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VANADIUM ALLOY CLADDING DEVELOPMENT
QUARTERLY PROGRESS REPORT
FOR THE PERIOD ENDING DECEMBER 31, 1969THIS DOCUMENT CONFIRMED AS
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Approved by:

P. Murray
P. MurrayContract AT(30-1)-3791
U. S. Atomic Energy Commission

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Westinghouse Electric Corporation
Advanced Reactors Division
P. O. Box 158
Madison, Pennsylvania 15663

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SECTION 1

INTRODUCTION

GENERAL

This is the tenth quarterly progress report on Vanadium Alloy Cladding Development Project being performed under AEC Contract AT(30-1)-3791, and covers the work performed during the second quarter of fiscal year (FY) 1970.

SUMMARY OF OBJECTIVES

The project objectives are to further the development and to characterize the properties of vanadium-based alloys that may offer certain advantages over stainless steel for fuel cladding in a liquid-metal-cooled fast breeder reactor (LMFBR). For this application, the alloys must:

1. Have sufficient strength to contain the fuel at temperatures up to 800°C
2. Be resistant to liquid-sodium corrosion
3. Be compatible with mixed uranium-plutonium ceramic fuels
4. Be resistant to embrittlement by fast flux irradiation
5. Be readily fabricated into high-quality tubing at reasonable cost
6. Have acceptable nuclear physics characteristics

This development project is being accomplished under Commission sponsorship to supplement and extend the favorable data on novel vanadium-based alloys obtained under AEC Contract AT(30-1)-3487. The work is specifically directed to the identification of an alternate LMFBR fuel cladding that has improved mechanical properties and irradiation damage resistance compared to stainless steels.

The project consists of the following seven technical tasks and one administrative task.

UPVA-105 Fuel Element Design Evaluation

VCAA-110 Alloy Selection and Fabrication

VCAA-120 Structural and Mechanical Properties Evaluation

VCBA-125 Alloy Optimization

VCBA-130 Sodium Corrosion Evaluation

VCBA-140 Cladding Alloy-Fuel Compatibility Evaluation

VCBA-150 Irradiation Performance Evaluation

VCBA-160 Project Administration

UPVA-105 is an assessment of the technical and economic potential of the successful development of an advanced vanadium alloy cladding for a prototypical LMFBR fuel element. This task has been completed.

In VCAA-110, five vanadium alloy compositions have been melted into 4-inch ingots from which sheet, rod, and tubing have been fabricated (see Table 1).

Table 1. Five Vanadium Alloy Compositions		
Nominal Composition	Original Designation	New Designation ^a
V-9Cr-3Fe-1.3Zr-0.05C	HSV 207	VANSTAR-7
V-6Fe-5Cb-1.3Zr-0.05C	HSV 209	VANSTAR-9
V-8Cr-10Ta-1.3Zr-0.05C	HSV 208	VANSTAR-8
V-15Cr-5Ti		
V-20Ti		
^a VANSTAR is the general term for the family of Westinghouse developed alloys.		

VANSTAR-7, VANSTAR-8, and VANSTAR-9 are Westinghouse compositions developed under AEC Contract AT(30-1)-3487. V-15Cr-5Ti and V-20Ti are alloys developed by the Argonne National Laboratory (ANL).

The VANSTAR alloys were selected for the characterization, compatibility, corrosion, and irradiation phases. A limited characterization of the other alloys (V-15Cr-5Ti, and V-20Ti) has been performed. A stock of all five alloys, in the form of rod, sheet, and tubing, has been made available to other Commission contractors. This task has been terminated.

VCAA-120 was aimed at characterization of the VANSTAR-8 and VANSTAR-9 alloys, including structure analysis, response to heat treatment, creep in vacuum, welding, and thermal stability of welds. In addition, limited characterization was performed on the other three alloys. This task was terminated due to absence of funding.

VCBA-125 objectives are to determine the degree of reactivity of selected vanadium alloys with interstitial elements at low interstitial activity levels, and to evaluate the effect of these interstitials on the mechanical behavior of the alloys.

VCBA-130 is an evaluation of corrosion resistance of the VANSTAR-7, VANSTAR-8, VANSTAR-9, and V-20Ti alloys in pumped sodium loops, investigating the variables of temperature, flow rate, and time.

VCBA-140 is an evaluation of the compatibility of the same alloys with single (UC) and mixed (U,Pu)C carbide fuels at varying fuel stoichiometry.

VCBA-150 is a preliminary examination of the effect of fast flux irradiation on the mechanical and structural properties of the two selected alloys, VANSTAR-8 and VANSTAR-9.

VCBA-160, Project Administration, coordination and control will be furnished for the timely within-budget completion of the contract, meeting of AEC contract administration requirements, and coordination with other Commission-sponsored LMFBR R&D projects.

