



# Retention of Sputtered Molybdenum on Ion Engine Discharge Chamber Surfaces

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Grit-blasted anode surfaces are commonly used in ion engines to ensure adherence of sputtered coatings. Next generation ion engines will require higher power levels, longer operating times, and thus there will likely be thicker sputtered coatings on their anode surfaces than observed to date on 2.3 kW-class xenon ion engines. The thickness of coatings on the anode of a 10 kW, 40-cm diameter thruster, for example, may be 22  $\mu\text{m}$  or more after extended operation. Grit-blasted wire mesh, titanium, and aluminum coupons were coated with molybdenum at accelerated rates to establish coating stability after the deposition process and after thermal cycling tests. These accelerated deposition rates are roughly three orders of magnitude more rapid than the rates at which the screen grid is sputtered in a 2.3 kW-class, 30-cm diameter ion engine. Using both RF and DC sputtering processes, the molybdenum coating thicknesses ranged from 8  $\mu\text{m}$  to 130  $\mu\text{m}$ , and deposition rates from 1.8  $\mu\text{m}/\text{h}$  to 5.1  $\mu\text{m}/\text{h}$ . In all cases, the molybdenum coatings were stable after the deposition process, and there was no evidence of spalling of the coatings after 20 cycles from about  $-60\text{ }^{\circ}\text{C}$  to  $+320\text{ }^{\circ}\text{C}$ . The stable, 130  $\mu\text{m}$  molybdenum coating on wire mesh is 26 times thicker than the thickest coating found on the anode of a 2.3 kW, xenon ion engine that was tested for 8200 hours. Additionally, this coating on wire mesh coupon is estimated to be a factor of  $> 4$  thicker than one would expect to obtain on the anode of the next generation ion engine which may have xenon throughputs as high as 550 kg.

## Introduction

Grit-blasted wire mesh on stainless steel backing is commonly used in ion engine discharge chambers to ensure adherence of sputtered coatings. One of the major sources of sputtered material is the molybdenum screen electrode that is bombarded by singly- and doubly-charged ions whose energies are in the neighborhood of 25 eV and 50 eV, respectively. Spalled coatings or flakes pose some risk in shorting the high-voltage ion optics. If spalling does occur, the fine wire mesh ensures the spalled material will have sufficiently small dimensions so electrode gaps cannot be bridged. The grit-blasted wire-mesh coating retention scheme was developed in the 1970s for mercury ion engines [1]. This method has been employed to retain sputtered coatings in many ion engine development programs [2-6].

A 30-cm diameter, engineering model ion engine was recently tested successfully for 8,200 hours to obtain component wear-rates and to gain insight into other life-limiting phenomena [7]. The ground-test

of this ion engine was one of the longest tests of an inert-gas ion engine and provided data on coatings and flaking within the discharge chamber. This test was run at full-power, 2.3 kW, and the engine had a xenon throughput of 88 kg. In this engineering model engine, the downstream 520  $\text{cm}^2$  of the discharge chamber anode was covered with grit-blasted wire-mesh. The upstream aluminum surfaces (1650  $\text{cm}^2$ ) were simply grit-blasted to retain the sputtered material. Post-test diagnostics [7] of the discharge chamber determined that a very adherent coating composed of molybdenum and stainless steel products was found on the grit-blasted wire mesh and aluminum surfaces (Table 1). The thickness of these adherent coatings ranged from 2  $\mu\text{m}$  to 5  $\mu\text{m}$ . Loose flakes were also found at the bottom of the discharge chamber. The thickness of these flakes was in the 2  $\mu\text{m}$  to 12  $\mu\text{m}$  range, and the composition was primarily molybdenum, stainless steel products, and tantalum. Molybdenum by far was the major constituent of flakes collected at any location in the discharge chamber. Figures 1a and 1b are scanning electron photomicrographs of the morphology of the surface and section-view of a

typical 12  $\mu\text{m}$  thick flake. The material appears very granular and "lumpy". The largest flake found in the cylindrical part of the chamber was 1.7 mm by 0.7 mm. In the vicinity of the cathode a 3.8 mm by 0.1 mm flake was found. All loose flakes that were analyzed had planar dimensions in the 100  $\mu\text{m}$  to 3800  $\mu\text{m}$  range. Since most of the wire mesh external surface is comprised of 51  $\mu\text{m}$  diameter wires, it is clear that these flakes did not spall from the wire mesh surface. The source of the loose flakes may have been the cathode/keeper-electrode or other surfaces that were not grit-blasted or covered with wire mesh. Even though the ion extraction grid gap is about 0.66 mm, the loose flakes did not produce any permanent grid shorts.

The best test of the effectiveness of chamber coating retention is operating the ion engine in the zero-g environment of space. The Deep Space 1 ion propulsion system was launched in October 1998 and will be thrusting until the end of this year. The Deep Space 1 ion engine has operated in zero-g conditions for over 13,000 hours with a xenon throughput exceeding 60 kg, and no grid shorting problems have been encountered [8]. The flight ion engines had the entire anode surface ( $\sim 2170 \text{ cm}^2$ ) covered with grit-blasted wire-mesh.

Next generation ion engines will likely have xenon throughput requirements in the 200 kg to 550 kg range, implying longer operation, higher power levels, and thicker sputtered coatings internal to the engine [9,10]. This paper describes the retention capability of thick molybdenum coatings processed using accelerated deposition rates and applied to grit-blasted aluminum, titanium, and wire mesh all of which are candidates for discharge chamber material. Coatings ranged in thickness from 8  $\mu\text{m}$  to 130  $\mu\text{m}$ . The coated specimens were subjected to vacuum thermal-cycle tests with temperatures from  $-60 \text{ }^\circ\text{C}$  to  $340 \text{ }^\circ\text{C}$ . Results of the thermal tests and post-test analyses are presented.

### **Apparatus and Procedure**

The goal of this effort is to attempt to document the retention capability of thick molybdenum films on grit-blasted surfaces. Thirty centimeter diameter thruster internal surfaces acquire a film thickness of about 5  $\mu\text{m}$  in 8,200 hours when operated at 2.3 kW.

