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COMPATIBILITY OF BOTH MIXED URANIUM -
PLUTONIUM OXIDES AND NITRIDES
WITH SODIUM AND 304 STAINLESS STEEL

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AND NITRIDES WITH SODIUM AND 304-STAINLESS STEEL

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May 30, 1969

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ABSTRACT

Out-of-reactor experiments to determine the compatibility of mixed uranium - plutonium nitrides (UN - 20 wt% PuN) and hypostoichiometric mixed oxides (UO_2 - 25 wt% PuO_2) both helium and sodium bonded to 304-stainless steel cladding have been performed. Capsules were heated for periods as long as 5000 hours at temperatures of 650°C and 1000°C. No reaction or corrosion layers were observed on adjacent fuel - clad surfaces in cases of both oxide and nitride fuels under the time - temperature conditions investigated.

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F. M. Smith

INTRODUCTION

In order to determine if there were gross incompatibility problems associated with irradiation testing of UO_2 - 25 wt% PuO_2 or UN - 20 wt% PuN either sodium or helium bonded to 304-stainless steel cladding, out-of-reactor tests using these materials were conducted and evaluated. Mixed oxide and nitride fuels were either sodium or helium bonded to welded 304-stainless steel capsules and, after adequate biological and oxidation protection, they were placed in a furnace to soak for periods up to 5000 hours at temperatures of 650 or 1000°C. The chemical compatibility of the tested components was analyzed through metallographic examination. Reference pellets and helium bonded capsules were used as controls in evaluating the compatibility of sodium bonded fuels.

The mixed oxide pellets were made of mechanically-blended PuO_2 and UO_2 powders, by pressing then sintering the green pellets at 1500 - 1550°C in an 8% hydrogen - 92% argon atmosphere. The mixed nitrides used were prepared by two different methods: metal-hydride reduction and carbo-thermic synthesis. A brief outline of the two types of preparations are shown in the reaction equations below. A more detailed description is given elsewhere by Weber et al.^(1,2)

a. Metal Derived Nitrides:

b. Carbothermic Reduction Nitrides:

The oxide pellets were substoichiometric with O/M values of 1.96 and 1.97 determined gravimetrically after heat treatment at 800°C for six hours in an argon - 8% hydrogen saturated with water vapor at 0°C.⁽³⁾ Densities ranged from 86 to 89% TD. The mixed nitride pellets prepared by the metal-hydride reduction method had an oxygen content of about 3000 ppm and carbon content of 300 to 3000 ppm. The oxygen concentration of the carbothermic nitrides ranged from 85 to 89% TD. Solid solutions of both oxide and nitride pellets were verified by X-ray diffraction and ceramographic techniques.

SUMMARY

Out-of-reactor experiments to determine the compatibility of mixed uranium - plutonium nitrides (UN - 20 wt% PuN) and hypostoichiometric mixed oxides (UO₂ - 25 wt% PuO₂) both helium and sodium bonded to 304-stainless steel cladding have been performed. Fuel pellets were double encapsulated in 304-stainless steel and in turn sealed in quartz envelopes under reduced helium pressure for protection from atmospheric oxidation. Heating occurred for periods as long as 5000 hours at temperatures of 650°C and 1000°C. After heating, the capsules were sectioned, mounted, ground, and polished. If the fuel was sodium bonded, metallography was done in two steps: first, with sodium in place in a dry nitrogen atmosphere using non-aqueous polishing lubricants and, second, after the sodium was removed by reaction with butyl alcohol. No reaction or corrosion layers have been observed on adjacent fuel - clad surfaces in the cases of both oxide and nitride fuels under the time - temperature conditions investigated.