SUMMARY OF PRIOR WORK

All work related to the present investigations and performed prior to the present period has been fully reported in:

WCAP-3487-16, W. Pollack, K. C. Thomas, K. R. Jordan, R. T. Begley, R. W. Buckman, and E. C. Bishop, "Development of High Strength Vanadium Alloys," (June 1967).

WCAP-3791-9, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending September 30, 1967."

WCAP-3791-13, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending December 31, 1967."

WARD-3791-17, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending March 31, 1968."

WARD-3791-20, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending June 30, 1968."

WARD-3791-25, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending September 30, 1968."

WARD-3791-30, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending December 31, 1968."

WARD-3791-34, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending March 31, 1969."

WARD-3791-38, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending June 30, 1969."

WARD-3791-42, E. C. Bishop, R. A. Nadler, and G. A. Whitlow, "Vanadium Alloy Cladding Development Quarterly Progress Report for the Period Ending September 30, 1969."

SUMMARY OF CURRENT PROGRESS

A summary of current progress is shown below. Detailed progress is reported in the individual task sections of this document.

1. Small double arc melted ingots have been prepared for use in the VCBA-125 task on interstitial effects in vanadium. Detailed plans for this program, using an ultra high vacuum system in conjunction with a microbalance, have been made. A kinetic analysis has been made of the reactions between vanadium and oxygen and nitrogen.
2. Superior Tube Company reported surprisingly poor fabricability for the vanadium alloys which they were processing. As a result, tubular material for the sodium corrosion task was produced (at WANL) by swaging material from material extruded at Argonne National Laboratory.
3. Sodium corrosion loop reconstruction is near completion. Lower interstitial absorption was observed in vanadium alloys exposed to sodium at 690°C (in VTL-2) than previously reported for samples exposed at higher temperatures.
4. In the vanadium alloy-fuel compatibility study, no reactions were observed in fuel samples examined. However, some incompatibility (reaction) was seen in the vanadium alloy samples in contact with stoichiometric UC and hyperstoichiometric (U,Pu)C. The samples in contact with chromium stabilized (U,Pu)C fuel exhibited little if any reaction. Microhardness evaluation will be conducted in an attempt to substantiate these findings.
5. The vanadium alloy samples inserted in EBR-II, in the WARD/BNW High Flux Program have reached approximately half their goal exposure.

SECTION 2

VCBA-125 ALLOY OPTIMIZATION

R. W. Buckman Jr., R. A. Nadler, and R. C. Svedberg

OBJECTIVE

The objectives of this program are to determine the degree of reactivity of selected vanadium alloys with interstitial elements at low interstitial activity levels, and to evaluate the effect of these interstitials on mechanical behavior of the alloys.

Oxygen, nitrogen, and carbon will be introduced into four vanadium alloys by gas-metal reactions at elevated temperatures (600-800°C) and low partial pressures (10^{-4} to 10^{-8} torr). The rate of interstitial pick-up by the alloys will be measured using vacuum microbalance techniques. The alloys to be investigated are:

1. Pure Vanadium
2. V-10Mo
3. V-10Cr
4. VANSTAR-7 (V-9Cr-3Fe-1.3Zr-0.05C)

PRIOR WORK

This portion of the vanadium alloy cladding development program was begun during the first quarter, FY-1970. It is an extension of the Structure and Mechanical Property Evaluation Program, VCAA-120.

Plans were made and equipment ordered for a program to determine the degree of reactivity of selected vanadium alloys with interstitial elements at low interstitial activity levels. An evaluation of the effects of these interstitials on the mechanical properties of these alloys will also be made.

Tube reducing and drawing of the vanadium alloys for AEC Contractors' use and for use in lining the corrosion loop in Task VCBA-130 has been completed.

CURRENT PROGRESS

Alloy Melting and Processing

The alloys to be evaluated during this program have been double arc melted under a partial pressure of high purity argon. The consumable electrodes used for the first melt were pressed from the seventeen pound lot (No. 1) of electro-refined vanadium, characterized in WARD-3791-42, and from another lot (No. 2) of electro-refined vanadium. Both lots were received from the

Bureau of Mines. The analyses of the two lots are presented in Table 2.

Table 2. Analysis of Electro-Refined Vanadium		
Element	Lot No. 1 Analysis (ppm)	Lot No. 2 Analysis (ppm)
Al	b	b
Ca	b	b
C	20	20
Cr	b	<6
Cu	98	b
H ₂	10	20
Fe	b	b
Mg	4	2
Mn	b	b
Mo	b	b
Ni	6	b
N ₂	10	10
O ₂	100	140
Si	15	<15
Hardness	53	56 Rf
^b Below Spectrographic detection limit		

It had been reported earlier that the vanadium crystals would be consolidated by trough melting. However, severe "splashing" occurred, with this splashed vanadium adhering to the tungsten electrode and subsequently dripping into the vanadium melt. Dripping is expected because a low melting alloy does occur in the tungsten-vanadium system at 1635°C. This low melting composition would contain tungsten and could contaminate the trough melt.

