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WANL-TME-1321
November, 1965

Westinghouse Astronuclear Laboratory

Westinghouse Astronuclear Laboratory
Contribution to the
Twenty-First High Temperature Fuel Committee Meeting
December 14, 15, and 16, 1965

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GENERAL INTRODUCTION

Advances in the high temperature fuel element program for the development of the NERVA nuclear rocket engine will be described. The principal topics will include:

- I. EXAMINATION OF NRX-A3 ELEMENTS AFTER HOT TEST
- II. ELECTRICAL CORROSION TEST DATA AND ANALYSIS
- III. NERVA FUEL DEVELOPMENT

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- 1 -
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I. EXAMINATION OF NRX-A3 ELEMENTS AFTER HOT TEST
 (M. Vogel, C. R. Simmons, D. Jacobs, R. Fiore, and C. Glassmire)

Introduction and Summary

NRX-A3 was the second reactor in the NRX-A series which was tested at full-power conditions. Although the NRX-A2 core was tested for less than four minutes at powers above 867 megawatts, the A3 core was operated and restarted for a total of approximately 17 minutes at full power plus 48 minutes at medium power (nuclear flux mapping experiments). The reactor was tested at Test Cell A at the Nevada Test Site in April and May, 1965, at the following conditions of total reactor power and run time.

<u>Power (megawatts)</u>	<u>Time at Power (minutes)</u>	<u>Date</u>
567	2.1	April 23
892	0.6	April 23
1113	3.5	April 23
545	1.5	May 20
872	0.5	May 20
1091	13.1	May 20
< 400*	7.0	May 28

The fuel elements in the core consisted of hexagonal cross section, 52-inch long extruded graphite shapes having 19 NbC coated coolant channels (bores) and containing pyrocarbon-coated UC_2 fuel particles.

Following the tests, the reactor was disassembled and the fuel elements were examined visually, photographed and weighed at the R-MAD building by the WANL post-operative examination team. The data obtained have been sorted and tabulated by means of

* Medium power test for approximately 48 minutes at a total integrated power of 4.3×10^5 Mw-sec.

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the IBM 7094 computer. Examination of the elements is continuing at the Los Alamos Scientific Laboratory and preliminary results have been obtained from metallographic examination, electrical resistivity measurements and flexure strength testing.

From an overall point of view, the elements lost considerably more weight in the A3 test than in the A2 test. The core average weight loss per element based on 1060 elements in the A3 core was 10.5 ± 2.0 grams as compared to 1.0 ± 0.3 grams for the A2 core. This increase in weight loss is proportionally larger than the increase in time at full power. It has been shown that significant corrosion occurred during the 48 minutes the A3 core was operated at low power for nuclear mapping experiments. In the future it may be necessary to define the goals of reactor testing in terms of separate reactor tests for the determination of fuel element lifetime and reactor system behavior.

The principal NERVA fuel element problem continues to be the hydrogen corrosion of the NbC coated bores. However, marked improvement in two of the corrosion effects on the external surfaces of the A2 elements were obtained in the A3 core. The in-leakage of hydrogen from between the pyrotile pressure plates which serve to "bundle" the core resulted in a heavy "tile-pattern corrosion" effect on 200 elements in the peripheral rows of elements of the A2 core as shown in Figure 1. The peripheral elements in the A3 core were coated with NbC on their external surfaces and this corrosion effect has been essentially eliminated as shown in Figure 2. A marked improvement in the external surface corrosion of the hot end tips of the A3 core is attributed to the use of non-fueled graphite tips cemented to the ends of the A3 elements. This resulted in an improved NbC coating on the external tip surface.

One new phenomenon observed in the NRX-A3 reactor which was not seen in NRX-A2 was inter-element bonding. This effect occurred primarily in the reactor periphery as shown in Figure 3, both between elements within clusters and between elements in adjacent clusters. In some instances the inter-elements bonds were so strong that elements were broken during attempts to separate them. Metallographic examination indicates that the bonding was a

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FIGURE 1 - NRX-A2 Core

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FIGURE 2 - NRX-A3 Core

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Position of Fuel Elements in NRX-A3
Involved in Inter-element Bonding

FIGURE 3

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consequence of "interstitial" pyrocarbon deposits between individual elements. Design and materials consideration must be given to the possible consequences of a large number of elements sticking together during longer time reactor testing. Particular attention must be given to potential thermal stress situations which may give rise to cracking of fueled or unfueled elements.

