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IONIZER SURFACE PROTECTION TECHNIQUES

By
C. R. Dulgeroff and D. E. Zuccaro

CONTRACT NAS 3-11527

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

HUGHES RESEARCH LABORATORIES
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ABSTRACT

A technique for applying a metallic coating to the surface of a porous tungsten ionizer of an ion thruster has been developed. The effect of a silver coating on the flow characteristics of the ionizer has been investigated. The technique was used on an operable ion engine and the effectiveness of the coating evaluated. A coating of 50 to 100 Å of silver did not provide the degree of protection desired.

SECTION I

SUMMARY

The objective of this program was to develop a procedure for applying a protective coating to the ionizer surface of a linear strip cesium contact ion thruster. The coating was to protect the ionizer surface during ground handling and testing. After serving its purpose, the coating should be removable in a flight environment using equipment that is integral to the thruster.

This program was divided into two tasks. Task I undertook the investigation of the selection and deposition of the coating. Three metals were considered as possible coating materials (silver, gold, and copper). Silver was chosen as the material for this study because of its high sputtering rate and ease of removal from the ionizer. Simple dc sputtering with magnetically confined electrons and an argon atmosphere was used. Task I studied the gas flow characteristics of ionizer material as a function of silver coating thicknesses. The ionizer could be sealed with a coating about 20 μm thick. It was found that after the silver was removed from the ionizer, the flow characteristics of the ionizer were the same as those prior to the coating. During the removal process, the silver formed small balls on the ionizer surface. This large amount of silver was considered hazardous in an actual engine configuration, so that thicknesses of 100 Å were used in Task II.

Task II was an extension of Task I to the linear strip engine. The sputtering technique was developed so that a protective coating could be placed on an ionizer surface without removing the ion engine from the vacuum chamber after the ion engine had been operated. In addition to the development of the technique, the effectiveness of the

silver coating was evaluated. An all-metal ion pumped UHV chamber was used for the evaluation. An ion thruster was operated until the ionizer displayed clean characteristics as determined by the critical temperature and neutral flux measurements. A silver coating of approximately 100 Å was sputtered on the ionizer and then evaporated. The silver coating did not affect the ionizer performance. A silver coating of approximately 100 Å was again placed on the ionizer, and this time the ionizer was exposed to air for three days. The thruster was then placed in the vacuum chamber and the silver removed. The engine was operated and critical temperature and neutral flux measurements were made. It was revealed that the critical temperature had increased by about 150°K. It was concluded that the investigation of the coating effectiveness was too limited to rule out the use of silver as a protective coating.

SECTION II

INTRODUCTION

An important problem in the development of contact ionizer thrusters is that of maintaining the "clean" surface conditions observed during laboratory operation of the thrusters. When the clean porous tungsten is exposed to atmosphere for modifications or shipment to the launch site, the ionizer immediately develops an oxide surface. Because of the higher critical temperature of the oxide surface, the thruster has different operating parameters until the oxide is desorbed. The goal of this program was to develop a method for applying a protective coating to the ionizer surface while the thruster is still in the vacuum system. The coating was to be removed by evaporation. Both the procedure and materials used in this investigation had to be compatible with a complete linear strip thruster. This report covers the results of two main tasks. The first task was devoted to the investigation of the effects of a silver coating on the flow characteristics of a tungsten ionizer. The silver was dc sputtered in an argon atmosphere. The second task was devoted to applying this technique to a fully assembled thruster. In an additional effort, the coating process was evaluated in terms of ionizer performance.

SECTION III

DEVELOPMENT OF A SPUTTER COATING TECHNIQUE

A. INTRODUCTION

The objective of Task I was to develop a technique for applying a protective coating to the surface of a porous tungsten ionizer. The coatings selected were: silver, copper, or gold. It was sepcified that the coating be applied by argon ion sputtering. The resultant coating was to be removed by heating the ionizer in vacuum with ionizer heaters used in the ion thruster. The goal was to obtain a coating which totally sealed the ionizer surface (i.e., reduced the transmission of gas to "zero" value).

Porous tungsten ionizer assemblies, of the type used in the model LE thruster, were used in this program. The transmission was measured by means of a nitrogen gas flow system.

A successful sputter cleaning and deposition technique was developed. Silver coatings up to 20 μm thick were deposited on the surface of the porous tungsten. The nitrogen transmission was reduced to 3% of the original value. To completely seal the ionizer would require much thicker coatings. Since the primary purpose of sealing the ionizer was the elimination of the propellant valve, it did not seem feasible to use this approach in view of the complexity involved in the removal of massive coatings at lower temperatures over a long period of time. Therefore, it was concluded that the technical objectives of Task I would be best satisfied by the development of an alternative protection scheme.* This involved the deposition of a thin

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*Technical directive from H. R. Hunczak, NASA LeRC, reference 9723-HRH, 18 June 1969.

(approximately 100 Å thick) silver coating over the ionizer to protect the surface from oxidation on exposure to the atmosphere. This was accomplished, and Task I was completed.

B. BACKGROUND

This section summarizes the technical background of the ion sputter deposition techniques and the gas transmission measurement. Its purpose is to provide a basis for the selection of a specific argon ion sputtering process. The physical details of the apparatus used in this program will be discussed in Section III-C.

1. Sputter Cleaning and Coating Technique

A typical procedure for cleaning and coating the ionizer is presented in Attachment I of Contract NAS 3-11527. This procedure is as follows:

"Place the ionizer assembly in a vacuum chamber equipped with a liquid nitrogen cold trap, and with the ionizer surface 2 inches from the ground plane. Evacuate the chamber and hold a vacuum, $< 10^{-6}$ torr for twenty (20) minutes. Be sure that the liquid nitrogen cold trap is activated at all times when an ionizer is in the chamber. Admit argon gas to the chamber to raise the pressure and establish the discharge on the ionizer surface at 2.0 kilovolts and 0.9 to 1.0 milliamperes for 20 minutes. Remove high voltage, ground the ionizer terminal and uncover the gold slug. Adjust argon pressure and establish discharge on the gold slug at 3.0 kilovolts and 0.9 to 1.0 milliamperes for two (2) hours. Remove the high voltage, vent the chamber to one atmosphere with nitrogen and inspect ionizer coating for uniformity."

A number of parameters determine the thickness of the deposit:

1. the target material
2. the gas used to form the plasma
3. the gas pressure
4. the diode (target-substrate) distance
5. the diode voltage
6. the diode current
7. the deposition time.

Other factors (such as the target temperature, the substrate temperature, and the character of the substrate surface) have a very limited influence on the deposition thickness. Several ion sputtering techniques can be applied. These are described below, following a brief discussion of the reasons for the selection of the target metals and the argon gas for the plasma.

The use of the coating materials to silver, copper, and gold is based on the following considerations. These metals are relatively inert with respect to chemical reactions with the atmospheric gases and with the porous tungsten. Their vapor pressure is sufficiently high that they can be completely removed from the porous tungsten by heating the ionizer to operating temperatures with the heaters used in the thruster's operation. An additional advantage is that these metals have the highest sputter yield of any of the elements when bombarded by argon ions. This is shown in the Fig. III-1, which is a graph of sputter yields for argon ions. Silver is preferable because it has a higher vapor pressure than the other two metals. The same vapor pressure is found at a 275°K higher temperature for copper and a 350°K higher temperature for gold.*

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*R.E. Horing, "Vapor Pressure Curves for the More Common Elements," RCA Laboratories, 1967.

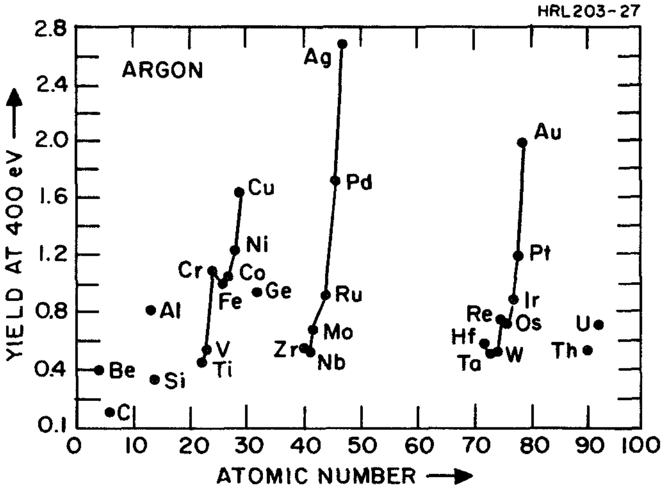

Fig. III-1.
Sputtering yields at 400 eV argon ion energy for 28 elements versus the element's atomic number (N. Laegreid and G.K. Wehner, J. Appl. Phys. 32 365 (1961)).

The choice of gas used to form the plasma is limited to the inert gases. Argon is by far the most abundant and least expensive, so that most of the previous studies have been performed with argon. Although the sputter yield is slightly greater for the more massive ions (i.e., krypton and xenon), the difference in the yield is not large enough to warrant the use of either of the other gases. For similar reasons, helium and neon are not used because of the low sputter yields.

The oldest and simplest sputtering method is the dc diode, shown in Fig. III-2(a). A stable plasma is formed between the target (cathode) and the substrate (anode) when the electrical field and the gas pressure are sufficient to cause collisional ionizations. For diode voltages of about 3 kV and with diode distances of the order of 5 cm, a gas pressure of about 500 μm is needed to sustain the plasma.

This would be a very inefficient method of coating the ionizer because the mean free path in the argon plasma is extremely short, resulting in scattering of the sputtered metal atoms and thus a very poor deposition rate. The mean free path L of a gas* in cm is given by the following expression:

$$L = 2.33 \times 10^{-20}\ T/P_{Torr}\ \delta^2$$

where T is the gas temperature in ^oK, P_{Torr} is the pressure in Torr, and δ is the atomic or molecular diameter (for Ar, $\delta = 3.67 \times 10^{-8}$ cm). If we estimate the gas pressure as 500 μm = 5×10^{-1} Torr and the temperature as 500^oK, the mean free path is

$$L = 2.33 \times 10^{-20} \times 5 \times 10^2 / 5 \times 10^{-1} \times (3.67 \times 10^{-8})^2 \doteq 0.018 \text{ cm}$$

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*S. Dushman, Scientific Foundations of Vacuum Technique, 2nd ed., J.M. Lafferty, Ed. (Wiley, New York, 1962).

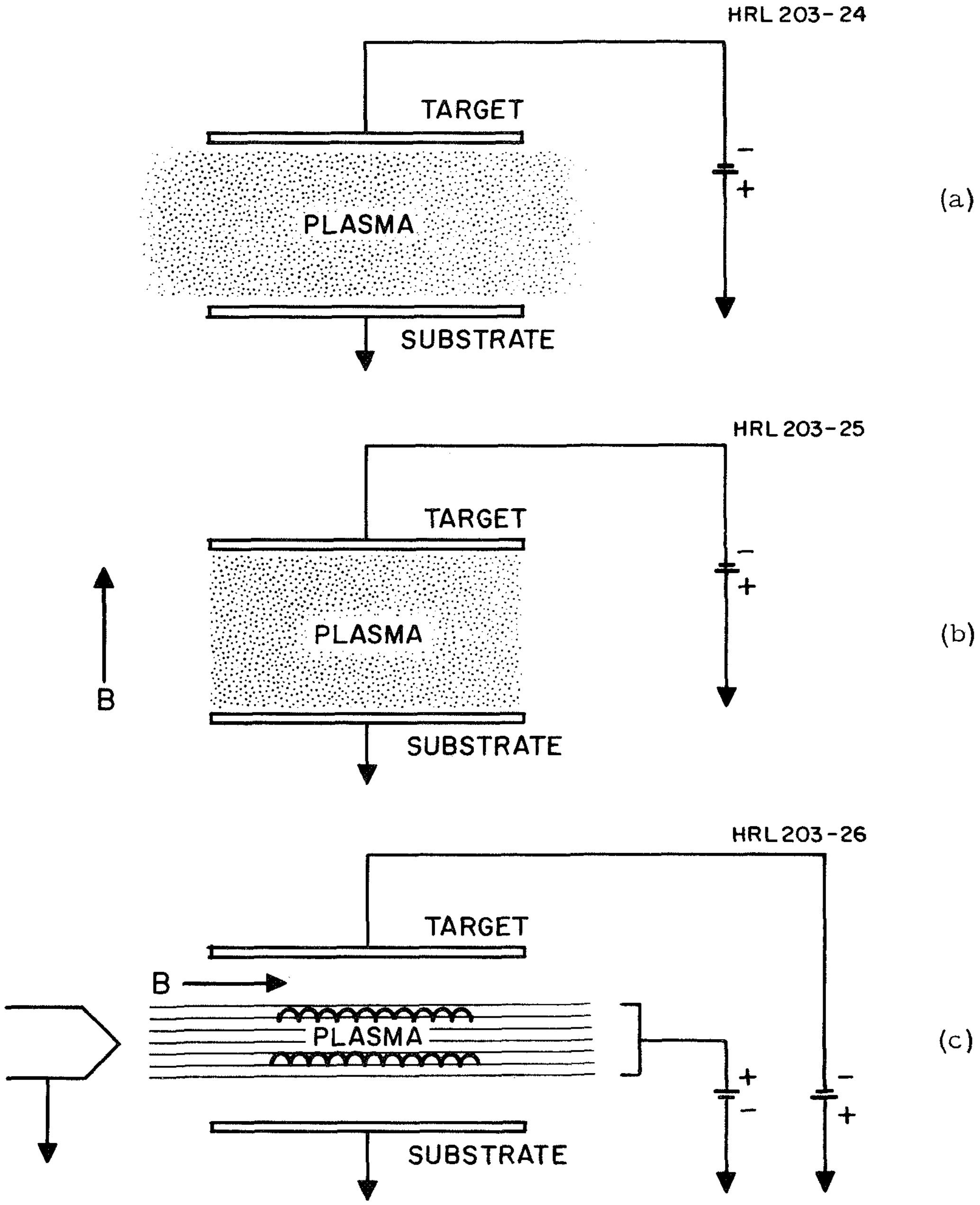

Fig. III-2. Types of plasma sputtering systems. (a) Simple diode. (b) Magnetically confined diode. (c) Triode or filament supported plasma.

The significance of this is that there will be considerable backscattering of the sputtered atoms, general scattering of the sputtering atoms, and collisional ionization of the sputtered atoms. These three loss mechanisms will mean that only a very small amount of metal will be deposited on the substrate. In addition, as a result of the high argon pressure, considerable argon gas will be trapped in the deposited metal.

The operating pressure of the diode can be lowered considerable by confining the plasma with an axial magnetic field, as shown in Fig. III-2(b). The magnetic field confines the electrons (rather than the ions) and thus stabilizes the plasma at a lower pressure. Depending on the electric field, this system can operate at pressures down to about 10 μm, where the mean free path in argon at $500^{\circ}K$ is 0.34 cm.

In addition to the magnetic confinement, it is possible to use rf excitation to generate a stable plasma at a lower pressure. Because the scattering is reduced, the efficiency is increased; this type of system is essential if the target material is a dielectric. The rf supported discharge can operate at pressures of 0.3 to 1 μm.

The triode or filament supported plasma type sputtering system is the remaining major type; it is also shown in Fig. III-2(c). Electrons, emitted by the cathode, produce the ions responsible for the sputter deposition. This system will operate to pressures of 0.1 μm.

The magnetically confined diode sputtering technique was selected for the following reasons. The simple diode system was ruled out because of the low efficiency resulting from the high gas pressure. The rf supported plasma and the triode cannot be applied to a fully assembled thruster and thus could not be considered for this development program. The magnetically confined diode system was chosen because of its greater efficiency.

Studies of the sputter coating of porous tungsten ionizers were performed previously under Contract NAS 3-6270; they are summarized briefly herein because the information is applicable. Rhenium and iridium metal were sputter deposited on Mod E porous tungsten in a coating about 1.5 μm thick. The pore size of this material is in the range of 3 to 4 μm. The 1.5 μm coating did not change either the nitrogen gas transmission or the cesium transmission. The ion emission parameters of the coated surface were characteristic of either the rhenium or the iridium metals. Photomicrographic analysis showed that the sputtered coatings had "piled" on the tungsten and did not seal the pores in the ionizer surface. From these studies it is concluded that a coating several times as thick as the pore size (e.g., 10 to 20μm) is needed to seal the ionizer surfaces.

In order to evaluate the results of the coating experiments, it is necessary to determine the transmission characteristics of the ionizer. For this program the reduction in transmission of the coated ionizer was expressed as the percentage of pores that are closed. This involves the measurement of the time interval for a specified pressure decrease in a constant volume of nitrogen (at a specified temperature). Accurate and reproducible measurements are possible using this technique which was developed under Contract NAS 3-6271.

C. EXPERIMENTAL PROGRAM

The experimental program involved the design, fabrication, and assembly of the sputter deposition apparatus, the nitrogen gas transmission measuring system, and the coating removal system. This section discusses the details of the experimental apparatus and the modifications made during the program.