Consequently, the crystals were compacted into 5/8-in. x 1-in. x 25-in. electrodes by a 400 ton load (32,000 psi) in a large hydraulic press. Alloying additions were made to the vanadium pressings in the following manner:

- V-10Cr - Electro-refined chromium particles were mixed with the vanadium and pressed into a composite electrode.
- V-10Mo - Molybdenum strips were fused to the flat sides of a pure vanadium electrode.
- VANSTAR-7 - Strips of zirconium, iron, and carbon cloth were laid in the vanadium-chromium powder mixture and pressed into an integral electrode.
- Pure V - Pressed from the electro-refined material.

The electrodes were arc melted in an argon atmosphere into 2-in. diameter ingots. Each of the four ingots from the first melting were quartered.

The four quarters were then welded into an electrode, and remelted in the consumable arc furnace.

The twice arc melted vanadium alloy ingots have been sectioned into 3/4-inch thick discs for further fabrication. Samples for chemical characterization have been taken from the ingots (as-cast) and have been submitted to the Westinghouse Research Laboratories for analysis.

Sections of each ingot are being polished to observe the as-cast macro and microstructure. A Vickers hardness traverse will then be made on a section from each ingot.

A 3/4-inch-thick disc, from the pure vanadium ingot, was flame sprayed with molybdenum, soaked at 500°C in argon, and forged on a Dynapak Forge to produce a pure vanadium disc about 0.250-inch thick. The molybdenum coating was then chemically stripped from the forged disc, and the vanadium was vacuum annealed at 900°C for one hour. The annealed sample was subsequently cold rolled (room temperature) into a 0.040-inch sheet without an intermediate anneal.

The remaining three alloys were canned in stainless steel for oxidation protection prior to forging. Higher forging temperatures will be required to break down the as-cast structure of these alloys.

Experimental Scoping

The diffusion coefficients of oxygen and nitrogen in pure vanadium have been calculated from the D_0 and Q values reported by Rostoker^[1]. These diffusion coefficients are presented in Table 3. At the temperatures of interest, the diffusion rates are fairly low, indicating that relatively long times are required to contaminate pure vanadium with the interstitials.

Table 3. Diffusion Coefficients of O ₂ and N ₂ in Pure Vanadium		
Temperature	Oxygen (cm ² /sec)	Nitrogen (cm ² /sec)
600°C	2.61 x 10 ⁻¹⁰	2.93 x 10 ⁻¹¹
725°C	2.00 x 10 ⁻⁹	3.70 x 10 ⁻¹⁰
825°C	6.64 x 10 ⁻⁹	1.65 x 10 ⁻⁹

From kinetic theory,^[2] the rate of impingement of the interstitial gases on a surface, at the pressures of interest, were calculated (Table 4). This table shows the amount of interstitial material that would be available to diffuse into the system assuming the sticking coefficient is 1.0. Comparing these values with the quantity of contaminant that could diffuse into the material under the constraint of diffusion control at each temperature, one can determine, upon assuming a suitable sticking coefficient, whether the system would form a surface scale (such as an oxide). This is the case when

the diffusion rate is slower than the rate of reaction of the interstitial with the surface. If the interstitial contamination rate is dependent on the rate of arrival of the gas at the surface, then such a scale should not form.

Table 4. Quantity of a Gas which Strikes a Surface Under the Conditions of the Experiment			
Pressure (torr)	Temperature		
	600°C	725°C	825°C
Oxygen (gram/cm ² -sec)			
10 ⁻⁴	1.117 x 10 ⁻⁶	1.044 x 10 ⁻⁶	9.996 x 10 ⁻⁷
10 ⁻⁶	1.117 x 10 ⁻⁸	1.044 x 10 ⁻⁸	9.996 x 10 ⁻⁹
10 ⁻⁸	1.117 x 10 ⁻¹⁰	1.044 x 10 ⁻¹⁰	9.996 x 10 ⁻¹¹
Nitrogen (gram/cm ² -sec)			
10 ⁻⁴	1.045 x 10 ⁻⁶	9.770 x 10 ⁻⁷	9.315 x 10 ⁻⁷
10 ⁻⁶	1.045 x 10 ⁻⁸	9.770 x 10 ⁻⁹	9.315 x 10 ⁻⁹
10 ⁻⁸	1.045 x 10 ⁻¹⁰	9.770 x 10 ⁻¹⁰	9.315 x 10 ⁻¹¹

Figure 1 shows the results of this calculation and analyses. The diagonal line shows the effect of pressure on the amount of oxygen and nitrogen which impinges on the surface of the vanadium in units of grams of gas per cm² of area per 100 hours. The band width shown describes the range of impingement for both oxygen and nitrogen over the temperature interval 600°C to 825°C. No good data has been located for carbon diffusion in vanadium.

