208
SEP 20 1972

3151
SC-M-72 0458

PROCEDURES FOR ION- PLATING
SPR II FUEL

J. S. Philbin
Reactor Source Applications Division

F. Gonzalez
Reactor Studies Division

THIS DOCUMENT CONFIRMED AS
UNCLASSIFIED
DIVISION OF CLASSIFICATION
BY JH/Kahm/amb
DATE 10/18/72

Printed: September 1972

SANDIA LABORATORIES

OPERATED FOR THE UNITED STATES ATOMIC ENERGY COMMISSION BY SANDIA CORPORATION | ALBUQUERQUE NEW MEXICO | LIVERMORE CALIFORNIA

17086
DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

*Issued by Sandia Laboratories,
a prime contractor to the United States Atomic
Energy Commission*

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, sub-contractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

SC-M-72 0458

PROCEDURES FOR ION-PLATING SPR II FUEL

Jeffrey S. Philbin
Reactor Source Applications Division, 5221

Francisco Gonzalez
Reactor Studies Division, 5222
Sandia Laboratories
Albuquerque, New Mexico 87115

September 1972

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

ABSTRACT

This report describes the method used for ion-plating fuel for the SPR II reactor. The method provides a means of coating uranium and its alloys with an adherent thin protective coating without the presence of the thin oxide film characteristic of conventional vacuum-depositing techniques. The report also contains procedures for operating the vacuum and high voltage equipment used in the ion-plating process.

Key words: aluminum plating, sputter cleaning, oxide removal, vacuum deposition

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

ACKNOWLEDGMENTS

The authors gratefully acknowledge the assistance of J. A. Snyder and V. E. James, who contributed their experience and technical advice on many techniques used in the ion-plating process.

TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	5
EQUIPMENT REQUIRED	6
BAKING THE FUEL PLATES	8
PREPARATION OF FILAMENTS	9
Equipment Required	9
Procedure	9
OPERATION OF THE VACUUM SYSTEM	11
The Rotary Pumps	11
The Diffusion Pump	11
Breaking the Vacuum	14
System Shutdown	15
PREPARATION OF THE VACUUM PLATING MACHINE	15
CLEANING THE FUEL	21
ION-PLATING THE FUEL	26
Placing the Sample Under High Vacuum	26
Controlling the Argon Environment	29
Sputter Cleaning the Sample	29
Ion-Plating the Sample	32
SUMMARY	33
REFERENCES	34

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Ion-plating Equipment	7
2	Preparation of Basic Filaments	10
3	Vacuum System Valves	12
4	Filament Support Stands	16
5	Fuel Plate Support Stand and High Voltage Feedthrough. .	17
6	Arrangement of Filaments and Support Stands for Ion-plating Fuel Plates	19
7	Fuel Plate in Acid Bath	23
8	Rubbing the Surface to Accelerate Chemical Reaction . .	23
9	Drying the Fuel Plate	24
10	Using Nitrogen Gas to Dry Small Holes in the Fuel . . .	24
11	Fuel Plate in Electropolishing Tank	25
12	Fuel Plate Properly Positioned in the Vacuum Chamber . .	27
13	Schematic of the Ion-plating Apparatus	28
14	Blue Glow Discharge at Cathode	31

PROCEDURES FOR ION-PLATING SPR II FUEL

INTRODUCTION

The procedures for ion-plating fuel for the SPR II reactor are described in this report. The SPR II reactor is a fast-burst reactor whose fuel is 93% enriched uranium alloyed with 10 weight percent of molybdenum. The fuel components consist of three control rods, a burst rod, and six cylindrical fuel plates. The rods are 10-5/8" x 7/8" dia. and the plates are each approximately 8" in diameter and 1-1/2" thick with various bolt holes, a glory hole, and alignment bosses. Because of the corrosive effect of air on uranium and its alloys, some type of protective coating is generally adopted as part of any design requirement for this element. The development of the "ion-plating" process¹ at Sandia Laboratories, Albuquerque, provided a possible means of coating uranium with an adherent thin protective coating without the presence of a thin oxide film which normally forms immediately after cleaning and before the coating can be applied with conventional vacuum-depositing techniques. In the ion-plating process the sample surface is sputter-cleaned by positive ion bombardment and maintained in a contamination-free state prior to and during the film deposition. In addition, the high energy flux to the surface promotes chemical reaction and diffusion at the interface. The optimum ion-plating processing conditions for maximum corrosion protection were determined by R. D. Bland,² in a study of the process parameters which influence the coating properties. The processing conditions recommended by R. D. Bland have been used in this report.

