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COMPATIBILITY OF NIOBIUM,
TITANIUM AND VANADIUM
METALS WITH LMFBR CLADDING

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C. N. Wilson

October, 1975

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COMPATIBILITY OF NIOBIUM, TITANIUM
AND VANADIUM METALS WITH LMFBR CLADDING

C. N. Wilson

ABSTRACT

A series of laboratory capsule annealing experiments were conducted to assess the compatibility of niobium, vanadium and titanium with 316 stainless steel cladding in the temperature range of 700°C to 800°C. Niobium, vanadium and titanium are candidate oxygen absorber materials for control of oxygen chemistry in LMFBR fuel pins. Capsule examination indicated good compatibility between niobium and 316 stainless steel at 800°C. Potential compatibility problems between cladding and vanadium or titanium were indicated at 800°C under reducing conditions. In the presence of $\text{Pu}_{0.25}\text{U}_{0.75}\text{O}_{1.98}$ fuel ($\Delta\bar{G}_{\text{O}_2} \approx -160$ kcal/mole) no reaction was observed between vanadium or titanium and cladding at 800°C.

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I. INTRODUCTION

Intergranular attack of austenitic stainless steel cladding at the fuel-cladding interface represents a potential limitation on goal exposure for FTR/LMFBR fuels. Fuel pin oxygen chemical potential ($\Delta\bar{G}_{O_2}$) and fuel-cladding interface temperatures appear to be the two most important parameters governing the extent of cladding attack. The cladding attack rate may be lowered by reducing the cladding operating temperature. However, with current LMFBR pin designs, a reduction in cladding temperature would have an adverse effect on overall thermal efficiency. Control of the fuel pin oxygen potential is theoretically possible by placing a material into the fuel pin to chemically buffer or getter oxygen. Irradiation tests in EBR-II (HEDL P-23 Special Pin Test and ANL O-8 Test) have been initiated to test the effectiveness of various concepts and candidate materials proposed for the purpose of controlling oxygen chemistry in LMFBR fuel pins. Among concepts to be tested are: 1) porous metallic gettering pellets placed at the ends of the fuel column, and 2) sputtered metallic coatings on the fuel pellet surface. Niobium, titanium and vanadium metals are candidate oxygen absorber materials to be tested.

Successful application of oxygen absorber concepts to high performance LMFBR fuel pins requires that the candidate oxygen absorber materials be compatible with austenitic stainless steel cladding for extended periods at the applicable cladding inner surface temperature. Peak cladding inner surface temperatures for high performance LMFBR fuel pins are generally expected to fall into the 600°C to 760°C temperature range. Initial peak cladding temperature for FFTF is predicted at approximately 650°C and FFTF rated peak cladding temperature is 757°C.⁽¹⁾ The peak cladding inner surface temperature predicted for the HEDL P-23 Special Pins Test in EBR-II is approximately 700°C at normal power and 750°C at 10% overpower.

Binary phase diagrams⁽²⁾ applicable to Nb, Ti or V used with Fe-Ni-Cr alloys were reviewed for indications of eutectic melting in the 700°C to 800°C temperature range. For Nb, the lowest temperature liquid phase was found in the Nb-Ni system and occurred at 53% Nb and 1175°C. Niobium - 316 stainless steel diffusion couples have been studied and only 15 microns of

significant interface inter-diffusion reported after 48 hours at 1100°C.⁽³⁾ Good chemical compatibility should therefore be expected between Nb and 316 stainless steel at LMFBF cladding temperatures. Binary phase diagrams indicated no apparent compatibility problems between V and 316 stainless steel, with the lowest temperature liquid occurring in the V-Ni system at 47.5% V and 1205°C. Eutectic melting occurs in the Ti-Ni system at 71% Ti and 943°C, and in the Ti-Fe system at 67% Ti and 1086°C, indicating potential compatibility problems between Ti and 316 stainless steel at LMFBF cladding temperatures. However, information obtained in an initial literature survey was inclusive regarding the expected compatibility of titanium and vanadium with stainless steel at LMFBF cladding temperatures.

This report describes a series of laboratory capsule annealing experiments conducted in order to evaluate any compatibility problems between 316 stainless steel cladding and candidate oxygen gettering materials prior to in-reactor testing.