The magnetically confined plasma diode sputtering apparatus was set up in a 6 in. diameter pyrex glass pipe vacuum chamber. The glass pipe was chosen because (1) it is inexpensive, (2) it allows the operator to view the entire system (which is important in an experimental sputtering operation), and (3) it permits the use of external Helmholtz coils to produce a magnetic field. This system is shown in Figs. III-3 and III-4. A cross sectional view and the details of the

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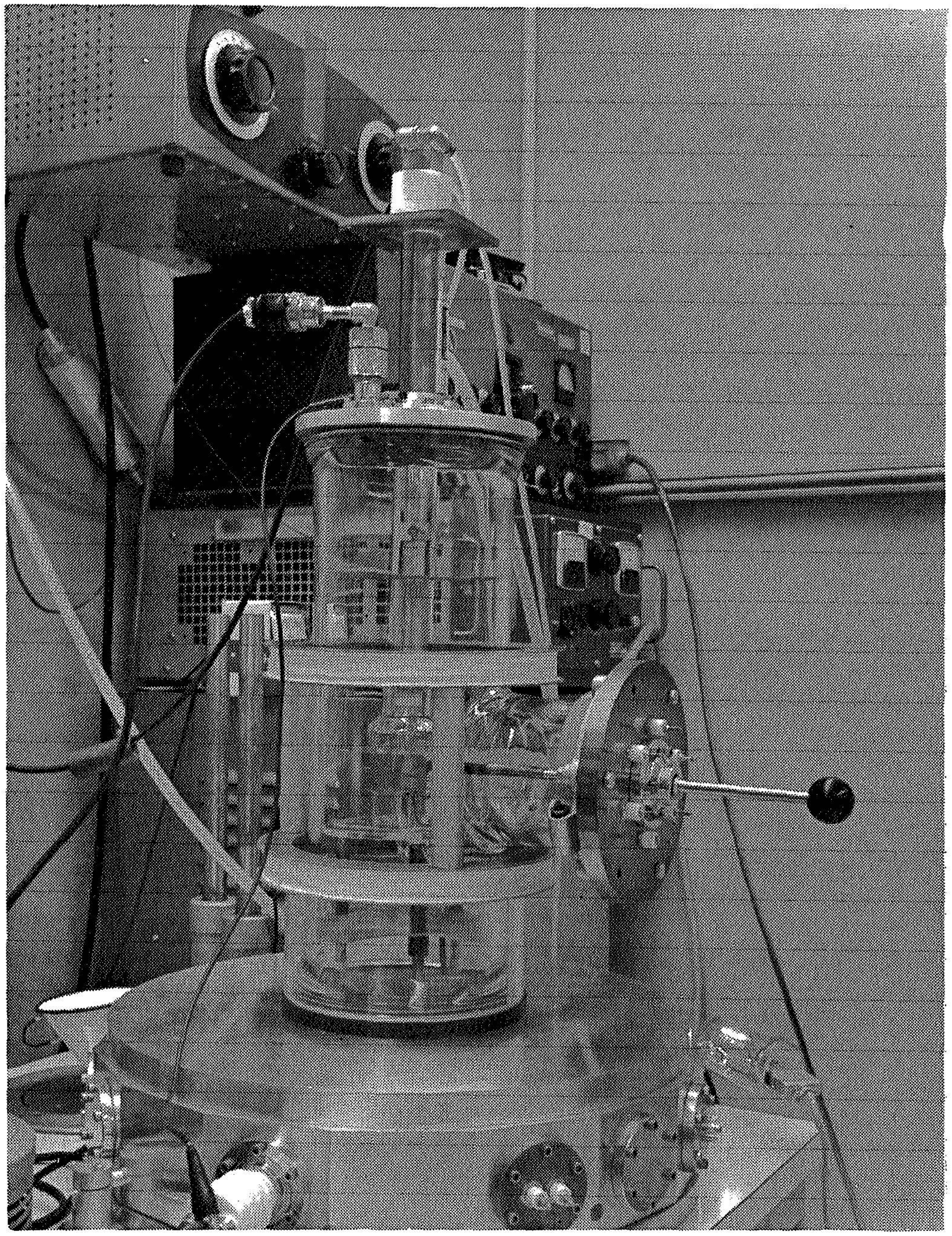

Fig. III-3. Complete sputter deposition system.

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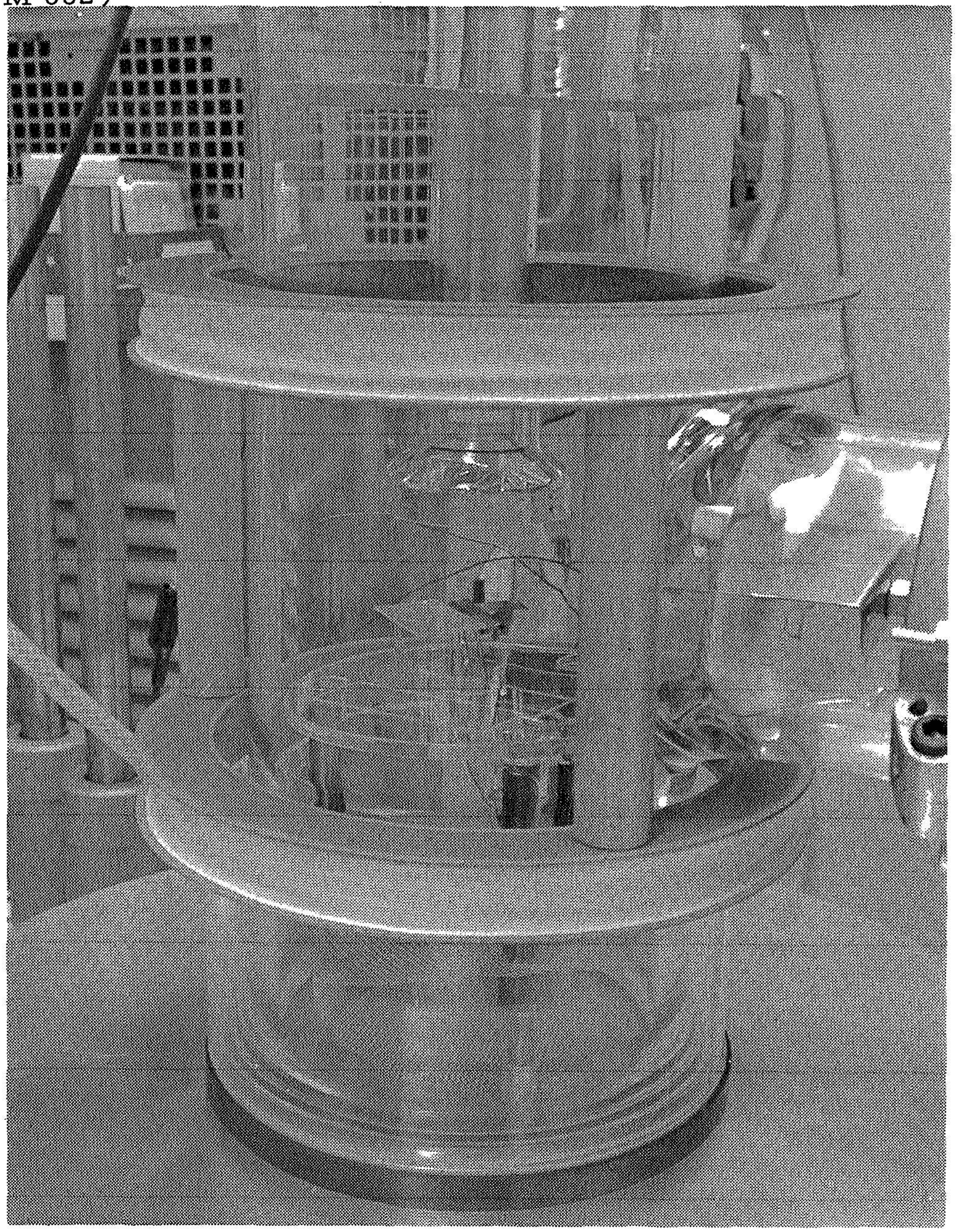

Fig. III-4. View of target and substrate area in sputter deposition system.

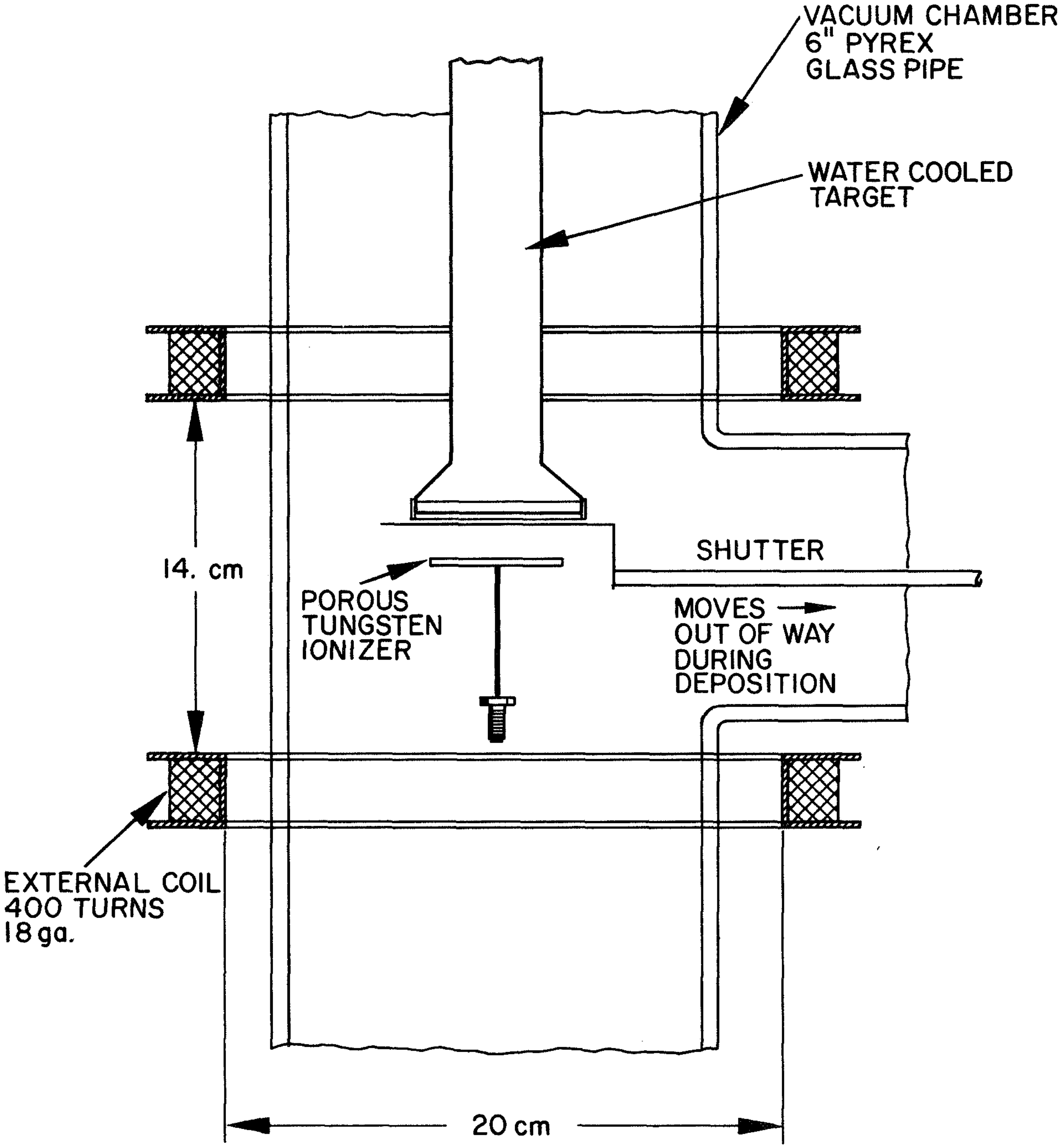

Fig. III-5. Drawing of details shown in Fig. III-4.

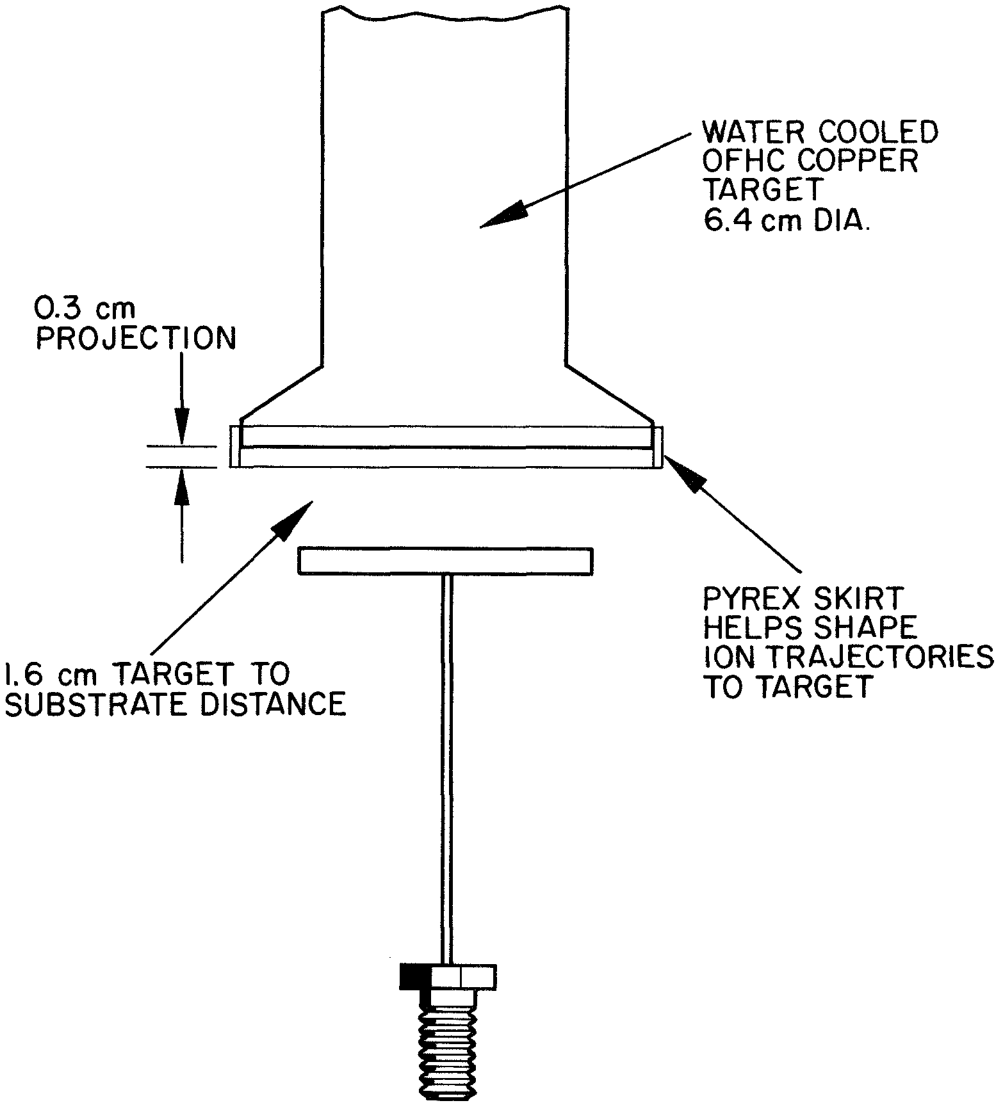

Figure III-6. Detail of sputter target and substrate (ionizer).

magnetic field coils and the diode are shown in Fig. III-5, and the detail of the diode (with the spacing used in runs 3 through 8) is given in Fig. III-6.

The target consisted of a 6.3 cm diameter water-cooled OFHC copper post over which metal foils were mounted. The foils were 99.99% pure metal (silver, gold, or copper). A glass skirt was placed around the edge of the target to help confine the plasma and thus increase the deposition rate. The target post was constructed with a vacuum seal that allowed it to be positioned at any specified distance from the ionizer (or substrate).

A stainless steel plate was used to mask the target during the sputter cleaning of the ionizer surface. It was mounted on a sliding vacuum seal that in a vacuum flange plate attached to a side arm to the glass pipe. This is seen in Figs. III-3 and III-5. When the sputter cleaning process was completed, the mask was pulled back (after the potentials were off) and the coating was sputter deposited after the diode potentials were reversed.

The substrate (the porous tungsten ionizer) was mounted on a glass plate below the target. The electrical leads to the substrate were shielded with glass and ceramic tubing to prevent discharge to any other part of the chamber. In order to measure the film thickness, a glass microscope slide with a cover glass as a mask was placed next to the substrate; it accumulates a metal coating equal to that deposited on the ionizer. After the run, the step height on the slide (i.e., the edge where the mask was located) is measured by means of an interferometer.

The vacuum pressure was measured with a Bayard-Alpert type ionization gauge tube. The argon pressure was measured with a thermocouple gauge (Hastings model DV5). The argon was a prepurified grade which is 99.998% pure. The flow rate was adjusted by means of a variable leak valve (Granville Phillips). The vacuum system is a standard 6 in. diffusion pumped station which uses DC705 fluid. The cold trap is a cold finger type.

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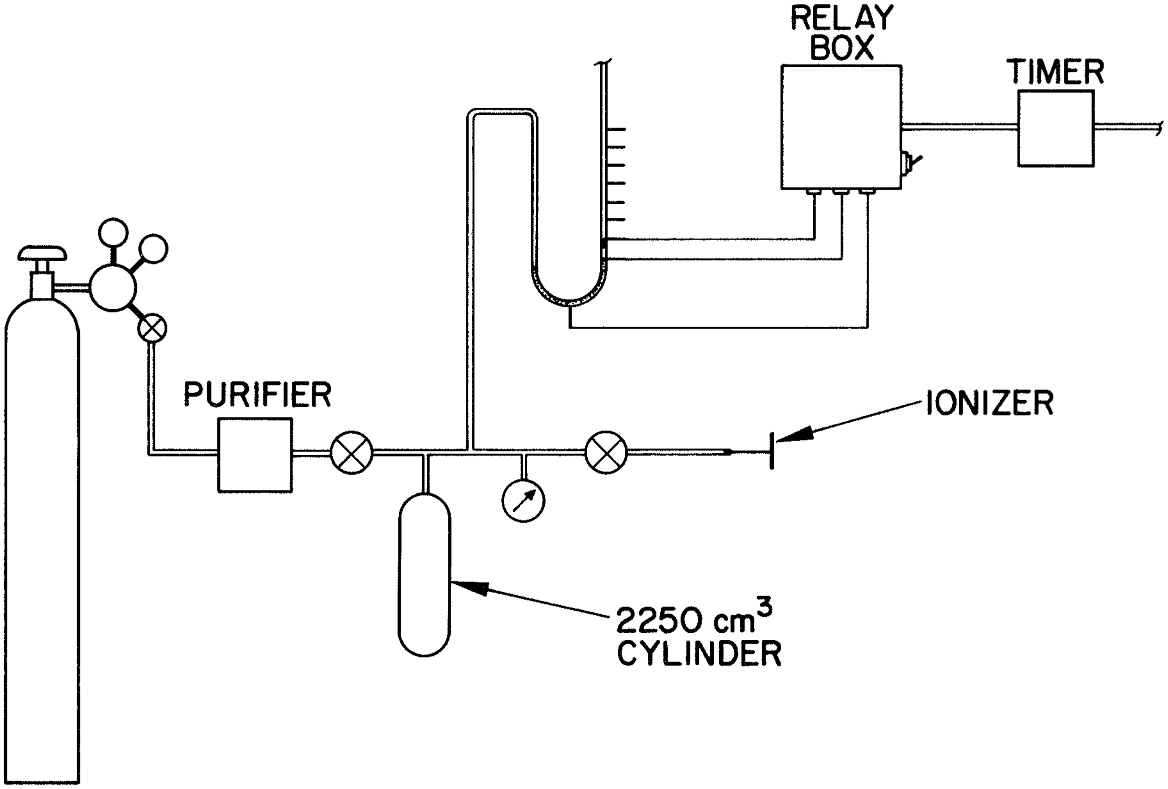

Fig. III-7. Mercury manometer with tungsten wire contacts for electronic timing.

