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AEC RESEARCH AND DEVELOPMENT REPORT

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Metals, Ceramics,
and Materials

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ALUMINUM ION PLATING OF URANIUM-MOLYBDENUM
ALLOY FAST-BURST REACTOR ELEMENTS

R. T. Bell

UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the ATOMIC ENERGY COMMISSION under U. S. GOVERNMENT Contract W-7405 eng 26

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Nuclear Division

Y-12 PLANT

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With the US Atomic Energy Commission

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ABSTRACT

Ion plating of thin (< 0.2 mil) aluminum coatings on fast-burst reactor parts fabricated from uranium-10 percent molybdenum alloy was evaluated as a method of protecting the alloy from oxidation and as a means of containing the fission products.

A number of core rings were plated with nickel while other rings were ion plated with aluminum so that a comparison between the two coatings could be made. During the ion plating of aluminum and subsequent heat treatment, certain uranium-aluminum intermetallic compounds (UAl_2 , UAl_3 , and UAl_4) were formed. During the process of testing to destruction, these rings encountered fissions in excess of 10^{17} and temperatures in excess of 700°C during which the nickel plating spalled severely while the aluminum and the uranium-aluminum intermetallics remained adherent to the alloy.

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SUMMARY

Thin (< 0.2 mil) aluminum coatings were ion plated on uranium-10 percent molybdenum fast-burst reactor rings and the plating was evaluated as a method of protecting the uranium alloy from oxidation. The effectiveness of the coating with regard to the release of fission products to the atmosphere was also evaluated. The reactor was assembled from a group of core rings, some of which were ion plated with aluminum and some were nickel plated. During its test to destruction, the reactor encountered pulse yields in excess of 10^{17} fissions, and temperatures above 700°C .

After testing, the reactor was disassembled and the plating protection evaluated. Results of the plating and testing were as follows:

1. Ion cleaning (sputtering) of the uranium alloy surface prior to plating removed the surface oxides and permitted the plating of the aluminum on a relatively clean, hot surface.
2. X-ray diffraction inspection of the plated rings before testing showed that uranium-aluminum intermetallics were formed during plating and subsequent heat treatment.
3. Nickel-plated rings, removed from the reactor after the test, had spalled severely. Aluminum-plated rings were still coated with the mixed aluminum-uranium intermetallics.

INTRODUCTION

Since the announcement of the ion plating process in 1964 by Sandia, ⁽¹⁾ Y-12 has been engaged in developing plating procedures and methods using this technique. A significant quantity of literature is now available concerning ion plating in general and a smaller quantity concerning the protection of uranium from corrosion; ⁽²⁾ however, there was no information concerning ionplating uranium-10percent molybdenum until this investigation began in November 1966. Since that time, components for a fast-burst reactor have been ion plated with aluminum by the Y-12 plant. In this investigation, a performance comparison was to be made between components conventionally plated with nickel and components ion plated with aluminum. During the cleaning of the part surfaces by argon ion bombardment, the temperature of the parts was to be elevated to the point where uranium-aluminum intermetallics (such as UAl_2 , UAl_3 , and UAl_4) would be formed during plating. It was the protection provided by these intermetallics that was to be evaluated as the reactor was tested to destruction.

ION PLATING FAST-BURST REACTOR ELEMENTS

PROCESS DESCRIPTION

The term "ion plating" designates a plating process in which the atoms of the material to be deposited are ionized, accelerated through a DC potential, and allowed to strike the substrate with a kinetic energy considerably greater than the kinetic energy of the vacuum-evaporation and sputtering techniques. The metal to be deposited is thermally evaporated from a filament located in the positive column of a DC inert-gas discharge. A portion of these evaporated atoms is ionized and accelerated to the cathode which is also the substrate. Ions of the inert gas are continuously bombarding the substrate, both before and after evaporation begins. This discharge sputtering of the substrate cleans it to the point that deposition begins. Beyond this point, this same discharge continues to sputter from the substrate a part of the material that is being plated. To achieve a plated thickness, the amount of metal removed by sputtering, after deposition begins, must be less than the amount of material deposited. Figure 1 shows a typical glass bell-jar installation suitable for ion plating a metal onto test coupons or small parts.