II. EXPERIMENTAL PROCEDURES

Sixteen capsules were assembled, annealed and examined during this investigation. The capsule test matrix is given in Table I. Four different capsule configurations (illustrated in Figure 1) were used. All of the compatibility capsules were sealed under argon by arc fusion welding around the end caps in an inert atmosphere glovebox. Capsules containing the metal-coated mixed-oxide fuel were encapsulated in an additional secondary capsule to provide double encapsulation. The assembled compatibility capsules were annealed in air using electrically heated furnaces. Temperature was controlled by power proportioning solid state controllers using calibrated type K thermocouples. Temperature accuracy is estimate at $\pm 5^\circ\text{C}$. Annealed capsules were prepared for metallography by drilling a hole into the capsule interior and vacuum impregnating the capsules with polyester metallographic mounting resin. The capsules were then sectioned through planes designated (A---A) in Figure 1 and polished for metallographic examination, which included SEM, electron probe,

TABLE I
OXYGEN ABSORBER COMPATIBILITY CAPSULES

<u>Capsule Designation</u>	<u>Capsule Description</u>	<u>Temp. (°C)</u>	<u>Time (Hours)</u>	<u>Approximate Reaction Depth ($\times 10^{-3}$ Inches)</u>
Nb	Nb powder - SS* pressure contact	800	137	0
Ti	Ti powder - SS* pressure contact	800	137	0.5
V	V powder - SS* pressure contact	800	137	0.5
$\overline{\text{Nb}}$	Nb foil - SS* pressure contact	800	137	-
$\overline{\text{Ti}}$	Ti foil - SS* pressure contact	800	137	0.5
$\overline{\text{V}}$	V foil - SS* pressure contact	800	137	-
Ti-1	Porous Ti pellets in cladding	800	89	0.5
Ti-2	Porous Ti pellets in cladding	700	568	0
Ti-3	Porous Ti pellets in cladding	800	568	1
Ti-V-1	Ti & V coated fuel pellets in cladding	800	89	0
Ti-V-2	Ti & V coated fuel pellets in cladding	700	568	0
Ti-V-3	Ti & V coated fuel pellets in cladding	800	568	0
Ti-4	316 SS* pellet on Ti Dynapack pellet	700	71	0
Ti-5	316 SS* pellet on Ti Dynapack pellet	800	71	0.5
Ti-6	316 SS* pellet on Ti Dynapack pellet	700	568	0
Ti-7	316 SS* pellet on Ti Dynapack pellet	800	568	1.0

*SS abbreviation for stainless steel.

and electrolytic etching. Capsules containing metal-coated mixed-oxide fuel were sectioned, mounted, and examined in a plutonium metallography glovebox.

A. Capsules with Pressure Contacted Powder or Foil

In the initial investigation six capsules were assembled to give pressure contact between niobium, titanium and vanadium powders, or metal foils and 316 stainless steel capsule components. Capsules designated Nb, Ti and V contained niobium, titanium or vanadium powders in the minus 200 to 500 mesh size range. Capsules $\overline{\text{Nb}}$, $\overline{\text{Ti}}$, and $\overline{\text{V}}$ contained niobium, titanium and vanadium foil. The metal powders and foils were loaded into the capsules in an argon inert atmosphere glovebox. The 3/8 x 16 threads per inch end bolts were then installed and torqued to 20 foot-pounds to provide pressure contact. The capsules were sealed by arc fusion of the capsule to the bolt under argon. The six sealed capsules were then annealed at 800°C for 137 hours.

The initial six capsules were designed to give extreme conditions of fine reactive powder or foil in pressure contact with stainless steel at a temperature above the maximum in-reactor cladding inner surface temperature. No interaction between the candidate oxygen absorber materials and stainless steel under these conditions would have indicated satisfactory compatibility and the study could have been terminated. However, results from the initial capsules indicated that further tests should be conducted to test porous Ti oxygen-gettering pellets and Ti and V sputter-coated mixed-oxide fuel for compatibility with 316 stainless steel cladding.

B. Capsules with Porous Ti Pellets in Cladding

In capsules Ti-1, Ti-2 and Ti-3 porous titanium pellets were placed in 316 20% cold worked split cladding halves. The porous titanium pellets used were prepared by Battelle-Northwest Laboratories using a Dynapack powder metallurgy process and are shown in Figure 2. Contact pressure between porous pellets and the cladding was due only to the pellet weight. The pellet diameter was less than the cladding I.D., providing a single narrow region of axial contact between titanium and the

(A)

CAPSULES:
 $\overline{\text{Nb}}, \overline{\text{Ti}}, \overline{\text{V}},$
 $\overline{\text{Nb}}, \overline{\text{Ti}}, \overline{\text{V}}$

(B)

CAPSULES:
Ti-4
Ti-5
Ti-6
Ti-7

(C)

CAPSULES:
Ti-V-1
Ti-V-2
Ti-V-3

(D)

CAPSULES:
Ti-1
Ti-2
Ti-3

FIGURE 1. Capsule Configurations, Orientation of Metallographic Sectioning Planes are Indicated as A---A.

cladding. Several serial sections (parallel to A---A Figure 1D) were examined metallographically. Two interface regions showing titanium-cladding interaction received additional examination using SEM equipped with energy dispersive X-ray fluorescence analysis capabilities (SEM-EDAX).

