanism and means of prediction for other related interstitial compounds.

The study program began with the layer-growth measurement of carbon diffusion in the niobium-carbon system. Brizes and Cadoff¹ determined the diffusivity of carbon over the temperature range from 1700° to 2200°C. Their data for the concentration independent diffusivity of carbon in NbC is given in a summary table of new data, table 2. The authors observed that the carburization of niobium in their experimental method led to a lower C/Nb ratio, NbC_{0.93}, on the surface than the phase diagram indicates, NbC_{0.98}. The conditions used in the study resulted in a reproducible surface composition at the lower stoichiometry. Marker movement experiments, using drilled micro-holes, showed that niobium diffusion toward the carburizing surface is negligible since the markers did not move significantly with respect to the niobium lattice. This was interpreted as evidence that the carbon moves interstitially through the niobium sublattice and the niobium atoms remain relatively fixed.

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TABLE 2

SUMMARY OF NEW CARBON DIFFUSION DATA

Carbide	T (°C)	D (cm ² /sec)	Q (kcal/mole)	Source	Ref.
TiC	1800-2700	0.1	58.7	Adelsberg, Cadoff, and Tobin	(5)
ZrC	2000-2860	0.95	78.7	Adelsberg, Cadoff, and Tobin	(4)
HfC	2300-3000	6.93	99.5	Adelsberg, Cadoff, and Tobin	(5)
VC	1800-2600	0.1	55.2	Adelsberg, Cadoff, and Tobin	(7)
NbC	1700-2200	7.6	88.2	Brizes, Cadoff, and Tobin	(1)
TaC	2100-2650	1.8	87.0	Brizes and Tobin	(6)

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The analysis of the multi-phased layer-growth kinetic data was refined by Brizes and Cadoff¹ by means of an application of the mathematical approach used previously by Kidson². Kidson successfully applied a Wagner³ diffusion expression to diffusion in multi-phase binary systems at phase boundary positions, and has shown that the diffusion coefficients can be related to the interphase (boundary) concentrations and movements.

The necessary experimental data for this analysis are the parabolic growth constants and the layer boundary concentrations. Kirkaldy⁸ has shown that for multiphase diffusion, the boundary concentrations at the intermediate phase (layer) boundaries are at the equilibrium values. The equilibrium diagrams may be used to obtain boundary concentrations where the data is available and accurate, except the concentration at the external surface of the monocarbide layer as noted previously.

The diffusivity of carbon in NbC reported by Brizes and Cadoff¹ was assumed independent of concentration. If we consider the diffusion to proceed by a random walk process, i.e., $D = a^2 N_v W$, where a is the lattice spacing, N_v = concentration of carbon vacancies, and W = jump frequency, it may be expected that the diffusivity would increase with decreasing carbon composition (an increase in vacant sites, N_v). For example, within the compositional boundary limits used in their report ($C/Nb = 0.93-0.70$), diffusivity may vary by a factor of 4. Other inter-related compositional dependent factors such as bonding strength and lattice parameter variation, which reflect lattice resistance to atomic movement (jump frequency) may operate in a compensating manner with the vacancy concentration term to reduce the overall dependence of diffusivity on composition. However, experimental evidence for a higher value of D in the low stoichiometry range of NbC was indicated by the observation of a large band of Widmanstätten structure. This indicates a very shallow concentration gradient in the low stoichiometry range and a high gradient near the carbon interface. A change in concentration gradient of this magnitude is not predicted where D is assumed constant. Another possible explanation for the large band of Widmanstätten structure is a possible error in the $NbC_x - (NbC_x + Nb_2C)$ phase boundary. It is clear that further investigations will be required to resolve this point.