EXPERIMENTAL

The capsule was designed after consideration of the necessary manipulations in the loading, bonding, and subsequent heating of plutonium-containing materials. Figure 1 shows a diagram of the steps involved from sodium bonding to the completed capsule for heating vertically in the furnace. The capsule unit, encasing a fuel pellet, was made from 304-stainless steel tubing with an 0.250 in. OD and 0.016 in. wall thickness. The connecting tubes between capsule units were 0.125 in. OD. The pellets were placed in the partially assembled capsule units and then all units with their respective pellets were welded together in series. The completed series was connected vertically into the sodium loading facility so that the double end caps were situated at the upper ends of their respective capsules. The string of capsules was evacuated and backfilled with sodium in a single operation at 200°C. The sodium-filled capsule string was allowed to soak at 425°C for three hours under a backpressure of helium, then cooled. The capsule units were separated from the series, weld sealed, and fitted with the plenum expansion chambers shown in Figure 1. All welding was done in a helium atmosphere. Each completed capsule was then welded into a second 304-stainless steel capsule which was 5/16 in. OD and 3-1/2 in. long. Before heating at the prolonged test temperature, the stainless steel double capsules were sealed in quartz envelopes under reduced helium pressure to protect them from air oxidation at the test temperatures. The helium pressure chosen was such that when the capsules were at the test temperature the total pressure inside the quartz envelope was approximately one atmosphere.

The capsules were placed in vertical tube furnaces and heated in a vertical position with the expansion plenums at the top so that the fuel pellets were submerged in sodium at all times. The furnace temperatures were controlled to $\pm 20^\circ\text{C}$. After heating, the capsules were examined by metallography to determine if a reaction had occurred at or near the fuel-cladding interface or if any noticeable change had occurred in the fuel material itself. Prior to the metallographic examination, it was necessary to first determine the location of the upper pellet end - sodium

FIGURE 1. Stages of Capsule Loading.

- Stage I - Capsule units welded in a string, sodium loading in progress
- Stage II - Capsule unit cut out of string, seal welded at bottom
- Stage III - Expansion plenum welded in place forming a completed capsule
- Stage IV - Completed capsule weld sealed in secondary 304-stainless steel capsule and then in quartz

interface in the capsule by radiography. Once determined, a tube cutter was used to score the capsule wall just above the interface to a depth so that only about one mil of the tube wall remained uncut. The capsule was then held in a vertical position with a fixed clamp and arranged so that the end containing the pellet was submerged in epoxy mounting resin just to the score lines. Once the resin had hardened completely, the capsule portion above the resin was "snapped off" showing the transverse face of the pellet. At this point, the specimen mount was placed in a large steel holder to facilitate manipulation in the glove box.

Hand polishing was done on kerosene-lubricated 2/0 and 3/0 emery paper. Use of coarser papers, 280-400-600 grit, was avoided since the coarse debris created during polishing lodged in the soft sodium. Even so, the sodium layer still entrapped a certain amount of pellet, stainless steel, and plastic particles. Some of these could be removed by ultrasonically cleaning the specimen in kerosene for 15 minutes. The specimen was then final polished on a vibratory polisher for 16 - 18 hours using kerosene as a lubricant and 0.3 micron alumina (Linde A) on a nylon cloth. After polishing, the sample was again cleaned ultrasonically and rinsed with clean kerosene. A cover glass was immediately placed over the polished surface while insuring that air bubbles were excluded between sample and cover glass. Sufficient adhesion existed between the sample mount, kerosene, and cover glass so that the entire assembly could be inverted for metallographic examination without danger of the cover glass falling off. This procedure serves to protect the sodium from corrosion due to normal atmospheric oxidation since the metallograph stage is outside the glove box. A maximum usable magnification of about 250X could be attained through the cover glass. Polarized light proved to be better than bright field since it gave more contrast among the different materials present. All filters were removed for maximum light and shorter exposure. Due to its softness, sodium polished faster than the surrounding fuel and cladding and, consequently, the polished surfaces were not coplanar. Two micrographs were required: one focused on the sodium and the other on the stainless steel and pellet (see Figure 5).