If one assumes that the sticking coefficient of the gases on vanadium is of the order of 0.1, which is in the range determined for oxygen on columbium, then the pressure and temperature conditions that would minimize oxidation or surface contamination can be readily chosen. The fractional uptake of oxygen and nitrogen by pure vanadium was calculated, assuming a surface concentration based on the solubility of oxygen and nitrogen reported to the author in a private communication with the Bureau of Mines Research Laboratories in Albany, Oregon. Comparing these values with the amount available, factored by a suitable sticking coefficient, the temperature-pressure combinations which separate a reaction system into one that is diffusion controlled, or surface reaction controlled, can be readily seen. At pressures higher and at temperatures lower than those indicated by the horizontal and vertical lines on Figure 1, the reaction is diffusion controlled. At pressures lower and temperatures higher than those indicated, the reaction is surface controlled.

Figure 1 Correlation of Diffusion Data and Exposure Pressure Indicating Experimental Conditions Where Reaction at Surface of Sample is Diffusion Controlled.

Experimental Technique

The ultra high vacuum microbalance system, described in the previous quarterly report (WARD-3791-42), was leak tested and several bellows-flange seals were found to be defective. These have been replaced and leak testing and initial bake-out is continuing.

The vanadium alloys will be exposed to oxygen and nitrogen pressures at several values within the range of 10^{-4} to 10^{-8} torr. Carbon will be introduced by exposing samples either to methane or carbon monoxide. Reaction of the sample surface with one of these gases will result in reduction of the gas and deposition of carbon.

The temperature increment of interest for this study will be 600°C to 825°C. Alloy strip specimens 2.81 cm x 12.7 cm x 0.76 cm (0.30 in.) will be exposed. This will allow two tensile test specimens, four bend test specimens, and a contingency sample to be obtained from a single exposure.

It is intended to expose the vanadium and the various alloys for 100 hours, and compare the weight gains and mechanical properties of each of the other alloys exposed under similar conditions. The object of this investigation is:

1. To determine which of the alloys exhibits the least reactivity (including solid solubility and internal compound formation) to the interstitial elements, oxygen, nitrogen, and carbon.
2. Determine the effects of interstitial elements on the tensile properties and ductile transition temperature of the alloys.

This study permits continuous weighing of the sample during the entire exposure to the interstitial contaminate. Also, the capacity of the microbalance will enable one to expose up to 10 grams of material in a single experiment with microgram sensitivities. This means:

1. All of the samples required to evaluate the mechanical properties can be exposed simultaneously, under identical conditions.
2. The flat surface area of the exposed sheet is large enough compared to the edge area so that the assumed models for diffusion into a slab are more easily applied.
3. Sufficient material will be available to characterize the mode of interstitial accommodation in the various alloys.

In addition, the UHV system can be used to determine the sticking probabilities of the various gases on the alloys, should this information be required in the future to better characterize the data on experimental behavior of the various alloy-interstitial systems.

Production of Tubing for Task VCBA-130 and for USAEC Contractors

Superior Tube Company completed work on the fabrication of VANSTAR-7, -8, and -9, single extruded tube blanks. The results were disappointing in that the yield of good material was much less than expected. The second 1.00-in. OD x 0.25-in. wall piece of VANSTAR-7, which was re-annealed in an effort to improve fabricability (WARD-3791-42), was reduced to 0.875-in. OD x 0.25-in. wall, but the piece was too defective to permit shipment to USAEC contractors or to process to a smaller size. This material may have contained defects which were carried over from the original cast billets. The fabricability of the second piece of V-15Cr-5Ti was as limited as that of the first piece, so that no useable tubing was made of this alloy.

The first pieces of VANSTAR-8 and VANSTAR-9 were annealed and given a second tube reduction to 0.875-in. OD x 0.25-in. wall. Both pieces were returned to WANL where they received a final anneal at 1200°C. Inspection after annealing showed the VANSTAR-9 to be completely sound, but there were some ID cracks in the VANSTAR-8. The AEC contractors for these materials have been informed as to the final status of the VANSTAR-8 and -9 tubing, and have been sent the VANSTAR-9, and offered the VANSTAR-8, which may be useable for the intended applications.

The second pieces of VANSTAR-8 and -9 were being processed to 0.386-in. OD x 0.050-in. wall for Task VCBA-130. Both pieces successfully negotiated the first tube reduction, but the VANSTAR-9 was completely lost, through cracking, on the second reduction. Because of excessive end losses in the first tube reduction, only six feet of VANSTAR-8 tubing was produced in the size required by WARD. Since this quantity was insufficient for the loops, the 0.500-in. OD x 0.050-in. wall tubing of VANSTAR-7 and -8 and -9, previously produced by ANL from double extruded tube blanks, was swaged to 0.386-in. OD x 0.062-in. wall, annealed at 1200°C, and ultrasonically inspected at WANL. Thickening of the tube wall during swaging necessitated the remachining of the WARD corrosion specimens (see VCBA-130).