C. Capsules with Coated Fuel Pellets

HEDL prepared mixed-oxide fuel pellets ($\text{Pu}_{0.25}\text{U}_{0.75}\text{O}_{1.93}$) were sputter-coated with titanium and vanadium for use in P-23 Special Pins Test in EBR-II.⁽⁴⁾ Approximately ten microns of titanium or vanadium metal was deposited on circumferential surfaces of the fuel pellets. Capsules Ti-V-1, Ti-V-2 and Ti-V-3 contained three of the titanium coated fuel pellets and three of the vanadium coated fuel pellets banded between two split cladding halves. Titanium and vanadium coated fuel pellets were separated by a 316 stainless steel pellet. The cladding halves were made from oversized cladding to provide a narrow axial region of fuel-cladding contact for the length of the capsule. The pellet-cladding assemblies were double encapsulated in stainless steel capsules.

After annealing, several serial sections (parallel to plane A-A in Figure 1C) were cut from each impregnated pellet-cladding assembly so as to give a section through each pellet. After initial metallographic examination, each specimen was ground down an additional 0.5 mm and re-polished to provide several additional section planes for examination. This process was repeated a third time for each section from capsule Ti-V-3. Sections from capsule Ti-V-3 were also examined by electron microprobe analysis.

D. Capsules with Planar Contact Interface

Capsules Ti-4 through Ti-7 were prepared to provide data on titanium-cladding reactions at two times and temperatures, with other conditions identical, to obtain information on Ti-cladding reaction kinetics. In these capsules, 316 stainless steel pellets were placed on top of porous titanium pellets to form an interface. Contact pressure was determined by the weight of the stainless steel pellets (~ 0.5 gm) on a circular interface 0.190 inches (4.83 mm) in diameter. Depths of reaction

FIGURE 2. Oxygen Absorber Pellets Used in HEDL P-23 Special Pins Test.

observed at the stainless steel pellet - titanium pellet interface were measured as functions of time at the two temperatures in order to derive an approximate reaction depth versus time relation at the two temperatures.

III. RESULTS AND DISCUSSION

Examination of the initial six capsules (Nb, Ti, V, $\overline{\text{Nb}}$, $\overline{\text{Ti}}$, $\overline{\text{V}}$), containing pressure-contacted metal powder (or foil) interfaced with 316 stainless steel, indicated potential compatibility problems between titanium and vanadium metals and cladding at 800°C. No reaction between niobium and 316 stainless steel was observed. Powder metal-316 stainless steel interfaces are shown in Figure 3. Interface interaction between titanium foil and 316 stainless steel is shown in Figure 4. Where the titanium foil contacted the stainless steel, etching revealed effects on the stainless steel microstructure to a depth of 10 microns below the interface. Interfacial regions shown in Figures 3 and 4 have been electrolytically etched (5 volts, 5 sec.) in a 10% oxalic acid solution to reveal the stainless steel microstructure near the interface. Cathodic protection of the stainless steel by vanadium precluded electrolytic etching of the stainless steel and heavy etching of the vanadium occurred. It should be noted that reaction depths in these capsules are exaggerated by a factor of two due to the approximate 30° angle between the metallographic section planes (A-A Figure 1A) and the actual interface.

After 137 hours at 800°C, vanadium powder particles sintered to the stainless steel surface. Porosity in the stainless steel to a depth of approximately 13 microns indicated a Kirkendall effect,⁽⁵⁾ in which diffusion of a stainless steel component into the vanadium occurred at a faster rate than vanadium diffusion into the stainless steel. SEM-EDAX analysis of capsule V indicated extensive diffusion of nickel from the stainless steel into the vanadium powder particles near the interface. Iron diffusion into the neck regions between vanadium particles and the stainless steel was also observed. Beam traces for chromium diffusion behavior were inconclusive due to overlapping of chromium K-alpha and vanadium K-beta X-ray energies. Chromium K-beta emission showed poor signal-to-noise ratio and could not be used for chromium analysis.

