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METALLURGY DIVISION

Quarterly Report

Period ending May 31, 1948

W. A. Johnson

June 1, 1948

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The new Metallurgy Laboratory building is scheduled for completion late in July; in the meantime, experimental work continues in a number of temporary laboratories scattered over the plant.

I. Corrosion

A. Beryllium Corrosion

Three beryllium samples have now been exposed to 5×10^{-3} M H_2O_2 in oxygen-saturated re-distilled water at 90°C for 143 days, with essentially no visible attack. Presumably this metal is protected quite well from attack by pure water plus peroxide.

Electron-diffraction pictures of the film formed on two beryllium samples in the above solution were taken after 14 and 58 days exposure. The same material was indicated, of greater thickness where the exposure was longer, but it could not be identified, since L. T. Newman does not have on file the pattern found. Presumably it is a substance which, because it has not been discovered before, has not been previously investigated by electron diffraction. It seems likely that the substance is some form of hydrated beryllium oxide.

After only two days exposure to oxygen-saturated distilled water

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(obtained from J. English) at 90°C, beryllium samples showed considerable amounts of white spotty deposit, and pits had been formed. After additions this water contained between 1.0 and 1.5 ppm chloride ion and 0.3×10^{-3} M H_2O_2. The marked increase in corrosion rate due to relatively small amounts of chloride ion is thus shown (copper was low in this solution, ~0.07 ppm). A similar effect due to copper, with possible acceleration due to chloride, has previously been reported.

A sample was submitted for electron-diffraction identification of the film after 42 hours exposure to the above conditions. Newman was unable to work with it, presumably because the coat was too bulky, and has sent it to the K-25 instrument people to attempt to get the diffraction pattern.

Apparatus to measure electrical resistance and effective capacity of surface films has been under consideration for some time. It is planned to have such equipment built within the next month.

B. Development of a Bath and Procedure for Electropolishing Uranium-Aluminum Alloys

This work was undertaken at the request of D. S. Billington in order to provide a metallographic polishing technique which could be applied to irradiated alloy samples. Electropolishing involves the controlled, anodic dissolution of surface metal, a technique well-adapted to remote control operation.

A variety of bath types were explored over a limited range of operating conditions. Samples of rolled, 5% uranium alloy sheet were used.

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(1) Caustic Baths

Over a concentration range from 2.5% to 50% and operating from 0-20 volts at room temperature, solutions of KOH produced an etched surface.

(2) Fluoboric Acid Bath

The Alcoa procedure for pure aluminum and solid solution aluminum alloys was tried without success. There appeared to be an excessive attack on the U Al_5 and eutectic.

(3) Phosphoric - Sulfuric - Chromic Acids

Samples anodically treated in baths of this type showed preferential attack on the eutectic and inclusions. No complete survey of this type bath was made.

(4) $KOH - K_2CO_3 - Na_3PO_4$ Baths

Several baths made up with varying amounts of these compounds were tested under varying conditions. A satisfactory bath and procedure were developed.

Recommended bath and procedure:

KOH	1.0%
K_2CO_3	2.5%
$Na_3PO_4 \cdot 12H_2O$	2.5%
Water	Balance
Temperature	80 - 100°C
Time	10 - 30 minutes
Voltage	5 - 15
Current Density	approx. 0.2 amps/cm^2
Stirring	mild, mechanical

This treatment works satisfactorily for specimens polished through 3/0 paper, but not too well when the rolled alloy sheet is processed without prior abrading. The polish obtained compares favorably with usual mechanical polishes. The main objection is that the eutectic and inclusions, although well defined, appear in slight relief. Since it would be desirable to bring out the aluminum structure and boundaries more clearly, some relatively pure aluminum sheet will be studied. It is hoped that work with this material will suggest modifications in the bath or in the procedure.