In this series of experiments the molybdenum is deposited rapidly on coupons so a 10  $\mu\text{m}$  film is accumulated in 2 h to 5 h. The coating microstructure may be somewhat affected by the rate of deposition of molybdenum, but the RF (Radio Frequency) and DC (Direct Current) sputter systems were used to expedite the process and gain some insight into the coating adherence. The type of surface preparation for the test coupons was the same as that used for the ion engines.

### **Test Coupons**

The coupons used in the RF sputter system were either wire mesh, aluminum, or titanium. One-half of each of the coupons was grit-blasted, and the other half was not. Dimensions are shown in Table 2. The coupons used as substrates in the DC sputter system were wire mesh and are defined in Table 3. The surface of these coupons was entirely grit-blasted. The stainless steel wire mesh is a Dutch weave using 71  $\mu\text{m}$  and 51  $\mu\text{m}$  wires which are diffusion-bonded to a stainless steel backing whose thickness is 89  $\mu\text{m}$ . Overall thickness of the wire mesh coupon is about 240  $\mu\text{m}$ .

All coupons were grit-blasted using alumina grit ejected through a 0.18 mm by 3.8 mm rectangular orifice. Nominal grit dimension was 50  $\mu\text{m}$ . The grit-blaster air-flow was about 75 standard  $\text{cm}^3/\text{s}$ , and the distance from the nozzle to the coupon was 2.5 cm. A motor-driven fixture ensured the coupon feed rate was 15 cm/min and that there was a grit-blasting overlap of 2.5 mm. Figure 2 shows the grit-blasted texture of the wires with microstructures having dimensions of the order of 10  $\mu\text{m}$ . The spacing between the two closely spaced wires in the Dutch-weave is about 3  $\mu\text{m}$ , while the distance between wire-pairs is nominally 30  $\mu\text{m}$ . Finally, the coupons were ultrasonically cleaned with acetone and then isopropyl alcohol.

### **RF Sputter System**

The RF sputtering system used a 7.6 cm target that was pure molybdenum. The layout of the coupon substrates is shown in Figure 3. The sputter-etch voltage was estimated to be 290 V. The molybdenum film was deposited for 7 h using 1 kW RF power at an argon pressure of 1.3 Pa. The base pressure of the sputter facility without argon gas load was  $4 \times 10^{-5}$  Pa. Prior to the deposition process,

the coupons were not ion-cleaned since it was desired to simulate a thruster discharge chamber condition prior to testing. A smooth witness coupon located 5.6 cm from the center of the main sputter deposition "spot". The molybdenum film thickness at that location was measured using a surface profilometer. The film thickness was 30  $\mu\text{m}$ . Test coupon temperatures were not recorded during the RF sputtering process.

### DC Sputter System

The DC sputtering system is shown schematically in Figure 4. The plasma generator is nearly identical to that used for early 30-cm diameter ion engines and inert gas ion sources [11,12]. The argon discharge was generally operated at 45 V to 50 V at current levels from 6 A to 12 A. The cylindrical target was stationed near the exit plane of the discharge chamber. The coupon table was usually separated from the molybdenum target by 1.8 cm. All insulators that electrically isolated electrodes were protected from the sputtered efflux by sputter shield cups.

The discharge was initiated using about 55 sccm argon flow through the hollow cathode and applying a 3-kV, 3- $\mu\text{s}$  pulse to a wire electrode near the hollow cathode orifice plate. After the discharge conditions were set, the target was biased -500 V relative to the grounded cathode assembly. Pressure in the vicinity of the DC sputtering system was between 9 mPa and 19 mPa. Molybdenum deposition times ranged from 3.2 h to 65 h. Basic parameters for these sputter deposition processes are shown in Tables 4 and 5.

### Thermal-Vacuum Test Facility

Thermal-vacuum cycle-testing was conducted in a bell-jar facility. The vacuum facility was pumped by an oil diffusion pump that provided a background pressure of  $9 \times 10^{-5}$  Pa. Above the diffusion pump was a cold trap of water-cooled baffles followed by liquid nitrogen-cooled baffles. The facility simulated temperature cycles experienced in a space environment in that heating and cooling occur through radiative processes. For the first part of a cycle, samples were located over a ceramic heater. A thermocouple on the sample holder was used to control the thermal cycling process. When a programmed upper temperature limit was reached, samples were physically moved, via a solenoid and

mechanical linkage, into a liquid nitrogen-cooled copper box. Heat from the sample holder was lost to the cold walls of the copper box until the controlling thermocouple reached the low temperature limit. At this point, the solenoid/linkage system moved the samples back to the heating environment for the next cycle. Temperature data for a second thermocouple on the sample holder was recorded through a computer data acquisition system. The controlling thermocouple was used to activate the solenoids that moved the coupons from the heater location to the cold box. Temperature limits for this thermocouple were +320  $^{\circ}\text{C}$  and -60  $^{\circ}\text{C}$  for Test 1 and +340  $^{\circ}\text{C}$  and -60  $^{\circ}\text{C}$  for Test 2 (Table 6). The temperature limits bound those used in the development of the Deep Space 1 ion engine [13]. In Test 1 and Test 2 the cooling times were about 3.5 hours and 2.3 hours, respectively. In all cases the heating time was about 15 minutes.

### Coating Diagnostic Hardware and Methods

Measurements of the mass of the molybdenum films were made by weighing coupons on a balance accurate at least to the nearest 0.1 mg. Coating masses ranged from about 30 mg to 210 mg. Knowing the coupon planar area and mass gain, the film thickness was estimated using the bulk density of molybdenum which is 10.2  $\text{g}/\text{cm}^3$  [14]. Attempts were made to measure the step in a masked silicon coupon to verify the deposited film thickness. This method was found to be unreliable for coatings  $\geq 25 \mu\text{m}$  since film spalling occurred on many of the smooth silicon coupons.

A scanning electron microscope (SEM) was used to characterize the surface features of the grit-blasted wire mesh before and after the coating process. Film cross-sections and film surface structure were documented at magnifications from 100X to 10,000X. An optical microscope (5X, 10X, 20X) was used to examine coatings after the thermal cycling tests.