Diffusion of vanadium into the stainless steel occurred to a depth of 13 microns corresponding to the 13 micron layer of porosity and nickel depletion. SEM-EDAX traces for elements Fe, Ni and V are shown in Figure 5.

Neck formation and the initial stages of sintering were observed between titanium powder particles after 137 hours at 800°C. Particles which interfaced with the stainless steel coalesced into a continuous layer averaging about 15 microns in thickness. Between the outer reaction layer and the stainless steel a band averaging 5 microns thick occurred which was heavily etched by oxalic acid. SEM-EDAX traces (0.5 μ /sec.) for elements Cr, Fe, Ni and Ti across the interface region are shown in Figure 6. The outer reaction layer contained titanium, nickel and iron and a small amount of chromium. The appearance of the interfacial region suggested the possibility of eutectic melting. However, the compositional inhomogeneity of the outer layer would suggest that a continuous liquid phase did not exist. A melting eutectic occurs in the Ni-Ti system at 943°C and 71 weight per cent titanium. Melting in the four-phase Fe-Ni-Cr-Ti system would be expected below 943°C, and 800°C is likely to be near the eutectic melting temperature.

Since the initial capsule tests indicated potential cladding compatibility problems with candidate titanium and vanadium oxygen absorber concepts, additional capsules were fabricated using porous titanium pellets and titanium and vanadium coated fuel pellets in contact with cladding in order to more accurately simulate conditions in the P-23 special pins irradiation test.

Examination of capsules Ti-1, Ti-2 and Ti-3 indicated reaction occurred between porous titanium absorber pellets and cladding at 800°C in an argon atmosphere. After 568 hours at 800°C reaction zones penetrating to a maximum depth of 30 microns into the cladding were observed. No reaction was observed at 700°C. Metallographic sections through 700°C and 800°C titanium - cladding interfaces are shown in Figure 7. Points of physical contact at porous titanium pellet - stainless steel interfaces were not observed during metallographic examination of 700°C capsules. Mechanical forces during mounting and sectioning most likely caused separation at

FIGURE 3. Reaction Interfaces Between 316 Stainless Steel and Pressure-Contacted Nb, V, and Ti Powders, 300°C, 137 Hours, 500X.

FIGURE 4. Titanium Foil - 316 Stainless Steel Interface after 137 Hours at 800°C. Etched with 10% Oxalic Acid, 1000X.

FIGURE 5. SEM-EDAX Traces for Fe, Ni and V Across 316 Stainless Steel - V Powder Interface. Extensive Ni Diffusion is Indicated by the Top Trace. (800°C, 137 Hours, 200 Sec. Traces.)

FIGURE 6. SEM-EDAX Traces for Cr, Fe, Ni and Ti Across 316 Stainless Steel - Ti Powder Interface. (800°C, 137 Hours, 200 Sec. Traces.)

FIGURE 7. Sections from Capsules Ti-2 and Ti-3. Formation of a Ti Coating on the Stainless Steel Surface Under Reducing Conditions at 800°C is Shown in the Lower Section. Etched with 10% Oxalic Acid.

interfaces which were not bonded by interfacial reaction. Localized interface contact during annealing was assumed as a result of capsule design and assembly conditions. Porous titanium pellets were centerless ground around their circumference and end ground. Capsule components were cleaned prior to assembly and care was taken to eliminate dust particles from the interface during assembly. All metallographic sections from porous titanium pellet-containing capsules annealed at 800°C showed regions of interface reaction. No regions of reaction were observed in identical capsules annealed at 700°C.

No reaction between cladding and vanadium or titanium coated mixed-oxide fuel was observed after 568 hours at 800°C. Metallographic sections from capsule Ti-V-3 are shown in Figure 8. The reduced reactivity of titanium and vanadium toward 316 stainless steel in the presence of mixed-oxide fuel may be explained by the difference in oxygen chemical potential in fuel-containing and non-fuel-containing capsules. In the non-fuel capsules, the oxygen inventory was limited to the oxygen contained in the oxide passive layers at metal surfaces. At 800°C, both vanadium and titanium would be reducing to the cladding (chromium oxide) passive layer. Once the cladding surface had been reduced at the cladding-vanadium or cladding-titanium interface, a defect occurrence in the vanadium or titanium oxide surface layer could allow metallic bonding to the cladding and reaction to occur. If oxygen equilibrium was approached in the non-fuel capsules, the oxygen potential would be determined by the most reducing surface in the capsule. Under these conditions the healing of any defect in oxide passive layers would be slow and reaction at points of contact between oxygen absorber pellets and cladding could occur. The equilibrium oxygen potential of titanium, vanadium and chromium oxide-metal couples,⁽⁶⁾ along with Woodley's data⁽⁷⁾ on 25%PuO₂ mixed-oxide oxygen potentials, are plotted as a function of temperature in Figure 9. Mixed-oxide fuel at an O/M of 1.98 and 800°C is oxidizing to titanium and should provide sufficient oxygen activity to maintain the integrity of an oxide passive layer at the cladding-absorber interface. Since fuel O/M and $\Delta\bar{G}_{O_2}$ at the fuel-cladding