Because of the reaction between sodium and any aqueous etchant, the sodium had to be removed if micrographs of the etched microstructure of stainless steel or pellet were required. Sodium was removed by controlled reaction with butyl alcohol and the pellet lifted from the mount. This method would not completely remove the sodium from all the interstices of the pellet; however, enough was removed from near the surface so that any reaction between the sodium and fresh mounting resin was minimized. The pellet was then remounted in resin in another mount using normal mounting procedures. The void left after the pellet removal from the original mount was filled with resin. The separated components were then polished and etched by normal procedures since no sodium problems now existed. Figure 2 shows a flow diagram of the overall procedure. Figures 3, 4, and 5 are photomicrographs of specimens taken at various stages of the procedure and were taken with polarized light through a cover glass at 250X.

Figure 3 shows the interface between stainless steel cladding, sodium, and a UO_2 - 25 wt% PuO_2 fuel pellet after being rough polished on 3/0 emery paper. The cladding is at the top; note the debris trapped in the sodium layer. Figure 4 shows a similar interface as in Figure 3, but here the sample has been ground with 280-400-600 grit papers prior to being rough polished with the 3/0 paper. Note how this treatment results in considerably larger grit being entrapped in the sodium layer. Figure 5 shows a similar interface as those just described except that the fuel here is UN - 20 wt% PuN, and the surfaces have been vibratory polished overnight on a Syntron. The upper photomicrograph is focused on the sodium layer while the bottom one is focused on the cladding and fuel.

RESULTS AND DISCUSSION

MIXED OXIDES

A summary of the hypostoichiometric mixed oxide compatibility tests is given in Table I. Reference mixed oxide pellets, i.e., pellets from the same batch as those used in the compatibility tests, were examined metallographically along with the tested samples. These reference pellets

FIGURE 2. *Ceramographic Procedures for Examination of Sodium Bonded Compatibility Capsule with Sodium in Place.*

FIGURE 3. Interfaces between Stainless Steel Cladding, Sodium, and $(U_{0.75}Pu_{0.25})O_2$ Fuel after being Polished on 3/0 Paper. Stainless Steel at Top, Fuel at Bottom. Note Debris Trapped in Sodium Layer. Polarized Light. Taken through Cover Glass. 250X.

FIGURE 4. Interfaces between Stainless Steel Cladding, Sodium, and $(U_{0.75}Pu_{0.25})O_2$ Fuel after being Ground with 280-400-600 Grit Papers then Polished with 3/0 Paper. Note Extra Large Grit Particles Trapped in Sodium Layer. Polarized Light. Taken through Cover Glass. 250X.

FIGURE 5. Photomicrographs after Polishing on Syntron. Sodium in Focus on Top and Stainless Steel Cladding and $(U_{0.8}Pu_{0.2})N$ Fuel in Focus on Bottom. Polarized Light. Taken through Cover Glass. 100X.

showed microstructures generally free of visible cracks at 75X magnification. Helium bonded pellets of the same material heated at 650°C for 1000 hours and 1000°C for 1000 hours showed essentially the same microstructure as the reference pellets, only with slightly more cracking. No interaction could be seen between the fuel and adjacent cladding. Sodium bonded pellets showed a little more cracking near the edges, probably due to sodium penetration, but still no interaction between the fuel and cladding. Figures 6 and 7 show as-polished micrographs of the sodium bonded couples. The peculiar layers seen near the cladding and fuel surfaces are actually successive layers of mounting resin. No reaction layer was present in any specimen examined, and no evidence of incompatibility has been found.