Figure 1. ION PLATING BELL JAR.

The high-voltage DC supply is of the high-reactance type which is able to control high current surges resulting from sparking in the system. The positive pole of the supply is at ground potential while the negative pole is fed through the base of the bell jar to the substrate holder and cathode of the circuit. The cathode lead from the base of the bell jar to the substrate holder is a 0.125-inch-diameter aluminum rod, enclosed in ceramic tubing, and shielded by a copper tube at ground potential. At the substrate holder end of the copper tube, an aluminum ground shield is attached at a distance of about 0.750 inch from the substrate holder. The lip on the bottom of the substrate holder provides support for up to three coupons, and there is no mechanical attachment of the coupons to the holder. The vacuum system is capable of obtaining a vacuum pressure of less than 5×10^{-6} torr.

Further detailed descriptions of the ion plating process are covered in several reports by Sandia.^(1, 3, 4)

A typical sequence for ion plating a metal onto a uranium coupon is:

1. Preclean the coupon with a nitric acid dip followed by an electropolish in a phosphoric acid solution.
2. Position the coupon in the vacuum system, using care not to handle the coupon with the bare hands, thereby contaminating the uranium surface. Care must be taken to remove all lint from the sample surface or the sample may end up with charred areas on its surface. The system is pumped to less than 5×10^{-6} torr.
3. Establish a high-voltage DC potential between the cathode (substrate) and the anode (filament).
4. Admit argon to the system through a micrometer valve until the desired current density and potential are established.
5. After a predetermined cleaning time, heat the filaments and begin the deposition. The filaments may be either tungsten or tantalum wire, or boats containing the metal to be evaporated.

PLATING URANIUM-MOLYBDENUM COUPONS

The uranium-10 percent molybdenum alloy is superior in its resistance to corrosion, compared to unalloyed uranium, but plating protection is desired to prevent the release of fission products into the atmosphere as well as to reduce oxidation. Previous fast-burst reactors have been electroplated with nickel and, in some instances, difficulty has been experienced with the nickel spalling after repeated thermal shocks. The decision was made to assemble this reactor with some aluminum ion-plated parts and some nickel-plated parts, test the reactor to destruction, and then

evaluate the effectiveness of aluminum versus nickel plating. The initial development work was the plating of one by two-inch coupons of uranium and uranium-10 percent molybdenum alloy with thin coats (0.2 mil) of aluminum. It was expected that, in addition to providing protection by the presence of the aluminum, uranium-aluminum intermetallic compounds would be formed either during plating or during subsequent heat treatments.

The first uranium-10 percent molybdenum alloy coupons to be plated and examined were acid etched, electropolished, and ion cleaned for a period of five minutes at five kilovolts and a current density of 0.3 ma/cm^2 prior to the aluminum deposition. X-ray diffraction was used to examine the resulting coating. A chart showing the major peaks of interest is presented in Figure 2. As expected, aluminum and uranium

Figure 2. X-RAY DIFFRACTION TRACE OF AN AS-PLATED URANIUM-MOLYBDENUM COUPON. (Original Preparation Procedure)

are shown as well as strong uranium dioxide and uranium trialuminide peaks, the aluminum being partly converted to the trialuminide as a result of depositing on a heated substrate. The presence of uranium dioxide indicates that the ion cleaning time and/or the current density were insufficient to remove all of the oxide from the coupon surface. Coupons such as these were then heat treated in a 10^{-6} torr vacuum for 45 to 60 minutes, after which X-ray diffraction showed essentially the same pattern.