The transmission measurements were made with the apparatus shown in Fig. III-7. Prepurified grade nitrogen gas (99.997% min; dew point = -85°F) was used in these measurements. The gas was passed through a purifier consisting of a drier and a filter. The drier produces gas with a dew point of -100°F, and the filter traps particles of 12 μm and larger. The mercury manometer was constructed with eight tungsten wire leads through the glass wall; the wires were placed about 5 cm apart. As the pressure decreased (and the mercury column fell), a relay circuit and a 6 V battery were used to start and stop an electronic timer; the time required for a specific pressure change was thus measured. The range of the timer was 0.1 to 1000 sec. An electric clock was used to determine the number of 1000 sec periods for longer decay times.

For evaporation of the coating the coated ionizer was mounted in a vacuum bell jar and heated with the heaters used in the ionizer when it is operated in the ion thruster. An optical pyrometer was used to measure the surface temperature. Following the removal of the coating, the ionizer was returned for the transmission tests.

D. RESULTS

A 99.99% gold foil was used as a target in run 1, in accordance with the specifications in the Statement of Work. A sputter deposition was made for the specified time, ion current, ion energy, and target-to-substrate distance. The operating conditions are presented in Table III-1. The resulting coating was much too thin, and was uneven from the end to the center of the ionizer. The target was changed to silver, which was used in all subsequent runs. The distance between the target and the substrate was reduced to 2.5 cm, and the argon plasma ion current to the target was increased to 3 mA.

These changes resulted in a 1.6 μm thick coating for run 2, representing about a threefold increase in thickness for a corresponding increase in the plasma ion current to the target. As a result, the target-to-substrate distance was again decreased, this time to 1.6 cm, where it was held for all subsequent runs. The plasma ion current was increased to 7 mA and the deposition time to 3 hours.

Run 3 thus represented a doubling of both the deposition time and the ion current. The increase in the silver film thickness to 6.75 μm is roughly proportional to that expected to result from the changes in the operating parameters.

The first three runs were used to establish the general relationship between the various operating parameters. The parameters that were kept constant during all eight runs were the diode voltage (2.0 kV during cleaning and 3.0 kV during deposition) and the magnet current (5.0 A during cleaning and deposition runs).

In order to obtain the maximum amount of data in the time available in this program, runs 4 and 5 were made by depositing a silver coating over the coatings deposited previously in runs 2 and 3, respectively. In run 4, ionizer number 2 (which had received a 1.6 μm silver coating in run 2) was sputter cleaned for 5 min (rather than the 20 min period used on the porous tungsten surface) at 2.0 kV and 0.95 mA. After this it was coated with a 9.2 μm layer of silver to produce a total of 10.8 μm. Gas transmission measurements indicated that about 33% of the surface was sealed, compared with 52% closure achieved in run 3 with a 6.75 μm deposit.

Run 5 consisted of a 15 min sputter cleaning of the previous deposit of 6.75 μm of silver. The plasma potential was 2.0 kV and the plasma current was 0.4 mA during the cleaning operation. Following this, an 8.1 μm layer was deposited over the previous run 3 coating of 6.75 μm. The transmission measurements showed that this combined layer had sealed 97% of the surface. This was the most successful of the coating experiments.

A single layer of 15.6 μm was deposited in run 6. This produced only a 60% closure of the surface, which was much less than had been expected. Photomicrographic analysis of the surface (Fig. III-8(a) was taken before the coating and Fig. III-8(b) was taken after the coating) failed to show any visible pores or defects. A bubble test was performed using chemically pure ethyl alcohol as a fluid. Gas flow was observed from the pores, most of them located near the ends of the ionizer.

Run 7 consisted of a single 20 μm deposit of silver on ionizer number 1. The gas flow tests indicated 90% closure of the pores. This was lower than expected based on the results of run 5. The ionizer was mounted in a vacuum system and heated gradually to evaporate the coating. At a brightness temperature of 980°C, the surface suddenly changed from a dull to a bright appearance as a result of the melting of the silver and its coalescence into a ball of about 0.15 cm diameter. It took about 15 min for the sphere to evaporate.

Based on this observation, it was decided that although it was possible to seal the surface with silver, the presence of a sphere of silver (formed in the removal process) could prove a serious problem in the thruster. For this reason heavy coatings received no further consideration. A second, and perhaps more important, application of the coating was to prevent the contamination of the surface of the porous tungsten by exposure to air. This could be accomplished by coating the surface with a thin (approximately 100 Å) coating which was attempted in the last run (number 9).

The difference between runs 5 and 7 may possibly be explained as follows. Run 5 underwent a mild sputter cleaning between the two depositions. This may have caused some silver to diffuse into the porous tungsten, thus increasing the sealing of the surface. This indicates the importance of testing the effect of substrate temperature on the degree of silver diffusion into the pores of the ionizer.

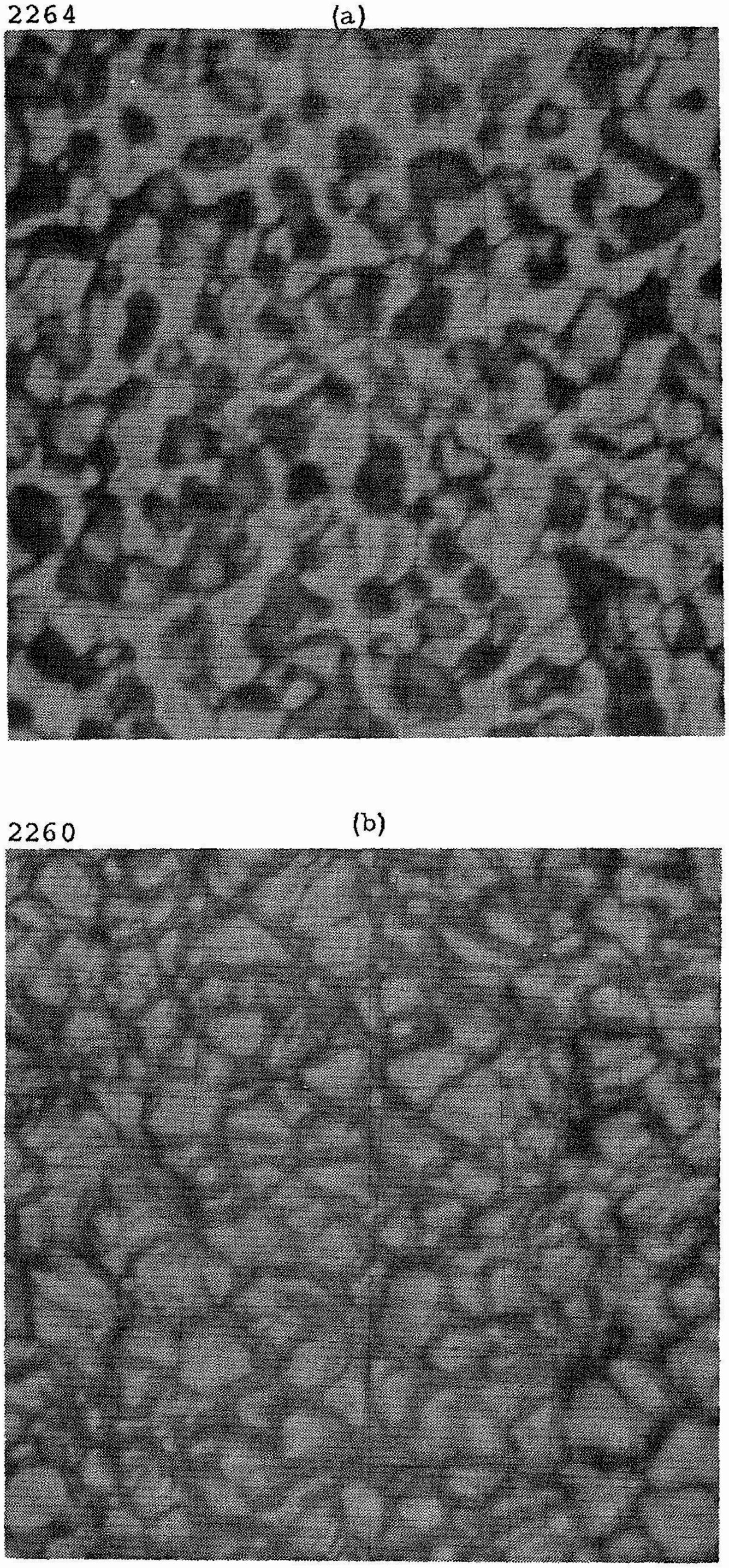

Fig. III-8. Photomicrographs of the surface (Run 6). (a) Before the silver deposition. (b) After the silver deposition.

E. CONCLUSIONS

A heavy coating can be used to seal a porous tungsten ionizer, but its effects can be detrimental to the thruster. The use of a light coating to prevent contamination by oxidation offers considerable promise.

TABLE III-1

Results of Sputter Deposition Runs

Run Number	Ionizer Number	N_2 *Transmission*[a] Before Test (t_1), sec	Target Material	Target-to-Ionizer Distance, cm	Vacuum System Pressure Before Run, Torr	Sputter Cleaning[b]			Sputter Deposition[c]			*Deposit* Thickness, µm	N_2 Transmission[a] After Coating Deposited (t_2), sec	N_2 Transmission[a] After Removal of Coating (t_3), sec	*Percentage*[d] of Closed Pores, %
						Pressure, µm	Current, mA	Time, sec	Pressure, µm	Current, mA	Time, min				
1	1	187.8	Gold	5	2.8×10^{-7}	10	1.2	20	13	1.0	120	0.3 to 0.6	190.3	194.0	1
2	2	196.8	Silver	2.5	5.9×10^{-7}	20	1.0	20	20	3.0	90	1.6	200.1	e	1.5
3	1	194.0	Silver	1.6	2.7×10^{-7}	11	0.4	20	38	7.0	180	6.75	400.1	f	52
4	2[e]	(200.1)[e]	Silver	1.6	4.5×10^{-7}	20	0.95	5	35	7.0	218	added 9.2 (total 10.8)	293.9	207.4	33
5	1[f]	(400.1)[f]	Silver	1.6	8×10^{-7}	12.5	0.4	15	38	7.0	210	added 8.1 (total 14.85)	6320.6	197.9	97
6	2	207.4	Silver	1.6	5.5×10^{-7}	14.5	0.4	20	35	7.0	342	15.6	514	g	60
7	1	201.9	Silver	1.6	4×10^{-7}	15	0.4	20	50 50	7 10	210 150	total 20	1998	204.6	90
8	1	204.6	Silver	1.6	5×10^{-7}	15	0.5	20	35	2	1	0.01	205.6	205.0	0.5

[a] N_2 transmission shown as time interval for a 50 Torr pressure decrease from an initial pressure of 169.5 Torr

[b] All sputter cleaning done at 2.0 kV potential and magnet = 5.0 A

[c] All sputter deposition done at 3.0 kV and magnet = 5.0 A

[d] The percentage of closed pores is given by $[(t_2 - t_1)/t_2] \times 100$

[e] *Ionizer 2 with run 2 coating used for run 4*

[f] Ionizer 1 with run 3 coating used for run 5

[g] This coating retained for further analysis

SECTION IV

DEVELOPMENT OF SPUTTERING TECHNIQUE FOR THE LINEAR STRIP THRUSTER

The main purpose of Task I was to evaluate the effects of the sputtered silver on the gas flow characteristics of the ionizer. Because complete sealing of the ionizer required considerable silver, representatives of Hughes Research Laboratories and NASA Lewis Research Center agreed that thicknesses of 50 to 100 Å should be used for the protective coating. It was believed that evaporating the large amounts of silver required for complete sealing of the ionizer could coat the high voltage insulators in the thruster and cause them to short out.

Task II was directed toward developing a method for sputter coating the ionizer of a complete thruster without breaking the vacuum after the thruster had been operated. The thruster configuration including electrodes was such that a redesign of the target as well as a quite different target to substrate separation was necessary to improve the target 'view' of the ionizer surface. Figure IV-1 shows a cross section of the strip engine geometry used on this program. The required sputtering parameters were determined by a series of experiments using a strip thruster assembly in a bell jar vacuum station. The thruster assembly was positioned in the bell jar with the ionizer facing up. Glass cover slides (0.006 in. thick) were set on top of the focus electrode so that the pattern and amount of sputtered silver could be observed.

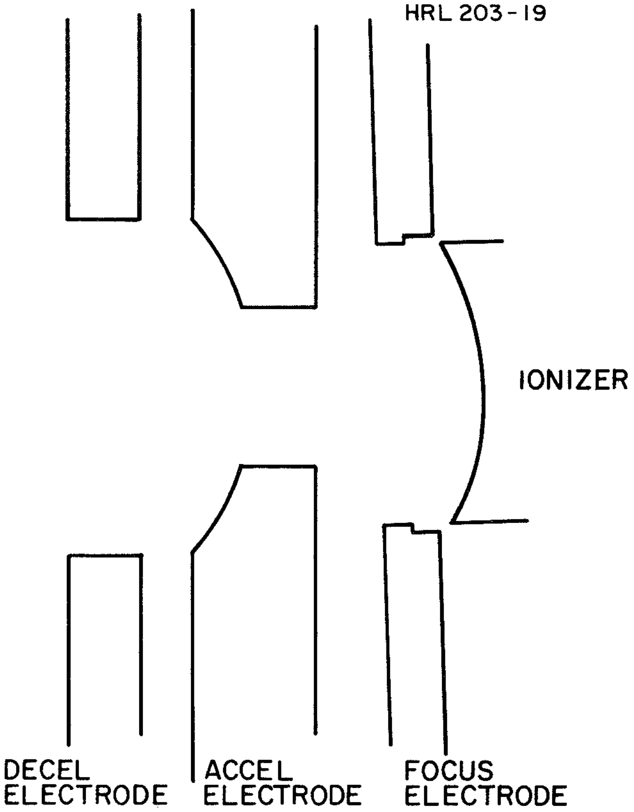

Fig. IV-1. Cross section of ionizer and electrodes.

The first tests used a 1/8 x 0.020 x 1.8 in. silver cathode located approximately 1 in. above the decel electrode. All parts of the thruster were grounded. Prepurified argon (99.998%) was bled into the chamber by means of a valve. Before any sputtering could take place all exposed parts of the lead that went to the silver cathode had to be either wrapped with teflon tape or covered with glass tubing. If this was not done, a plasma could not be formed at the cathode surface. The objective of these first tests was to obtain a stable plasma during the sputtering process. After this was accomplished, the patterns on the glass cover slides were studied. The best pattern (i.e., one that would indicate 100% coverage of the image) was obtained with the cathode in the vicinity of the accel electrode. A glass block eventually was used to keep the cathode centered with respect to the accel electrode, as shown in Fig. IV-2. For the geometry shown in Fig. IV-2, it was found that a stable sputtering plasma was obtained with -750 V placed on the cathode in an argon pressure of 350 μm. as determined by a thermocouple gauge. After these parameters were determined, several glass slides were sputtered as a function of time. The sputtering times were 60 sec, 30 sec, 15 sec, 7-1/2 sec, and 3 sec. These samples were covered with a thin layer of gold, and their thicknesses were measured with an interferometer. The results of the sample thickness measurements are shown in Fig. IV-3. Only the samples with exposure times of 15 sec or more could be measured. For shorter times the thicknesses were less than 200 Å and could not be measured by the interferometer.

Because the glass cover slides were not on the ionizer surface a correction factor was applied to determine the amount of silver that would be deposited on the ionizer. This correction factor was an approximation obtained by treating the silver cathode as a line source. This assumption yields the result that the deposition on the ionizer is inversely proportional to the distance from the silver cathode. The results obtained using this correction factor are shown in Fig. IV-4. It can be seen that a sputtering time of 5 sec should be used to obtain a thickness of approximately 100 Å. It is estimated that the error in the thickness measurement can be as large as 10%; i.e., a 5 sec exposure could give an actual thickness of 90 to 110 Å.

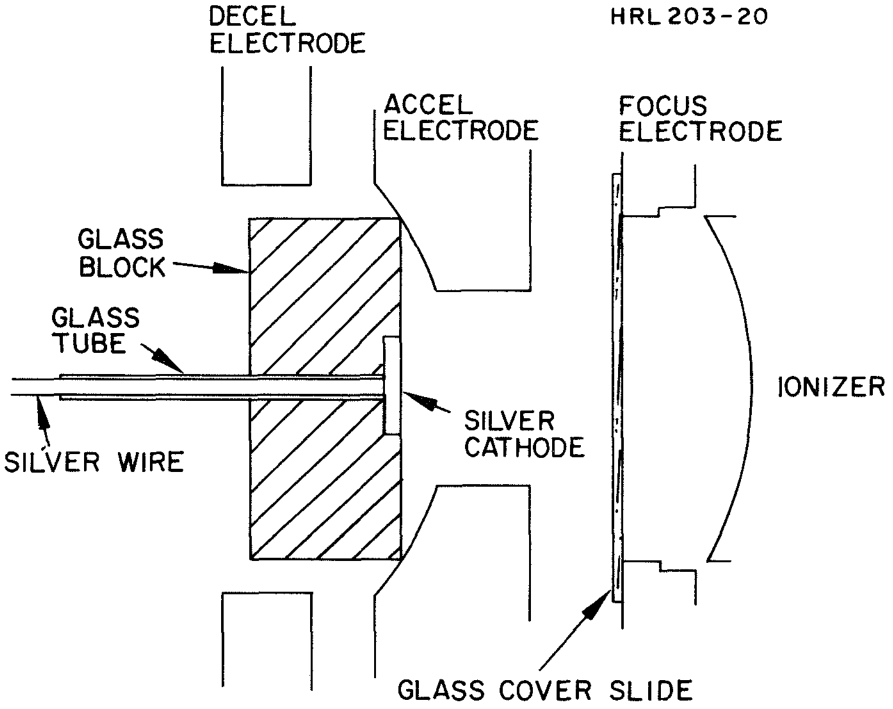

Fig. IV-2. Cross section of ionizer, electrodes, and cathode.

