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MEMORANDUM TO FILE

FROM: R. R. HERRIES

R. R. Herries/psa

Trip Report
BATTELLE MEMORIAL INSTITUTE
February 7-8, 1955

W. J. O'Leary and R. R. Herries visited BMI to review the progress of subcontract research on extended surface fuel elements and on consolidation of Horizons' thorium.

SUMMARY

1. Corrosion samples, 1" diameter X 0.180" thick nickel-plated uranium, which had been press-clad with aluminum at 510°C and 6000 psi, have been on test for 18 days with no evidence of undercutting. Similar samples press-clad with 10,000 psi have been exposed for 7 days with no failures.
2. The hydrogen and chlorine content of as-received Horizons' electrolytic thorium was greatly reduced during vacuum sintering and arc melting.
3. A second lot of Horizons' thorium has been compacted and sintered to provide electrodes for two 9-10 lb. melts.
4. Diffusion couples are being a ~~Classification Canceled/Changed~~ determine the rate of solid solution diffusion of thorium in aluminum.

DISCUSSION

Uranium

Mechanical Hot Die Pressing

Since corrosion tests of uranium-nickel-aluminum bonds, press-clad with pressures of 5000 to 8000 psi, have shown non-reproducibility in results, a series of samples has been

DPW 55-24-3

(SRTH-798)

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prepared to investigate the effects of higher bonding pressures. These "half-dollar" nickel-plated uranium samples, 1" diameter X 0.180" thick, were assembled in 1½" diameter picture frames and 30-mil aluminum covers. Ten samples, press-clad at 950°F (510°C) with pressures of 6000 psi, and a second group pressed at 10,000 psi, were "pinch-welded" on their circumferences, pinholed with a 15-mil drill, and placed on corrosion test. A similar set of 10 specimens, pressed with 15,000 psi, has not yet been completed.

The desirability of using larger specimens which more closely simulate SRL press-cladding conditions was discussed, since at a given "average" bonding pressure, the uniformity of pressure actually applied to increments of the uranium surface can be greatly affected by the size of the area under pressure. Since large variations in pressure over a large unit of a rough surface may result in minute discontinuities in the bond, which then act as corrosion paths, the corrosion resistance of clad elements is probably lowered. For this reason, larger samples would be more indicative of the behavior of SRL step-pressed fuel elements. It was decided that until a baseline was established with the smaller samples, no large ones would be pressed.

BMI plans for design of a new press-cladding die were discussed. With the die now in use, it is difficult to estimate the average pressure and variations in pressure applied to the flat sides of a plate assembly. This is due to certain design features incorporated to insure edge bonding of samples, with the situation further complicated because the die is of the bottoming type. These subjects of die design and pressing conditions will be discussed with SRL personnel visiting BMI on February 9, 1955.

Extrusion Cladding

A report covering the work on extrusion cladding of uranium plates with aluminum is being prepared.

Corrosion

A set of ten aluminum-clad uranium samples, nickel-coated at the Mound Laboratory by the decomposition of nickel carbonyl, have been under corrosion test at BMI. Two of the ten were coated with AlSi before press-cladding. As previously reported in DPW-55-242, the 3" X 5" specimens were found to have several areas of non-bond prior to their exposure in boiling distilled water. These were attributed to the fact that there was a 4-5 weeks delay

between nickel coating of the samples and their press-cladding. Within 14 days eight of the ten had failed by undercutting corrosion from the pinhole. The two samples with the additional coating of AlSi showed better corrosion resistance; one failed at the end plug after 14 days and the other began to undercut at 34 days.

The "half-dollar" samples, for the investigation of higher bonding pressures, as described in the discussion of mechanical hot die pressing, have shown good corrosion resistance to date. These are the first "half-dollar" size samples prepared with 30-mil aluminum; all the former ones had 100-mil aluminum picture frames. The 10 samples, bonded with 6000 psi to serve as a basis for evaluating the others, have been on test in boiling distilled water for 432 hours (18 days) with only "pimpling" at the pinholes. One specimen had separated at a defective aluminum-to-aluminum "pinch-weld" after 96 hours, and was removed from test after 432 hours. Seven days (168 hours) of exposure produced no change in the 10,000 psi group.