## Results and Discussion

As indicated in Table 1, discharge chamber wire mesh coating thicknesses up to 5  $\mu\text{m}$  have been measured on a 2.3 kW ion engine that processed 88 kg of xenon propellant during the course of a 8200-hour test. The Deep Space 1 flight spare ion engine has processed more than 150 kg of xenon,

and from a simple extrapolation based on xenon throughput, the chamber coating thickness might be as much as 9  $\mu\text{m}$ . Next generation engines may process up to 550 kg of xenon, and discharge chamber coating thickness may be in the vicinity of 22  $\mu\text{m}$  to 31  $\mu\text{m}$ . Since the extrapolations of coating thickness are very crude, it was decided to examine the retention capability of molybdenum to wire mesh for coating thicknesses in excess of 100  $\mu\text{m}$ .

The RF and DC coating methods provide very rapid deposition of molybdenum compared to deposition rates on ion engine anodes. The RF and DC sputter deposition rates were 5.1  $\mu\text{m}/\text{h}$  and 1.8  $\mu\text{m}/\text{h}$  to 3.3  $\mu\text{m}/\text{h}$ , respectively. These accelerated deposition rates are roughly three orders of magnitude more rapid than the rates at which the molybdenum screen grid is sputtered in a 2.3 kW ion engine [7]. Determining the coating stability of ion engine molybdenum deposited at a rate of only 8  $\mu\text{m}$  in 13,000 hours is beyond the scope of this effort. The high-rate sputter methods use wire mesh substrates prepared in similar fashion as the ion engine material. The high-rate DC sputter system had substrate temperatures of about 240  $^{\circ}\text{C}$ , and this is close to a typical ion engine chamber temperature of 300  $^{\circ}\text{C}$  or less. The DC sputter system pressure was about 0.02 Pa, and that is very similar to the ion engine chamber environment (Table 4).

The following sections describe the characteristics of the molybdenum coatings after the deposition process followed by discussions of the coating integrity after the thermal cycle tests.

### **Characteristics of the Coatings after the Deposition Process**

The six RF sputter coated coupons were about 1.2 cm by 2.5 cm, and each coupon experienced a mass gain of 0.10 g to 0.13 g after 7 hours of sputtering (Table 2). If it is assumed that the coating density is the same as bulk molybdenum, then the coating thickness ranged from 34  $\mu\text{m}$  to 39  $\mu\text{m}$  (Table 5). The witness coupon, located 1.8 cm beyond the target outside diameter, had a film thickness of 30  $\mu\text{m}$  as measured by a surface profilometer. Since the coupons of interest are in the main deposition zone, it is highly likely that their coating thickness is  $\geq 30 \mu\text{m}$ . Additionally, at sputtering pressures of 1 Pa or less, others have

found molybdenum sputtered coatings exhibit bulk-like properties with densities ranging from 80% to 100% of bulk density [15,16]. If the coatings are 80% dense, the thickness range is from 42  $\mu\text{m}$  to 49  $\mu\text{m}$ .

The RF-coated coupons were made of either stainless steel wire mesh, titanium, or aluminum. One-half of each coupon was grit-blasted and the other half was not. After these coupons were coated and returned to room temperature, there was no evidence of spalling on any of the coupon surfaces. The nominal 35  $\mu\text{m}$  coatings were even retained without spalling on the non grit-blasted surfaces.

All seven DC sputter-coated coupons were made of stainless steel wire-mesh diffusion bonded to stainless steel sheet metal. Coupon mass gain after the deposition process ranged from 8.1  $\text{mg}/\text{cm}^2$  to 130  $\text{mg}/\text{cm}^2$  (Table 3). If it is assumed the coating density is the same as bulk molybdenum, the coating thickness varied from 8.0  $\mu\text{m}$  to 130  $\mu\text{m}$ . In two cases the thickness based on mass gain was compared with the coating thickness measured by a profilometer on step at the coating interface on a silicon witness coupon. In the case of the wire mesh-2 coupon, the coating measurements were both determined to be 22  $\mu\text{m}$  (Table 3). For wire mesh-3 coupon, the two types of coating thickness measurements differed by about 30% for the nominal 10  $\mu\text{m}$  films. Thicker films of 25  $\mu\text{m}$  to 130  $\mu\text{m}$  spalled on the smooth silicon witness plate. After coating at a temperature of about 240  $^{\circ}\text{C}$ , the wire mesh coupons were returned to room temperature with no indication of spalling of coatings.

Figure 5 shows the 51  $\mu\text{m}$  diameter wires coated with 25  $\mu\text{m}$  of molybdenum. The coatings exhibit a rough structure that is dictated by the grit-blasted substrate that is shown in Figure 2. Energy dispersive analysis using X-ray diagnostics verified the coating was pure molybdenum with trace amounts of chromium, zirconium, and possibly phosphorus. Figure 6 is a highly magnified photograph of the 51  $\mu\text{m}$  diameter wire coated with 8  $\mu\text{m}$  of molybdenum. This photograph indicates that the coating may be comprised of "macro-columns" whose cross-sections have dimensions in the 6  $\mu\text{m}$  to 17  $\mu\text{m}$  range.



Figure 7 shows the structure of a 58  $\mu\text{m}$  molybdenum coating. Large columns with 1  $\mu\text{m}$  to 3  $\mu\text{m}$  dimensions are comprised of many smaller columns with submicron dimensions. Molybdenum coatings with densely-packed, fiber-like columns having submicron dimensions have been reported by others using magnetron sputter deposition methods.[17] In this reference it was found that "the overall formation of the column microstructure begins at the scale of tens of microns, though the formation of a small number of cone-like structures is already observed for the 23 nm thick film." Since the coatings applied to the wire mesh are not continuous, stresses are probably more readily relieved because of the discontinuous columnar structure.

#### **Integrity of the Coatings After the Thermal-Vacuum Tests**

The RF sputter-coated coupons were tested for 20 cycles per the thermal limits indicated in Table 6. After the test, visual inspection showed no changes in coating appearance. The coupons were nominally 1.2 cm by 2.5 cm with a coating mass in the 100 mg to 130 mg range. After thermal cycling, the change in coating mass, in all cases, was less than 1%. Mass measurements after the thermal testing were only made to the nearest tenth of a milligram. Microscope inspections and the mass measurements made before and after thermal cycle testing indicated there was no evidence of spalling of the nominal 35  $\mu\text{m}$  thick molybdenum coatings on wire mesh, aluminum, or titanium substrates.