Additional alloy coupons were precleaned in the same way, ion cleaned at the same current density and voltage but with a 20-minute cleaning cycle, cooled in a vacuum for 30 minutes, ion cleaned for 20 minutes more, and plated on the hot substrate. Figure 3 shows the X-ray diffraction trace produced from such a coupon. In this trace, uranium dioxide is noticeably lacking while uranium tetraaluminide is present. The coupon used to produce the trace shown in Figure 3 was heated in a vacuum for one hour at 600° C and examined a second time. The X-ray diffraction trace for this coupon is presented in Figure 4. All of the available aluminum was converted to the trialuminide and the surface of the coupon was quite dark, not at all like the usual ion-plated aluminum coatings.

A number of these plated coupons were cross sectioned and photographed. Figure 5 shows a cross section of one of the unalloyed uranium coupons that was aluminum ion plated and totally converted to the uranium-aluminum intermetallics. All of the conversion occurred during plating, a time of about two minutes. After ion plating, nickel was plated onto the coupon to protect the surface during preparation for metallographic examination. The scratch across the surface from upper right to lower left gives an indication of the relative hardness of the two metals and the two intermetallic compounds. Knoop hardness measurements on similar coupons indicated that the dialuminide is about twice as hard as the uranium alloy and twenty times as hard as aluminum. Kiessling⁽⁵⁾ reports the hardness of the three aluminides (UAl_2 , UAl_3 , and UAl_4) as 630, 300, 260 Hvp/50 gms/15 sec, respectively. Figure 6 presents a typical X-ray diffraction trace from one of these coupons.

A cross section of a thicker plate of aluminum (about one mil), plated on another alloy coupon, is given in Figure 7. As before, the conversion was accomplished while the plating was being done. In this particular instance, all of the aluminum was not converted. There appears to be a porous area, or at least a poor-contact area, between the aluminum and the trialuminide phase which, according to Wilkerson,⁽⁶⁾ may arise from the aluminum atoms diffusing through the trialuminide much more rapidly with temperature than the rate of diffusion of uranium atoms through the dialuminide. From these considerations, unconverted aluminum could be expected to flake from a substrate under some conditions.

Figure 3. X-RAY DIFFRACTION TRACE OF AN AS-PLATED URANIUM-MOLYBDENUM COUPON. (Modified Preparation Procedure)

PLATING REACTOR PARTS

The reactor parts that were plated consisted of the following: (1) the safety block, a cylinder that is three inches in outside diameter and nine inches high; (2) rings, nine inches in diameter with individual thicknesses from 0.380 to 1.340 inch; and (3) mass-adjustment rods and bolts, one inch in diameter and about ten inches long.

Figure 4. X-RAY DIFFRACTION TRACE OF A CONVERTED URANIUM-MOLYBDENUM COUPON. (Modified Preparation Procedure with Additional Heat Treatment)

Plating the Safety Block

Figure 8 gives an overall view of an ion plating station and the safety block after having been plated. A closer view of this plating may be seen in Figure 9 which shows the part supported on three metal feet that are insulated from ground by alumina insulators. The high-voltage direct current is fed to one of these feet through the high-voltage feedthrough in the left of the picture. During plating, the insulators are protected from being coated by a short section of glass tubing. In plating this particular part, two AC-filament supplies were used, one for the inside-diameter filaments and one for the outside-diameter filaments. The filaments consisted of three tungsten wires wrapped together with a 30-mil aluminum core. In addition,

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Figure 5. CROSS SECTION OF AN ALUMINUM ION PLATE ON A URANIUM-MOLYBDENUM COUPON SHOWING INTERMETALLIC COMPOUNDS. (Bright Field Illumination; 1,000X; Cathodic Vacuum Etch)

Figure 6. X-RAY DIFFRACTION TRACE OF TOTALLY CONVERTED ALUMINUM ON A URANIUM ALLOY COUPON.