C. Plating of Uranium Bars for Protection from Oxidation During Heating and Working

Several samples of uranium metal were plated for A.G.H.Anderson. The procedure is described by Gray in CT-2116. The production of a non-adherent electrodeposited, metal envelope about the uranium was readily accomplished by using anodic alkaline cleaning followed by anodic treatment in concentrated H_2SO_4 at 30-40°C. with 15 volts applied between the uranium and the graphite cathode. A "flash" current is produced and drops off to nearly zero in twenty seconds. Following rinsing , the sample is immediately plated in essentially standard baths. Samples with silver, nickel, and composite nickel-silver plates were prepared. Silver plates, of this non-adherent type, gave adequate protection from oxidation but were too malleable under forging conditions since they pulled free from the uranium and tended to split. Nickel did not show satisfactory protection when the plated uranium was heated in air.

Materials have been received which permit the preparation of plated uranium samples having an adherent deposit of electro deposited metal. This procedure, which is also fully described in CT-2116, is based on a pretreatment etch using trichloro-acetic acid. The function of the etch is to remove worked metal and produce a film free surface. Samples are currently being plated by this procedure.

II. Blistering and Warping of Hanford Slugs

A temporary laboratory within the structure of the permanent Metallurgy Laboratory building was built and equipped, induction heating equipment, furnaces, creep testing machines and simple metallographic equipment installed and put into service.

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The design of the apparatus to reproduce Hanford type radial thermal gradients has been completed and construction of this equipment begun; it is scheduled to go into operation in August.

Much difficulty was experienced in devising a method for joining slugs end to end by brazing. Zinc was chosen as the brazing material, but satisfactory brazes in air using Alcoa flux could not be obtained at temperatures under 600°C. Since this temperature is above the melting point of the Al-Si bond in Hanford slugs, this procedure was abandoned. Efforts to pre-tin the aluminum with zinc were also unsuccessful because of the high brazing temperature required. A successful procedure was finally evolved in which the aluminum surface was coated uniformly with zinc by placing a coin of rolled zinc on a slug heated to 500°C and abrading the surface while the zinc was in the molten condition. The slugs could then be brazed at a temperature of 450°C by abrading the ends of the slugs against one another. Joints prepared in this way show excellent bonding and the hazard of flux inclusions is obviated since flux is not used. Equipment is now under construction which will permit eight slugs to be brazed end to end into a straight smooth rod.

Equipment to rapidly and automatically record slug profiles has been constructed using a lathe as a holding and indexing fixture; a microtorque potentiometer mounted on a dial gage as a measuring device; and a Brown recorder to record the potentiometer signal and translate this into a magnified profile. This equipment has already been used to determine the profile reproducibility of a special tool designed to machine skin cuts following slug contours.

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Two creep testing units have been installed and modified by the addition of an oil bath for testing uranium in the range of 100 to 350°C. Test runs have been started with uranium specimens machined from Hanford extruded bar material. In addition, tensile tests in the same temperature range are being made.

Mathematical analysis of the temperature gradients in slugs heated by different methods is being continued. Calculations have been made using data from the literature on the variation of thermal conductivity and electrical resistivity with temperature for the heat generation at Hanford and for heating by the passage of electric current. It has been shown that the skin effect at 60 cycles is negligible and the calculations already carried out for direct current are directly applicable. Assuming a constant surface temperature of 100°C and a center temperature of 500°C, the maximum discrepancy between the temperature distribution produced by electric heating and that arising in pile operation does not exceed 2°C. For a center temperature of 300°C the maximum deviation does not exceed 1°C. On the other hand, if the temperature distribution is approximated by a simple parabola, this deviates from the actual case by as much as 22°C when the surface and center temperatures are respectively 100 and 500°C. Thermal stresses were calculated for the temperature distribution under Hanford heating and for a simple parabola, and were found to differ by at most 5 percent. The small differences were not significant in view of the present uncertainty in the expected amount of stress relief by plastic flow. An effort will be made to interpret these "elastic" stresses in terms of the creep to be expected because of the high stresses. No effort was made to take into account the probable decrease in thermal conductivity at Hanford as a result of radiation.

III. Radiation Effects

The work of the Metallurgy Division on radiation effects is described in the report of the Physics Division, Physics of Solids.

IV. Other Projects

Preliminary work has been started on diffusion in solid metals, particularly the gold-silver system, and on the plastic behavior, as revealed by tensile loading, of a number of very pure coarse grained metals.

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