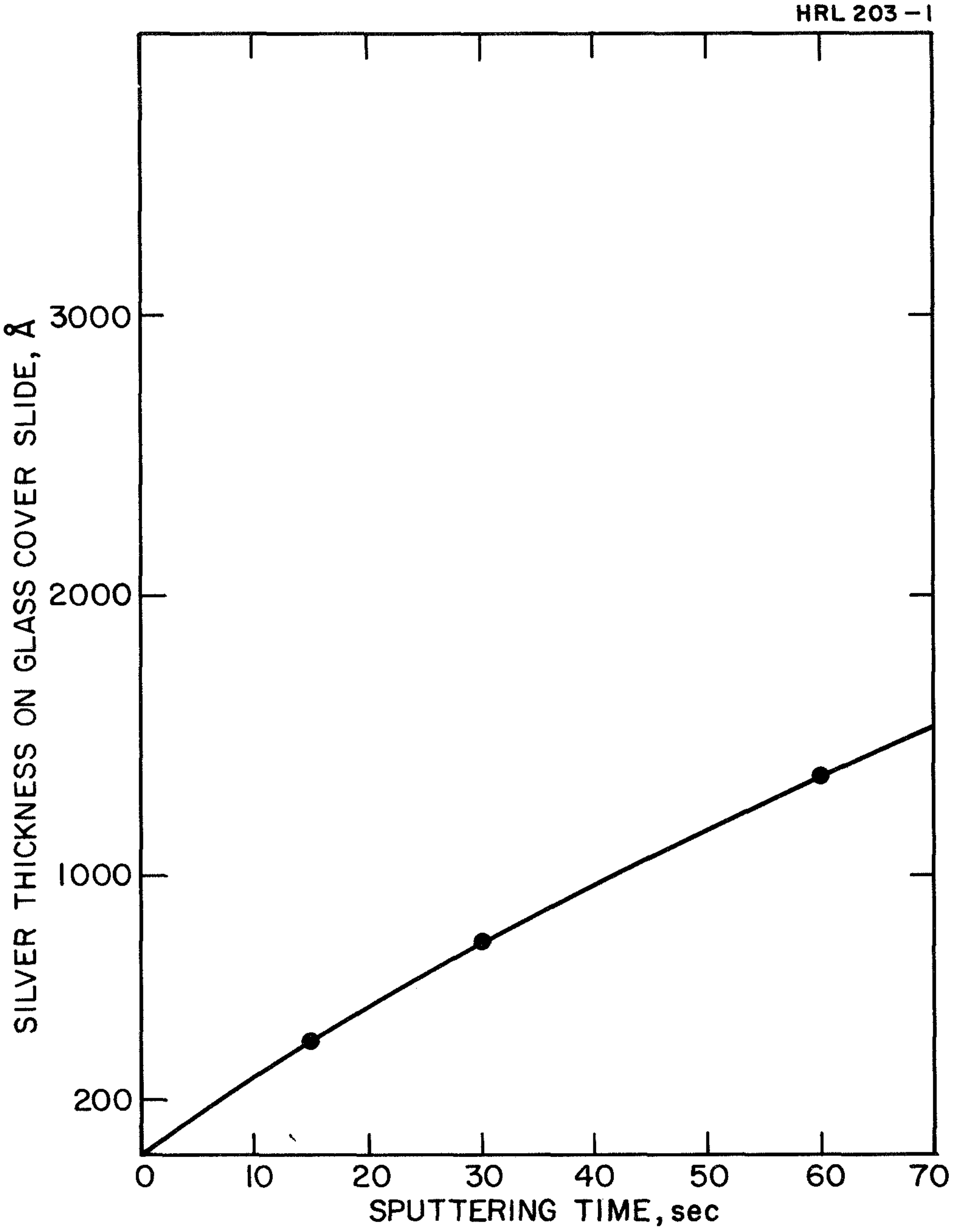

Fig. IV-3. Silver thickness on glass side versus sputtering time.

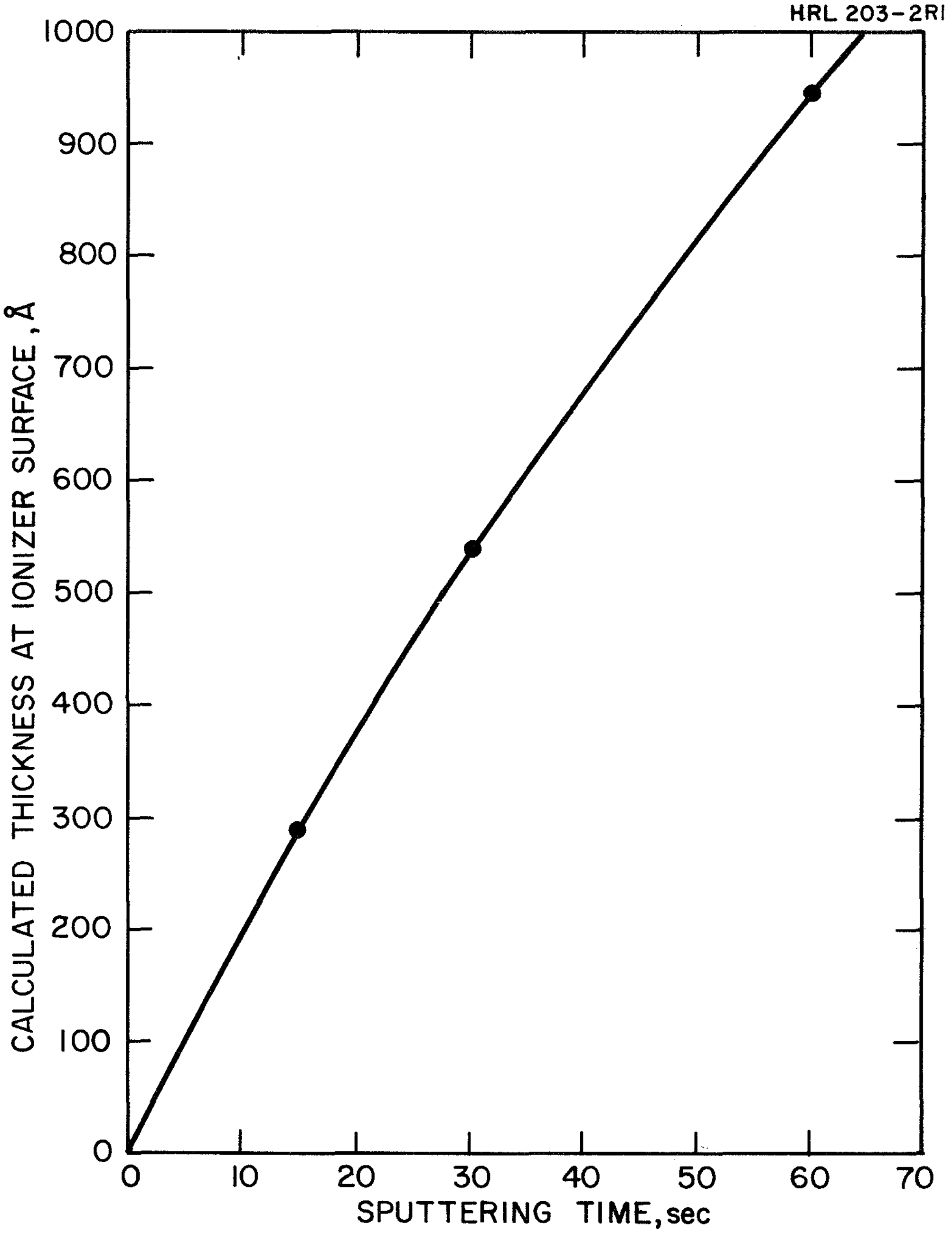

Fig. IV-4. Calculated silver thickness at ionizer surface versus sputtering time.

The above measurements were made at the center of the silver deposit. Examination revealed that the thickness across the glass slide of the 60 sec sputtering sample was fairly uniform. The thickness at the center of the deposit was 1350 Å, while that at the edge was 1200 Å; this is a variation of only 9%. A final sputtering test was made by placing a copper foil on the surface of the ionizer. Silver sputtered on the copper foil, covered it completely, indicating that the ionizer would be completely covered. Once the parameter for sputtering had been established, it was necessary to adapt the cathode so that it could be manipulated in the vacuum chamber without breaking the vacuum. A manipulator was constructed so that the cathode could be moved in and out of position by a rotatory magnetic coupling; see Fig. IV-5.

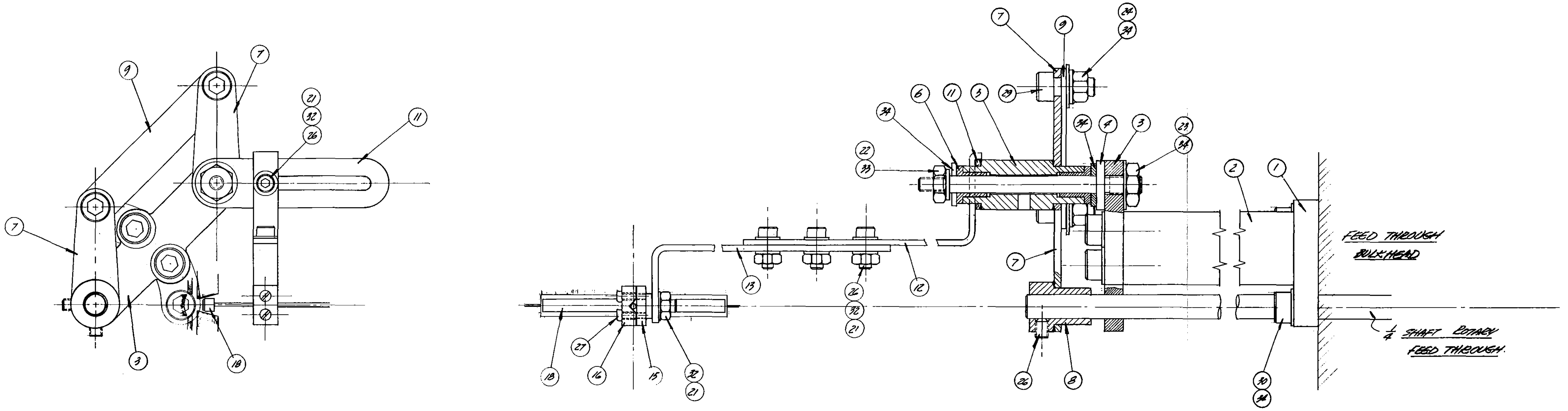

Fig. IV-5. Assembly of cathode and manipulator.

				35
9			WASHER - FLAT #10 CRES	34
1			WASHER - FLAT #6 CRES	33
9			WASHER - FLAT #4 CRES	32
				31
2			SCREW - HEX SOCKET - CAP 10-32 x 1½ LONG CRES	30
4			SCREW - HEX SOCKET - CAP 10-32 x 3/16 LONG CRES	29
4			SCREW - HEX SOCKET - CAP 6-40 x 5/32 LONG CRES	28
2			SCREW - SLOTTED - FILL HEAD 0-80 x 1/4 LONG CRES	27
2			SCREW - SOCKET - SET - FLAT PT 4-40 x 3/16 LONG - CRES	26
				25
2			NUT - SELF LOCKING - 10-32 CRES	24
1			NUT - HEX 10-32 CRES	23
1			NUT - HEX 6-32 CRES	22
5			NUT - HEX 4-40 CRES	21
				20
				19
1		1024950	ASSEM - SPUTTER SOURCE	18
				17
1		1024944	HOLDER - SOURCE	16
1		1024949	MOUNT -	15
				14
1		1024948	ARM - EXTENSION - MOUNT	13
1		1024947	ARM - EXTENSION	12
1		1024946	ARM - BUSHING	11
				10
1		1024943	LINK	9
1		1024942	BUSHING - ARM	8
2		1024941	ARM - CRANK	7
2		1024940	BUSHING - BEARING	6
1		1024939	BUSHING	5
1		1024938	SHAFT	4
1		1024937	PLATE	3
1		1024936	COLUMN	2
1		1024935	BASE	1

SECTION V

EVALUATION OF THE PROTECTIVE COATING

After the sputtering technique for the linear strip thruster was developed, the effect of the coating on the ionizer properties was investigated. This investigation used an ultrahigh vacuum chamber, single strip ionizer assembly, and a neutral particle detector.

A. APPARATUS

The apparatus used for this program was used several years ago on Contract NAS 3-6271 for the evaluation of high work function ionizers. This single strip thruster was designed for high perveance operation. The design perveance for the ionizer strip geometry was 41×10^{-9} A/V$^{3/2}$. The choice of the strip geometry permitted a large ionizer area to be seen unrestricted by electrodes when viewed from off-axis; this made it possible to use a neutral detector that viewed only the ionizer surface and thus had a high signal strength.

An all-metal copper-gasketed chamber (see Fig. V-1) of approximately 14 in. diameter and 40 in. length was used to appropriate an actual space environment. This chamber was bakable and ion pumped. The ionizer assembly was mounted at one end of the chamber; it can be seen from Fig. V-1 that the axis of the neutral detector is located 45° off the normal to the ionizer surface. A photograph of the chamber is shown in Fig. V-2; the left end shows the LN$_2$ feedthroughs for the ion beam collector and cryowall, both of which were grounded during the ionizer testing.

The ionizer used for the evaluation was constructed from material made at Hughes Research Laboratories from 3.9 μm spherical powder. The ionizer was 4.6 cm long and had an area of 2.73 cm^2.

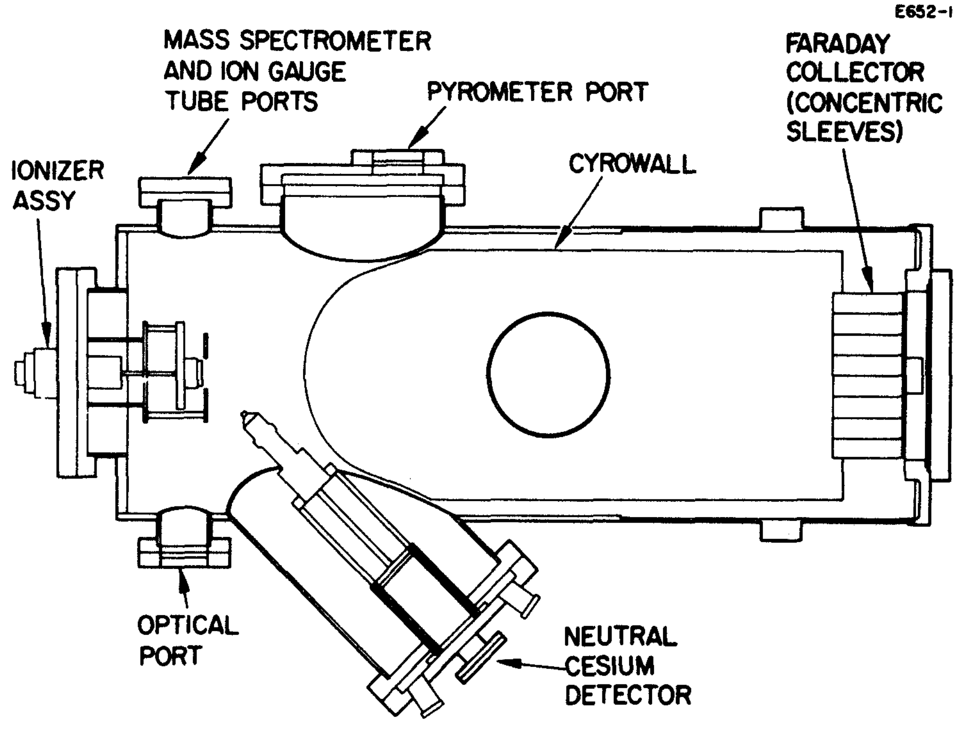

Fig. V-1. Cross sectional view of vacuum chamber.

M 3788

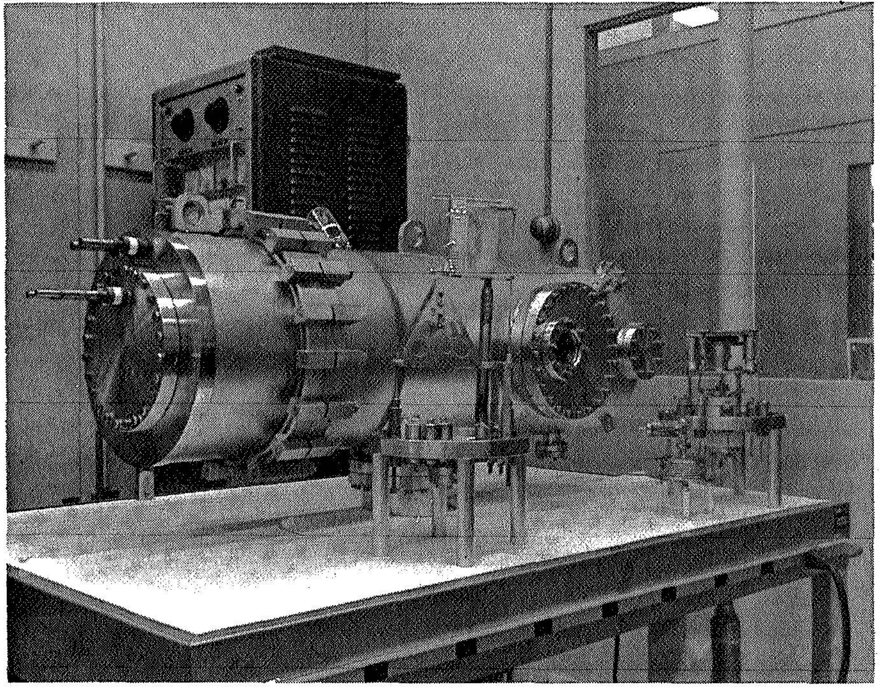

Fig. V-2. View of the vacuum chamber from collector end (neutral detector, cesium reservoir, and ionizer assemblies also shown).

