

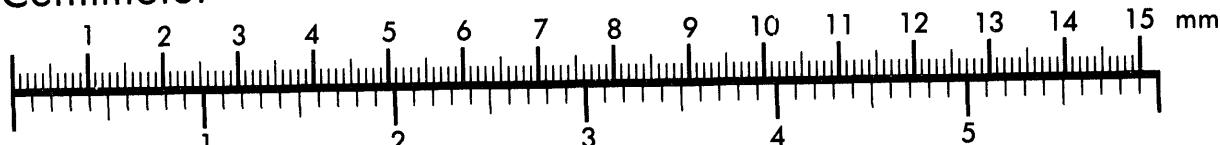


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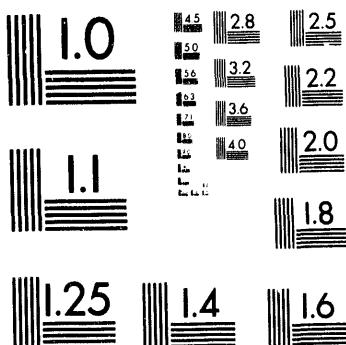
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~~MEMORANDUM TO FILE~~

FROM: R. R. HERRIES

March 3, 1955

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Trip Report  
BATTELLE MEMORIAL INSTITUTE  
February 21, 1955

W. J. O'Leary and R. R. Herries visited BMI to discuss the results of subcontract research on the extended surface fuel element program, and to review progress on the consolidation of Horizons' electrolytic thorium.

#### SUMMARY

1. Nickel-plated uranium samples, 1" diameter X 0.180" thick, press-clad with 30-mil thick aluminum at 6, 10, and 15 thousand psi, have been on corrosion test for periods of 10 to 31 days. Six specimens from the 6,000 psi group have failed by undercutting after 21 to 31 days; this is another good record.
2. "Pinch welds" have proved very effective as a means of sealing corrosion samples.
3. Two 9-10 pound virgin ingots and three smaller remelts were made by consumable electrode arc melting of Horizons' electrolytic thorium. Physical and chemical data on the remelts are being obtained.
4. Diffusion coefficients have been determined, at two sets of time-temperature conditions, for two diffusion couples of aluminum and a 0.5 w/o thorium in aluminum alloy, after high temperature annealing. The data show that the rate of interdiffusion of thorium and aluminum is very slow.

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DISCUSSION

Uranium

Extrusion Cladding

A report covering the extrusion cladding of uranium with aluminum at BMI is nearing completion. This work has shown that, with the techniques and equipment employed, nickel-plated uranium rods of small circular, or rectangular (0.375" X 0.180"), cross-section can be clad with 30-mil aluminum. A plate of MTR size, approximately 3" X 0.180", can be clad with 5/32" aluminum sheathing.

It was agreed that one or two BMI personnel will attend a discussion of the extrusion cladding of uranium fuel elements with Du Pont and General Cable Corp. at a date in the near future.

Corrosion

Since results of corrosion tests on uranium-nickel-aluminum bonds, mechanically pressed at pressures from 5 to 8 thousand psi, have not been reproducible, a series of "high pressure" samples was prepared to evaluate the effects of higher bonding pressures on the corrosion resistance of press-clad assemblies. The higher pressures are expected to produce more continuous bonding between the relatively rough metal interfaces. Thirty uranium pieces, 1" diameter X 0.180" thick and electroplated with 0.5 mil of nickel, were press-clad with 30-mil aluminum, were pinch-welded on their periphery, and were pinholed with a 15-mil drill. Of these specimens, ten were press-clad at the standard conditions of 510°C and 6000 psi to serve as a basis for evaluating the groups pressed with 10 and 15 thousand psi.

Although one sample from each of the 6 and 10 thousand psi groups failed at the aluminum-to-alumirum pinch-weld during testing, this method of sealing has given far better results than the method of conventional welding previously used. Three samples of the 6000 psi group remain on test in boiling distilled water after 31 days; 3 having failed after 21-25 days, 2 after 25-28 days, and 1 after 28-31 days. In all cases, a pimple formed at the pinhole and slowly grew in diameter until it reached the edge of the uranium specimen, from which edge the corrosion spread rapidly over the opposite side of the core. This behavior indicates that at 6000 psi the edge bonding is poorer than bonding on the flat surface.

With the exception of the one pinch-weld failure, the 10,000 psi group has shown only pimples at the pinhole after 21 days on test. Similar results were reported for the 15,000 psi samples after 10 days of exposure.

Thorium

Consolidation of Thorium

Five ingots were cast from consumable electrode arc melted Horizons' thorium at BMI. Two of these were 9-10 pound virgin melts, and three were remelts made from eight smaller ingots previously cast and analyzed as reported in DPW-55-24-3. The original eight melts had been made with varying conditions of atmosphere and electrode preparation.

The two 9-10 pound melts were made from the second lot of Horizons' thorium from Nuclear Metals. After screening, the minus 250 mesh particles, comprising 10% of the as-received metal were removed, and the metal was sampled for chemical analysis. The fines were found to have approximately twice the  $H_2$  and  $O_2$  content that the coarse material had. The metal was cold-pressed into electrodes, 1" X  $\frac{1}{2}$ " X 6", under a pressure of 50 tsi. These electrodes were then vacuum sintered at  $1250^{\circ}C$  for 15 to 30 minutes. It was found that the ultimate reduced pressure attained during sintering was a greater factor in  $H_2$  removal than was time at temperature. Electrodes held for 15 minutes at a minimum pressure of 150 microns contained less than 4 ppm  $H_2$ , while electrodes held for 30 minutes at a minimum pressure of 350 microns contained 12 ppm  $H_2$ .