It was not possible, within the scope of the present investigations, to positively identify the reason or reasons for the limited fabricability of the VANSTAR single extruded alloys, or of V-15Cr-5Ti. However, since high yields of excellent quality tubing had been made of all these alloys by double-extrusion at Argonne National Laboratory, it is suspected that the single extrusion at Oak Ridge did not sufficiently work the as-cast extrusion billets for optimum ductility. It is anticipated that commercial production of such alloys from larger ingots would entail double extrusion to commercial size tube blanks.

SECTION 3

VCBA-130 SODIUM CORROSION EVALUATION

R. J. Hornak, S. L. Schrock, and G. A. Whitlow

OBJECTIVES

The objectives of this task are to evaluate the effects of flowing sodium in the temperature range of 600 to 800°C on the corrosion and mechanical properties of the vanadium-based alloys (VANSTAR-7, VANSTAR-8, and VANSTAR-9) and the V-20Ti alloy, and to gain some understanding of the mechanism of the corrosion process. The study is being conducted in pumped loops, investigating the variables of temperature, flow rate, and time. In addition, the effects on the loop containment materials under these conditions are being examined.

PRIOR WORK

All work related to the present investigation and performed prior to the present period was fully reported in the nine previous Vanadium Alloy Cladding Development Quarterly Progress Reports (see page 3).

In the first two loop runs (VTL-1 and VTL-2), it was found that when exposed to flowing sodium containing less than 10 ppm oxygen at 675 to 800°C, samples of the VANSTAR-7, -8, -9, and the V-20Ti alloys gained weight. The increases in weight were accompanied by microstructural changes and hardness increases.

Chemical analyses of corroded VANSTAR-9 and V-20Ti samples revealed that large quantities of the interstitials (nitrogen, carbon, and oxygen) were absorbed during corrosion, resulting in the observed weight gains. Parabolic kinetics were assigned to the corrosion processes up to 1500-hours exposure time, and "activation energies" for VANSTAR-7, -9 and V-20Ti were calculated. However, this was not the case for samples exposed to sodium for 2650 hours.

Due to the interstitial absorption, considerable reductions in mechanical properties of corroded samples, especially at room temperature, were observed. Analysis of the stainless steel loop tubing revealed carbon and nitrogen depletion at the sodium exposed surface. At the air exposed surface, results indicated that nitrogen had diffused into and through the tubing. Calculations based on diffusion data confirmed these results.

The design of the next two loop runs (VTL-3 and VTL-4), in which the test sections will be lined with vanadium alloy tubing, and in which stabilized stainless steel containment tubing will be used (VTL-4), was completed and fabrications started. The design and the philosophy behind these changes was fully described in two previous quarterly reports (WARD-3791-34 and WARD-3791-38).

CURRENT PROGRESS

Loop Reconstruction

Problems which arose in the fabrication of the vanadium alloy tubing, required for the loop test section liners, at Superior Tube Company, have necessitated a change in plan. Material for the liners had to be produced at WANL from stock 0.500-in. OD x 0.050-in. wall tubing. Fabrication of nominal 0.386-in. OD x 0.050-in. wall tubing by swaging has been successfully achieved in each of the three VANSTAR alloys. However, since the maximum tubing length produced was 68 in., it will be necessary to join two pieces prior to insertion in the 92-in.-long test section. Trial welds of these vanadium alloy test section liners have proved to be satisfactory. Machining of the vanadium alloy strip samples is underway for both loops. In order to fit these samples in the test section, the nominal width of a sample has been reduced to 0.25 in. Alignment and fixturing of samples in the test section will be achieved by means of pins in the end thickness of each sample. After completion of this stage, the samples will be dimensioned, cleaned, weighed, and assembled into the loop system. It is anticipated that final assembly of the test loops VTL-3 and VTL-4 will be completed early in the next reporting period.

Interstitial Analysis

The interstitial layer analysis results for the vanadium alloy samples from the high velocity loop system VTL-2, reported in the last quarterly report, have been confirmed on additional samples.

After 1500 hours exposure to flowing sodium at 690°C (VTL-2), no oxygen gradients were observed across the thickness of a VANSTAR-9 sample (Figure 2). However, the bulk oxygen level had increased from 600 ppm in the unexposed material to 800-850 ppm in the sodium exposed sample. This result suggests that the oxygen supply to the sample at this temperature was limited, although diffusion rates at 690°C must be high enough to permit diffusion to occur across the sample width. The absence of the extremely high nitrogen and carbon surface levels, seen after 790°C exposure temperatures, may also have facilitated diffusion of oxygen through the sample thickness.

Analytical results for a V-20Ti sample exposed under similar conditions (Figure 3) revealed high surface levels of carbon and nitrogen ($\geq 38,000$ ppm), but little increase at distances of a few mils (10^{-3} in.) from the exposed surface. The absence of any oxygen gradient was again observed. The oxygen increases are small ($\sim 30\%$) compared to those measured for samples exposed at higher temperatures ($\sim 790^\circ\text{C}$), but are reasonably consistent with data reported by other workers in this area.