The DC sputter-coated coupons were also tested for 20 cycles. Coupon temperatures at the minimum and maximum temperatures were  $-60\text{ }^{\circ}\text{C}$  to  $-54\text{ }^{\circ}\text{C}$  and  $+297\text{ }^{\circ}\text{C}$  to  $+340\text{ }^{\circ}\text{C}$ , respectively. The spread in temperature measurements is likely due to the fact that the non-controlling thermocouple was probably not firmly joined to the coupon holder. As indicated in Table 3, the coupon planar areas were 1.6  $\text{cm}^2$  to 3.7  $\text{cm}^2$ , and the molybdenum coating masses varied from about 30 mg to 214 mg. After thermal cycling the coupons were weighed, and the masses decreased by  $< 1\%$  in the case of the 8  $\mu\text{m}$  coating to  $< 0.2\%$  in the case of the 130  $\mu\text{m}$  coating. Since mass measurements were made to the nearest tenth of a milligram, these variations in mass are close to the measurement uncertainty. Visual inspections

were made at 5X, 10X, and 20X with an optical microscope. There was no evidence of spalling of the molybdenum films ranging in thickness up to 130  $\mu\text{m}$ . Detailed analysis of the coatings, after the thermal-cycle test, using SEM diagnostics was beyond the scope of this work.

In summary, the accelerated deposition of molybdenum on wire mesh, aluminum, and titanium was followed by thermal cycle testing, and there was no evidence of film spalling as indicated by coating mass measurements and optical microscope inspections. Coating thicknesses ranged from 8  $\mu\text{m}$  to 130  $\mu\text{m}$ , and deposition rates were from 1.8  $\mu\text{m}/\text{h}$  to 5.1  $\mu\text{m}/\text{h}$ . Coupon temperatures during the DC sputter deposition were in the  $200\text{ }^{\circ}\text{C}$  to  $240\text{ }^{\circ}\text{C}$  range which is close to the anode temperatures for the Deep Space 1 ion engine [13]. The 130  $\mu\text{m}$  thick molybdenum coating on wire mesh is estimated to be more than a factor of four thicker than one would expect to obtain on the anode of the next generation ion engine which may have xenon throughputs as high as 550 kg.

#### **Concluding Remarks**

Grit-blasted titanium, aluminum, and wire mesh with stainless steel backing are commonly used in ion engines to ensure adherence of sputtered coatings. Next generation ion engines will require higher power levels, longer operating times, and thus there will likely be thicker sputtered coatings on their anode surfaces than observed to date on 2.3 kW-class xenon ion engines. The thickness of coatings on the anode of a 10 kW, 40-cm diameter thruster [18], for example, are estimated to be 22  $\mu\text{m}$  or more after extended operation.

Grit-blasted wire mesh, titanium, and aluminum substrates were coated with molybdenum at accelerated rates to establish coating stability after the deposition process and after thermal cycling tests. These accelerated deposition rates are roughly three orders of magnitude more rapid than the rates at which the screen grid is sputtered in a 2.3 kW-class, 30-cm diameter ion engine. Using both RF and DC sputtering processes, the molybdenum coating thicknesses ranged from 8  $\mu\text{m}$  to 130  $\mu\text{m}$ , and deposition rates from 1.8  $\mu\text{m}/\text{h}$  to 5.1  $\mu\text{m}/\text{h}$ . Coupon temperatures during the DC

sputtering were in the 200 °C to 240 °C range which is close to anode temperatures for the Deep Space 1 ion engine. In all cases, the molybdenum coatings were stable after the deposition process, and there was no evidence of spalling of the coatings after 20 cycles from about -60 °C to +320 °C. Integrity of the coatings was determined by measurement of coating mass to detect spalled material and by optical microscope examinations.

The stable, 130 μm molybdenum coating on wire mesh is 26 times thicker than the thickest coating found on the anode of a 2.3 kW, xenon ion engine that was tested for 8200 hours. Additionally, this coating on wire mesh coupon is estimated to be a factor of >4 thicker than one would expect to obtain on the anode of the next generation ion engine which may have xenon throughputs as high as 550 kg.

## References

- [1] Power, J.L. and Hiznay, D.J., "Solutions for Discharge Chamber Sputtering and Anode Deposit Spalling in Small Mercury Ion Thrusters," NASA TMX-71675, March 1975.
- [2] Bechtel, R.T., Trump, G.E., and James, E.J., "Results of the Mission Profile Life Test," AIAA Paper 82-1905, November 1982.
- [3] Beattie, J.R., Matossian, J.N., and Robson, R.R., "Status of Xenon Ion Propulsion Technology," AIAA Paper 87-1003, May 1987.
- [4] Francisco, D.R., Low, C.A., and Power, J.L., "Successful Completion of a Cyclic Ground Test of a Mercury Ion Auxiliary Propulsion System," IEPC Paper 88-035, October 1988.
- [5] Sovey, J.S., Hamley, J.A., Haag, T.W., Patterson, M.J., Pencil, E.J., Peterson, T.T., Pinero, L.R., Power, J.L., Rawlin, V.K., Sarmiento, C.J., Anderson, J.R., Becker, R.A., Brophy, J.R., Polk, J.E., Benson, G., Bond, T.A., Cardwell, G.I., Christensen, J.A., Freick, K.J., Hamel, D.J., Hart, S.L., McDowell, J., Norenberg, K.A., Phelps, T.K., Solis, E., Yost, H., and Matranga, M., "Development of an Ion Thruster and Power Processor for New Millennium's Deep Space 1 Mission," AIAA Paper 97-2778, July 1997.
- [6] Kitamura, S., Hayakawa, Y., Miyazaki, K., Yoshida, H., Yamamoto, Y., and Akai, K., "1000-Hour Test of a 35-cm Diameter Xenon Ion Thruster," IEPC Paper 99-142, October 1999.
- [7] Polk, J.E., Anderson, J.R., Brophy, J.R., Rawlin, V.K., Patterson, M.J., Sovey, J.S., and Hamley, J.A., "An Overview of the Results from an 8200-Hour Wear-Test of the NSTAR Ion Thruster," AIAA Paper 99-2446, June 1999.
- [8] Polk, J.E., Kakuda, R.Y., Anderson, J.R., Brophy, J.R., Rawlin, V.K., Patterson, M.J., Sovey, J.S., and Hamley, J.A., "Validation of the NSTAR Ion Propulsion System on the Deep Space One Mission: Overview and Initial Results," AIAA Paper 99-2274, June 1999.
- [9] Anderson, J.R., Goodfellow, K.D., Polk, J.E., Rawlin, V.K., and Sovey, J.S., "Performance Characteristics of the NSTAR Ion Thruster During an On-Going Long Duration Ground Test," *Aerospace Conference Proceedings, 2000 IEEE*, Vol. 4, March 2000, pp. 123-148.
- [10] Patterson, M.J., Haag, T.W., Foster, J.E., Rawlin, V.K., Roman, R.F., and Soulas, G.E., "Development Status of a 5/10 kW Class Ion Engine," AIAA Paper 2001-3489, July 2001.
- [11] Sovey, J.S., "Characteristics of a 30-cm Diameter Argon Ion Source," AIAA Paper 76-1017, November 1976.
- [12] Curren, A.N. and Jensen, K.A., "Textured Carbon on Copper: A Novel Surface With Extremely Low Secondary Electron Emission Characteristics," NASA TP-2543, 1985.
- [13] Rawlin, V.K., Patterson, M.J., and Becker, R.A., "Thermal Environmental Testing of NSTAR Engineering Model Ion Thrusters," IEPC Paper 97-051, August 1997.
- [14] Weast, R.C. (Editor), CRC Handbook of Chemistry and Physics, CRC Press, Inc., Cleveland, OH, 1977.