D373

Figure 7. CROSS SECTION OF A THICK ALUMINUM ION PLATE ON A URANIUM-MOLYBDENUM ALLOY COUPON SHOWING UNCONVERTED ALUMINUM. (Bright Field Illumination; 750X)

short lengths of aluminum wire were wound around these filaments and the aluminum was premelted on the filaments, in vacuum, prior to installing the workpiece.

Plating the Reactor Rings

One of the large rings in the ion-plating equipment may be seen in Figure 10. Each ring was first cleaned in a one-to-one nitric acid solution, electropolished in phosphoric acid, then cradled in two aluminum support blocks that were insulated from ground. The insulators were shielded by Pyrex glass tubes. The high voltage was fed by a bare tungsten wire from the high-voltage feedthrough to one of the aluminum blocks and the part was surrounded by four sets of tungsten filaments, each filament having an aluminum core. Ion cleaning was continued for 20 minutes at 5 kv and 200 ma, followed by a pumpdown time in a 10^{-6} torr vacuum of about 30 minutes, an additional 20-minute clean, and a four-minute aluminum deposition time. This procedure left two small unplated areas on the rim of the part that had to be coated in a second run. After the first plating, the part was allowed to cool for four hours in a vacuum of 10^{-6} torr before the vacuum system was opened to the atmosphere and the part rotated to new support points. The system was again pumped to less than 2×10^{-6} torr and the ion cleaning and deposition cycles repeated. In an effort to minimize the unplated areas created during the first plating, the other rings were supported by three vertical rods insulated from the base plate and extending into three holes in the part. This modification proved to be superior. A part ready for plating can be seen in Figure 11; the plated parts are shown in Figures 12 and 13.

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Figure 8. ION PLATING STATION WITH A PLATED PART IN POSITION.

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Figure 9. ALUMINUM ION-PLATED SAFETY BLOCK.

120842

Figure 10. REACTOR RING TO BE PLATED.

123007

Figure 11. REACTOR RING IN POSITION TO BE PLATED.

123008

Figure 12. ALUMINUM-PLATED RING IN THE EQUIPMENT.

124506

Figure 13. ALUMINUM ION-PLATED REACTOR RING.

Since the surface area of a ring was on the order of 650 cm^2 , using a current of 200 ma provides a current density of about 0.3 ma/cm^2 of part surface, enough to effect the conversion of a portion of the aluminum as it was being plated. X-ray diffraction inspection of some of the rings verified the presence of the trialuminide on the as-plated rings. During cleaning and plating, a particular value of current density was maintained at approximately five kilovolts rather than maintaining a certain argon pressure. In Figure 14, a representative plot of system pressure versus high voltage for various current levels is presented. During sputter cleaning, a recorder was connected across a ten-ohm resistor in the high-voltage circuit, as indicated in Figure 1, to record the cleaning current as the plating progressed. It has been noted that the color of the glow discharge and the current density change during cleaning and that stabilization of these parameters may indicate that the surface of the workpieces has been freed of contaminants. Bland⁽⁴⁾ has investigated this phenomenon and reports that the time necessary for cleaning a substrate is directly related to the cathode current density, extent of the initial substrate contamination, and the contamination level in the vacuum system, assuming a given pressure and high voltage.

In plating these reactor parts, not only was there an interest in cleaning the surface but in raising the temperature of the part to a degree sufficient for the formation of the uranium-aluminum intermetallics during plating. Subsequent to plating, each

Figure 14. GLOW DISCHARGE VOLTAGE AS A FUNCTION OF THE ARGON PRESSURE.

part was removed to a high-vacuum furnace and given a heat treatment at 600° C for 30 minutes at a pressure of 10^{-5} torr to insure conversion. When cooled and removed from this furnace, aluminum could be flaked from some of the parts that had plating thicknesses in excess of about two mils. The appearance of the parts, after flaking, was dark and discolored like the surfaces of the totally converted coupons. X-ray diffraction showed these parts to have the tri and tetraaluminide reaction products.