TABLE I. *Mixed Oxide Compatibility Tests*

<u>Identification Number</u>	<u>Temperature °C</u>	<u>Time hr</u>	<u>O/M</u>	<u>Bond</u>
H-1-2	650	1000	1.97	He
H-1-6	1000	1000	1.96	He
1-2	650	100	1.97	Na
3-6	650	100	1.96	Na
3-2	650	1000	1.97	Na
1-6	650	1000	1.96	Na
2-2	1000	100	1.97	Na
4-6	1000	100	1.96	Na
4-2	1000	1000	1.97	Na
2-6	1000	1000	1.96	Na

The grain growth in the 304-stainless steel cladding is of interest. Those capsules heated to 650°C for 1000 hours show about 10 to 12 grains across the 16-mil capsule wall which corresponds to ASTM grain size 6. The capsules heated to 1000°C exhibited a grain growth such that only two to three grains spanned the same wall thickness, which corresponds to ASTM grain size 2 (see Figure 8). All of the oxide and nitride compatibility capsules examined have shown these same results with respect to cladding grain size.

FIGURE 6. *As-Polished Photomicrograph of Specimen 1-6. Diffusion Couple of $(U_{0.75}Pu_{0.25})O_{1.96}$ Sodium Bonded to 304-Stainless Steel and Heated to $650^{\circ}C$ for 1000 Hours. The Sodium Has Been Replaced by Epoxy Resin and Separates the Cladding on the Left from the Fuel on the Right. No Reaction Layers Are Present on Either the Fuel or the Cladding. Layers Seen Are Result of Mounting Technique. 750X.*

FIGURE 7. As-Polished Photomicrograph of Specimen 2-6. Diffusion Couple of $(U_{0.75}Pu_{0.25})O_{1.96}$ Sodium Bonded to 304-Stainless Steel and Heated to $1000^{\circ}C$ for 1000 Hours. Sodium Has Been Removed and Replaced by Epoxy Resin Which Separates Cladding Left and Fuel Right. No Reaction Layers Present on Either Fuel or Cladding. Layers Seen Are Result of Mounting Technique. 750X.

(a) 150X

(b) 75X

FIGURE 8. Typical 304-Stainless Steel Cladding Microstructure after being Heated: (a) 1000°C for 1000 Hours; (b) 650°C for 1000 Hours.

MIXED NITRIDES

Table II summarizes the out-of-reactor compatibility tests for mixed nitrides (UN - 20 wt% PuN) prepared by the metal-hydride-nitride reaction method. All were sodium bonded with pellets from three separate process batches involving different carbon contents, and were either centerless ground or unground on the cylindrical surfaces.

TABLE II. *Mixed Nitride Compatibility Tests*

Identification Number	Temperature °C	Time hr	% TD	Bond	Remarks
A-1	1000	1000	85.6	Na	3000 ppm C, ground
A-2	1000	1000	~85	Na	300 ppm C, unground
A-4	1000	1000	88.6	Na	320 ppm C, ground
A-5	1000	100	~85	Na	300 ppm C, ground
A-6	1000	100	88.6	Na	320 ppm C, unground
A-7	1000	100	85.6	Na	3000 ppm C, ground
A-9	650	1000	~85	Na	300 ppm C, ground
A-10	650	1000	88.6	Na	320 ppm C, unground
A-11	650	1000	~85	Na	300 ppm C, ground

The same results noted for the mixed oxide tests above apply here also, i.e., there was no evidence of reaction between the fuel and cladding. Figure 9 shows a typical as-polished radial section of a UN - 20 wt% PuN fuel/sodium/304-stainless steel interface. Both the fuel and cladding are completely free of any evidence of reaction or mass transport.

Some of the cladding showed small cracks along the grain boundaries emanating from the inner clad surface after electrolytically etching with 10% chromic acid; however, after re-polishing and electrolytically etching with 10% oxalic acid, these cracks do not reappear. Since the chromic etch attacks grain boundary carbides much more rapidly than does oxalic acid, it was assumed that the cracks originally seen were due to removal of carbides precipitated at grain boundaries.