[15] Andritschky, M. and Teixeira, V., "Residual Stress and Adhesion of Molybdenum Coatings produced by Magnetron Sputtering," *Vacuum*, Vol. 43, Nos. 5-7, 1992, pp. 455-458.

[16] Klabunde, F., Lohmann, M., Blasing, J., and Drusedau, T., "The Influence of Argon Pressure on the Structure of Sputtered Molybdenum: From Porous Amorphous to a New Type of Highly Textured Film," *Journal of Applied Physics*, Vol. 80, No. 11, December 1996, pp. 6266-6273.

[17] Drusedau, T.P., Klabunde, F., Veit, P., and Hempel, T., "Investigations on Microstructure, Surface Topography, and Growth Process of Sputtered Molybdenum Showing Texture Turnover," *Physica Status Solidi, A: Basic Research*, vol. 161, No. 167, 1997, pp. 167-184.

[18] Patterson, M.J., Foster, J.E., Haag, T.W., Pinero, L.R., Rawlin, V.K., Soulas, G.C., Doehne, M.S., and Roman, R.F., "Next-Generation 5/10 kW Ion Propulsion Development Status," IEPC Paper 01-089, October 2001.

Table 1. Background information on the extended operation of ion engines.

	NSTAR Life Demonstration Test of a 2.3 kW ion engine	Extended Life Test of the Deep Space 1 flight spare ion engine	Deep Space 1 ion engine	Next Generation Ion Engine, 5 kW/10 kW
Reference	7	9	8	10
Approximate xenon throughput, kg	88	153 (as of 08-27-01)	60 (as of Aug. 2001)	550
Maximum thickness of coating on the discharge chamber wire mesh, $\mu\text{m}$ (composition)	5 (measured)  (Mo, Fe, Ni, Cr)	9 (estimated)	3 (estimated)	22 to 31 (estimated)
Maximum thickness of loose flakes in the discharge chamber, $\mu\text{m}$ (composition)	12  (Mo, Fe, Ni, Cr, Ta)	--	--	--
Evidence of spalled coatings on the anode causing high voltage shorts?	No	No	No	--

Table 2. Substrate and molybdenum coating characteristics using the RF sputter deposition system.

Substrate	Dimensions, cm	Mass gain after coating, g	Calculated coating thickness assuming bulk density of molybdenum, $\mu\text{m}$
Wire mesh - 4C	1.27 X 2.62 X 0.024	0.12607	37
5052 Al - 6C	1.22 X 2.58 X 0.061	0.11569	36
5052 Al - 7A	1.19 X 2.43 X 0.081	0.10121	34
4901 Ti - 4C	1.27 X 2.60 X 0.077	0.13014	39
4901 Ti - 6C	1.24 X 2.54 X 0.077	0.11775	37
4901 Ti - 7A	1.26 X 2.65 X 0.074	0.11526	34

Table 3. Substrate and molybdenum coating characteristics using the DC sputter deposition system.

Coupon	Planar area, cm <sup>2</sup>	Mass gain per coupon area, mg/cm <sup>2</sup>	Mass gain after coating, g	Calculated coating thickness assuming the bulk density of molybdenum, μm	Measured coating thickness on a silicon witness coupon, μm
Wire mesh - 1	3.74	8.1	0.0304	8.0	--
Wire mesh - 2	3.26	22	0.0716	22	22
Wire mesh - 3	2.99	10	0.0298	9.8	13
Wire mesh - 4	3.05	25	0.0770	25	Spalled coating
Wire mesh - 6	2.89	59	0.1705	58	Spalled coating
Wire mesh - 8-2	1.65	130	0.2139	130	Spalled coating

Molybdenum deposition rates ranged from 1.8 μm/hr to 3.3 μm/hr. Coupon thickness is about 240 μm.

Table 4. Typical parameters for the molybdenum sputter-deposition process.

Sputtering method	Pressure, Pa	Substrate temperature, °C	Estimated deposition rate assuming coating is 100% dense, μm/h
RF	1.3	--	5.1
DC	0.009 to 0.019	200 to 240	1.8 to 3.3
Typical operation of the ion engine	~ 0.02	~ 250 to 300	6×10 <sup>4</sup>

Table 5. Characteristics of coupons coated with molybdenum.

Coupon	Coating method	Estimated thickness of Mo coating, μm	Evidence of spalling after coating?	Change in coating mass after the thermal cycle test	Evidence of spalling after the thermal test?
WM-4C	RF sputter	37	No	< 0.8%	No
Al-6C	RF sputter	36	No	< 0.9%	No
Al-7A	RF sputter	34	No	< 1%	No
Ti-4C	RF sputter	39	No	< 0.8%	No
Ti-6C	RF sputter	37	No	< 0.9%	No
Ti-7A	RF sputter	34	No	< 0.9%	No
WM-1	DC sputter	8.0	No	< 1%	No
WM-2	DC sputter	22	No	< 0.3%	No
WM-3	DC sputter	9.8	No	< 0.7%	No
WM-4	DC sputter	25	No	< 0.7%	No
WM-6	DC sputter	58	No	< 0.6%	No
WM-8-2	DC sputter	130	No	< 0.2%	No

Table 6. Thermal cycle test parameters.