EQUIPMENT REQUIRED

<u>Quantity</u>	<u>Item</u>
1	18" x 12" x 3" KIMEX glass soaking dish
1	medium size beaker
1	medium size (or large size) glass or teflon funnel
1	box Alconox detergent
1	box pipe cleaners
2	bottles of nitric acid (gallon size)
1	electropolishing tank
	phosphoric acid for electropolishing
	Electro-Glo solution for electropolishing
1	holding tool for plates
4	small bottles of sodium hydroxide (lye)
	assorted tools
	assorted mounting hardware for rods and/or plates
1	tank argon gas
1	tank nitrogen gas
1	large dewar of liquid nitrogen with feed tube
1	gettering furnace
1	pyrometer for thermocouple readout
	sufficient tungsten filaments with tantalum wetter strips and aluminum clips
1	box of lint-free cloth
1	TV monitor for security
1	portable vacuum cleaner for contaminated waste
1	hole brush
1	Moseley Autograf (Model 680) strip chart recorder
1	CVC vacuum machine
1	high voltage power supply
1	high current power supply for filaments

A view of the vacuum machine and some associated equipment used in the ion-plating process is shown in Fig. 1.

Figure 1. Ion-plating equipment

BAKING THE FUEL PLATES

The fuel plates but not the rods must be baked in an evacuated oven to remove cutting oils and other impurities which might have contaminated the surface during the casting and machining processes. The ovens must be scheduled about a week in advance by contacting Division 7121-2. R. H. Altherr has been coordinating this operation. It is also necessary to pick up the fuel and handling equipment early in the morning from the storage vault in Building 868. These arrangements can be made through A. W. Porter, Division 4611, who must accompany all visitors to the storage vault. The handling equipment is in a plastic bag on a shelf outside the vault. This equipment includes:

- 1 pair of asbestos gloves
- 1 pair of rubber gloves
- 2 clay boats
- 1 sign, "Radioactive Experiment in Progress"

Note: Loosen the bolts on the bird cage storage container before leaving the vault or bring a speed wrench and 1/2" socket along for this purpose as tools are not available at Building 834.

It is necessary to register in the log book at the vault upon entering.

The fuel plate must be delivered to Building 834 early in the morning so the plate can be baked, cooled, and returned to the vault before 5:00 p.m. The two clay boats should be placed in the oven first. The fuel plate is placed on top of the clay boats and the thermocouple can then be positioned on the top of the fuel plate. The technician at the facility will then close the oven and begin evacuating the chamber. The fuel plate will be baked at 200°C for a period of about 1-1/2 to 2 hours. The bird cage and equipment bag should be left at Building 834 until the fuel plate is retrieved in the late afternoon. About one hour before the fuel plate is to be removed from the oven,

the technician may purge the oven with argon gas to accelerate the cooling, if necessary. Asbestos gloves should be worn to remove the fuel plate from the oven since the plate will still be warm ($\sim 50^{\circ}\text{C}$).

Special care should be exercised to avoid contaminating the plate with oils from the skin or other sources of contamination such as dirty table tops, etc. Arrangements should be made to have a health physics monitor take swipes of the oven after the fuel plate is removed.

After the baking operation is completed, arrangements should be made to have the fuel transported to Area V where the ion-plating operation is performed.