Its surface was contoured as required for the high perveance optics. This ionizer was used in the assembly shown in Figs. V-3 and V-4. This assembly comprised a focus ionizer plate, an accel electrode, and a decel electrode, each mounted individually with a set of three insulators from a base plate. The base plate, in turn, was attached to a vacuum flange that went on the end of the UHV chamber.

The accel electrode was made from OFHC copper and had a sheath heater brazed to the upper surface. The heater was used to evaporate any cesium that might come in contact with the accel electrode.

Cesium was supplied to the thruster by a laboratory feed system through a manual valve attached to the small flange seen in Fig. V-4. The feed system was filled with cesium in a nitrogen atmosphere.

A neutral detector was an important tool for the evaluation of the protective coating. A reproducible, high signal was desirable to determine neutral flux from the ionizer. The detector was placed as close to the ionizer as reasonable; this distance turned out to be 8 cm at a 45° angle to the beam axis. The detector comprised a mechanical shutter, three collimator slits, a set of ion deflector plates, a neutral cesium ionizer and collector, and a liquid nitrogen reservoir. The detector assembly is shown in Fig. V-5. Of the 1.28 cm^2 of ionizer area visible at a 45° angle, the neutral cesium detector viewed an umbra area of 0.11 cm^2 and a penumbra of 0.96 cm^2 of the ionizer surface.

The neutral detector was aligned optically with respect to the ionizer by means of positioning screws from the support bracket. The position of the detector could be adjusted from outside the vacuum system.

M 3793

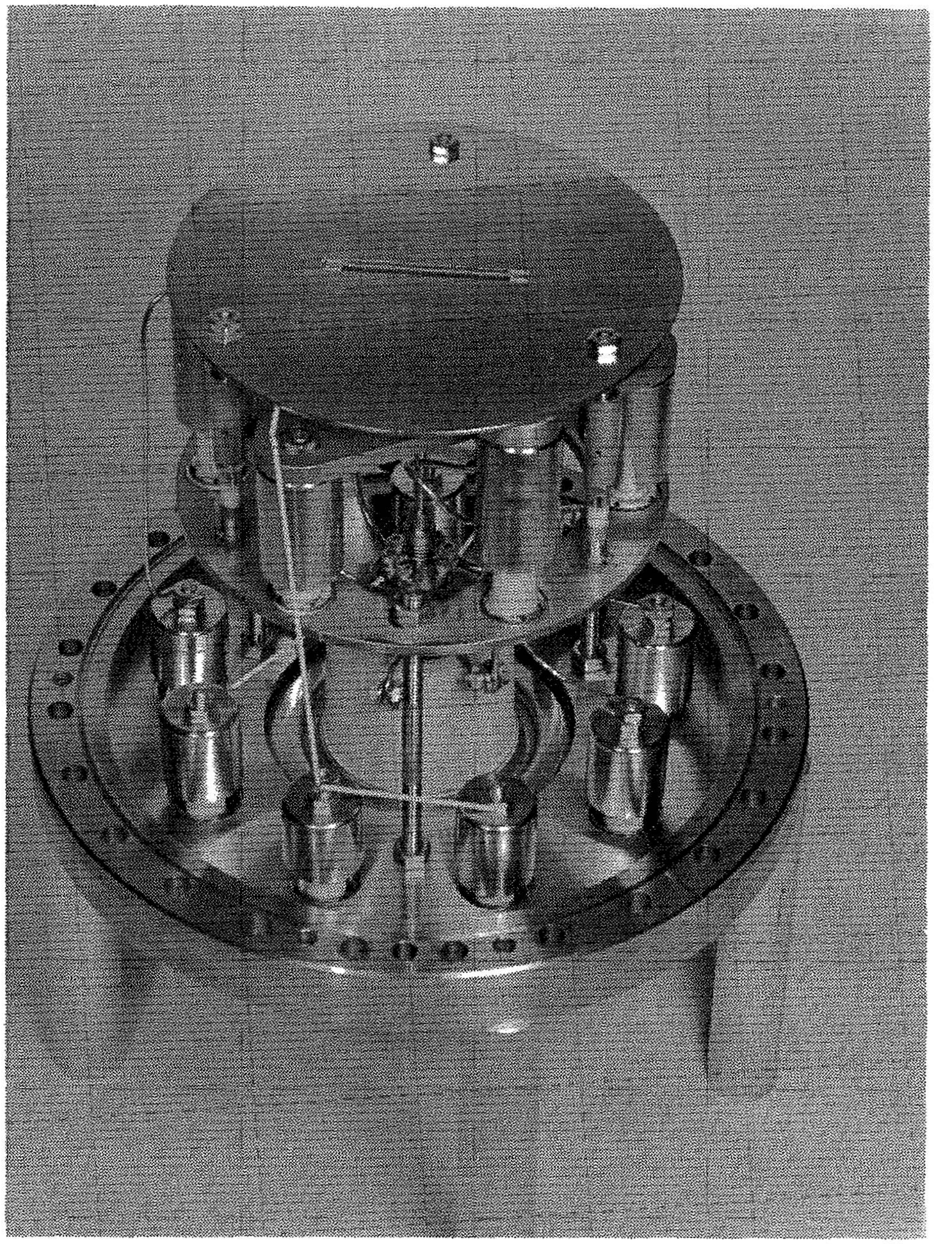

Fig. V-3. Top view of the ionizer assembly.

M 3792

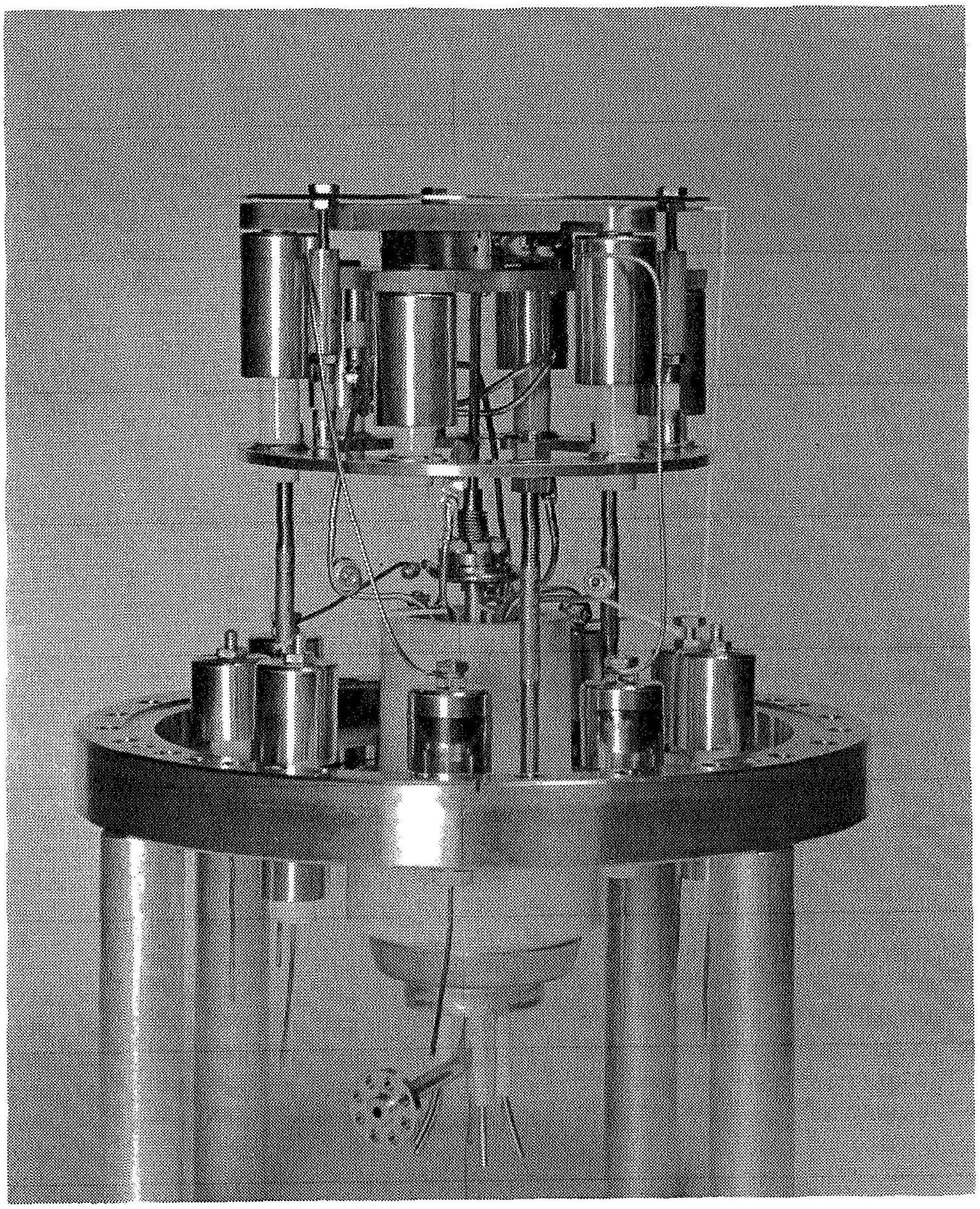

Fig. V-4. Side view of the ionizer assembly.

M 3791

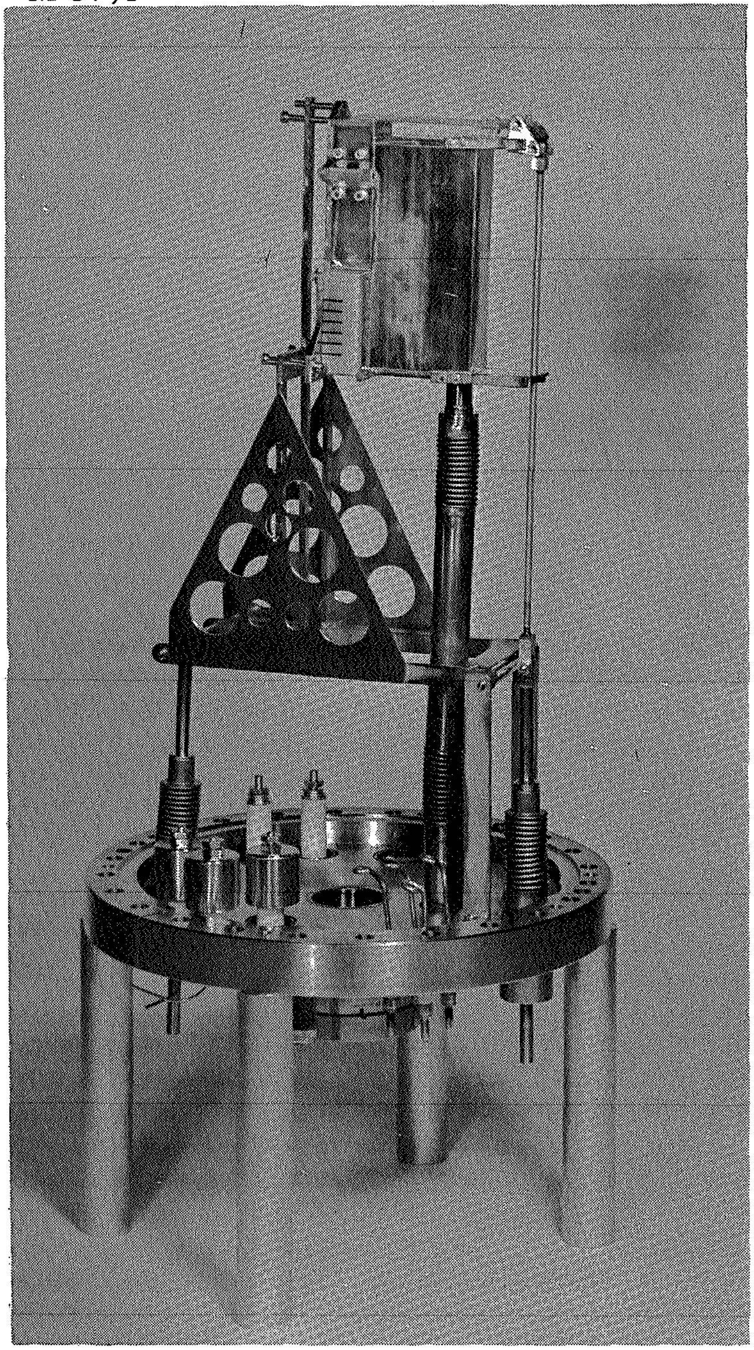

Fig. V-5. Neutral detector assembly.

B. EXPERIMENTAL RESULTS

For these experiments the chamber pressures were in the low 10^{-7} Torr or high 10^{-8} Torr range, depending upon the beam intensity. Before any cesium was allowed to pass through the ionizer, numerous temperature measurements were made. The ionizer temperature was determined by means of a brightness optical pyrometer and a rhenium-tungsten alloy thermocouple. The brightness temperatures were corrected for adsorptive and reflective losses for the viewing window. The emissivity correction of 0.6 (at 0.65 μm) was used. The thermocouple used for monitoring was located in a well near the center rear of the ionizer manifold. The thermocouple output was calibrated with respect to the optical pyrometer corrected values. Thus the temperature readings were obtained from a calibrated meter during the critical temperature measurements.

The temperature measurements were accurate to ±30°K based on the reproducibility, accuracy of the meter, and possible variation of the ionizer emissivity correction. As a result of end conduction losses the ionizer displayed a nonuniform temperature distribution of less than 25°K. Temperature measurements with the optical pyrometer were always made at the center of the ionizer. The relative values for the measurements made during the entire experiment are of course much less (±5°K).

The neutral cesium detector was calibrated by means of ion beam-on beam-off measurements. These were made after either the neutral detector or ion source was moved during the experiment.

Values for the ion beam current were obtained by subtracting the accel drain current from the current readings on the positive high voltage power supply. In some cases, when leakage to ground from the ionizer was measurable, prior to beam start-up, an additional current value also was subtracted from the positive supply reading.

Because the effect of the coating on the critical temperature was the object of the study, this was the most important measurement made. Neutral flux measurements were used to substantiate the results of the critical temperature measurements.

1. Order of Taking Data

To evaluate the coating process the experiment was run in the following order.

a. Operate thruster until ionizer displayed a clean surface

b. Expose thruster to laboratory environment

c. Operate thruster until ionizer displayed a clean surface

d. Sputter-coat ionizer with 50 to 100 Å of silver

e. Remove coating by evaporation and run thruster

f. Sputter-coat ionizer with 50 to 100 Å of silver

g. Expose thruster to laboratory environment for > 24 hours

h. Operate thruster and measure critical temperature.

2. Initial Tests

The first tests made on the strip thruster were ionizer temperature measurements, perveance measurements, and neutral fraction measurements. These were performed to compare the thruster performance with that recorded several years ago when it was used under Contract NAS 3-6271. The 3.9 μm HRL ionizer used in the thruster was that known as unit 10 under the ionizer study of Contract NAS 3-6271. Several perveance curves were obtained at different beam levels. These results, along with the results from the previous work, are shown in Fig. V-6. The design perveance for the 2.73 cm^2 ionizer used in this thruster is 41 x 10^{-9} $A/V^{3/2}$. Both groups of data fall near this perveance line in Fig. V-6. This result was expected because no changes have been made on the thruster since it was last used.

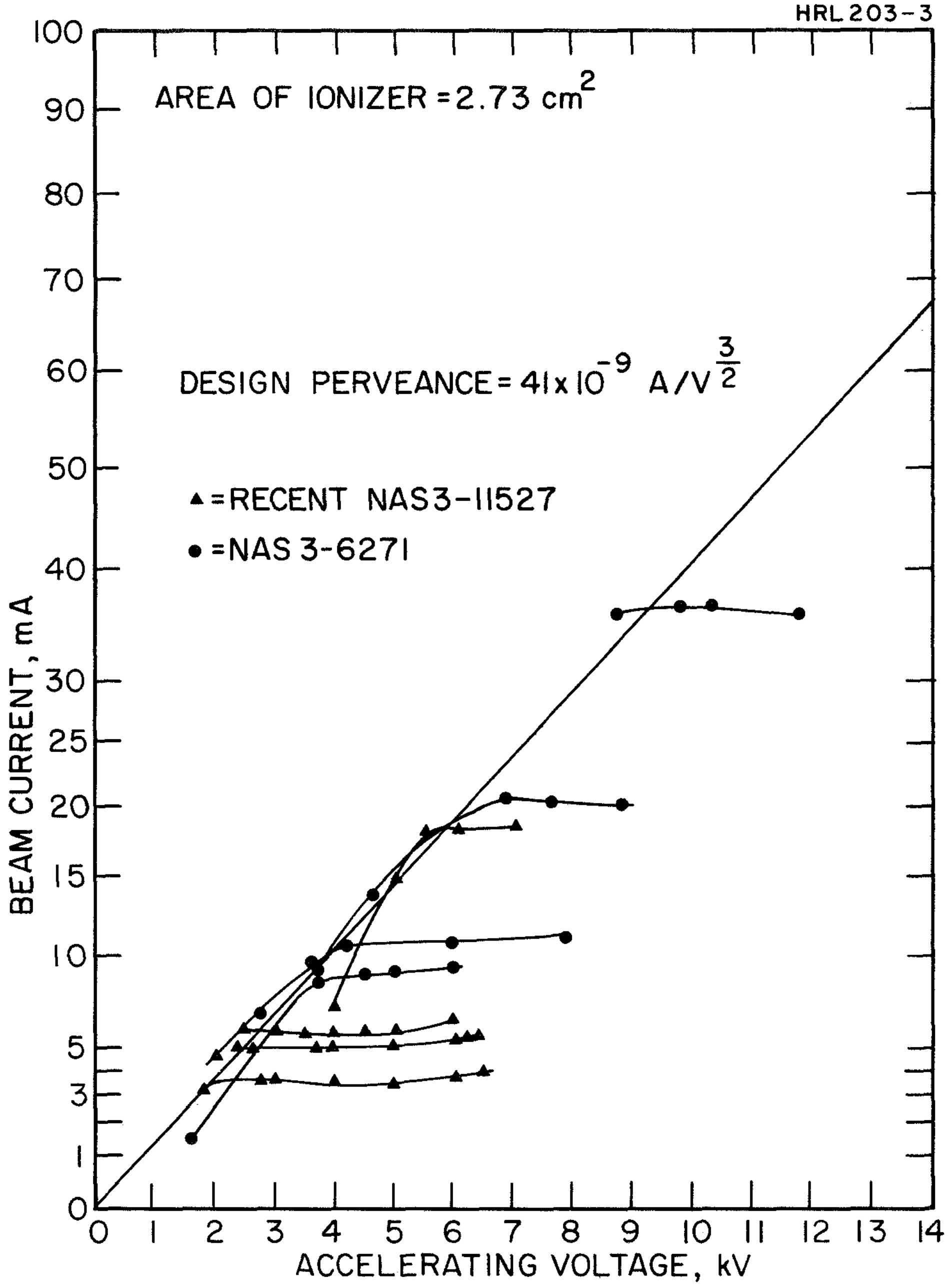

Fig. V-6. Beam current versus accelerating voltage for strip engine.