Although the non-fueled tip successfully improved the NbC end-coat performance, there appears to be a problem of corrosion at the cemented joint of the tip to the element. Leak tightness or porosity of this joint has been improved for the A4 elements, and the development of a joint brazing process is continuing.

The electrical corrosion test for corrosion performance of individual elements has been shown to provide an excellent criterion for judging in-reactor behavior of fuel elements.

A. Summary of Visual Observations

In general, there were three types of visible corrosion phenomena observed on fuel element surfaces. These were: corrosion in the region from the hot end undercut to the cemented joint (Stations 51 1/4 to 51 3/4)*, pinhole formation, and longitudinal surface corrosion along fuel element flats. Corrosion in the uncoated undercut region usually extended at least to the first row of coolant channels so as to expose NbC bore coating. Pinholes were observed at nearly all axial locations but with considerably fewer occurrences at the cold and hot ends. In many cases pinhole formation resulted in exposure of NbC coating in the coolant channels. Longitudinal surface corrosion generally consisted of light streaks and deposits associated with the high temperature reaction between interstitial hydrogen and graphite. In some cases this reaction was sufficient to expose the NbC in the coolant channels. Surface corrosion on peripheral elements attributable to hydrogen in-leakage between pyrotils was much less severe than that observed on A2 elements. Pinhole associated liner exposure is believed to have occurred from hydrogen emerging from coolant channels to fuel surfaces; whereas other liner exposure was caused by interstitial flow of hydrogen along fuel element flats.

* Station 51 1/4 is defined as 51 1/4 inches from the cold end of the 52 inch long element. It is the location of the non-fueled tip joint.

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- 7 -
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Unfueled cemented tips were generally in good condition, the NbC coating being still very adherent both on the flats and the end faces. Two or three elements were noted with missing tips, however, it is not known whether they fell off during operation or during subsequent hot cell handling.

1. Pinhole Distribution

Visual examination established that 943 of the 1604 elements had 1/64 to 1/16 inch diameter pinholes through the external flat surfaces to the bores. Only 58 elements of the A2 core were found to have pinholes.

Figure 4 shows the radial distribution of the fraction of elements with pinholes (excluding peripheral elements). The pinholing maximizes at the radial center of the core, which is consistent with the greater weight loss at the center described below. A maximum in the axial distribution was found at Station 38 as shown in Figure 5. It was observed that pinhole frequency extends over a longer axial band at the center of the core than at the periphery.

Considering those peripheral elements which were surface coated to reduce "tile-pattern" corrosion, improved surface corrosion was observed, particularly with respect to hot-end undercut corrosion. However, the surface coating did not inhibit pinhole formation. The outer row of whole (19 hole) elements had a pinhole frequency of 41% and lost an average of 4.5 grams; whereas the second row of peripheral elements showed 46% of the elements pinholed and lost an average of 5.4 grams.

In addition to pinhole formation, the external surface corrosion effects observed included NbC bore exposure, which resulted from excessive graphite corrosion. The radial distribution of the channel exposures follows that of the pinhole and weight loss distribution; it is at a maximum at the core radial center but is essentially constant in frequency from 10 to 40 cm radius. The axial distribution shows an increasing frequency of channel exposures towards the hot end. The maximum at Station 50 reflects the heavy corrosion at the fuel element undercut end.

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FIGURE 4

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FIGURE 5 - Total Axial Distribution of Pinholes in NRX-A3 Fuel Elements

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TABLE I

Summary of Fuel Element End Behavior

Sector	No. of Fuel Elements	Liner Exposure			Joint Behavior	
		Fuel Tip	Unfueled Tip	Through Joint Area	Holes At Joint*	Corrosion At Joint
1	272	165	53	5	46	9
2	263	209	59	47	44	5
3	259	202	63	82	30	6
4	267	179	55	93	65	8
5	263	208	34	63	17	4
<u>6</u>	<u>259</u>	<u>207</u>	<u>83</u>	<u>23</u>	<u>42</u>	<u>55</u>
Total	1583	1170	347	313	244	87
%		74	22	20	15	5.5

* Similar in appearance to pinholes.