PREPARATION OF FILAMENTS

Equipment Required

- Spool of 3 x .020 twisted tungsten wire
- Spool of 20 mil tantalum wire
- Jar of 65 mil aluminum clips
- Yardstick or tape measure

Procedure

The preparation of filaments used in the ion plating of fuel elements varies with the mass and geometry of the fuel element being plated. The basic filaments used with fuel rods have fewer aluminum clips than the filaments used with fuel plates. Three filaments are required for an ion-plating operation. A basic filament (for rods) consists of a 40-inch tungsten wire, eight 3-inch pieces of tantalum wire, and sixteen aluminum clips. The eight tantalum wires are spiraled around the tungsten wire beginning at 2", 7", 12", 17", 22", 32" and 37" from one end. The tantalum should be wound so each spiral is about one inch long, leaving about four inches between the spirals (see Fig. 2). Two aluminum clips (one near each end) are then firmly pressed around each spiral. On plate filaments four 1-1/2 inch pieces of tantalum wire and four additional clips are also required. The tantalum wire strips are wound in spiral fashion halfway between spirals 1 and 2, 3 and 4, 5 and 6, 7 and 8 of the

Figure 2. Preparation of basic filaments

basic filament described above. These extra spirals will be about 1/2" long. The four extra clips are then centered on these new tantalum spirals. As the aluminum melts it will wet the tantalum wire. It is very important to have the filaments cut to the same length so all of the aluminum clips will melt at the same time. It is also important to have the spirals on all three filaments closely aligned so the filament supports can be positioned properly. Use of these filaments is described in a subsequent section.

OPERATION OF THE VACUUM SYSTEM

The Rotary Pumps

There are two mechanical vacuum pumps associated with the ion-plating vacuum system. The smaller of these pumps is called the holding pump and the larger is called the roughing pump. The holding pump must always be turned on before the diffusion pump is turned on.

1. Before starting the holding pump, close all valves on the vacuum system (see Fig. 3). Valve 1 is the air-release valve (turn clockwise to close); valves 2 and 3 are ball-cock valves from the roughing pump to the foreline and bell jar respectively (turn counterclockwise to close - handle vertical, pointing down). Valve 4 is a gate valve (or sliding valve) between the diffusion pump and the bell jar (turn clockwise to close).
2. The holding pump is turned on by throwing the master and holding - pump switches on the chassis control panel.
3. The roughing pump is turned on by a single-pole switch on the base of the roughing pump.

The Diffusion Pump

4. Connect inlet and drain hoses to the diffusion pump coolant line and turn on the water coolant.

Figure 3. Vacuum system valves

5. Turn on the diffusion pump heating coil (switch is located on the chassis control panel). NOTE: NEVER run the diffusion pump without turning on the water-coolant (see Step 4).
6. Open valve 2 (turn clockwise 1/4 turn - handle horizontal, pointing left).
7. (If the diffusion pump has been in operation before one starts, this step can be skipped.) Allow sufficient time (~ 45 minutes) for the diffusion pump to warm up. The vacuum in the foreline can be monitored during this period by turning the Pirani thermocouple gauge switch to position "FL." The pressure in the foreline should be about 25 microns (25×10^{-3} mm of Hg) or less by the time the diffusion pump is warmed up. Do not turn on the discharge vacuum gauge at this time.
8. Use the nitrogen gas tank, liquid nitrogen dewar, and feed tubes to fill the nitrogen cold trap after the diffusion pump has warmed up.
9. The bell jar and base plate should be cleaned to obtain a good vacuum. The cleaning procedure is discussed in another section (see "Preparation of the Vacuum System").
10. Position the bell jar on the base plate and tighten the clamps to the guide posts. Be sure to ground the bell jar if the High Voltage is to be used.
11. Close valve 2 (turn counterclockwise 1/4 turn - handle vertical, pointing down).
12. Open valve 3 (Note: valves 2 and 3 should never be open at the same time - do not reverse steps 11 and 12). The roughing pump will now pull a vacuum on the bell jar. The bell jar pressure can be monitored by turning the thermocouple vacuum gauge switch to position "BJ." Do not turn on the discharge vacuum gauge at this time.

13. Wait until the pressure in the bell jar is pumped down to below 50 microns (about 5 minutes).
14. Close valve 3.
15. Open valve 2 (Note: valves 2 and 3 should never be open at the same time - do not reverse steps 14 and 15).
16. Turn the slide valve counterclockwise to open. The bell jar is now open to high vacuum and should reach an ultimate pressure of 1×10^{-6} mm of Hg in about 4 hours.