During the early testing a few critical temperature and neutral fraction measurements were made. These were not considered part of the data used for evaluation of the coating material, but they did reveal the condition of the ionizer surface following exposure to air for five years. The critical temperature and neutral fraction were first measured after the temperature calibrations were made; therefore, the ionizer was elevated to temperatures up to 1600°K for a few hours before cesium was allowed to pass through it. Two other curves were taken, but the system was exposed to air between the runs. The critical temperature curves are shown in Fig. V-7. The Taylor-Langmuir, porous tungsten, and oxygenated tungsten lines are drawn in Fig. V-7. The first curve displayed a surface condition between the clean porous tungsten and oxygenated tungsten surface. The second and third tests revealed critical temperatures approaching that of oxygenated tungsten. Because the ionizer was exposed to air between tests, this contaminated surface is not unexpected. The neutral fraction measurements for these three curves are shown in Fig. V-8. It can be seen that all curves are near the 5.0 eV work function line, indicating a contaminated surface. Because these were checkout tests, no effort was made to clean the ionizer. In addition, it was thought that the thruster would operate with less noise if it were cleaned prior to the step-by-step plan of testing outlined earlier.

3. Evaluation of the Coating

Following the initial tests, the thruster was disassembled and cleaned. The accel electrode was exchanged for one with an operating heater. The heater was intended to operate the accel at a temperature near 700°K to keep cesium from condensing on it. In turn, this would decrease any electrode drain associated with cesium coverage. The first step in the evaluation was to operate the thruster until the ionizer was clean. The starting time/zero time reference was the point where the ion beam was first observed. Neutral fraction measurements were

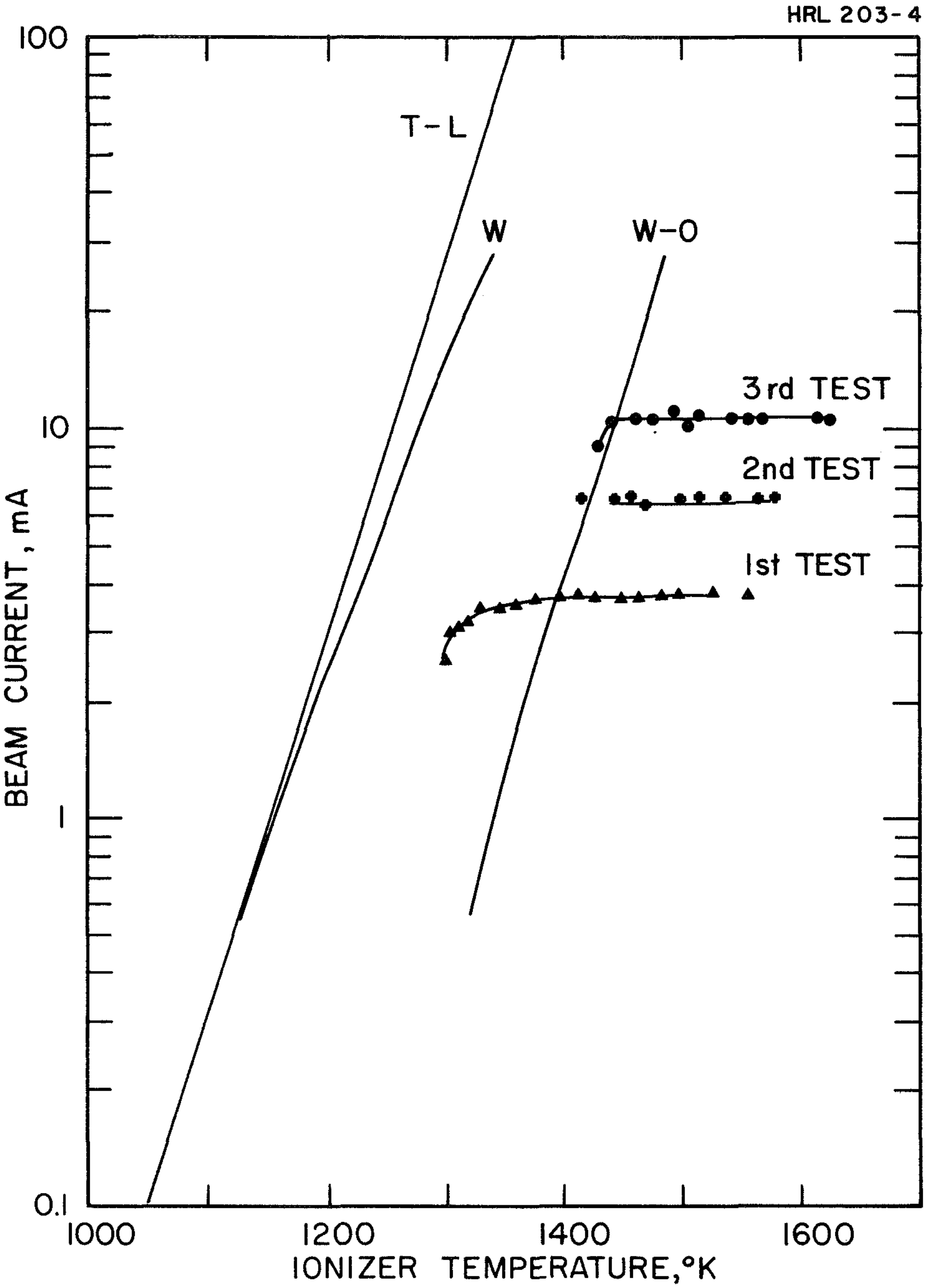

Fig. V-7. Critical temperatures from initial testing for strip engine.

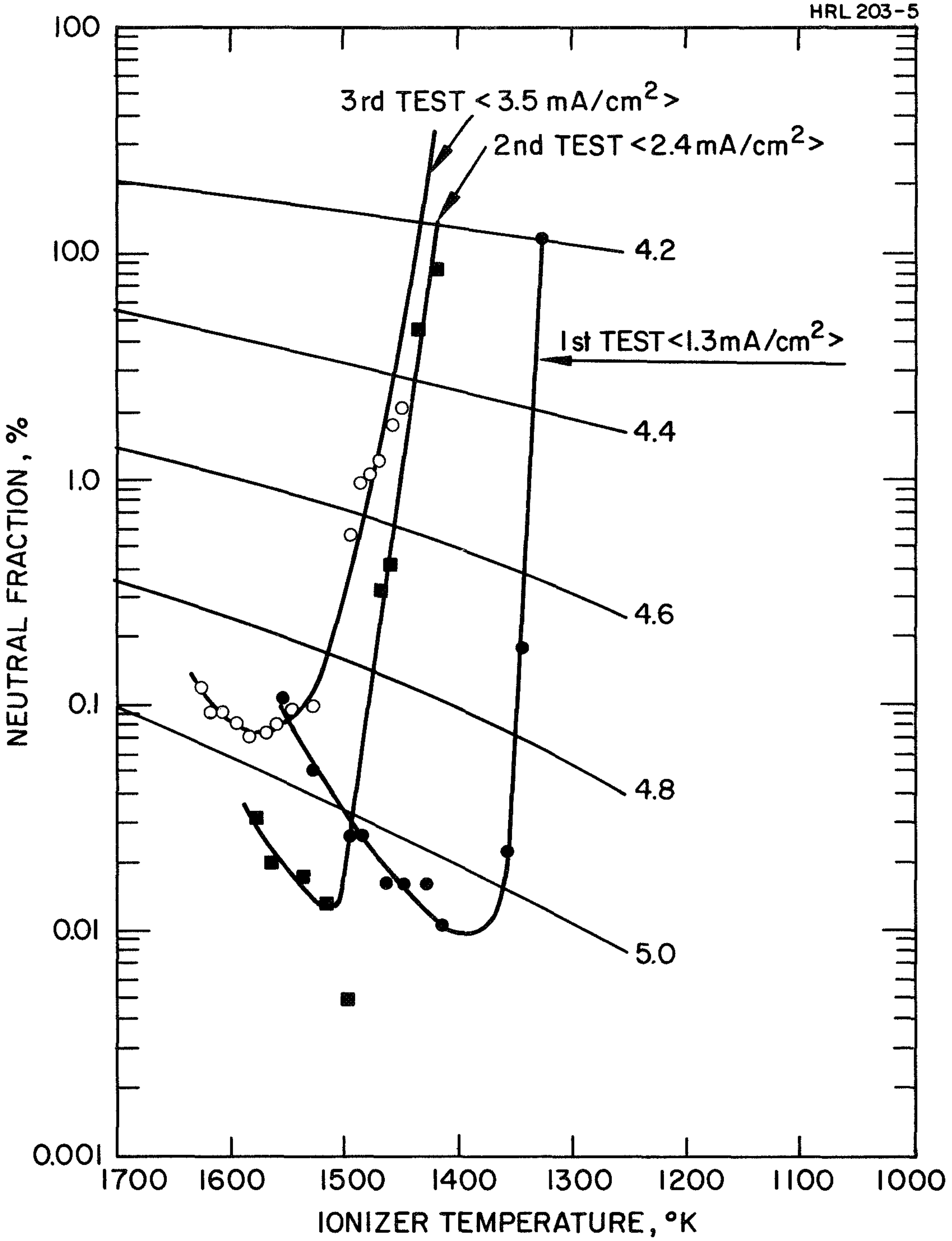

Fig. V-8. Neutral fraction versus ionizer temperature for HRL 3.9 μm material.

taken as the beam increased from 3 mA to 19 mA. There was no measurable neutral fraction signal, indicating that the neutral fraction was less than the detection sensitivity of the neutral detector. Critical temperature curves were taken at the end of 11-1/2, 19, 35-1/2, and 50-1/2 hours of operation. The neutral fractions increased from virtually zero up to 1.5% at the end of 50-1/2 hours. The critical temperature was measured as follows: The ionizer was initially at 1600°K. The temperature was lowered, and beam current and neutral fraction data were recorded. Lowering of the temperature continued until the beam current started to drop. In most cases, when the drop began it was so rapid that a data point could not be recorded. For this reason, a plot of the data shows the last point before the rapid drop occurred. Figure V-9 shows the critical temperature curves. The criteria for a clean ionizer was taken to be a nearly constant beam current to within 50°K of the clean porous tungsten line shown in Fig. V-9. This condition was measured at the end of 50-1/2 hours. The last point measured before the beam dropped was at 1362°K. Although there is only a 25°K difference between the 35-1/2 hour and 50-1/2 hour curves, the neutral fraction differs by more than an order of magnitude. The neutral fraction data corresponding to the critical temperature curves are shown in Fig. V-10. The clean ionizer curve was taken with an average current density of 6.99 mA/cm^2. It can be seen that this curve falls between the 4.5 and 4.6 eV work function lines.

The performance of the ionizer is compared with that under Contract NAS 3-6271 in Fig. V-11. The recent data were taken at an average current density of 6.99 mA/cm^2; the earlier data were taken at average current densities of 8.8 and 13.4 mA/cm^2. The results of the present work have the same general shape but lie closer to the 13.4 mA/cm^2 than the 8.8 mA/cm^2 curve. Another comparison for the same ionizer is given in Fig. V-12, which shows the neutral fraction versus current density for a clean ionizer. In terms of neutral fractions, the recent work gives neutral fractions about twice as large

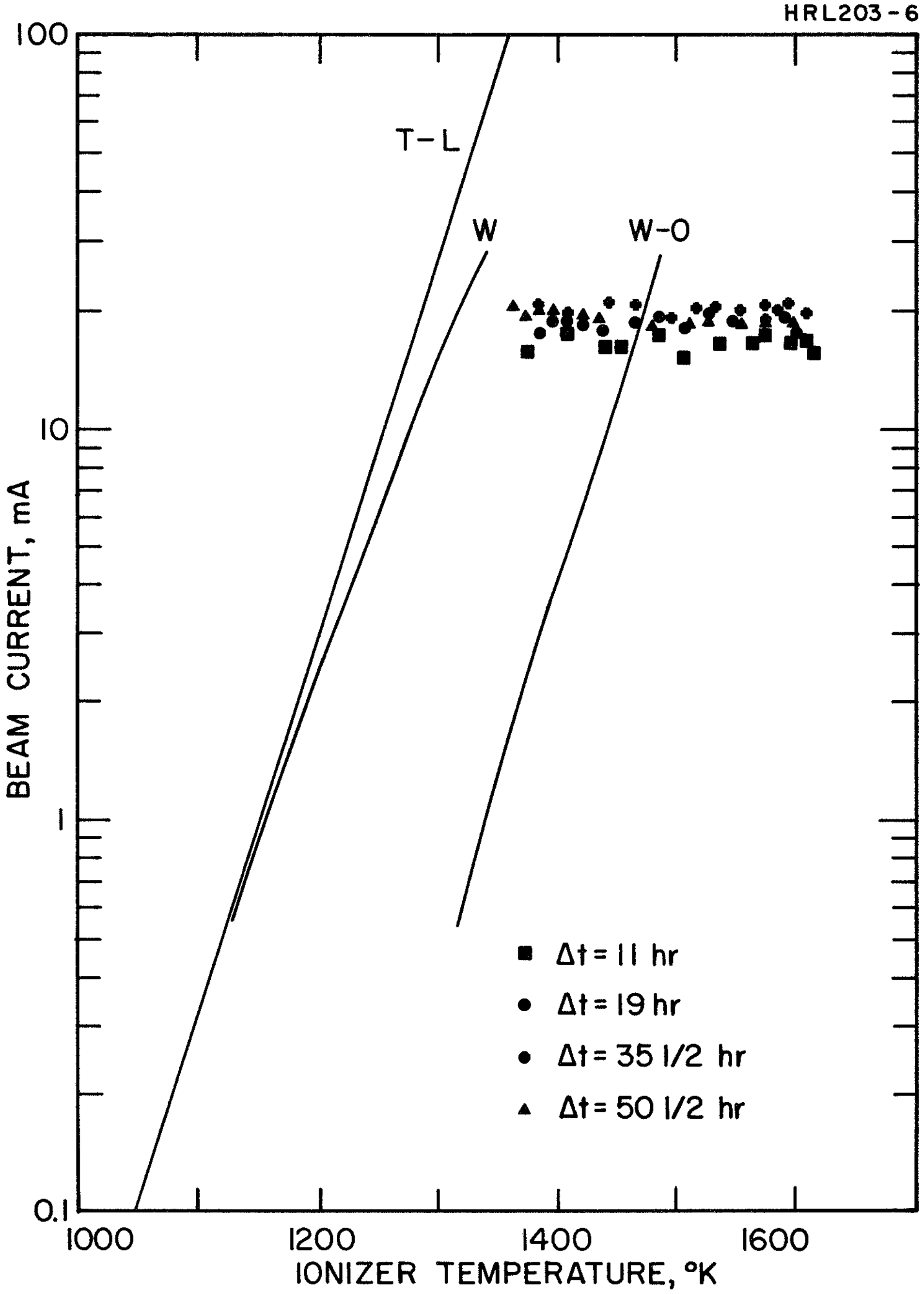

Fig. V-9. Critical temperature curves for 3.9 μm HRL ionizer.