Metallography

Previous metallographic, microhardness, and analytical data have indicated that, as a result of sodium exposure, vanadium alloys form a hard surface skin. The skin composition is mainly vanadium carbide/nitride. However,

Figure 2 Interstitial Absorption in a VANSTAR-9 Sample Exposed to Sodium Flowing @ 14 fps and Containing <10 ppm Oxygen for 1500 Hours @ 690°C

3519-3

Figure 3 Interstitial Absorption in a V-20Ti Sample Exposed to Sodium Flowing @ 14 fps and Containing <10 ppm Oxygen for 1500 Hours @ 700°C

3519-2

(a) UNEXPOSED

10 μ

(b) EXPOSED

10 μ

Figure 4 Scanning Electron Micrographs of the Surfaces of VANSTAR-7 (V-Fe-Cr-Zr-C) Alloy (a) Unexposed and (b) Exposed to Sodium Flowing @ 5 fps for 1500 Hours @ 710°C

3519-4

(a) UNEXPOSED

10 μ

(b) EXPOSED

10 μ

Figure 4 Scanning Electron Micrographs of the Surfaces of VANSTAR-7 (V-Fe-Cr-Zr-C) Alloy (a) Unexposed and (b) Exposed to Sodium Flowing @ 5 fps for 1500 Hours @ 710°C (Continued)

3519-5

(a) UNEXPOSED

1 μ

(b) EXPOSED

1 μ

Figure 4 Scanning Electron Micrographs of the Surfaces of VANSTAR-7 (V-Fe-Cr-Zr-C) Alloy (a) Unexposed and (b) Exposed to Sodium Flowing @ 5 fps for 1500 Hours @ 710°C (Continued)

3519-6

by visual examination and low magnification microscopy little if any change in surface morphology was apparent. Recently, some samples of the VANSTAR-7 (V-Fe-Cr-Zr-C) alloy have been examined, using a scanning electron microscope. Some of the preliminary observations of the actual surface exposed to sodium (no prior preparation) are illustrated in Figure 4. Some changes in surface topography have occurred, taking the form of surface obtrusion on a micro-scale. This is consistent with previous findings in which no surface scale was observed.

SECTION 4

VCBA-140 CLADDING ALLOY-FUEL COMPATIBILITY EVALUATION

R. C. DeKlever, M. French, M. Hodge, and G. A. Whitlow

OBJECTIVE

The objective of this task is to assess the compatibility of the VANSTAR alloys and V-20Ti with mixed uranium-plutonium carbide fuels and with sodium bonded uranium carbide fuel for times up to 5000 hours. The V-20Ti alloy has been selected as a reference so that these results may be compared with prior ANL work in this field.

PRIOR WORK

Two compatibility capsules have been tested. The first (#7), containing three pellets of unmodified hypostoichiometric UC and six pellets of unmodified hyperstoichiometric (U,Pu)C fuel with samples of VANSTAR-8 and -9, and V-20Ti, accumulated 4,650 hours at 800°C. The second capsule (#12), contained nine pellets of Cr₂₃C₆ modified (U,Pu)C fuel and samples of VANSTAR-8 and -9, and V-20Ti, accumulated 2,500 hours at 800°C. Both capsules were sodium bonded. Disassembly of both capsules was completed during the last reporting period, and evaluation of the first capsule initiated.

CURRENT PROGRESS

Metallographic examination of the fuel and vanadium alloy samples from both compatibility capsules has now been completed. Based solely on metallographic examination (microhardness will be performed in the near future), certain general observations which are valid for these particular experimental conditions (> 2500 hours at 800°C) may be made. In the first capsule, containing hypostoichiometric UC and hyperstoichiometric (U,Pu)C, no reaction was observed in the fuel at the alloy/fuel interface. All the vanadium alloy samples showed reaction generally in the form of a harder acicular type phase near the contact surface. The degree of incompatibility (in decreasing depth of reaction) was VANSTAR-9, V-20Ti, and VANSTAR-8. No significant difference was detected between reactions with the single or mixed carbide fuel.

In the second capsule, containing chromium stabilized (U,Pu)C fuel, no reaction was seen in the fuel at the contact surfaces. In addition, very little reaction was seen in the VANSTAR-8 and VANSTAR-9 samples in contact with its fuel. However, the V-20Ti alloy did exhibit some incompatibility (reaction) at the fuel contact surface.

Microhardness evaluation of the vanadium alloy sample will commence at WARD Cheswick as soon as the instrument is available for use.

SECTION 5

VCBA-150 IRRADIATION PERFORMANCE EVALUATION

P. J. Levine and G. A. Whitlow

OBJECTIVE

The objective of this task is to examine the effects of fast flux irradiation on the mechanical properties and structures of the alloys VANSTAR-8 and VANSTAR-9. Exposures of 1 to 2×10^{22} n/cm² above 0.1 Mev will be achieved in EBR-II.