Test	Number of cycles	Low temperature, °C	High temperature, °C	Nominal cooling time, hours	Nominal heating time, hours
1	20	-60 ± 2	322 ± 3	3 to 4	0.22
2	20	- 60 to -54	297 to 340	2.3	0.25

Tests 1 and 2 used RF and DC sputtered coupons, respectively.

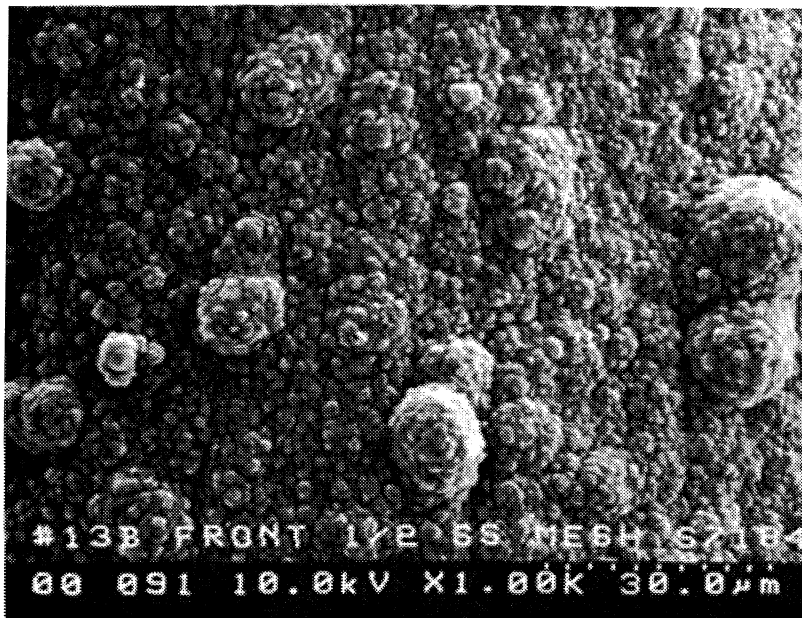


Figure 1a – Photomicrograph of the surface structure of a metal flake found in the discharge chamber of the 30 cm diameter ion engine that completed the 8200 hour Life Demonstration Test. (Courtesy of the Jet Propulsion Laboratory)

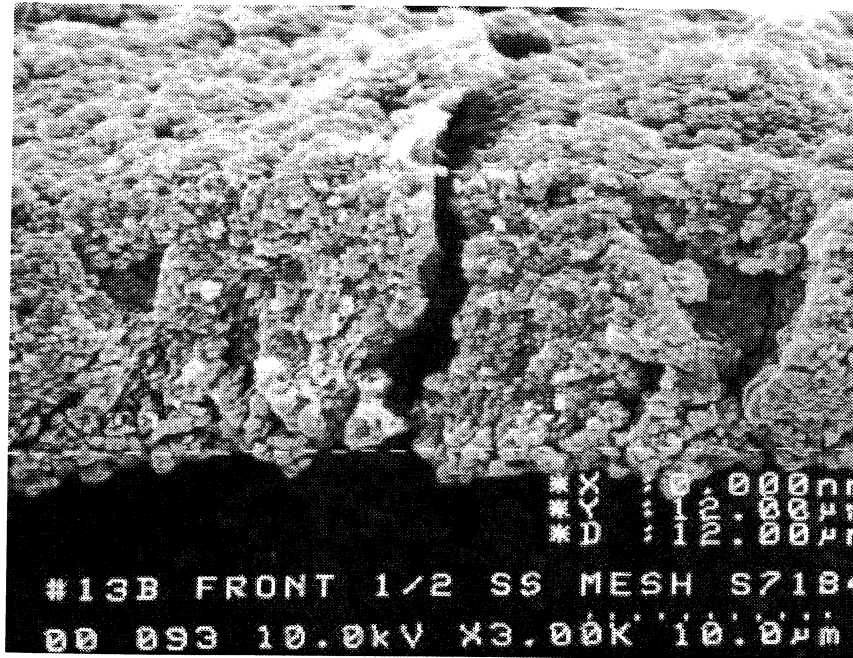


Figure 1b – Photomicrograph of the cross-section of a flake found in the discharge chamber of the 30-cm diameter ion engine that completed the 8200 hour Life Demonstration Test. Flake thickness is about 12  $\mu\text{m}$ . (Courtesy of the Jet Propulsion Laboratory)

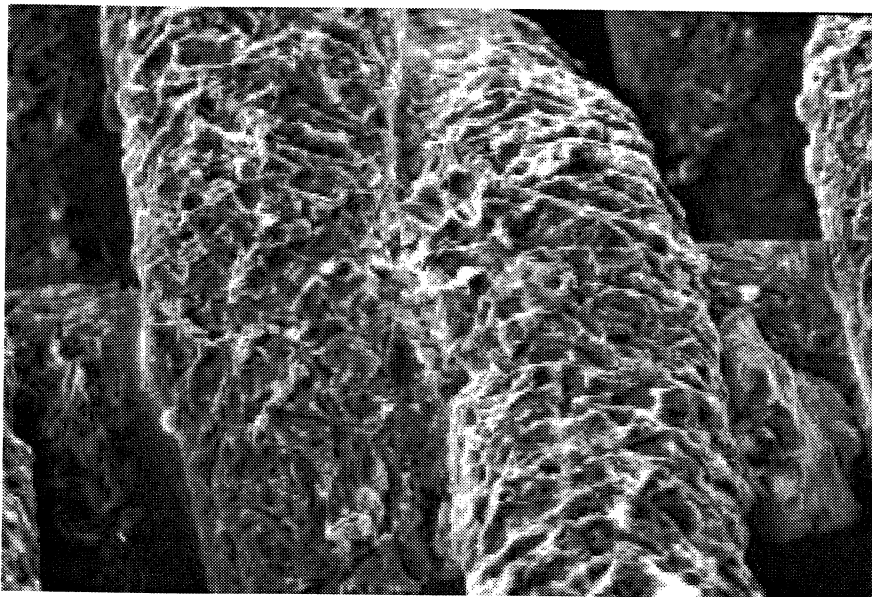


Figure 2 – Photomicrograph of grit-blasted wire mesh. The wire diameter is 51  $\mu\text{m}$ .