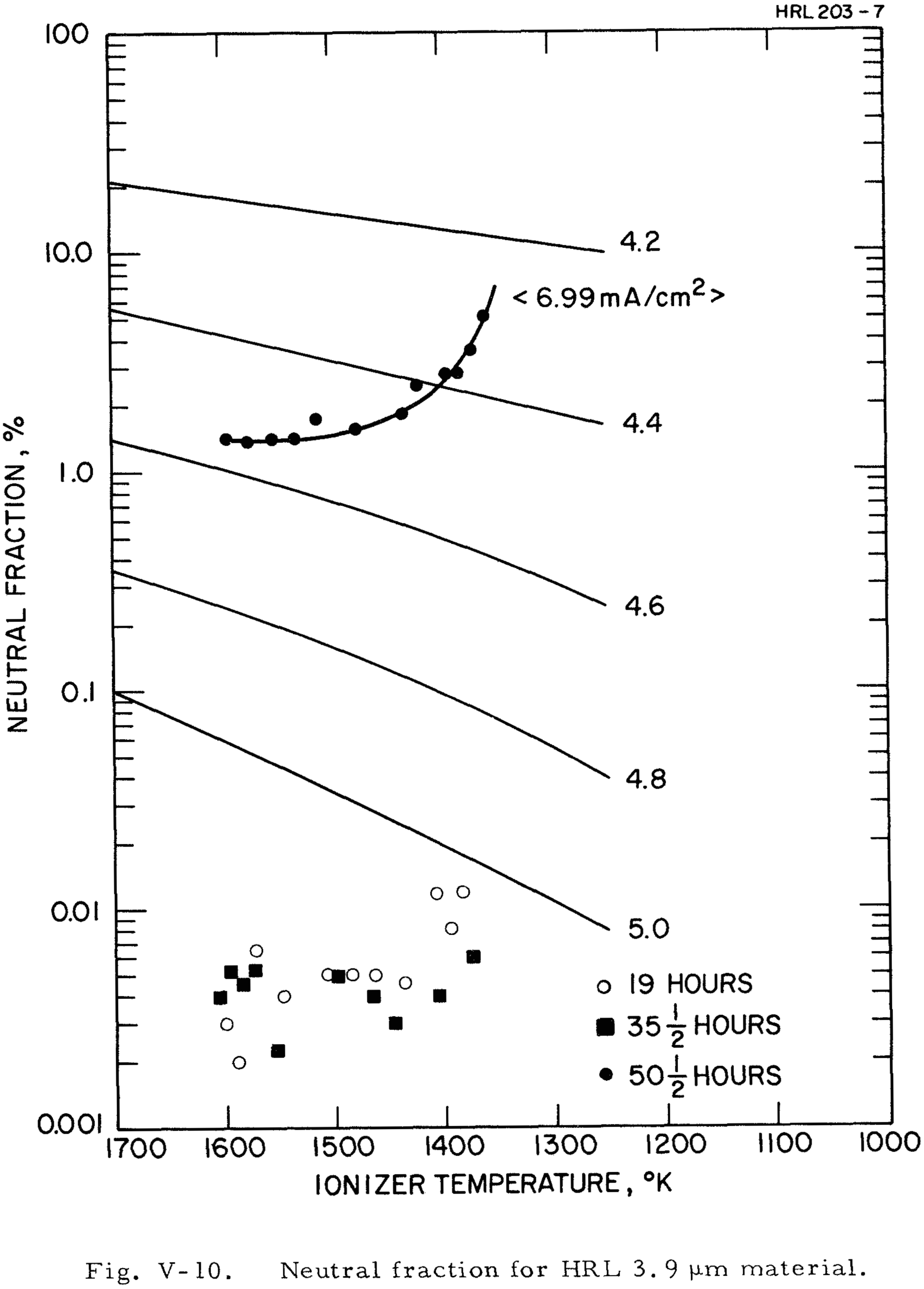

Fig. V-10. Neutral fraction for HRL 3.9 μm material.

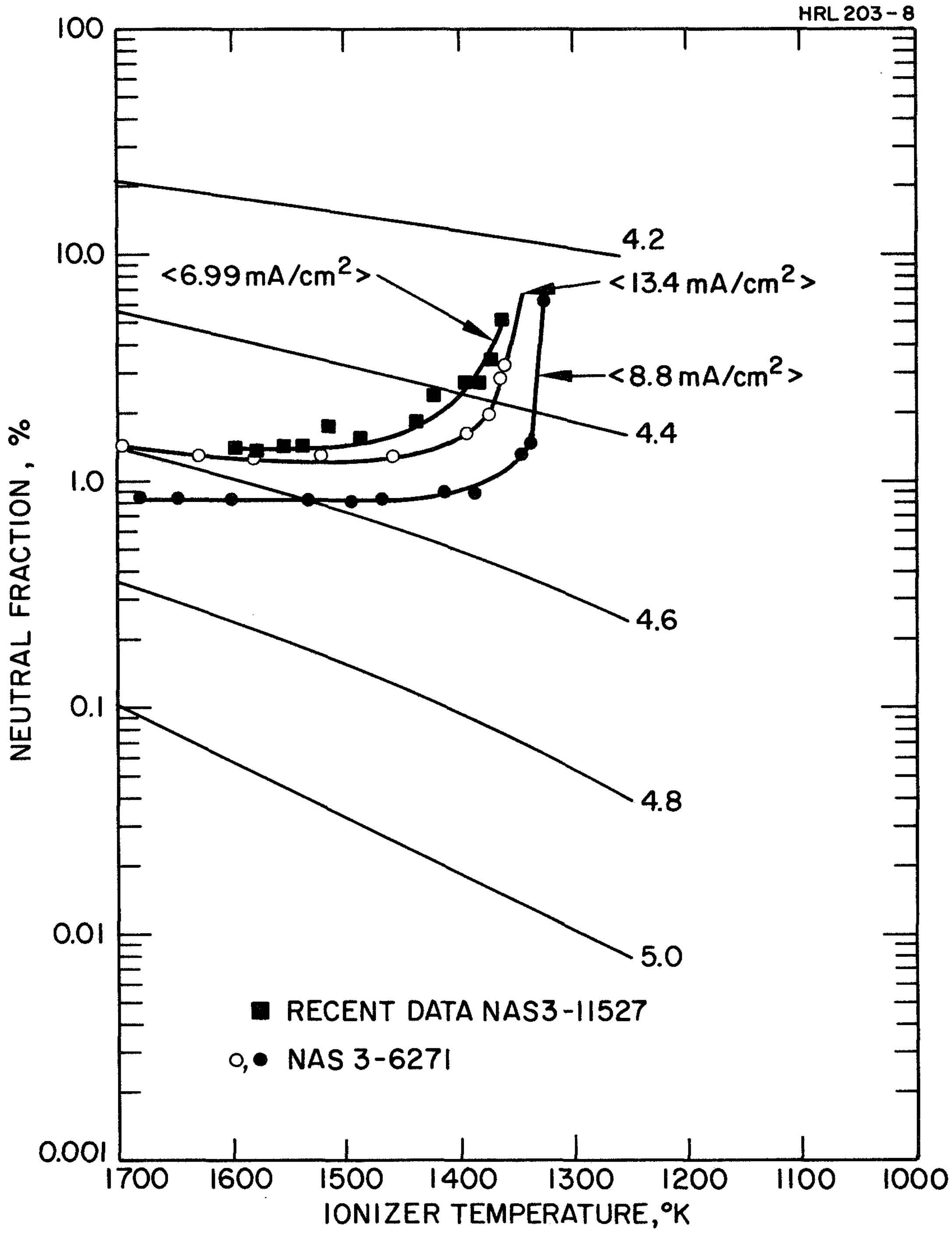

Fig. V-11. Neutral fractions versus ionizer temperature for 3.9 μm HRL material.

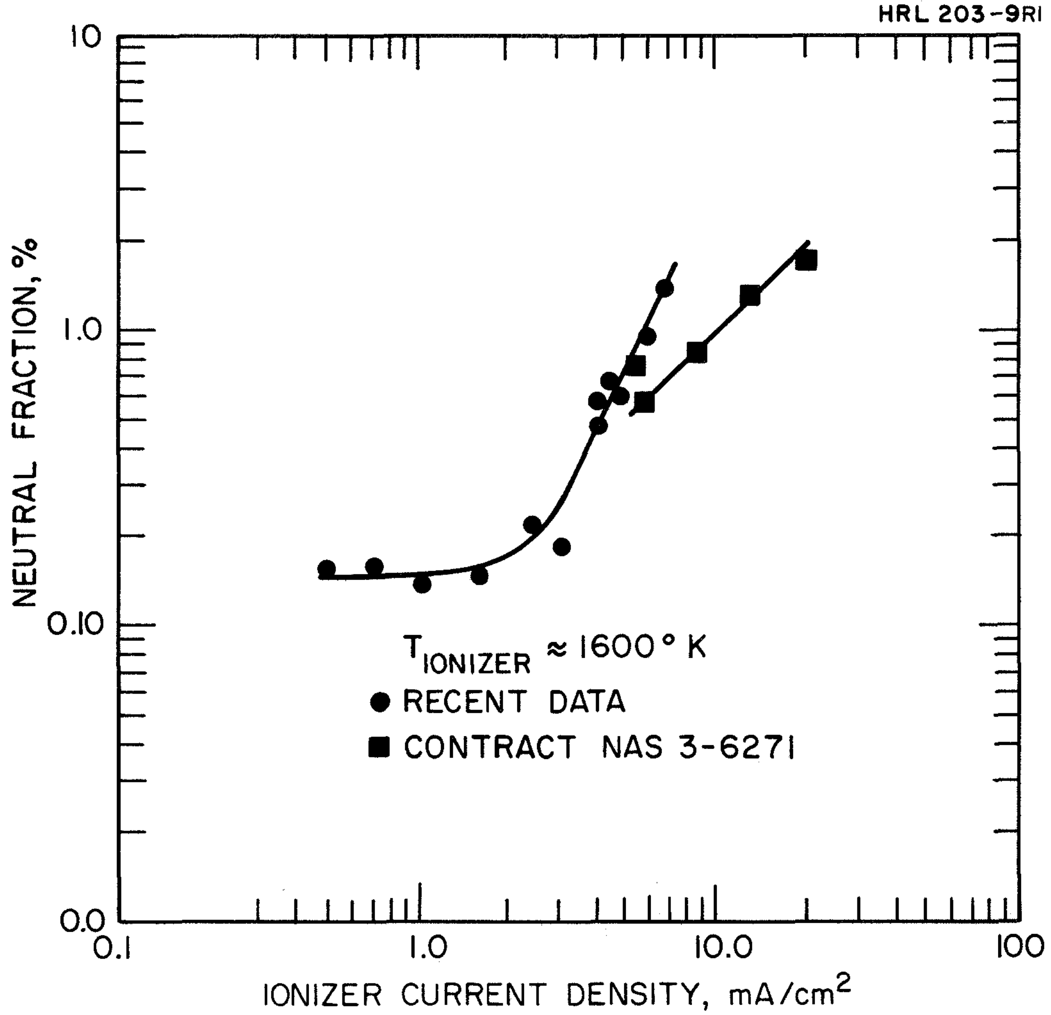

Fig. V-12. Neutral fraction versus ionizer current density for 3.9 μm HRL material.

as the earlier results for a current density of 7 mA/cm^2. This difference is caused either by a systematic error in the measurements or by new characteristics of the ionizer. Because the identical thruster and neutral detector were used, it is plausible to rule out the error. The ionizer had been stored on a shelf in a warehouse, unprotected from the natural environment, for five years, so that it is conceivable that a change in properties could occur. In any case, exact agreement between both sets of data is not imperative for evaluation of the silver coating. The performance of the ionizer was evaluated by comparison with the critical temperature curves taken on this program. After the clean ionizer performance at 50-1/2 hours was obtained, the thruster was turned off and exposed to the laboratory environment for approximately four days. It was then reinstalled in the UHV chamber and the ionizer critical temperature was measured 9 hours after the ionizer reached operating temperature. This curve is shown Fig. V-13 with the 50-1/2 hour curve also plotted as a reference. The ionizer displayed a clean surface in a much shorter time (i.e., 8-1/2 hours rather than 50-1/2 hours). The condition of the surface immediately after exposure to air is questionable. Fortunately, neutral fraction data were taken every 1/2 hour before the critical temperature data were taken, and showed approximately when the surface approached the clean condition. This can be seen in Fig. V-14. The beam current was increased in steps up to approximately 16 mA. No neutral fraction was detected until the test had been under way for 5-1/2 hours. At this time, the beam was at the 12 mA level for 1 hour. Neutral fraction measurements made at the end of 4-1/2 and 5 hours were below the sensitivity of the detector. It can be seen that there was a measurable neutral flux at the 5-1/2 hour mark. As the beam was increased to about 16 mA, the neutral fraction also increased. The critical temperature was taken at the point shown in Fig. V-14. This short cleanup time should not be taken as typical, since prior experience has indicated cleanup time is not directly related to exposure time.

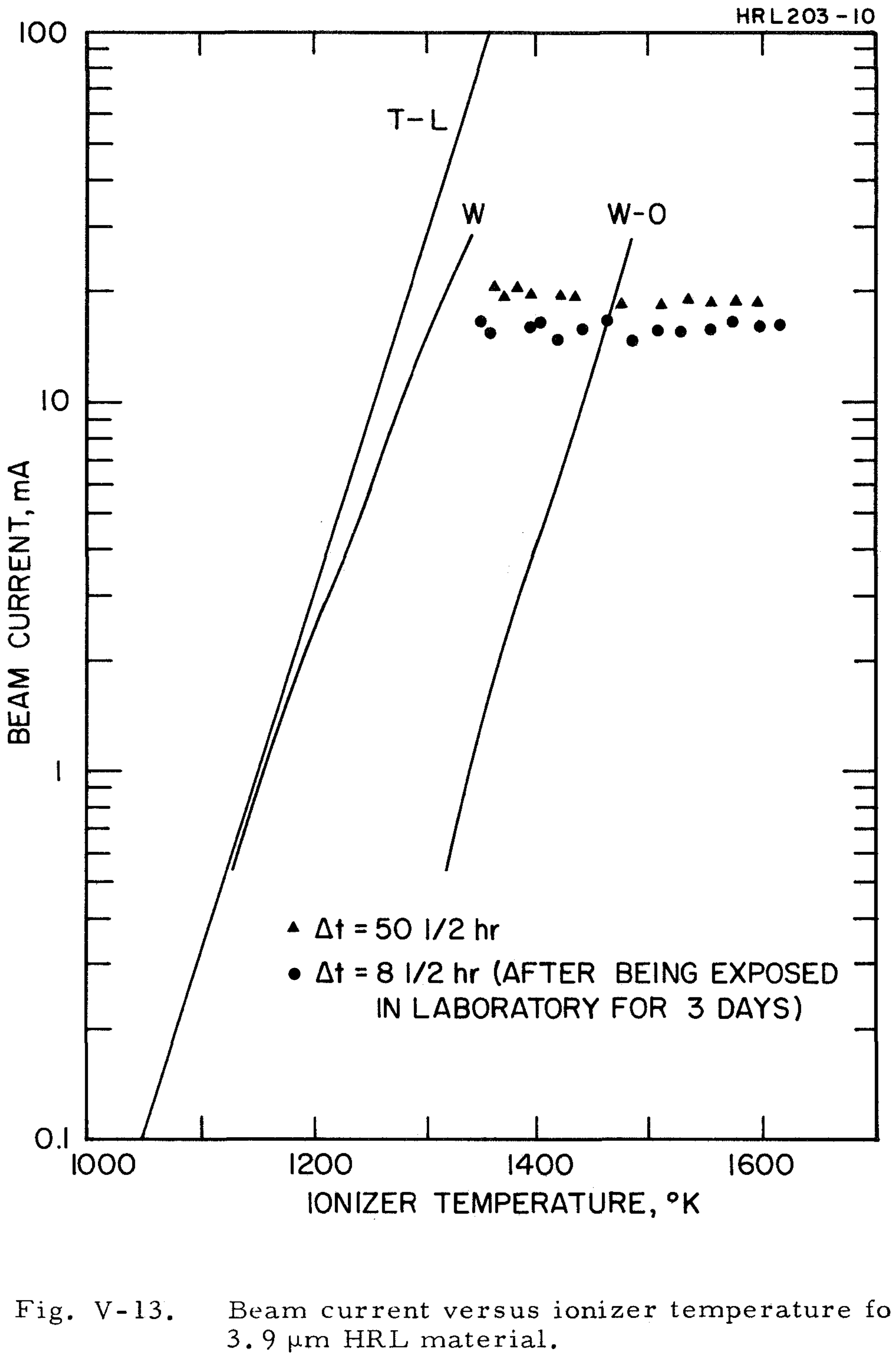

Fig. V-13. Beam current versus ionizer temperature for 3.9 μm HRL material.

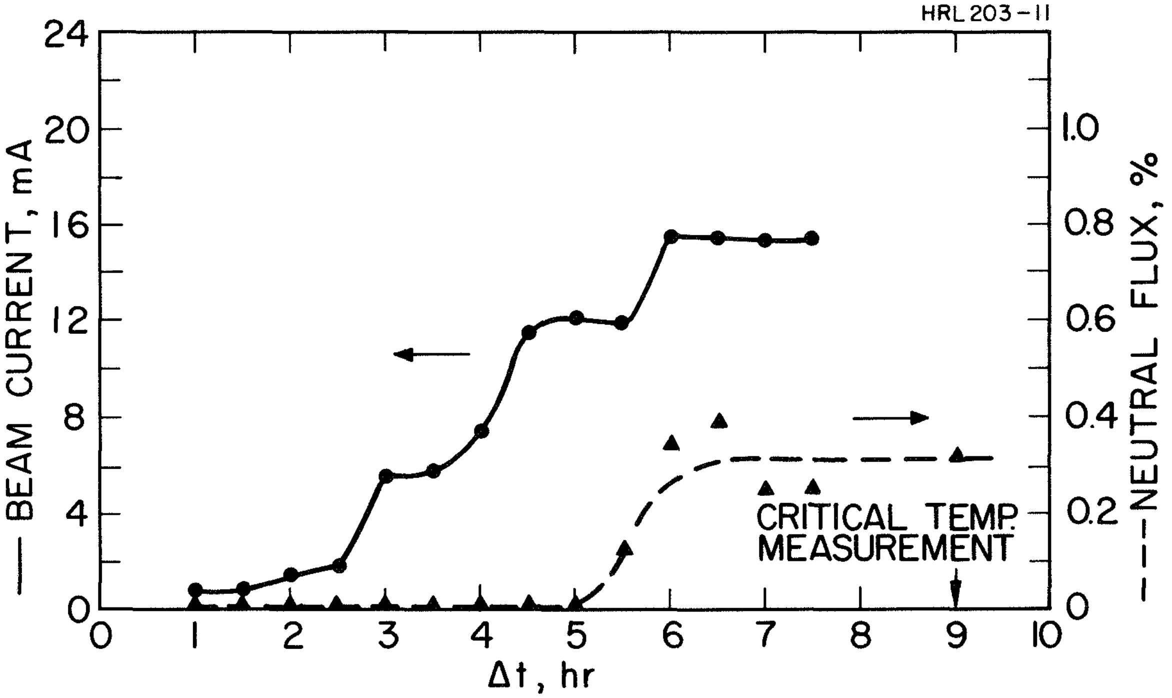

Fig. V-14. Beam current and neutral fraction versus time for 3.9 μm HRL material after thruster was exposed to air for three days.

4. *First Silver Coating*

After the ionizer was thought to be clean, the thruster was turned off and allowed to cool. The ion pump was turned off and a mechanical pump was used to pump the chamber through a zeolite trap to prevent oil from diffusing into the chamber. Argon was bled into the system until a steady 350 μm pressure (as read by a thermocouple gauge) was obtained. The silver cathode was placed in its proper position by means of the magnetic rotary feedthrough. Once in position, the voltage on the cathode was decreased to -750 V for 5 sec. The argon gas flow was then turned off, and the system was pumped in for about 4 hours with the mechanical pump. The mechanical pump was then valved off, and the pressure was decreased further with two sorption pumps. The system reached operating pressure about 10 hours after the silver was sputtered onto the ionizer surface. The ionizer was turned on and kept at 1600°K for 2 hours before the feed system was turned on; this was more than enough time to evaporate 50 to 100 Å of silver from the ionizer.

