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# METALLURGY DIVISION

Quarterly Report

Period Ending March 1, 1948

W. A. Johnson

April 5, 1948

Date Received: 4/5/48 Date Issued: 4/6/48

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# METALLURGY DIVISION

## Quarterly Report

## Period Ending March 1, 1948

Although the Metallurgy Division still lacks adequate experimental facilities, this situation is expected to be remedied in the very near future. The design work on the new laboratory and office building for the Division, temporarily halted in November as a result of termination of the Kellex Corporation contract, was resumed late in December by a new architect-engineer, Patchen & Zimmerman. Progress has been quite satisfactory, and all drawings should be completed in April. Construction work was resumed in January by J. A. Jones Construction Company and is proceeding concurrently with design. The building is expected to be ready for occupancy in June or July. Substantially all major items of equipment for the laboratories have been acquired or are on order.

The acquisition of these experimental facilities together with a modest increase in personnel will permit vigorous prosecution of the research program proposed for the Metallurgy Division (issued March 23, 1948). This program outlines attacks on the following problems:

1. Anisotropy in properties of uranium
2. Aqueous corrosion of beryllium



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3. Effect of radiation on metallurgical reactions
4. Diffusion in solid materials
5. Mechanical strength and atomic properties of metals
6. Ductility of beryllium
7. Mechanical working by extrusion

Such experimental work as has been possible with the limited facilities available is described in the following paragraphs:

## I. Corrosion

### A. Preparation and Properties of Oxide Films

Initial attempts to prepare oxide films on beryllium by corroding in hydrogen peroxide solutions met with some difficulty, resulting from the presence of copper in the distilled water supply sufficient to cause the formation of copper oxide deposit on the surface of the specimen and very rapid decomposition of the peroxide. By the use of redistilled water, this difficulty was essentially eliminated. No copper oxide was deposited on the samples and the decomposition of hydrogen peroxide, though erratic, was reduced to a reasonable rate.

Four beryllium specimens have now been corroding for 54 days in oxygen-saturated, redistilled water containing $3.3 \times 10^{-3}$M $H_2O_2$ at 90°C. The only visible change in the samples after 50 days exposure was a slight decrease in lustre. Since that time some local attack, accompanied by the formation of a white corrosion product, has occurred in areas coincident with surface imperfections on the samples.

It is planned to measure the electrical resistance and the effective capacity of the oxide films on these samples by means of



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special apparatus now being assembled. Attempts will be made to determine their structure using X-ray and electron diffraction techniques.

With the acquisition of the new laboratory facilities, the number of corrosion tests will be expanded to permit the investigation of a larger number of corrosion variables and these over wider ranges

B. Reduction of the Thickness of Aluminum Coating on Slugs

In connection with the experiments on the resistance heating of Hanford slugs, it is desired to reduce the thickness of the aluminum coating in a controlled, uniform manner from the original value of around .030" to approximately .010". Since bow in the slugs made machining extremely difficult, electrochemical etching was attempted.

For a survey of various etching solutions, test specimens in the form of 2" x 4" panels of .032" 1/2H 2S sheet coated with Ucilon to expose 1, 2.25, or 4.5 sq. in. of surface were employed as anodes in an electrolytic cell.

1. $H_2SO_4$ - $H_3PO_4$ Electrolytic Polishing Bath

A small range of composition and other conditions of operation were tried without indication of success. Efforts to obtain a more rapid rate of solution of the metal by increasing the amount of water or by adding HCl appeared promising, but anodic removal of the aluminum was irregular and a completely satisfactory procedure would probably require considerable development work.

2. $Na_2CO_3$ - $Na_3PO_4$ Bath

These solutions attacked the aluminum anodes but only under conditions which required high voltages (10-18 volts) and which produced an anodized coating. Addition of NaOH to the bath

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increased the rate of attack, but metal removal was not uniform and some specimens acquired a hydrated iron oxide deposit.

3. NaOH Bath

In tests with 10 per cent NaOH solution the aluminum anodes acquired rather heavy deposits of hydrated iron oxide at applied voltages greater than 3 volts. Upon raising the bath temperature to 75°C, decreasing the applied voltage to 1.5 - 2.5 (giving a current density of 1 - 5 amp./sq.in.) and stirring the bath, the aluminum was found to acquire a very fine, loose, black coating of iron rather than the hydrated iron oxide and to be etched in a uniform manner.