NOTE: The "BJ" thermocouple gauge is incapable of resolving pressures below about 10 microns and it is not uncommon for this gauge to indicate a pressure of ~ 10 microns when the actual pressure is several orders of magnitude less.
17. Periodically replenish the supply of liquid nitrogen in the cold trap to maintain the best vacuum. Remove the gas feed tube from the N_2 gas bottle after each use to relieve the back pressure.
18. Turn on the discharge vacuum gauge when the "BJ" thermocouple gauge indicates a pressure of about 40 to 50 microns (1 micron = 10^{-3} mm of Hg) or less. Operation of the discharge gauge at higher pressures could easily damage the gauge.

Breaking the Vacuum

19. TURN OFF THE DISCHARGE VACUUM GAUGE.
20. Close the slide valve by turning clockwise until the valve seats firmly.
21. Open the air release valve 1.

22. To lift the bell jar the seal must be broken. This is accomplished without ruining the rubber gasket by manually applying a small horizontal force to the upper part of the bell jar.

System Shutdown

23. Turn off the diffusion pump heating coil and allow the system to cool for about 2 hours.
24. Lower the bell jar and close air release valve 1.
25. Close valve 2.
26. Open valve 3 and rough bell jar down to 50 microns.
27. Close valve 3.
28. Turn off roughing pump and bleed roughing pump hose.
29. Turn off holding pump and bleed holding pump hose.
30. Turn off cooling water and store the hose behind the roughing pump.

PREPARATION OF THE VACUUM PLATING MACHINE

Before each plating operation it will be necessary to clean and mount the insulators and holding fixtures which will be used in the vacuum system. Exploded views of a filament support stand, a thermocouple-high-voltage (HV) feedthrough, and a fuel plate support stand are shown in Figs. 4 and 5. Fig. 6 shows the arrangement of the fixtures for the fuel plates. Note that there are two height adjustment positions for the top two filaments. The lower positions are used for fuel plates, and the upper positions are used for fuel rods. The thermocouple - HV feedthrough for the fuel plates also serves as one of the support fixtures (on extreme right). Note the locating bushing, the double

Figure 4. Filament support stands

Figure 5. Fuel plate support stand and high voltage feedthrough

insulator shield and the hex nut on the thermocouple-HV support which are not shown in the exploded view of Fig. 5. The four fuel plate support fixtures shown in Fig. 6 are not used for the rods. For rods, a similar, but shorter, thermocouple-HV feedthrough is used which fits into the center hole of the cover plate. The rod mounting adapter on the extreme right side of Fig. 5 is used for mounting the rods on the thermocouple-HV feedthrough. The adapter used to support the fuel plate is shown next to the rod adapter in Fig. 5.

The conductive aluminum coatings from previous operations must be removed from the insulators. This is particularly important for the unshielded insulators on the filament stands.

For the following operations wear a lab coat, rubber gloves, and a face shield. Chemicals and solvents are stored in cabinets underneath the ventilation hood.

1. Prepare a clean working area on the stainless steel table and clean out the tubs.
2. Fill one tub with approximately 6 inches of hot water and add enough Alconox detergent to form suds.
3. Place the insulators in a medium size beaker with a small amount of lye (50% NaOH) and let soak for approximately 1/2 hour.
4. Scrub the insulators with a Scotch Brite scouring pad until all of the aluminum is removed.
5. Wash the insulators in the detergent solution, rinse with warm water, and place on a towel to dry. Acetone may also be used to rinse the insulators.
6. Caution: when cleaning the aluminum shields and support rods do not place these items in the lye solution. These parts can be placed in the detergent solution, scrubbed with a scouring pad and rinsed with

Figure 6. Arrangement of filaments and support stands for ion-plating fuel plates

warm water and/or acetone before each series of plating operations or more frequently if necessary. Normally, a lint-free cloth soaked in acetone can be used to wipe off loose particles between successive plating operations.