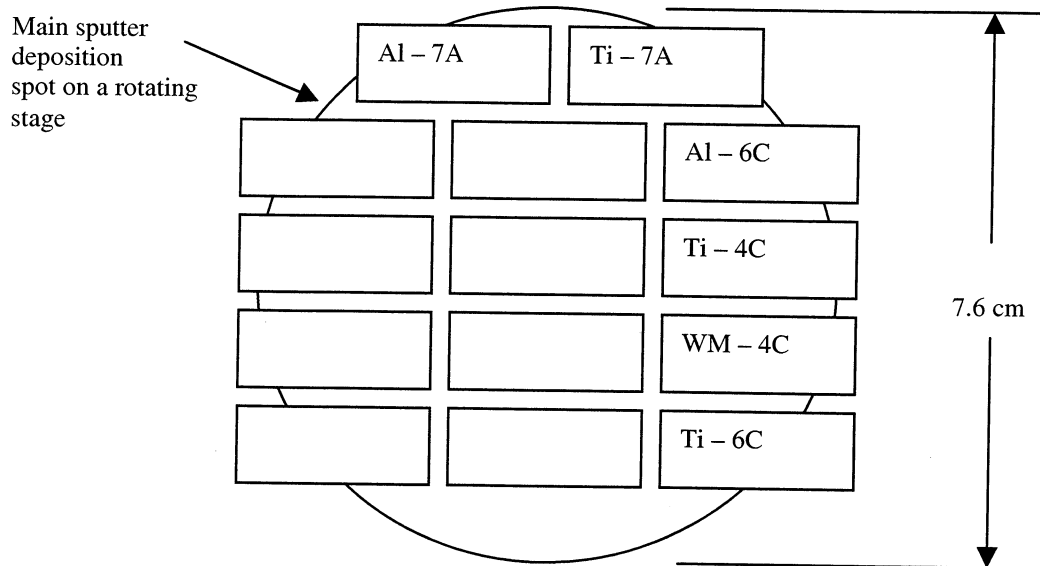


Figure 3 – Layout of coupons on the RF sputtering system stage.

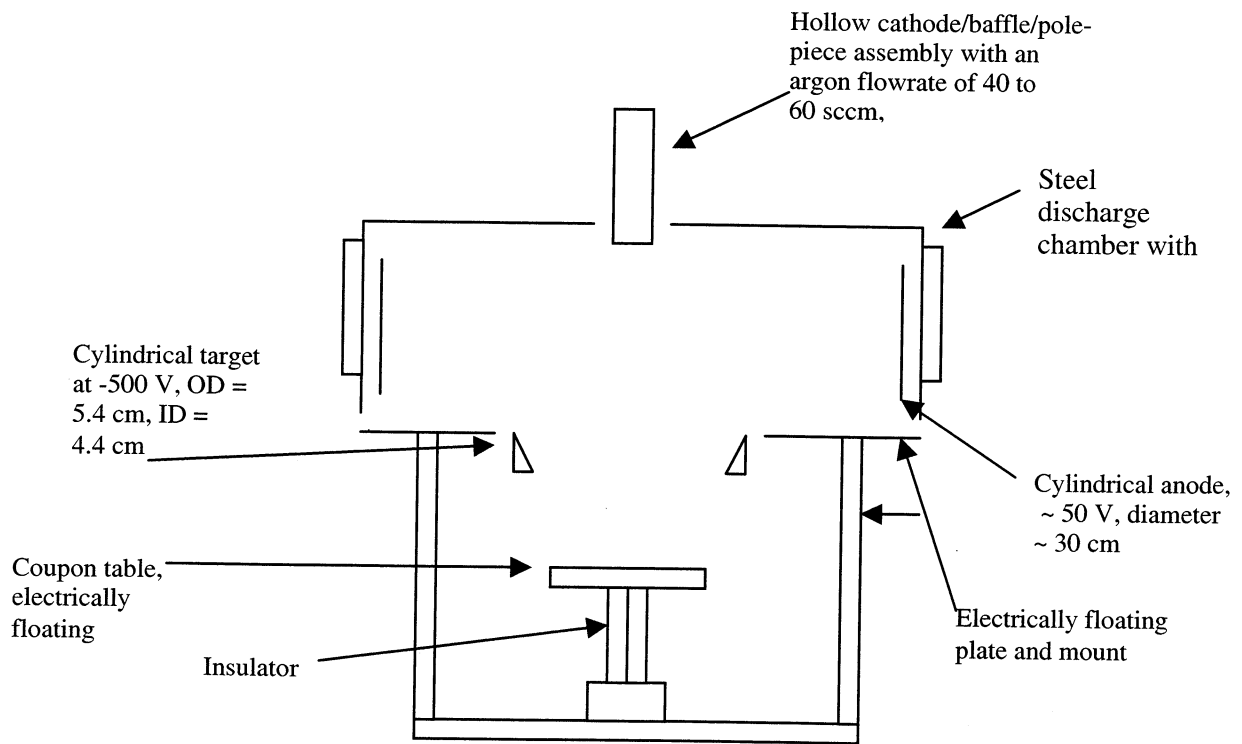


Figure 4 – DC sputtering hardware (not to scale).

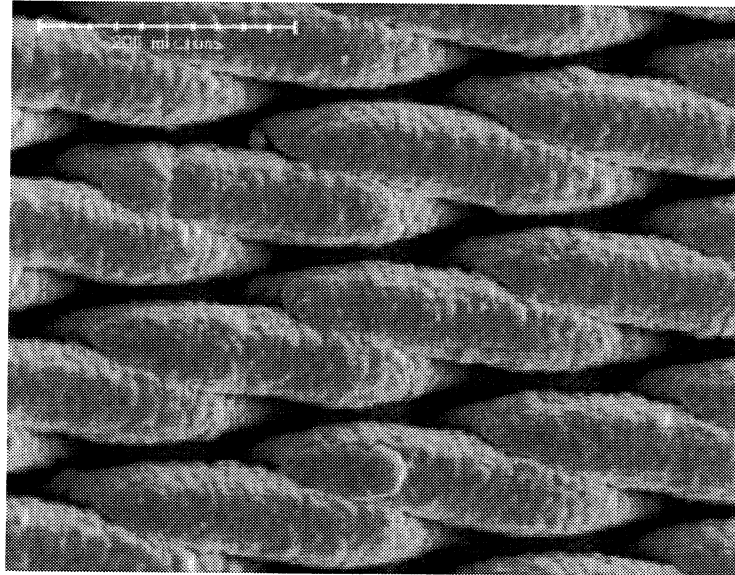


Figure 5 – Photomicrograph of the wire mesh with a 25  $\mu\text{m}$  thick molybdenum coating. The wire diameter is 51  $\mu\text{m}$  prior to coating.