Further development revealed the following conditions to give quite satisfactory metal removal:

| | | |
|---|---|---|
| Solution | - | 10% NaOH |
| Cathode | - | Copper |
| Temperature | - | 70°C |
| Current Density | - | 0-5 amps/sq.in. of anode |
| Stirring | - | Desirable but not necessary |
| Rate of Metal Removal | - | .0005"/min. |

The uniformity was good: Variations as little as $\pm$.0002" were measured over an area 1.5" x 3" after removal of .025" of aluminum. There was no difference in uniformity whether current was employed or not, but greater currents gave higher rates of metal removal. Intermittent dips produced the same uniformity of attack as a single dip which permits the accurate control of coating thickness. Aluminum-silicon brazing alloy showed



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a behavior similar to that of 1/2H 2S aluminum.

The first attempt to use this method to reduce the thickness of the aluminum on Hanford slugs failed because of severe pitting. It was apparent that an exceedingly resistant film protected the aluminum since neither the hot alkali nor 5 per cent hydrofluoric acid would remove it. Evidence for the belief that the film is responsible for inactivity rather than the condition of the metal is found in the visible opaque glaze on the slugs as received and the satisfactory behavior of aluminum cans before brazing two slugs. A canned slug was lightly polished with fine emery paper to remove the film and than treated by the procedure recommended above for dissolving aluminum. General removal of aluminum resulted but pitting continued. Further work to remove this film will be required before the problem can be considered to have been solved.

## II. Warping and Blistering of Hanford Slugs

### A. Experimental Studies

In order to determine whether the observed dimensional instability of Hanford slugs is caused by thermal stresses rather than neutron bombardment, a special apparatus has been designed to simulate the temperature gradients and temperature cycles of slugs in the Hanford piles. This is to be accomplished by the simultaneous high current electric resistance heating and high velocity water cooling of slugs singly or in groups. The following specific phases of this investigation have been undertaken:

1. The design of the apparatus has been largely completed; procurement of assembly parts is actively under way.

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2. A temporary laboratory, within the structure of the new metallurgical laboratory building, has been designed and constructed to house this equipment. This will permit its actual use at least three months prior to time the entire laboratory becomes available.

3. The problem of brazing single canned slugs into long straight bars has been greatly simplified by the use of zinc foil which melts at a considerably lower temperature than the Al-Si alloy bond of the can, and zinc plating the aluminum ends to eliminate the necessity for fluxing. Further developments are needed in connection with jigs for alignment and the use of a low oxygen content brazing atmosphere, such as purified hydrogen, requisite for producing sound brazed joints.

4. Equipment to rapidly measure, magnify and record bowing and surface irregularities of slugs has been designed and procurement of component parts initiated.

5. Creep testing equipment has been purchased to determine the propensity of uranium to slow flow, under small loads, at elevated temperatures in the range of those characteristic of pile operation. The heating furnace of this equipment has been completely redesigned to permit the testing of uranium.

B. Mathematical Analysis

A theoretical analysis of the problem of stress distribution in electrically heated round bars is nearly complete. Equations for the temperature distribution under the assumption that the electrical resistance and thermal conductivity are linear functions of temperature have been derived with appropriate expressions for the average electrical



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resistance, the power, and the voltage for any temperature difference between center and surface. These calculations were based on heating by direct current, and extension to the case of 60 cycle alternating current, differing slightly because of the skin effect, is underway.

Additional studies on the calculation of the thermal stresses and strains under the above conditions of heating for elastic media have been completed; this work will have to be extended to plastic materials since the calculated stresses far exceed the predicted creep strength of uranium.

In connection with this general problem theoretical considerations have been undertaken concerning the stresses set up by gradual temperature changes in polycrystalline aggregates of orthorhombic crystals. This work again has been limited to the elastic case and will have to be extended to plastic materials since the calculations show a maximum microscopic stress in the case of uranium of 1000 lbs./sq.in. per 4°C change in temperature.

## III. General Considerations

In addition to the two major projects described above, a large variety of minor problems are under consideration. These include constant and detailed review of the progress of design and construction of the new laboratory, work on radiation effects (described in the Quarterly Report of the Physics Division, CNL-35), completion of the design and installation of vacuum melting equipment, preparation of alloys of uranium with the transition metals, forging and drawing of uranium bars of small scale cross section preliminary to the production of such shapes from enriched uranium, and planning for more extensive experimental work upon completion

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of the laboratory.

## IV. Personnel

The staff of the Division numbers eleven technical and three non-technical.

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