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The other effects such as soot and pyrocarbon deposition have been summarized with the channel exposure data in Figure 6 showing their relative frequency of occurrence as a function of axial position.

2. Fuel Element End Behavior

A photograph of a heavily corroded A3 fuel element tip is shown in Figure 7. This picture shows most of the features observed in the corrosion of the ends of the A3 elements. The non-fueled tip was cemented to the fuel element with a carbonaceous cement. Porosity or leakage at the joint has resulted in corrosion at the joint. Exposed NbC bore coating is visible at the end of the undercut region. The second most significant effect visible is the NbC bore coating exposed at the cemented joint. This particular channel exposure may have resulted from the defect hole in the NbC liner visible in the photograph. The general corrosion undercutting and lack of adherence of the NbC end coating is also visible. A summary of the observations of joint corrosion and liner exposure is given in Table I.

B. Summary of Weight Losses

The average core weight loss based on 1060 elements was 10.5 ± 2.0 grams. Data on 566 fuel elements were not obtained because the automatic weighing device became inoperative. Figure 8 is a plot of the relative radial distribution of weight loss excluding the peripheral surface coated elements. The curve was obtained by averaging the weight loss of all the elements at discrete radii. The data clearly shows weight losses decreasing from 17.5 grams at the center to 6.0 grams at the core periphery. (The absolute magnitude of the average weight at each radius is estimated to be accurate to ± 2.0 grams. However the radial trend is accurate to ± 0.6 grams.) Most of this radial variation is attributable to the difference in maximum fuel temperature between the core center and periphery. Five of the six sectors had overcooled peripheral elements.

Several comments will be made regarding a comparison of the expected and actual weight losses of fuel elements from the NRX-A3 reactor. For a full-power run time of approximately 17 minutes, the expected NRX-A3 weight loss based on electrical corrosion

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FIGURE 6
Axial Distribution Surface Corrosion, Carbon Deposition
And Channel Exposure in NRX-A3 Fuel Elements

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Nozzle End View, A Face Up, of Element 49-14917, Core Position 2J5K, Before (Top) and After the A-3 Test. Note Corrosion to Channels at Shoulder on F Face In Area of Missing NbC and Hole at Joint in Channel 3.

FIGURE 7 - Example of Hole at Joint in Element 2J5K

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FIGURE 8 - Radial Distribution of Weight Loss in NRX-A3

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tests was 4 to 7 grams per element. The difference in predicted and observed weight loss can be quantitatively explained by the differences between testing at 100% power versus testing at combinations of 100% power and reduced power.

C. Results of Additional Studies

An incremental weight loss examination of an electrically tested fuel element typically shows the weight loss is greatest between 15-25 inches from the cold end rather than at the hot end tip as might be expected. The reasons for this are described in Section II of this report. The corrosion in this range is called mid-range corrosion and is currently under investigation. Incremental electrical resistance measurements and a limited number of incremental weight loss measurements along the length of the element show that the major corrosion attack in the NRX-A3 fuel elements is essentially identical to that seen in electrical testing.

Nineteen of the A3 elements were sectioned and examined for corrosion effects. The summary of the results is shown in Figure 9. The greatest weight change per inch of element length corresponds to the region where heavy binder attack and crack corrosion are observed. Figure 10 shows the major corrosion attack near Station 20-21. A minor peak near Station 40 results from the low power operation of the core and the consequent downstream shift of mid-range corrosion.

Figure 11 shows the post-test strength of A3 elements is greatest at the cold end of the elements. The strength may be 50% greater than pre-test strength. The cold-end increase in strength is attributed to radiation effects on the graphite. Note the reduction in flexure strength at axial stations where mid-range corrosion attack is observed.