PRIOR WORK

All work related to this phase of the investigation was reviewed and fully reported in previous quarterly progress reports^[3-11]. The irradiation program on this project consists of two parts:

1. The WARD/BNW High Flux Program - An irradiation pin composed of six subcapsules containing vanadium alloys, and designed in conjunction with BNW for insertion in Row 2 of EBR-II, was assembled and shipped to the reactor site. This pin was inserted in the reactor in Run 33 during March 1969. It was placed in position 2BI and designated as subassembly No. X057.
2. The CDAA-500^c, EBR-II Program - Vanadium alloy tensile specimens (one per capsule) were contained in the bottom of each Phase I - EBR-II fuel capsule under Task CDAA-500. These specimens were contained in stainless steel capsules filled with sodium, and were to be irradiated at reactor coolant ambient temperature (360°C). Two fueled capsules, each containing a VANSTAR-9 sample, were inserted in EBR-II in March 1969.

Eight additional carbide fueled capsules, each containing one vanadium alloy sample, were assembled, shipped, and were awaiting insertion in EBR-II.

CURRENT PROGRESS

WARD/BNW High Flux Program

The irradiation pin inserted in EBR-II in March 1969 continued to accumulate fluence. As of October 15, 1969, the capsule accumulated exposure was 7211 MWd compared to an estimated goal exposure of 15000 MWd.

^cCDAA-500 Fast Flux Irradiation Studies Task of Uranium Plutonium Carbide Development.

CDAA-500, EBR-II Program

It is anticipated that the first two capsules (W2X and W8X) will be removed from the reactor in mid January 1970.

An additional capsule (W7F) will be inserted in pile on January 1, 1970; the remaining seven fueled capsules are still awaiting insertion.

SECTION 6

VCBA-160 PROJECT ADMINISTRATION

F. A. Brassart, W. B. Heubel, and P. Murray

OBJECTIVE

The objectives of this task are to assure that the project is successfully completed, on schedule, within the budget, and to the satisfaction of the Atomic Energy Commission; to assure compliance with contracted obligations; and to coordinate this project with other AEC-sponsored and Westinghouse-sponsored LMFBR development projects.

Overall project direction and day-to-day administration will be provided under this task. Plans and controls will be established and maintained; periodic reviews will be held with the Commission; correspondence and reports will be coordinated; and day-to-day technical and administrative liaison with the Commission will be provided.

CURRENT PROGRESS

Work programs for FY-1970 were submitted to the Commission in June 1969. Technical work has continued on the basis of those work programs.

The quarterly progress report (WARD-3791-42) for the period ending September 30, 1969, was prepared and distributed.

SECTION 7

REFERENCES

1. William Rostoker, The Metallurgy of Vanadium, John Wiley and Sons, Inc., p. 81, (1953).
2. S. Dushman, J. M. Lafferty, Scientific Foundations of Vacuum Technique, John Wiley and Sons, Inc., p. 14, (1962).

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Division of Reactor Development and Technology
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Division of Naval Reactors
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Assistant Director Pacific Northwest Laboratory
U. S. Atomic Energy Commission
P. O. Box 550
Richland, Washington 99352

AEC-RDT Site Offices
Argonne National Laboratory
U. S. Atomic Energy Commission
Building 2
Argonne, Illinois 60439

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USAEC Fast Breeder Reactor Information Exchange Arrangements

Argonne National Laboratory
U. S. Atomic Energy Commission
P. O. Box 2108
Idaho Falls, Idaho 83401

Atomics International
U. S. Atomic Energy Commission
P. O. Box 1446
Canoga Park, California 91304

General Electric Company, NSP
U. S. Atomic Energy Commission
P. O. Box 15132
Cincinnati, Ohio 45215

General Electric Company
U. S. Atomic Energy Commission
310 DeGuigne Drive
Sunnyvale, California 94086

Gulf - General Atomic
U. S. Atomic Energy Commission
P. O. Box 2325
San Diego, California 92112

Oak Ridge National Laboratory
U. S. Atomic Energy Commission
P. O. Box X
Oak Ridge, Tennessee 37830

Pacific Northwest Laboratory
U. S. Atomic Energy Commission
P. O. Box 550
Richland, Washington 99352

Westinghouse Electric Corporation (2)
U. S. Atomic Energy Commission
Advanced Reactors Division
P. O. Box 158
Madison, Pennsylvania 15663

LABORATORIES

Director, LMFBR Program Office (2)
Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439

Director, Metallurgy Division (2)
Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439

Manager, FFTF Project (2)
Pacific Northwest Laboratory
P. O. Box 999
Richland, Washington 99352

FTTF Fuels Department (2)
Pacific Northwest Laboratory
P. O. Box 999
Richland, Washington 99352

Manager, Chemistry and Metallurgy Division
Pacific Northwest Laboratory
P. O. Box 999
Richland, Washington 99352