During melting, however, since relatively little advantage had been realized by the use of lower pressures (less than 100 microns) in the eight earlier melts, the two large virgin ingots were melted in a  $H_2$  atmosphere at a partial pressure of 20 in Hg. The ingots obtained were very porous, being similar in appearance to the previous eight 3-pound melts. Since the Bureau of Mines at Albany had reported a concentration of oxides at the surface of arc-melted ingots, these two ingots were scalped 1/8" on their diameters in preparation for remelting. Analyses from the second lot of material and from the resulting melts are not yet reported.

Pieces of the first eight ingots were welded together to form electrodes for three remelt heats. These were melted under conditions of electrode preparation and of atmosphere corresponding to those of the original melts, in order that the degree of purification could be compared. The remelt of the ingot produced from a green electrode was checkered with a network of fine cracks upon solidification, whereas the other two remelts were sound. The cracks in the first remelted ingot were attributed to the higher  $H_2$  content of the ingots made from unsintered electrode. Although analyses are not complete for the remelts, the following table shows the purification obtained during initial melting of these eight small ingots.

ANALYSIS OF ARC MELTED HORIZONS THORIUM

| <u>Impurity</u>                                 | <u>As-received</u> | <u>Sintered</u> | <u>Melted (sintered electrode)</u> |                               | <u>Melted (green electrode)</u> |                               |
|---|--------------------|-----------------|------------------------------------|-------------------------------|---------------------------------|-------------------------------|
|   |                    |                 | <u>&lt;100 microns</u>             | <u>He atmos.<br/>(20" Hg)</u> | <u>&lt;100 microns</u>          | <u>He atmos.<br/>(20" Hg)</u> |
| H <sub>2</sub>                                  | 74-85              | 4-12            | <6                                 | <10                           | 30                              | 50                            |
| Cl <sub>2</sub>                                 | 600                | 600             | <60                                | <60                           | <60                             | <60                           |
| C   | 300-400            | 300-400         | 200                                | 300-400                       | 300                             | 200                           |
| HCl insolubles                                  | 0.75-0.88 w/o      | 3.0 w/o         | 2.5 w/o                            | 3.0 w/o                       | 3.5-4.7 w/o                     | 3.27 w/o                      |
| Na  | 250-300            | 250-300         | <57*                               | <57*                          | 57                              | <57*                          |
| O <sub>2</sub> (vacuum fusion)                  | 3000               | 3000            | 3000                               | 3000                          | 3000                            | 3000                          |
| O <sub>2</sub> (calculated from HCl insolubles) | 1050-1100          |                 |                                    |                               |                                 |                               |
| N <sub>2</sub>                                  | 200-300            | 200-300         | 200-400                            | 200-400                       | 200-400                         | 200-400                       |

NOTE: All values are in ppm except HCl insolubles.

\* - lowest limit detectable.

DPM-55-24-4

The results show that a major benefit was achieved in sintering the electrodes prior to melting. The H<sub>2</sub> content of the as-received metal (74-85 ppm) was greatly reduced in sintering and was decreased even farther on arc-melting. Consequently, all melts made from sintered electrodes contained less than 10 ppm H<sub>2</sub>, whereas melts made from green electrodes contained 30-50 ppm H<sub>2</sub>.

The Cl<sub>2</sub> content of the metal was reduced tenfold on melting, and the Na content was also substantially lowered. Very little improvement was found in the C values. HCl insolubles in the as-received metal increased from 0.75-0.88 w/o to 3.0 w/o, probably because the water was driven off in sintering, fixing the hydroxides. The oxygen content calculated on the basis of the values for the HCl insolubles was 1050 ppm in the as-received thorium powder. Vacuum fusion determinations gave an oxygen content of 3000 ppm, which remained constant through the sintering and melting operations. From these results it is indicated that only slight advantages are realized with the use of a lower pressure, i.e., less than 100 microns; the HCl insoluble content of the vacuum-melted metal was 2.5 w/o as compared to 3.0 w/o after melting in the helium atmosphere. Correspondingly, the H<sub>2</sub> content was less than 6 ppm in vacuum-melted metal as compared to less than 10 ppm for the metal melted in He. In view of these data and the fact that the arc is less stable at lower pressures, future melts will be made in a He atmosphere.

The hardness of virgin melts was 50-60 Vickers, but values for remelts were 70-80 Vickers. This increase in hardness is attributed to the probable increase in oxygen content from the excessive welding necessary to piece electrodes together for remelting. Since the two 9-10 pound melts were scalped, and will require less welding, this large increase in hardness is not anticipated for the second melting of the larger ingots.

It is emphasized that all the data have been obtained from small scale melts; consequently, they may not be valid for larger production methods. Similar work should be carried out with ingots several inches in diameter and weighing from 100 to 200 pounds, before final conclusions are drawn.

Diffusion of Thorium into Aluminum

In order to determine the extent of diffusion of thorium into aluminum that occurs through the solid solution phase, a series of diffusion couples was prepared and annealed. Cylinders of an alloy containing 0.5% thorium in aluminum, which is below the limit of solid solubility, were bonded to aluminum cylinders and annealed at four different combinations of time and temperature. One couple annealed at 644°C, and one annealed at 577°C have been sectioned and chemically analyzed to provide data for a determination of the diffusion coefficients. The results from the 644°C couple were in good agreement with results from previous work on thorium diffusion, showing that the rate of diffusion is slow.

Two couples, annealed at lower temperatures for correspondingly longer times, have yet to be analyzed. Duplicate couples are available for each time-temperature condition, and will be used as a check if necessary. The complete results will show whether any diffusion takes place beyond that limit which can be detected by metallography.

RRH/jss

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