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ELECTRODEPOSITION OF ALUMINUM AND ZIRCONIUM ON URANIUM

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CHEMISTRY DIVISION

ELECTRODEPOSITION OF ALUMINUM AND ZIRCONIUM ON URANIUM

M. H. Lietzke

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ELECTRODEPOSITION OF ALUMINUM AND ZIRCONIUM ON URANIUM

M. H. Lietzke

Abstract

A method has been found for the electrodeposition of adherent deposits of both aluminum and zirconium on uranium. The plating baths consist of aluminum bromide or zirconium tetrachloride dissolved in fused ethyl pyridinium bromide. The current efficiencies are extremely good for aluminum deposition but low for the zirconium bath.

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Contribution from the Oak Ridge National Laboratory
Chemistry Division

ELECTRODEPOSITION OF ALUMINUM AND ZIRCONIUM ON URANIUM(1)

M. H. Lietzke

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(1) This document is based on work performed for the Atomic Energy Commission at the Oak Ridge National Laboratory.

Introduction

Electroplating has been considered as a possibility for protecting uranium surfaces in reactor units(2). The protective coating is necessary to prevent

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(2) R. Wehrmann, Electroplating, CT-443, June 1945.

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oxidation of the uranium and to entrap the fission products formed during operation of the reactor. A satisfactory electrodeposited coating must be adherent and must be capable of being built up to a satisfactory thickness. It should have a low cross section for neutron capture and show good resistance to corrosion. Both aluminum and zirconium satisfy the latter two requirements; hence it seemed of interest to determine whether adherent, reasonably thick electrodeposits of these metals could be made on uranium.

Previous attempts to electroplate these metals on uranium were unsuccessful(2). Although satisfactory deposits of aluminum could be obtained on a variety of cathode materials, no deposits could be obtained on uranium. Recently the use of a fused

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organic bath containing ethyl pyridinium chloride or bromide was reported for aluminum deposition.(3) Attempts to deposit aluminum on uranium from the bath as described

(3) F. H. Hurley, U.S. 2, 446,331, August 3, 1948.

were unsuccessful. It was also found impossible to deposit aluminum at room temperature from a bath described by Wier and Hurley.(4) This bath consisted of a solution of a

(4) T. P. Wier, Jr. and F. H. Hurley, U. S. 2,446,349.

N-substituted alkyl pyridinium chloride or bromide and $AlCl_3$ in the presence of an excess of benzene or one of its derivatives. It was found possible, however, to develope a similar type of fused bath which gave very adherent deposits of either aluminum or zirconium on uranium cathodes.

This report describes the preliminary experiments which show that adherent plates of both aluminum and zirconium can be electrodeposited on uranium and gives the conditions under which these electroplates are obtained.

Experimental Data and Results

The experiments were performed on a small scale and were merely intended to demonstrate that the electroplating could be accomplished. The plating baths were contained in 10 ml. beakers which were supported in a larger beaker filled with glycerine. The glycerine bath was heated on a hot plate. In this way the temperature was held to within a few degrees of the desired value. The electrodes were supported from above and passed through a cork which fit the beaker containing the plating bath. A motor generator was used as the source of direct current.

The strips of uranium metal used as cathodes were cleaned just prior to use by

the preferred alternate pretreatment cycle described in CT-1793[5]. After degreasing

(5) A. G. Gray and R. G. Wehrmann, Quarterly Report on Electroplating pertaining to coatings for Tuballoy, Metallurgical Project CT-1793, June 1944.

the strips were pickled in 50% nitric acid for 3-4 minutes at room temperature. The pickle treatment was followed by a cold running water rinse. The strips were then etched completely black with concentrated hydrochloric acid and again rinsed in cold running water. Finally the strips were again dipped in 50% nitric acid until the uranium surface appeared bright, rinsed thoroughly in cold running water, blotted dry with Kleenex, and immersed immediately in the plating bath. Since several different strips of uranium metal were used in the plating experiments the actual area plated varied from 1.1 to 1.5 cm^2 in the case of the zirconium plating. In the case of the aluminum experiments the area plated was 3.2 cm^2.

(1) The Aluminum Plating Bath. The aluminum bath contained about 70 mole percent anhydrous aluminum bromide dissolved in 30 mole percent ethyl pyridinium bromide. The solution was electrolyzed at 130-135°C with a cathode current density of about 0.4 amp/dm^2. In calculating the current density both sides of the cathode were considered since both sides appeared plated equally well. Since only one anode was used the bath had extremely good throwing power. The surface of the aluminum anode became pitted during operation of the bath. However, the anode current efficiency was not determined.

Excellent aluminum plates were obtained on the uranium cathodes both in short electrolyses (5-15 minutes) and in longer runs ($1_1/_2$ hours). No attempt was made to determine how thick a deposit could be obtained. The plates were bright and silvery and extremely adherent to the basis metal. Analysis of a plate obtained in a 1/2

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hour run indicated a cathode current efficiency of 100%. The deposit was 2.5×10^{-4} cm. thick. In a $1_1/_2$ hour run the cathode current efficiency was about 80%. The deposit was 5×10^{-4} cm thick. The current efficiency calculations were based merely on the length of run, the current, and the weight of deposit. The potential difference across the cell was 3-4 volts.

(2) The Zirconium Plating Bath. The zirconium bath consisted of about 37 mole percent anhydrous zirconium tetrachloride dissolved in 63 mole percent ethyl pyridinium bromide. It was necessary to use the relatively low concentration of zirconium tetrachloride since zirconium tetrachloride was not as soluble in ethyl pyridinium bromide as was aluminum bromide. The bath was operated at a temperature of 160-170°C with a platinum anode and the uranium cathode. A series of plates was made at various cathode current densities (C.C.D.). All runs were of one hour duration. The cathode current efficiencies (C.C.E.) and the thickness of deposit in each case are shown in Table 1.

Table 1

Electrodeposition of Zirconium as a Function of Cathode Current Density

C. C. D	C. C. E.	Thickness of Deposit
0.9 amp/dm^2	0.7 %	ca. 1.1×10^{-5} cm
1.6 amp/dm^2	0.6%	1.2×10^{-5} cm
3.5 amp/dm^2	0.3%	1.2×10^{-5} cm
5.2 amp/dm^2	0.2%	1.0×10^{-5} cm

As the cathode current density increased the cathode current efficiency decreased. The deposits obtained at the lower current densities were silvery and metallic, while those obtained at 5.2 amp/dm^2 were dark grey. The throwing power of this bath also appeared to be very good since both sides of the cathode appeared plated equally well

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even though only one anode was used. No attempt was made to determine how thick a deposit could be obtained at a given current density. The cathode current efficiencies were very much lower than for the aluminum bath.

Conclusions and Discussion

Because of the relative simplicity of operation electroplating offers a convenient method for coating uranium. Conditions under which it is possible to obtain adherent deposits of either aluminum or zirconium on uranium have been established. Further experiments will have to be performed to determine how thick a deposit can be obtained in each case and the resistance of the deposits to corrosion. It is possible that some other similar ionizing organic solvent might be found which would allow the use of a higher current density and thus permit more rapid coating of the uranium. The current efficiency for the deposition of zirconium might also be higher in some other solvent. Since the use of $AlBr_3$ in place of $AlCl_3$ gave satisfactory deposits of aluminum it is possible that the use of $ZrBr_4$ instead of $ZrCl_4$ might give better deposits of zirconium. In view of the very low current efficiency for the deposition of zirconium from ethyl-pyridinium bromide it is possible that thick deposits could not be obtained even upon long electrolysis.