FIGURE 9. *Radial Section of Compatibility Capsule, A-1, Showing Fuel and Cladding Edges in Contact with Sodium. Fuel Is $(U_{0.8}Pu_{0.2})N$, 85.6% TD, Containing 3000 ppm C. Capsule Was Heated 1000 Hours at 1000°C. 250X.*

Additional out-of-reactor compatibility experiments were performed using fuel pellets sintered from mixed nitrides prepared by carbothermic synthesis methods. Pellets from two batches (UPCN-2S, UPCN-4S) were used, and were synthesized through the following steps.⁽⁴⁾ Starting material for both batches was synthesized by carbothermic reduction of $UO_2 + PuO_2 +$ carbon mixtures under nitrogen at temperatures near 1600°C. Carbon content of the starting material for the UPCN-2 was higher than that of UPCN-4, and both exceed the carbon concentration required for stoichiometric reduction. After synthesis, the powders were analyzed for anion content with the following results (average of two determinations):

	<u>N</u>	<u>C</u>	<u>O</u>	<u>Lattice Parameter</u>
UPCN-2	4.48 wt%	0.81 wt%	0.12 wt%	4.899 Å
UPCN-4	5.22 wt%	0.17 wt%	0.10 wt%	4.893 Å

The nitrogen content of pure, stoichiometric $(U_{0.8}Pu_{0.2})N$ is 5.55 wt%. Total anion content for UPCN-4 is ~ 5.5 wt% and for UPCN-2 is ~ 5.4 wt%. However, the uncertainty in the analytical results for nitrogen and oxygen was $\pm 10\%$ of the amount present; for carbon the uncertainty was $\pm 20\%$. Thus, no firm statement as to the exact stoichiometry of the material can be made.

After ball-milling, pressing, and sintering to densities near 87% TD for UPCN-2S and 94% TD for UPCN-4S, the analytical results for anion content were as follows:

	<u>N</u>	<u>C</u>	<u>O</u>	<u>Lattice Parameter</u>
UPCN-2S	4.24 wt%	0.60 wt%	0.89 wt%	4.898 Å
UPCN-4S	5.27 wt%	0.11 wt%	0.30 wt%	4.891 Å

Oxygen content of both batches increased due to conditions under which the powders were handled during the operations prior to sintering. The microstructure of samples from the two sintering batches indicate a two-phase structure. A mixed nitride or carbonitride solid solution is the major phase with a minor oxide phase present as inclusions, mostly on grain boundaries. Qualitatively, the volume of oxide phase present appears to be proportional to total oxygen content. Insufficient data were obtained to allow identification of the precise composition and stoichiometry of the two phases. However, the relatively high lattice parameter of the nitride or carbonitride phase in UPCN-2S compared to UPCN-4S reflects the effect of increased carbon, and possibly oxygen, content in the solid solution phase. The compatibility tests using pellets from these two batches are summarized in Table III.

Here again, metallography showed the couples to be completely compatible. No reaction layers were evident nor was any attack or corrosion found on the cladding. In addition, there were no differences in compatibility between the two batches of nitrides having different impurity contents. Also, no noticeable differences were found between nitrides prepared by the metal-hydride-nitride or carbothermic synthesis routes.

TABLE III. Carbothermic UN - 20 Wt% PuN Compatibility Tests

<u>Capsule Number</u>	<u>Nitride Fuel Batch</u>	<u>Temperature °C</u>	<u>Time hr</u>
B-1	UPCN-2S	650	1000
B-2	UPCN-2S	1000	1000
B-3	UPCN-4S	1000	1000
B-4	UPCN-4S	650	5000

CONCLUSIONS

Out-of-reactor tests on both hypostoichiometric mixed oxides and mixed nitrides sodium bonded to 304-stainless steel cladding were completed. The test capsules were heated at either 650 or 1000°C for times ranging from 1000 to 5000 hours. Examination of these capsules showed no noticeable evidence of reaction or corrosion layers on adjacent fuel - clad surfaces. Sodium logging occurred in every case where the fuel was sodium bonded; however, very little, if any, fuel spalling was seen because of any interaction between the fuel and sodium.

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