Plating Bolts and Mass-Adjustment Rods

Bolts and mass-adjustment rods were supported vertically in the center area of the ion plating chamber, mounted so that > 90 percent of their area was plated during the first run. They were then supported from different points and plated a second time. Usually, three of the bolts were plated at one time.

TESTING THE REACTOR PARTS

The reactor was assembled with both aluminum ion-plated rings and nickel-plated rings for testing to destruction. Pulse yields greater than 3×10^{17} fissions were achieved during testing and temperatures in excess of 700° C were recorded. A comparison of nickel plating and aluminum ion plating is shown in Figure 15, a view looking upward into the center of the stack of rings. The bottom three rings were aluminum ion plated, the fourth ring was nickel plated, the next three rings were ion plated, and the final 0.380-inch-thick ring was nickel plated. In areas where the aluminum is totally converted, the rings are a dark brown or gray color on the surface; while, in small areas on some rings, there appears to be some free aluminum that had not been converted. In Figure 16, an aluminum-plated ring is shown as it was upon removal from the reactor. The uranium alloy was still protected by the uranium-aluminum intermetallics, as verified by X-ray diffraction. By comparison, note in Figure 17 a nickel-plated ring after removal from the reactor showing exposed base metal in some areas and nickel that can be peeled from most of the remainder of the part. Upon completion of the testing program, some design modifications were made in some of the rings; the remaining nickel was stripped from the plates, and all were replated with aluminum and heat treated.

Table 1 shows an estimate of time and manpower required to plate a typical reactor part initially. If 100 percent part coverage is desired, Steps 1 and 6 through 12 would have to be repeated. Subsequent to the plating of this reactor in the Y-12 Plant, another reactor has been plated by the Dow Chemical Company and a report⁽⁷⁾ covering the plating is available.

ORNL-74625

Figure 15. STACK OF CORE RINGS AFTER TESTING.

ORNL-74651

Figure 16. ION-PLATED RING REMOVED FROM THE REACTOR.

ORNL-74646

Figure 17. NICKEL-PLATED CORE RING REMOVED FROM THE REACTOR.

Table 1
TIME AND MANPOWER ESTIMATE FOR PLATING A RING

Step	Operation	Time Required	Manhours
1	Installation of Tungsten Filaments, HV, Fixtures, etc	3 hrs	3
2	Pumpdown, Filament Premelt	10 min	1/6
3	Acid Etch, Water Rinse	20 min	2/3 ⁽¹⁾
4	Electropolish	20 min	2/3
5	Acetone Rinse, Dry	20 min	2/3
6	Install Part in Vacuum System and Pump to $< 5 \times 10^{-6}$ Torr	20 min	1/12
7	Pump in 10^{-6} Vacuum	20 min	-
8	Ion Clean (5 kv - 0.3 ma/cm ²)	20 min	1/3
9	Pump in 10^{-6} Vacuum	20 min	-
10	Ion Clean (5 kv - 0.3 ma/cm ²)	20 min	1/3
11	Ion Plate	5 min	1/12
12	Cool in 10^{-6} Vacuum	4 hrs ⁽²⁾	-
	Total	9 hrs 55 min	6

(1) Nuclear safety considerations requires two men during Steps 3 through 5.

(2) Part will still be too hot to be handled without protection.

The ion plating process, using aluminum ions of some five-kilovolt potential, produced an adherent coating on a uranium-10 percent molybdenum alloy surface. Uranium-aluminum intermetallics were formed. If an adequate vacuum system were available, the necessary fixtures for plating would be minimized, consisting of little more than a DC supply, an AC supply, and a jig to support the part and hold the filaments. The cost of ion plating would then be comparable to conventional plating techniques. Further development of the process and an evaluation of the rings in service may indicate the desirability of a plating metal other than aluminum or the use of multimetal coatings.

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