II. ELECTRICAL CORROSION TEST DATA AND ANALYSIS (M. Blinn, G. Kilp, R. Leasure, A. Boltax)

The goal of the NERVA fuel element development program is to produce fuel elements with a 60 minute full-power lifetime with a corrosion weight loss rate of less than 0.5 grams/minute/element. The progress towards this goal is shown in Figure 12.

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FIGURE 9

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FIGURE 10

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FIGURE 11

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FIGURE 12 - NERVA Fuel Lifetime for NRX-A Reactors
(fuel element weight loss rate is shown for each case)

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Figure 13 presents a summary of electrical hydrogen corrosion data obtained on NRX-A fuel elements. Individual data points are shown for 1B test times above 30 minutes. The data indicate a generally linear increase in weight loss with time and can be described by a weight loss rate of 0.4 ± 0.15 grams per minute. The available data for test times above 30 minutes are on the high side of the weight loss spread. However, it is expected that the data spread will move toward lower weight losses as improvements are introduced to fuel element processing.

A. Corrosion Data Analysis (Weight Losses)

The corrosion data have been analyzed in terms of two major mechanisms of carbon weight loss; mid-range and high temperature corrosion. The details of the analysis were first reported in WANL-TME-1067, December, 1964. The current version of this analysis will be briefly reviewed.

The current equation used to describe carbon weight loss by mid-range corrosion at a given temperature is given by:

$$\Delta w_m = K \Delta(\Delta l/l) \exp (-30,000/RT) \quad (1)$$

where Δw_m is the incremental weight loss in grams per minute per inch of fuel element, K a constant, $\Delta(\Delta l/l)$ a semi-empirical term obtained from analysis of the difference in thermal expansion data of the NbC coating and the graphite matrix substrate, and T the test temperature in $^{\circ}K$. The total fuel element weight loss by mid-range corrosion is given by integrating the incremental weight losses over the length of the fuel operated below $1900^{\circ}C$. Thus, the total mid-range weight loss is given by:

$$\Delta W_m = \int_0^L \Delta w_m dL \quad (2)$$

where L is the length of fuel element operating below $1900^{\circ}C$. For the 1B hydrogen corrosion test Δw_m is taken to be 0.2 grams per minute which yields a value for K of 2.6×10^5 grams per minute.

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FIGURE 13 - Summary of Electrical Hydrogen Corrosion Data
For the 1B Test

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The term $\Delta(\Delta l/l)$ is a function of temperature and is obtained by:

1. Assuming most NbC coating cracks are closed at operating temperatures above 1600°C .
2. Determining values for $\Delta(\Delta l/l) = [(\Delta l/l)_{\text{NbC}} - (\Delta l/l)_{\text{fuel}}]$ below 1600°C from thermal expansion data after superimposing expansion curves at 1600°C .
3. Empirically selecting $\Delta(\Delta l/l)$ values above 1600°C to account for the coating cracks open between 1600°C - 1900°C .

The values of $\Delta(\Delta l/l)$ in current use are given in Table II. It is expected that these values will change as more quantitative corrosion data is accumulated.

The current equation describing the weight loss by carbon diffusion through the NbC coating is the same as that presented previously in WANL-TME-1067, namely:

$$\Delta w_h = k_h \exp(-76,000/RT) \quad (3)$$

where Δw_h is the carbon weight loss in grams per minute per inch of fuel element, k_h a constant dependent on NbC coating thickness and carbon concentration gradient, and T the test temperature in $^{\circ}\text{K}$.

Recently, measurements have been made of the incremental weight loss of corrosion tested fuel elements. Two examples of this type of data are shown in Figures 14 and 15 after the 1B10 + 20 corrosion test. Also shown are the normalized incremental electrical resistance change data and the calculated incremental weight loss obtained from equations (1) and (3). The diffusion portion of the weight loss was calculated assuming a two mil NbC coating thickness. The temperatures used in the calculation were the measured surface temperatures (in the 1B corrosion test) except for the region from 38-52 inches (from the cold end) where a nominal axial variation in NbC temperature was selected.