Similar refinements of the layer-growth technique and analysis to those described by Brizes and Cadoff² were applied by Adelsberg and Cadoff to the determination of carbon diffusivities in the three group IVB monocarbides, TiC^5 , and ZrC^4 , and HfC^5 . Data for these carbides are listed in table 2. The authors found two advantages to the use of molten transition metal in a graphite crucible for layer-growth measurements; a reduction or elimination of cracking of the scale, and an increase in the useful temperature range of study. Carbon diffusion was studied at temperatures up to the monocarbide-carbon eutectic temperatures.

Brizes⁶ measured the carbon diffusion in the tantalum-carbon system and his data are listed in table 2. He determined the carbon concentration profile in a carburized tantalum layer-growth specimen by means of electron micro-probe analysis for carbon. The carbon concentration gradient was found to be non-linear in the TaC layer as reported for NbC^1 .

Adelsberg and Cadoff⁷ reported the rapid diffusion of carbon through the remaining Group VB monocarbide, VC , thus completing the experimental phase of the comparative diffusion study. Their results are also listed in table 2.

In reviewing and summarizing the experimental results of the comparative diffusion study, it should be noticed that the principal errors in this study, as in all layer-growth studies, are the result of:

- 1) the assumption of a concentration independent diffusivity,
- 2) the use of incomplete equilibrium phase diagrams to obtain the carbon concentration gradient,
- 3) the lack of accurate pyrometry at the extremely high temperature ($+ 30^\circ$ to 50°C errors),
- 4) the difficulties in maintaining a constant and reproducible carbon stoichiometry at the carburized surface, particularly at lower temperatures (below 2100°), and
- 5) the competition of grain boundary diffusion with volume diffusion, particularly at lower temperatures.

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U-Zr-C Phase Diagram

The carbon rich section of the (U-Zr-C) ternary system is being studied by equilibrium thermal analysis with the aid of metallography and X-ray diffraction. The results of the study to date are shown in pseudo vertical section through the ternary system, figure 11. The three phase region ($UC_2 + (U-Zr)C_{ss} + C$) forms from the liquid + carbon phases at $2410^{\circ}C$ at a composition (80 UC - 20 ZrC) saturated with carbon.

Cubic UC_2 can take zirconium in solution thereby lowering the peritectic reaction temperature from $2475^{\circ}C$ on the binary to $2410^{\circ}C$ at the ternary reaction isotherm. In the (Zr-C) binary system it was confirmed that ZrC solidifies eutectically when saturated with carbon. Composition of the Zr rich end of the ternary reaction isotherm was found to be 19 a/o UC-81 a/o ZrC by X-ray analysis and is in good agreement with that found by thermal analysis as shown in figure 11. Two temperatures were determined for the solidus, carbon + (Zr-U) $C_{ss} + C$ drops sharply to $2410^{\circ}C$ when the solution contains more than 15 percent UC as can be seen in figure 11.

SiC Coatings on Graphite

The feasibility of using SiC as protective coating on graphite in the $1000^{\circ}-1900^{\circ}C$ temperature range was examined. The hydrogen corrosion rates of unfueled NERVA graphite with and without SiC carbides have been compared. The SiC coatings were deposited by pyrolysis of a methyl silane class vapor by Dow-Corning Corporation. The SiC is the beta or cubic form.

Weight loss of samples tested at 500 psi H_2 and temperatures between 1000° and $1900^{\circ}C$ indicate generally lower corrosion rates for the coated material. The weight loss of SiC coated samples is about 10 times lower than that of uncoated graphite at about $1300^{\circ}C$.

Preliminary metallographic information suggests that the weight loss of the coated samples arises from reaction of the SiC with hydrogen, not from corrosion of the graphite substrate. Thus, SiC would afford protection as a sacrificial coating. However, the relatively high rate of SiC corrosion in hydrogen at $1500^{\circ}C$ does not indicate any practical application of this coating material.

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Figure 11. Pseudo - vertical section through the (U-Zr-C) ternary system between terminal compositions (ZrC+C) and (UC₂+C). The eutectic valley temperatures at the specified U/Zr mole ratio are given (These preliminary data were obtained by equilibrium thermal analysis)

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