FIGURE 8. Interface Between Cladding and Coated Fuel Pellets After 568 Hours at 800°C. (500X)

interface tend to increase with burn-up, the probability of reaction between oxygen absorbers and cladding would be expected to be reduced with burn-up.

Mixed-oxide fuel at an O/M of 1.98 and 800°C is slightly reducing to vanadium oxide. However, the $\Delta\bar{G}_{O_2}$ difference between vanadium and O/M = 1.98 fuel at 800°C is only about 4 kcal/mole O_2 , and the vanadium-cladding interface was probably not sufficiently reduced to allow metallic bonding across the interface in the capsules containing vanadium coated fuel. Vanadium-cladding reaction may be possible in fuel pins containing initially low O/M fuel and operated at the highest predicted LMFBR cladding temperatures. However, the effect of initially low O/M fuel on $\Delta\bar{G}_{O_2}$ at the fuel-cladding interface should tend to be compensated for by oxygen redistribution along the radial temperature gradient in an operating fuel pin.

Additional effects of capsule oxygen potential were observed in all non-fuel titanium containing capsules annealed at 800°C. In these capsules, a surface layer of titanium was observed coating the stainless steel surface in regions not contacted by titanium. The titanium layer is shown as a bright ribbon extending along the stainless steel surface in Figure 7. The presence of the titanium ribbons indicates either increased titanium vapor pressure or increased titanium surface diffusion under reducing conditions.

Capsules Ti-4 through Ti-7 indicated that under reducing conditions the titanium-cladding reaction rate in terms of cladding penetration decreases with time. Interfaces from capsules Ti-4 through Ti-7 are shown in Figures 10 and 11. An eight-fold increase in annealing time at 800°C caused a two-fold increase in reaction penetration depth. Using the relationship of depth of reaction proportional to the cube root of time, 639 days would be required for a reaction depth of 0.003 inches (75 μ) at 800°C based on reaction depths observed in capsules Ti-5 and Ti-7. This would provide adequate time for irradiation testing of the HEDL P-23 Special Pins containing porous titanium absorber pellets. Furthermore, cladding penetration due to reaction with titanium absorber pellets in a typical LMFBR application should be less than that observed in the 800°C

FIGURE 9. Equilibrium Oxygen Potentials of Metals and Mixed-Oxide Fuel. (References 6 and 7)

FIGURE 10. Sections from Capsule Ti-4 and Ti-6, 10% Oxalic Acid Etch.

FIGURE 11. Sections from Capsules Ti-5 and Ti-7, 10% Oxalic Acid Etch.

capsules in this investigation. Predicted peak cladding temperatures in LMFBR high performance fuel pins will be less than 800°C and will probably be in the 650° to 750°C temperature range. Oxidation of the titanium pellets and reaction with fission products at the titanium-cladding interface should further reduce titanium-cladding reaction. Absorber pellet reaction with oxygen and fission products is also expected to reduce cladding-vanadium reaction rates. While some reaction between cladding and titanium and vanadium is possible at the highest predicted LMFBR cladding inner surface temperatures, the extent of reaction, and the effects of fission products and irradiation damage cannot be predicted quantitatively. Experience from initial irradiated fuel pins containing these oxygen absorber materials should provide the basis for more detailed predictions.

IV. CONCLUSIONS

Based on data obtained from analysis of laboratory compatibility test capsules, niobium, titanium and vanadium candidate oxygen absorber concepts are expected to show adequate compatibility with 316 cladding for irradiation testing of these materials in fuel pins. The compatibility rating, in order of decreasing compatibility potential, would be niobium, vanadium, titanium. At 800°C the compatibility of 316 cladding with titanium and vanadium is considered marginal, with the presence or absence of reaction dependent upon a fuel pin oxygen chemical potential adequate for maintenance of an oxide layer at the oxygen absorber-cladding interface. For cladding inner surface temperatures less than 700°C, no compatibility problems between 316 cladding and vanadium or titanium coated fuel or porous titanium pellets is foreseen.

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