7. The remaining non-aluminum pieces should be treated with the lye solution and cleaned thoroughly before each series of plating operations or more frequently, if necessary. Normally a lint-free cloth soaked in acetone can be used to wipe off loose particles between successive plating operations.
8. Tape your gloves to your lab coat sleeves for the following operation.
9. Apply a small amount of lye solution to a Scotch Brite pad by placing the pad over the bottle and tilting the bottle and pad together until the pad absorbs some of the solution. Do not pour the solution on the pad.
10. Clear off a window area on the inside of the bell jar by rubbing the pad in a circular motion around the area to be cleaned. Be careful not to let any lye drip from the pad to the aluminum base plate of the vacuum system as it will attack the finely finished surface of the base plate and could cause vacuum leaks.
11. Let the lye react with the aluminum coating on the bell jar for a few minutes and then wipe clean with a water-damp lint-free cloth.
12. Wipe the area dry with an acetone-damp lint-free cloth. (Note: Occasionally, it may be necessary to clean the entire bell jar when flaking appears. The bell jar will have to be removed for a complete cleaning. It is best to do this prior to a rod plating rather than prior to plating a fuel plate.)

13. Use a special vacuum cleaner (labeled "for contaminated waste") to clean up loose particles and flakes from the chamber, the chassis, and the floor around the vacuum plating machine.
14. Rinse the prepared filaments with acetone and blow dry with dry nitrogen gas.
15. Mount the filaments and support fixtures as shown in Fig. 6. The ends of the filaments are clamped in place so that the filaments form a circle resting on the insulators of the three filament support stands.
16. Two of the four fuel plate supports are adjustable. An aluminum mock-up fuel plate can be used to adjust the supports for good contact. It is particularly important that the fuel plate make good contact with the high voltage post.

CLEANING THE FUEL

The purpose of the cleaning operation is to remove the oxide and surface contamination from the fuel. The cleaning must be thorough and uniform and the fuel should be placed under vacuum as soon as possible to retard the formation of a new oxide film.

For the following operations wear a lab coat, rubber gloves and a face shield.

1. Pour a sufficient amount of water and concentrated nitric acid (HNO_3) (1 part water, 3 parts acid) into the KIMEX 18" x 12" x 3" glass dish to completely immerse the fuel to be cleaned. The acid is to be poured into the water and not vice versa.
2. Place the fuel in the acid bath for about one to two minutes (for fuel plates use the handling tool and position the handling tool so that the bottom surface of the plate is not flush with the dish). Rub the surface with fingers to accelerate the chemical reaction

(see Figs. 7 and 8). As a precaution against criticality, no more than one fuel piece should ever be placed in the acid bath at one time.

3. Inspect the fuel surface for uniform appearance and remove from acid bath if satisfactory.
4. Place in a hot water, detergent mixture in the sink and rub the surface vigorously with a Scotch Brite pad. Use sandpaper to clean slots, pipe cleaners to clean thermocouple holes, and a stiff brush to clean control rod holes.
5. Rinse with hot water.
6. Dry with lint free cloth thoroughly and quickly (see Fig. 9). After drying fairly well use a dry pipe cleaner to clean the thermocouple holes and then blow dry nitrogen gas on the fuel to further dry any wet spots. Dry any water streaks immediately (see Fig. 10).
7. For rods proceed to step 12.
8. Place a fuel plate (no rods) in a phosphoric acid and Electro-Glo* solution (3 parts acid, 1 part Electro-Glo) in the electropolishing tank. Support the fuel plate with the holding tool as shown in Fig. 11. The holding tool must be positioned in a control rod hole rather than the glory hole in order to completely submerge the sample. Do not try to push the holder all the way through the control rod hole.
9. Attach the positive electrode to the holding tool as shown in Fig. 11 and turn on the power supply on the shelf below the tank. The current should be set at 20 amps. Electropolish for about 45 seconds. A bubbling action will be observed in the solution during the electropolishing treatment.
10. Remove from the electropolishing tank and repeat steps 4, 5, and 6.