Several general comments will serve to review the data presented in Figures 14 and 15:

1. The calculated weight loss for 30 minutes of 1B testing is 9.0 grams. This may be compared to the range of values obtained in all 1B30 tests of 7.5 to 17.5 grams and

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FIGURE 14 - Comparison of Measured Incremental Weight Loss
And Resistance Change with Calculated
Incremental Weight Loss (F172/F127)

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FIGURE 15- Comparison of Measured Incremental Weight Loss
And Resistance Change with Calculated Incremental
Weight Loss (C239/C237)

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TABLE II - Values of $\Delta(\Delta l/l)$ Used in Computer Analysis of Mid-Range Corrosion

Temperature (°C)	$\Delta(\Delta l/l)$ ($\times 10^5$)
1000	184
1100	154
1200	123
1300	92
1400	62
1500	31
1600	3*
1700	2*
1800	1*
1900	0

* Empirical values selected to account for open cracks between 1600° and 1900° C

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an average value of about 11 grams. Note that the two examples of incremental weight loss bracket the average 1B30 weight loss.

2. The measured mid-range incremental weight loss peak occurs at 17-19 inches from the cold end whereas the calculated peak occurs at 22 inches. Uncertainties in the axial temperature values in the electrical corrosion test can give rise to an error of about ± 2 inches.

3. In general, the areas under the calculated and measured mid-range corrosion portions of the curve (10-25 inches) are in fair agreement whereas the calculated integrated weight losses from 27-47 inches are significantly less than the measured values. This result indicates that the analytical model does not account for a major weight loss phenomenon occurring between 1600° and 2200°C. Examination of axial sections of fuel elements indicates that corrosion rings and general carbon loss occur in this region.

4. The normalized values of incremental electrical resistance change provide an excellent correlation with the measured incremental weight loss. This correlation appears valid to 42-45 inches where a large decrease in electrical resistance is observed due primarily to uranium migration effects.*

Further measurements will be made of incremental weight loss and resistance change. Modifications will be made to the analytical corrosion model to bring it into better agreement with the actual measurements.

B. Metallographic Examination of Electrical Test Results

Detailed examination of axially sliced sections of corrosion tested fuel elements has revealed characteristic modes of corrosion damage along the length of the fuel element.** Starting from the cold end of the element the observed regions of corrosion damage may be summarized as follows:

* It is of interest that the electrical resistance peaks obtained on Code 9 fuel elements from the NRX-A3 reactor fall between Stations 19 and 22. Thus, the electrical and reactor tests position mid-range corrosion in the same axial region of the fuel element.

** See WANL-TME-1067, December, 1964, and LASL N-1-1585, July, 1965.

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1. The first sign of matrix corrosion is preferential attack of the matrix binder phase. This form of corrosion attack in the 1B test is generally observed starting at about Station 17 and ending at Station 23. A schematic representation of this form of attack is shown in Figure 16A. From similar observations of the RPT-2250°C test, LASL reports observing preferential binder attack (punky graphite) starting at about 10 inches and ending at 17 inches. The rather large difference in the WANL and LASL axial positions of this form of corrosion attack is somewhat unexpected, but may be partially accounted for by the known differences in the corrosion tests.

2. Starting at about Station 20, corrosion appears as specific attack of the graphite in the regions where the niobium carbide liner is cracked. This form of attack has the appearance of circumferential rings that is often referred to as corrosion rings. Corrosion rings are generally observed between Stations 20 and 40. LASL reports observing corrosion rings between Stations 15 and 28. Figure 16B shows a schematic representation of this form of corrosion attack. Often the corrosion rings link up to form a region of undercutting beneath the liner as illustrated in Figure 16C. Note that the maximum observed incremental weight loss in Figures 14 and 15 occurs in the region where binder attack and corrosion rings overlap.

3. External pinholes or interchannel communication holes are often seen between Stations 30 and 45. Figures 16D and 16E illustrate two mechanisms for pinhole formation. One mechanism involves the extension of corrosion rings to either the external surface or an adjacent channel. The other involves localized attack at high permeability regions or other inhomogeneities.