Thorium

Consolidation of Thorium

The consolidation and purification of Horizons' electrolytic thorium by consumable electrode arc melting is being investigated at BMI. Eight melts have been made under varying conditions from both green and sintered electrodes, which had been cold-pressed at 50 tsi. Four of the ingots were melted under an atmosphere of helium at a partial pressure of 20 inches Hg, and the other four were melted in a vacuum less than 100 microns. In all cases the ingots after a single melting operation were quite porous, with more or less adhering globular metal caused by spattering as melting occurred.

Analytical results are not complete on the as-sintered or on the melted material. However, results show that melting and/or sintering greatly reduced the H_2 and Cl_2 content of the metal. Chlorine dropped from 600 ppm, as-received, to less than 60 ppm in the ingot. Hydrogen was lowered from 74-85 ppm to less than 10 ppm in ingots melted from sintered electrodes. Green electrodes produced ingots with approximately 30 ppm of hydrogen.

HCl insolubles in the as-received metal were reported as 0.75% to 0.88 w/o, which increased to 3.0% on sintering and/or melting. The oxygen content, calculated on the basis of the values for the HCl insolubles, was 0.1 w/o

in the as-received thorium powder. Vacuum fusion determinations on the as-received metal gave an oxygen content of 0.3 w/o, which remained constant through the sintering and melting operations. From the analytical results it appears that only slight advantages are realized by the use of lower pressures, i.e., less than 100 microns. The HCl insoluble content of the metal, which was about 3.0 w/o after melting under a helium atmosphere at a pressure of 20 in. Hg, was 2.5 w/o when a pressure less than 100 microns was used. Correspondingly, the H₂ content was reduced from less than 10 ppm to less than 6 ppm.

No non-metallics were detectable by microscopic examination of the as-received metal; however, star-shaped inclusions in the ingots were found by X-ray diffraction to contain mostly oxides and carbides. It was reported that ingots cast at the Bureau of Mines had a concentration of the oxides at their surface; consequently, $\frac{1}{4}$ inch is scalped from the ingots. The ingots are then remelted to produce larger ingots of higher purity and improved soundness. The metal at BMI will be remelted and again analyzed to determine the extent of such improvements. Hardnesses of the first melts were in the range of 50-60 Vickers (approximately 60 Brinnell).

The second lot of Horizons' metal has been cold-pressed into 1" X $\frac{1}{2}$ " X $\frac{1}{8}$ " electrodes and sintered, as before, in vacuum at 1250°C for 15 minutes. These electrodes will be joined to make 2 larger melts (9-10 lbs. each) which will be analyzed and remelted.

Diffusion of Thorium into Aluminum

Diffusion couples are being annealed and examined in order to determine the extent of diffusion of thorium into aluminum that occurs beyond that limit that can be detected by metallography. An alloy of thorium in high-purity aluminum had been prepared, machined, and bonded to aluminum in preparation to annealing, when it was discovered that the alloy contained only 0.1% thorium rather than the desired 0.5%. Therefore, an available alloy of the correct thorium content in commercially pure aluminum was machined for use in couples.

Some trouble had been encountered previously with the couples sticking in the die during bonding. This has been eliminated by the use of a stainless steel sleeve around the cylinders. This sleeve is then machined off after the bonded couples are removed from the die.

One couple, annealed at 644°C for 38 hours, is being machined for chemical analyses. Others are being annealed at lower temperatures for correspondingly longer times. Evaluation will consist of chemical analyses of 2-mil cuts at the interface; the samples will increase to 5 and 10-mil cuts as the distance from the interface increases. Since the thorium in aluminum alloy is below the limit of solid solubility, this method will determine the amount of diffusion that occurs through solid solution.

RRH/jss

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