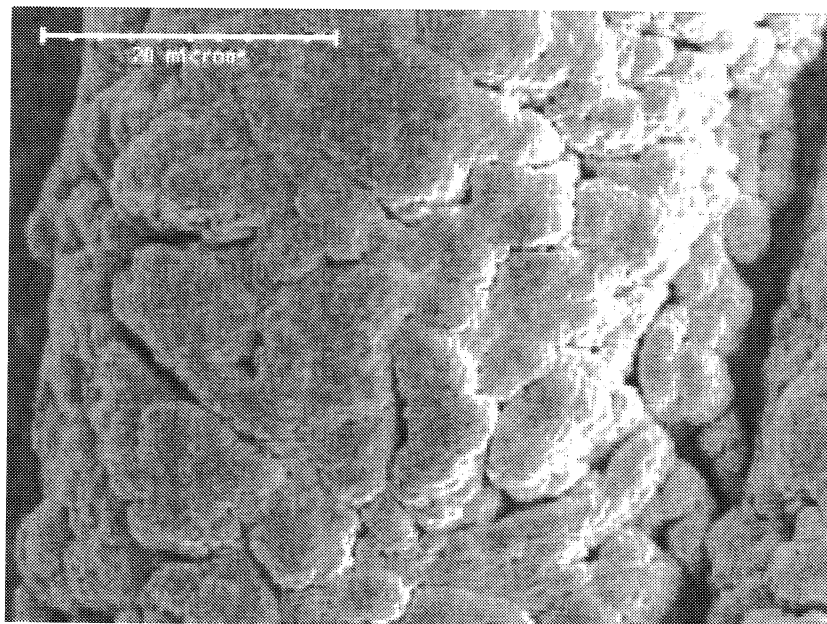


Figure 6 – Photomicrograph of a 51  $\mu\text{m}$  wire contained in the wire mesh with an 8  $\mu\text{m}$  coating of molybdenum.



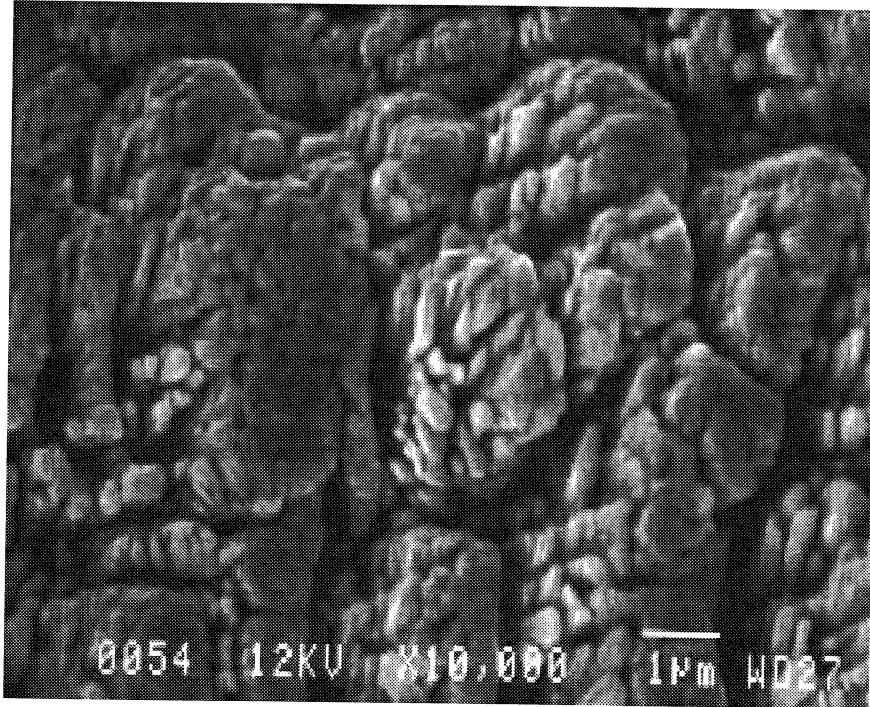


Figure 7 – Photomicrograph of a 51  $\mu\text{m}$  wire surface with a 58  $\mu\text{m}$  thick coating of molybdenum.

# REPORT DOCUMENTATION PAGE

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<b>13. ABSTRACT</b> ( <i>Maximum 200 words</i> )  Grit-blasted anode surfaces are commonly used in ion engines to ensure adherence of sputtered coatings. Next generation ion engines will require higher power levels, longer operating times, and thus there will likely be thicker sputtered coatings on their anode surfaces than observed to date on 2.3 kW-class xenon ion engines. The thickness of coatings on the anode of a 10 kW, 40-cm diameter thruster, for example, may be 22 μm or more after extended operation. Grit-blasted wire mesh, titanium, and aluminum coupons were coated with molybdenum at accelerated rates to establish coating stability after the deposition process and after thermal cycling tests. These accelerated deposition rates are roughly three orders of magnitude more rapid than the rates at which the screen grid is sputtered in a 2.3 kW-class, 30-cm diameter ion engine. Using both RF and DC sputtering processes, the molybdenum coating thicknesses ranged from 8 to 130 μm, and deposition rates from 1.8 to 5.1 μm/h. In all cases, the molybdenum coatings were stable after the deposition process, and there was no evidence of spalling of the coatings after 20 cycles from about -60 to +320 °C. The stable, 130 μm molybdenum coating on wire mesh is 26 times thicker than the thickest coating found on the anode of a 2.3 kW, xenon ion engine that was tested for 8200 hr. Additionally, this coating on wire mesh coupon is estimated to be a factor of > 4 thicker than one would expect to obtain on the anode of the next generation ion engine which may have xenon throughputs as high as 550 kg.				
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