4. At axial stations between 40 inches and the hot end of the element, corrosion may take on the appearance of preferential binder attack or general channel enlargement. The behavior in this region of the fuel element is quite varied. In some cases little or no damage is seen in the 40 to 52 inch region of the element. Theoretically, one would expect to find a small carbon loss due to decarburization of the niobium carbide and undercutting of the graphite beneath the liner. The hot end damage, when visible, is generally greater than that expected to be accounted for by this diffusion mechanism.

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FIGURE 16-Schematic Illustration of Characteristic Corrosion Modes:
 A - Preferential Binder Attack, B - Corrosion Ring,
 C - Linked Corrosion Rings or Undercutting,
 D - Corrosion Ring Intersecting Surface,
 E - Corrosion Ring Plus Local Attack at Matrix Inhomogeneity

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III. NERVA FUEL DEVELOPMENT

A. Coated Particle Fuel Development

The principal goals of the WANL fuel particle development program have stressed increasing thermal stability of coated particles fuels and improving the mechanical strength of the coating to maintain particle integrity through element fabrication. During the past year the WANL fuel particle coating program has included the following experimental phases:

- (1) Development of single, duplex, and triplex pyrocarbon coatings
- (2) Pyrocarbon coatings doped with varying concentrations of Zr which reside as ZrC in the coating
- (3) PyC coatings in which aluminum was provided as a fugitive additive in the coating gas stream to catalyze graphitization of the PyC coating.

On the basis of numerous tests, a two step PyC coating procedure was developed to combine desirable features of both the low and high temperature coating into a duplex migration resistant coating. A low temperature (1200° - 1400° C) coating was deposited first to provide a medium density and isotropic laminar structure about 6-12 microns thick. Following this, a high temperature (1800° - 2200° C) coating was deposited directly on top of the low temperature coating to a thickness of about 12-15 microns. The outer coating was essentially a fine grain high density (>2 g/cc) isotropic structure. This combination coating has provided excellent uranium migration resistance at 2300° C for four hours.

To develop a particle capable of withstanding long times at high temperatures, experiments were initiated to evaluate the possibility of doping PyC coatings with a suitable additive to increase migration resistance. Zirconium and aluminum were selected because of desirable nuclear and chemical characteristics. Equipment was modified to permit the introduction of zirconium and aluminum in controlled quantities as $ZrCl_4$ and $AlCl_3$ vapor added to the main CH_4 -He stream in the inlet section of the fluidized bed. A total of 17 runs were made with gradually increasing aluminum additions and a total of 45 runs were made adding zirconium in a wide range of concentrations. Additives were incorporated only in the outer coating whereas the inner PyC coating was deposited in the usual manner.

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It was found that small additions of the zirconium in the range of 1-5 w/o (based on particle weight) provided significantly increased resistance to uranium migration. The majority of particles survived 2500°C in a two hours treatment without appreciable migration. The employment of this technique in a controlled production operation should provide coated UC₂ particles having a 100°-200° higher temperature capability than the best available coated particles.

The experimental runs using aluminum additions did not produce satisfactory coatings.

B. Diffusion of Carbon in Refractory Metal Carbides

The rates of carbon diffusion in the refractory-metal carbides of Nb, Ta, Ti, Zr, and Hf were determined. The diffusion equations obtained in the temperature range of the measurements are given below:

Nb

$$D_{NbC}^C = 1.0 \exp (-76 \text{ kcal/mol/RT})$$

$$D_{Nb_2C}^C = 0.09 \exp (-55 \text{ kcal/mole/RT})$$

Ta

$$D_{TaC}^C = 1.8 \exp (-90 \text{ kcal/mole/RT})$$

$$D_{Ta_2C}^C = 49 \exp (-98 \text{ kcal/mole/RT})$$

Zr

$$D_{ZrC}^C = 0.95 \exp (78.7 \text{ kcal/mole/RT})$$

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Ti

$$D_{TiC}^C = 9.9 \exp (-58.7 \text{ kcal/mole/RT})$$

Hf

$$D_{HfC}^C = 2 \exp (-94 \text{ kcal/mole/RT})$$

where D is in cm^2/sec and T , $^{\circ}\text{K}$. In general, D was assumed independent of concentration over the various stoichiometry ranges.

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