Division Leader, Chemistry and Metallurgy Division (CMB)
Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico 87544

Director, Metallurgy and Materials Science Division
Brookhaven National Laboratory
Upton, New York 11973

Director, Metals and Ceramics Division (2)
Oak Ridge National Laboratory
P. O. Box X
Oak Ridge, Tennessee 37830

Division Chief, M & S Division
NASA - Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135

Director, Atomics International
Liquid Metal Engineering Center
P. O. Box 309
Canoga Park, California 91304

Division Leader, Inorganic Materials
Chemistry Department
Lawrence Radiation Laboratory
P. O. Box 808
Livermore, California 94551

Manager, Advanced Development Activity
General Electric Company
Knolls Atomic Power Laboratory
P. O. Box 1072
Schenectady, New York 12301

General Manager, Westinghouse Electric Corporation
Bettis Atomic Power Laboratory
P. O. Box 79
West Mifflin, Pennsylvania 15122

CONTRACTORS

Director, LMFBR Technology Program
Atomics International
P. O. Box 309
Canoga Park, California 91304

Associate Manager, Materials Engineering Department
Battelle Memorial Institute
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Director, Nuclear Development Center
The Babcock and Wilcox Company
Atomic Energy Division
Lynchburg, Virginia 24501

Manager, Nuclear Laboratories
Combustion Engineering, Inc.
Nuclear Division
Prospect Hill Road
Windsor, Connecticut 06095

Laboratory Assistant Director
Gulf-General Atomic
P. O. Box 608
San Diego, California 92112

Manager, Plutonium Chemistry and Ceramics Fuels Development
Plutonium Laboratory
Nuclear Materials and Equipment Corporation
Leechburg, Pennsylvania 15656

Manager, Sodium Reactor Technology
General Electric Company
Breeder Reactor Development Operation
210 DeGuigne Drive
Sunnyvale, California 94086

Manager, Research, United Nuclear Corporation
Research and Engineering Center
Grasslands Road
Elmsford, New York 10523

Head, Fuels and Materials
Atomic Power Development Associates
1911 First Street
Detroit, Michigan 48226

K-2 Group Leader
Reactor Division
Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico 87544

Irradiations Manager
EBR-II Project
Argonne National Laboratory
P. O. Box 1096
Idaho Falls, Idaho 83401

Director, Vallecitos Nuclear Center
General Electric Company
P. O. Box 846
Pleasanton, California 94566

EXTERNAL DISTRIBUTION LIST

Pacific Northwest Laboratory

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Richland, Washington 99352
Attn: A. Bement
E. Evans

Oak Ridge National Laboratory

P. O. Box X
Oak Ridge, Tennessee 37830
Attn: W. Harms
P. Patriarca
J. Weir
J. Devan

Combustion Engineering Corporation

Nuclear Division
Windsor, Connecticut 06095
Attn: S. S. Christopher
J. Stewart

Battelle Memorial Institute

505 King Avenue
Columbus, Ohio 42301
Attn: H. R. Ogden
R. Bauer

General Electric Company

Advanced Products Operation
310 DeGuigne Drive
Sunnyvale, California 95103
Attn: E. L. Zebroski
C. Spalaris

United Nuclear Corporation

Grasslands Road
Elmsford, New York 10523
Attn: K. Goldmann

Nuclear Materials and Equipment Corporation

Apollo, Pennsylvania 15613
Attn: Z. Shapiro

Babcock and Wilcox Company

Lynchburg, Virginia 24505
Attn: C. Baroch

Babcock and Wilcox Company

1725 K Street N. W.
Washington, D. C. 20006
Attn: L. R. Weissert

National Aeronautics and Space Administration
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: G. M. Ault
C. A. Barrett
W. D. Klopp

National Aeronautics & Space Administration
1520 H. Street, D. C.
Washington, D. C. 20546
Attn: G. Deutsch

Department of Navy
Naval Air Systems Command
Washington, D. C. 20360
Attn: I. Machlin
R. Schmidt

Brookhaven National Laboratory
Upton, Long Island, New York 11973
Attn: D. Gurinsky
C. Klamut

Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439
Attn: M. V. Nevitt
J. H. Kittel
P. Shewmon
L. Kelman
N. Carson
Che-Yu-Li

Ames Laboratory
Iowa State University
Ames, Iowa 50010
Attn: O. H. Carlson
F. A. Schmidt

Mine Safety Appliance Research Corporation
Evans City, Pennsylvania 16033
Attn: R. Werner

Wah Chang Corporation
Albany, Oregon
Attn: S. Worchester
E. Baroch

Albany Metallurgy Research Center
U. S. Bureau of Mines
Albany, Oregon 97321
Attn: H. G. Poole
H. Kato

Boulder City Metallurgy Research Laboratory
U. S. Bureau of Mines
500 Date Street
Boulder City, Nevada 89005
Attn: T. A. Sullivan