*Trademark - Electro-Glo Corporation

Figure 7. Fuel plate in acid bath

Figure 8. Rubbing the surface to accelerate chemical reaction

Figure 9

Drying the fuel plate

Figure 10

Using nitrogen gas to dry small
holes in the fuel

Figure 11. Fuel plate in electropolishing tank

11. Repeat steps 2 through 10 if surface is not uniformly clean and bright.
12. Place fuel in the vacuum plating machine. A rod is screwed on to the threaded adapter. A fuel plate is positioned on the four post supports (see Fig. 12). Check to see that the fuel plate makes good contact with all four supports.
13. After placing the fuel under high vacuum (see next section) carefully pour the nitric acid back into its container(s) using a funnel. Do not dispose of acid in the sink. The uranium in solution must be sent to A. W. Porter, Division 4611.

ION-PLATING THE FUEL

An ion-plated aluminum coating of 0.3-0.8 mils with a deposition rate of 0.2 mil/min provides the most effective corrosion protection for uranium.² The quality of the final coating can best be assured by thoroughly precleaning the sample as described above, and by maintaining close control over the discharge voltage, current and pressure during the sputter cleaning and deposition phases of the ion-plating described in the following sections. The fuel piece should be placed under high vacuum as soon as possible after the cleaning is completed. A schematic of some ion-plating apparatus described in this section is shown in Fig. 13.

Placing the Sample Under High Vacuum

1. Lower the bell jar and secure it with two clamps on the guide posts.
2. Clamp the high voltage positive lead to the bell jar guide post flange on the left side to form a positive ground.
3. Close the air release valve (#1) to the vacuum chamber.
4. Place the sample under high vacuum by following steps 11 through 18 under "Operation of the Vacuum System."

Figure 12. Fuel plate properly positioned in the vacuum chamber

Figure 13. Schematic of the ion-plating apparatus

5. Wait until the vacuum system reaches its ultimate vacuum of 1×10^{-6} mm of Hg (about 4 hours).
6. Connect the pyrometer to the thermocouple leads. The yellow lead is positive. Insulate the pyrometer from the chassis since the chassis will be positive high voltage.

Controlling the Argon Environment

7. Set the Variac on the gettering furnace at 60 for a furnace temperature of 600°C . Turn the furnace on about 15 minutes before the plating operation is to begin.
8. Open the stop valve on the argon tank and set the regulator to read 5 pounds pressure.
9. Flush the argon through the gettering furnace before connecting the furnace up to the leak valve on the stand. This will not be necessary for subsequent operations if the line is not bled in the meantime. Connect the hoses as shown in Fig. 1.
10. Turn the discharge gauge down scale and open the variable leak valve to about 113 or 114. The pressure will increase to $25 - 40 \times 10^{-3}$ mm of Hg (25-40 microns). The foreline pressure will read about 50 microns.
11. Wait about 5 minutes for the air from the argon feed lines to diffuse from the system.

Sputter Cleaning the Sample

12. Set the Moseley Autograf (model 680) strip-chart recorder for a chart speed of 8 divisions per minute and a range of 5 volts. This instrument will record the plasma current by monitoring the voltage drop across a resistor (see Fig. 13). A full-scale reading on the 5 volt range corresponds to a plasma current of 500 ma.

13. Turn on the Moseley Autograf stripchart recorder and zero it.
14. Turn on the high voltage supply by closing the throw switches and pressing the "High Voltage On" switch.
15. Increase the Variac setting on the high voltage supply until the voltage reading (high scale) reaches 6 kilovolts for fuel rods or 6.5 kilovolts for fuel plates. The voltage will decrease slowly during the sputter-cleaning operation.
16. Replenish the liquid nitrogen in the nitrogen trap if necessary.
17. Slowly close the slide valve (#4). A blue glow discharge will appear around the substrate or cathode (see Fig. 14) as the plasma begins to conduct. Continue to close the slide valve until the plasma current reaches 120 ma for fuel rods or 475 ma for fuel plates. Arcing may occur as the current is increased if the insulators are not clean. Wait until arcing stops before further increasing the current.
18. The plasma current will decrease to a stable value as the surface is sputter cleaned (this takes about 5-10 minutes). The temperature will increase markedly during this time ($T > 300^{\circ}\text{C}$). Do not heat the sample above 350°C (see step 20).
19. If fuel plates are being ion-plated the filament power supply should be turned on and a current of about 20 amps should be applied to the filaments by slowly turning the crank arm on top of the unit as soon as the sputtering begins. This will prevent contamination of the filaments during sputtering. This action is not necessary for fuel rods. Make certain that the filament current is at a minimum when the supply is turned on to avoid breaking the filaments.
20. If the temperature of the sample exceeds 350°C the sample will have to be allowed to cool before proceeding. In this case:
 - a. open slide valve;

Figure 14. Blue glow discharge at cathode

- b. decrease filament supply to a minimum and then turn off filament supply, if on:
- c. close the argon variable leak valve and stop valve;
- d. turn off the gettering furnace;
- e. decrease the high voltage:
- f. turn off the high voltage;
- g. turn off recorder:
- h. allow 3 to 4 hours to cool below 200°C;
- i. repeat steps 10 through 20.

Ion-Plating the Sample

- 21. The plasma current should level off at about 95 ma for fuel rods and 450 ma for fuel plates. It may be necessary to throttle (close) the slide valve to increase the currents to the above values which are based on an optimum current flux of 0.5 ma per square centimeter.² The plasma voltage should be no lower than 5 kilovolts at this time. Do not ion plate if the temperature of the sample is above 350°C (see step 20).
- 22. If not already on, turn on the filament supply and slowly increase the current to avoid damaging the filaments. Make certain that the current is at a minimum when supply is turned on to avoid breaking the filaments. The plasma current may increase slightly at this time and then stabilize again.
- 23. Slowly preheat the filaments to a red glow (about 40 amps) and hold this temperature until all contamination from sputtered materials is flushed from the chamber. By monitoring the ion current it is easy to tell when stability is reached, and the evaporation can be started.
- 24. A relatively low evaporation rate produces the most consistent results. Begin the deposition by increasing the filament current slowly and then increase the current at a faster rate as vaporization and deposition begin. Deposition rates on the order of 0.2 mils/min are de-

..
..
sirable (2 to 3 minutes total deposition time). Carefully monitor the plasma voltage and current during the deposition and make adjustments if necessary to maintain a voltage of about 5 kV and a current of 95 ma for rods or 440 ma for plates.

25. After completion of the deposition, open the slide valve, decrease the filament current to a minimum and then turn off the filament supply. Next turn off the argon supply, the gettering furnace, the high voltage, and the recorder, in that order.
26. Allow about 5 hours or longer for the sample to cool below 100°C.
27. Break the vacuum (see "Operation of the Vacuum System") and remove the fuel.

SUMMARY

Aluminum ion-plating is an effective process of coating SPR II fuel, providing both corrosion protection and retention of fission products. The quality of the final coating is influenced by the precleaning process and various process parameters, such as the discharge voltage, current, and vacuum pressure during the sputter-cleaning and deposition phases of the ion-plating process.

REFERENCES

1. D. M. Maddox, Film Deposition Using Accelerated Ions, Sandia Laboratories, Albuquerque, SC-DR-63-281, November, 1963.
2. R. D. Bland, A Parametric Study of Ion-Plated Aluminum Coatings, Sandia Laboratories, Albuquerque, SC-DR-67-709, September, 1967.

DISTRIBUTION:

B. F. Hefley, 3141
A. W. Porter, 4611-1
A. M. Clogston, 5000
A. Narath, 50
O. E. Jones, 5100
L. C. Hebel, 5200
J. V. Walker, 5220
R. M. Jefferson, 5221
L. L. Bonzon, 5221
J. C. Conant, 5221
B. F. Estes, 5221
V. E. James, 5221
F. M. Morris, 5221
J. S. Philbin, 5221 (5)
J. B. Rivard, 5221
J. A. Snyder, 5221
R. L. Coats, 5222
J. A. Horak, 5222
F. Gonzalez, 5222
D. J. Sasmor, 5222
J. E. McDonald, 5300
R. D. Bland, 5332
L. M. Berry, 5500
A. Y. Pope, 5600
R. H. Altherr, 7121
L. S. Ostrander, 8232
Central Files, 3141-1 (15)
L. C. Baldwin, 3152
Technical Publications Div., 3151 (2)
For AEC/TIC