A critical temperature curve was taken 2-1/2 hours after the ionizer was at operating temperature. This curve is plotted with the previous curves in Fig. V-15. There was only about a 25°K difference between the curve taken prior to the silver deposit and that taken after the coating was deposited and removed. Therefore, it was concluded that the silver did not affect the ionizer characteristics.

The neutral fraction data in Fig. V-16 substantiate the above conclusion. The curve taken after the silver was removed is in better agreement with the 50-1/2 hour curve than that taken prior to silver deposition. This implies that the ionizer was less clean than desired prior to deposition, or it cleaned up after the deposition was removed. It is also possible that the ionizer became clean after the critical temperature data were taken and prior to the silver deposit because the ionizer was at operating temperature for 2 hours after the data

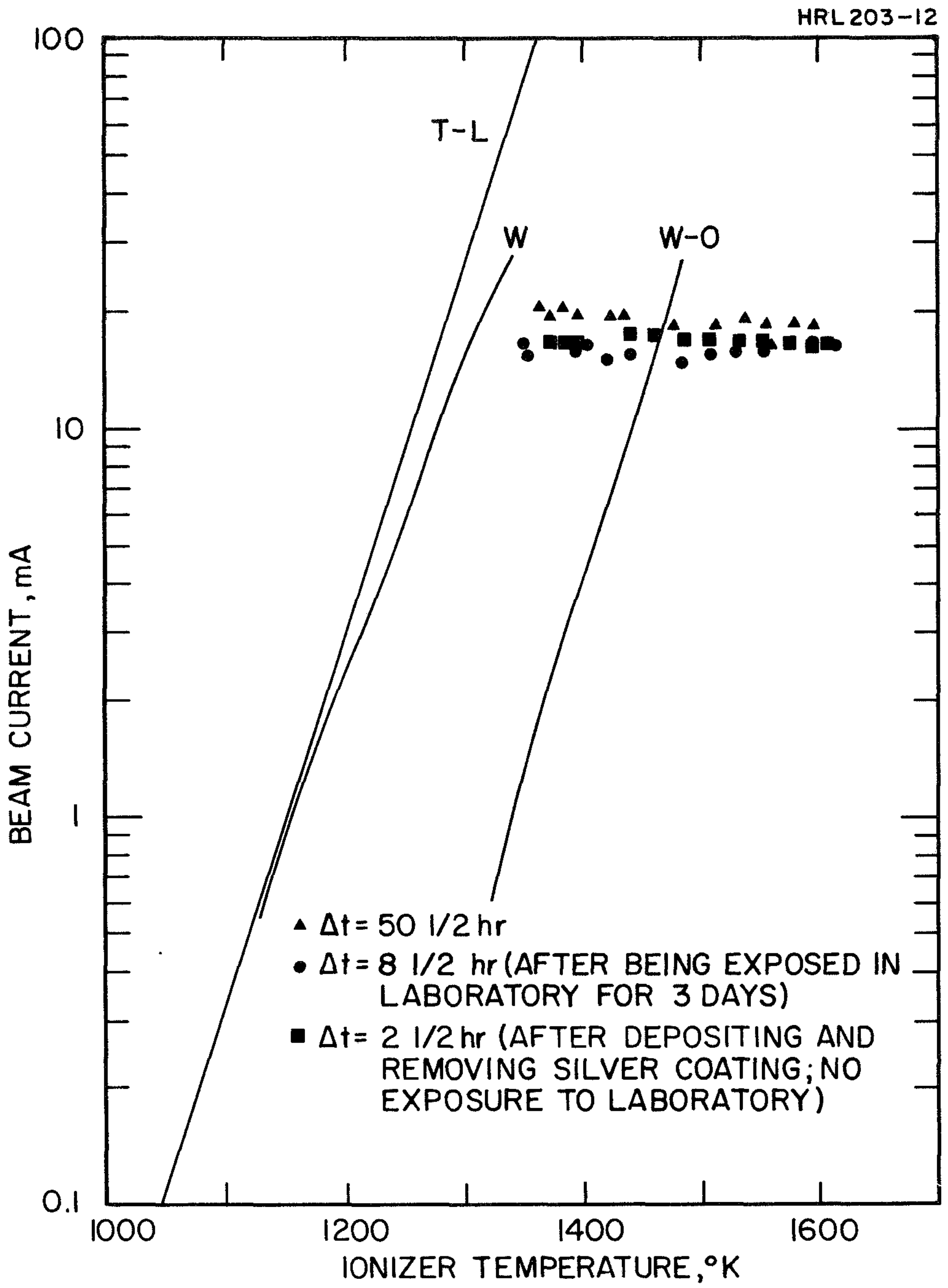

Fig. V-15. Critical temperature curves for HRL 3.9 μm material.

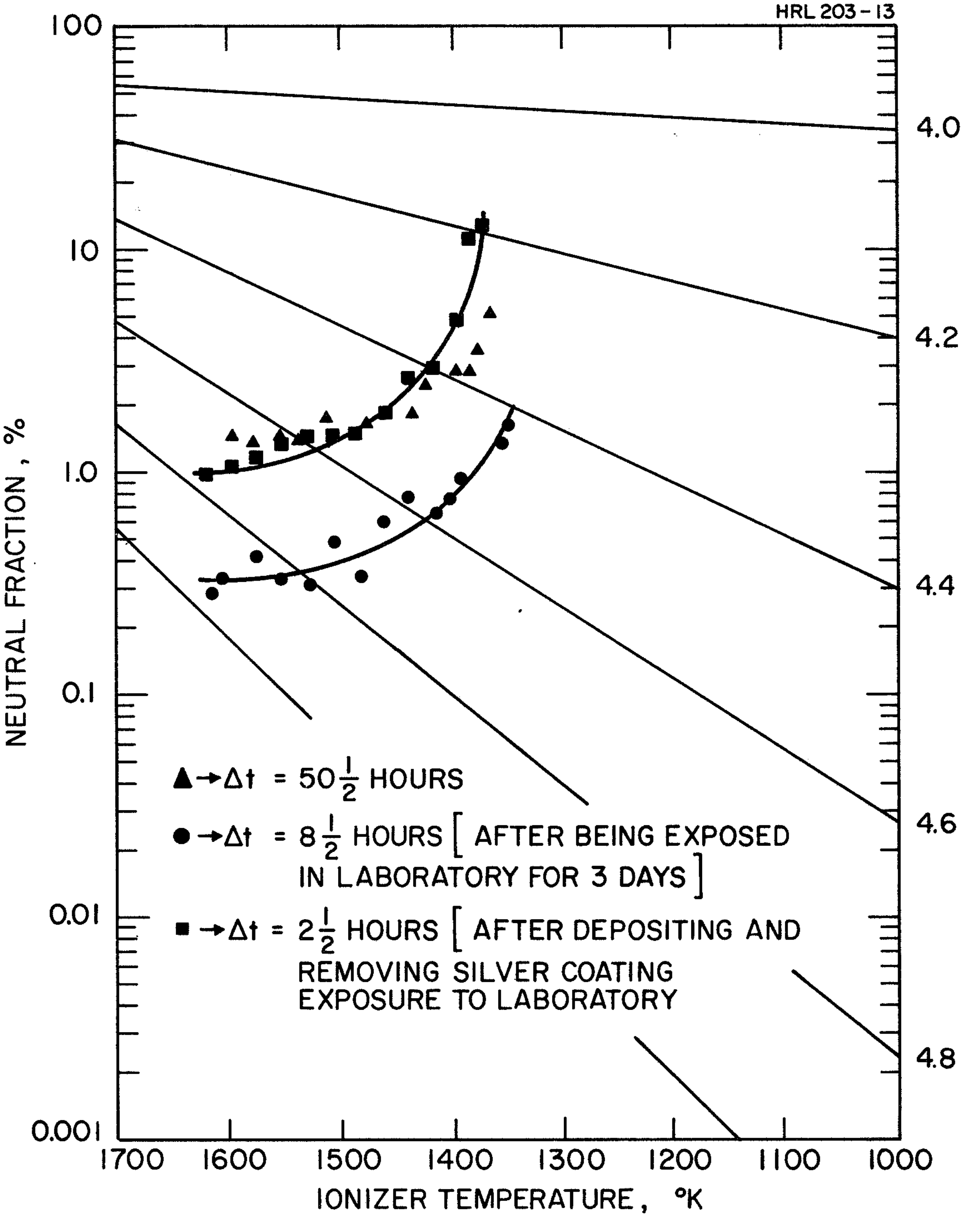

Fig. V-16. Neutral fraction versus ionizer temperature for HRL 3.9 μm material.

were taken. This 2 hour period was necessary to assure that all cesium was removed from the ionizer prior to sputtering. It can be concluded that the silver coating did not deteriorate the ionizer performance when exposed to 350 μm of argon pressure during the sputtering process.

The second, and final, coating of silver on the ionizer was made after the thruster was cooled. This time, however, the chamber was vented to the laboratory atmosphere and the side flanges were removed from the chamber. The thruster was exposed in this manner for 54 hours. The system was then closed and pumped down. The ionizer was at 1600°K for 1 hour before the feed system was turned on. At 1600°K, it takes a fraction of a second to evaporate 100 Å of silver. The first critical temperature curve was taken 3 hours after the ionizer reached operating temperature. This critical temperature curve is shown in Fig. V-17, along with the previous results. It is obvious that the ionizer characteristics have changed dramatically. The critical temperature has increased about 150°K, which is an unexpected result. The neutral fraction data for this first run are shown in Fig. V-18 and are compared with the results for a clean ionizer. It can be seen that the neutral fractions are quite different at lower temperatures. It is interesting to note that the work functions are nearly the same at higher temperatures. This is unexpected for an oxygenated porous tungsten surface. It appears that the characteristics of the ionizer have changed, but not as the result of an oxygen-tungsten reaction. Seven critical temperature curves were taken to determine whether the characteristics would change. These curves are shown in Figs. V-19 and V-20. The last critical temperature curve was taken 8 hours after the ionizer had reached operating temperature. There were no signs that the ionizer was cleaning up. The neutral fraction data for all seven runs are shown in Fig. V-21 along with the clean ionizer curve; they all show the same general shape.

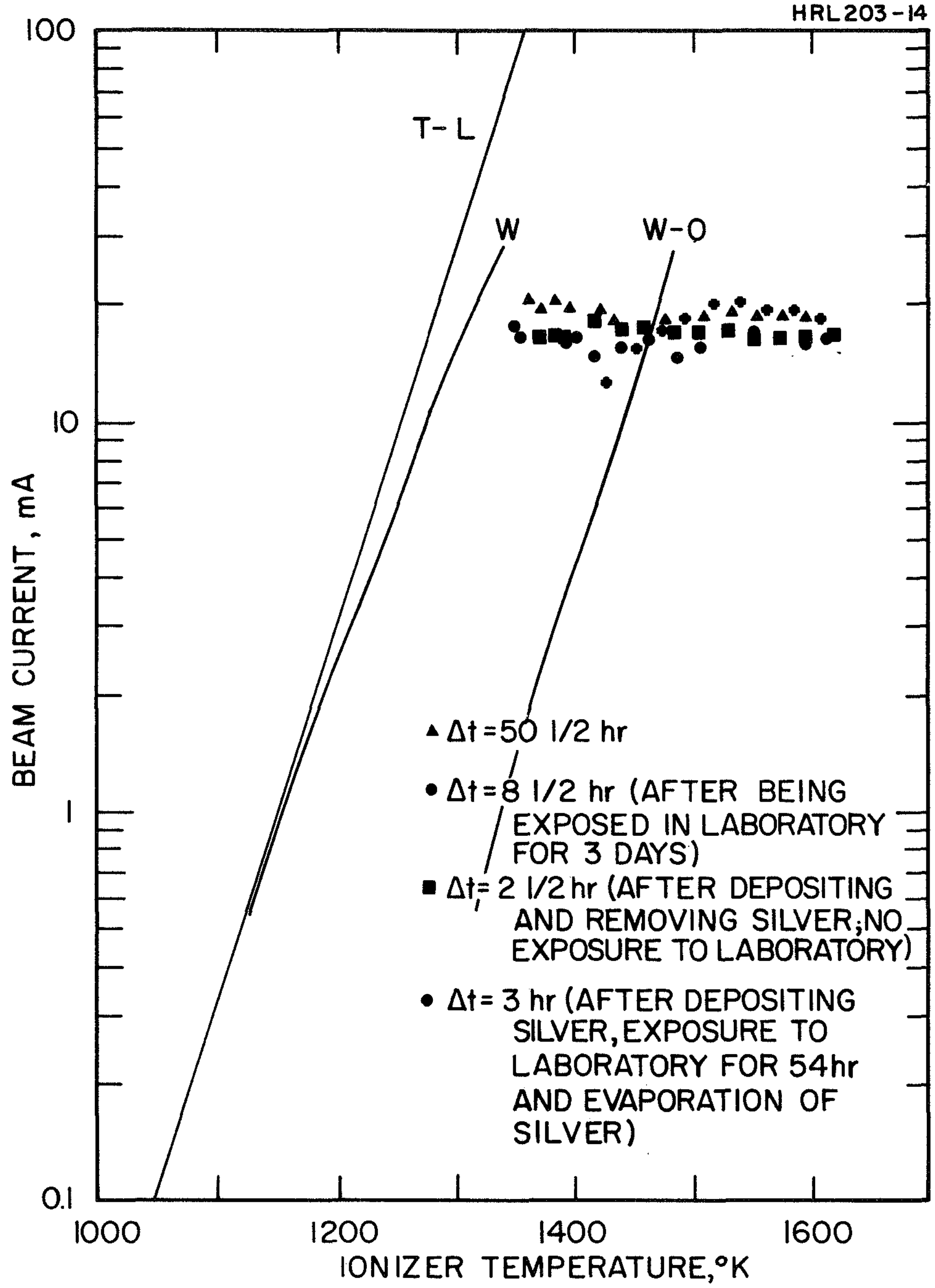

Fig. V-17. Critical temperature curves for 3.9 μm HRL material.

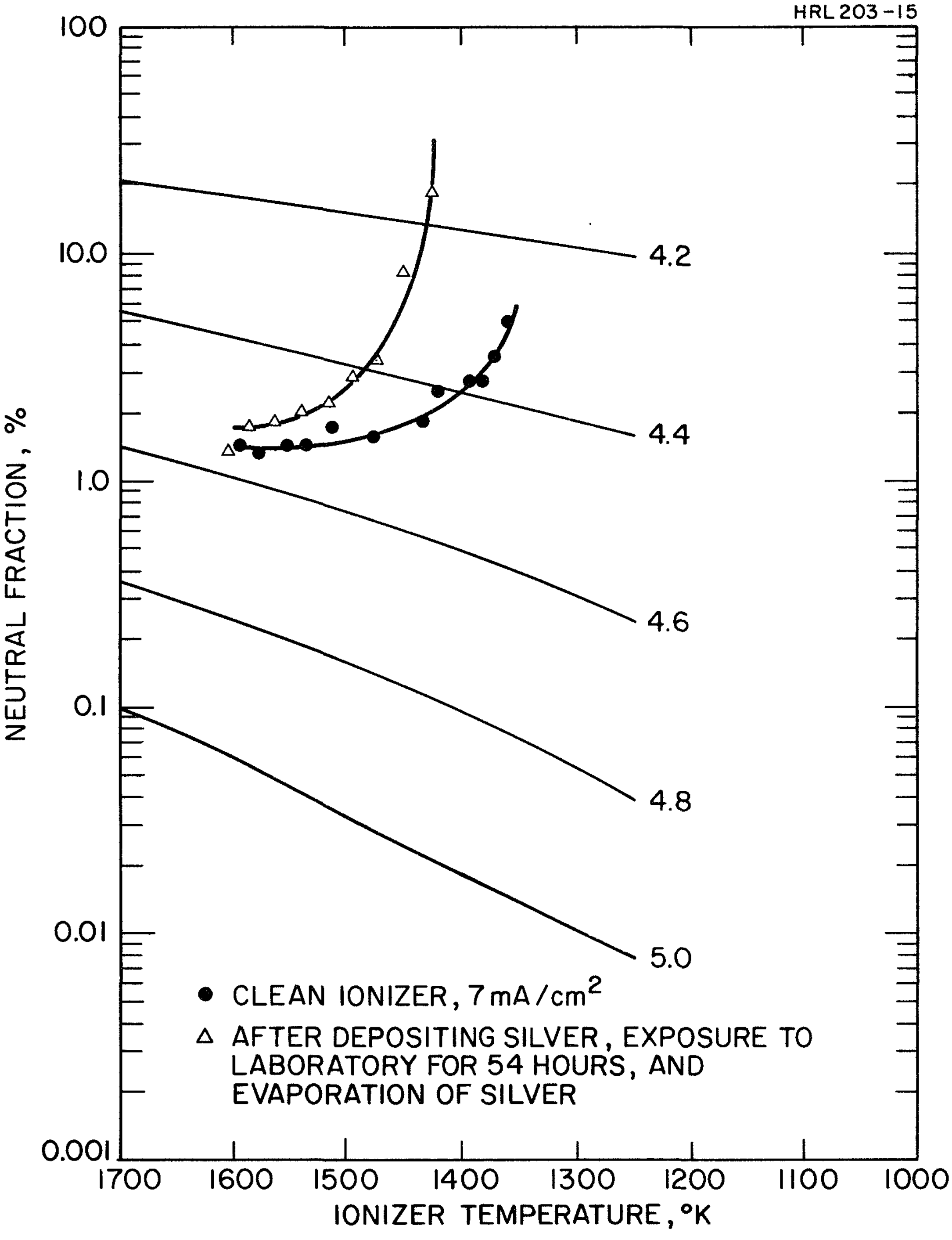

Fig. V-18. Neutral fractions for HRL 3.9 μm material.

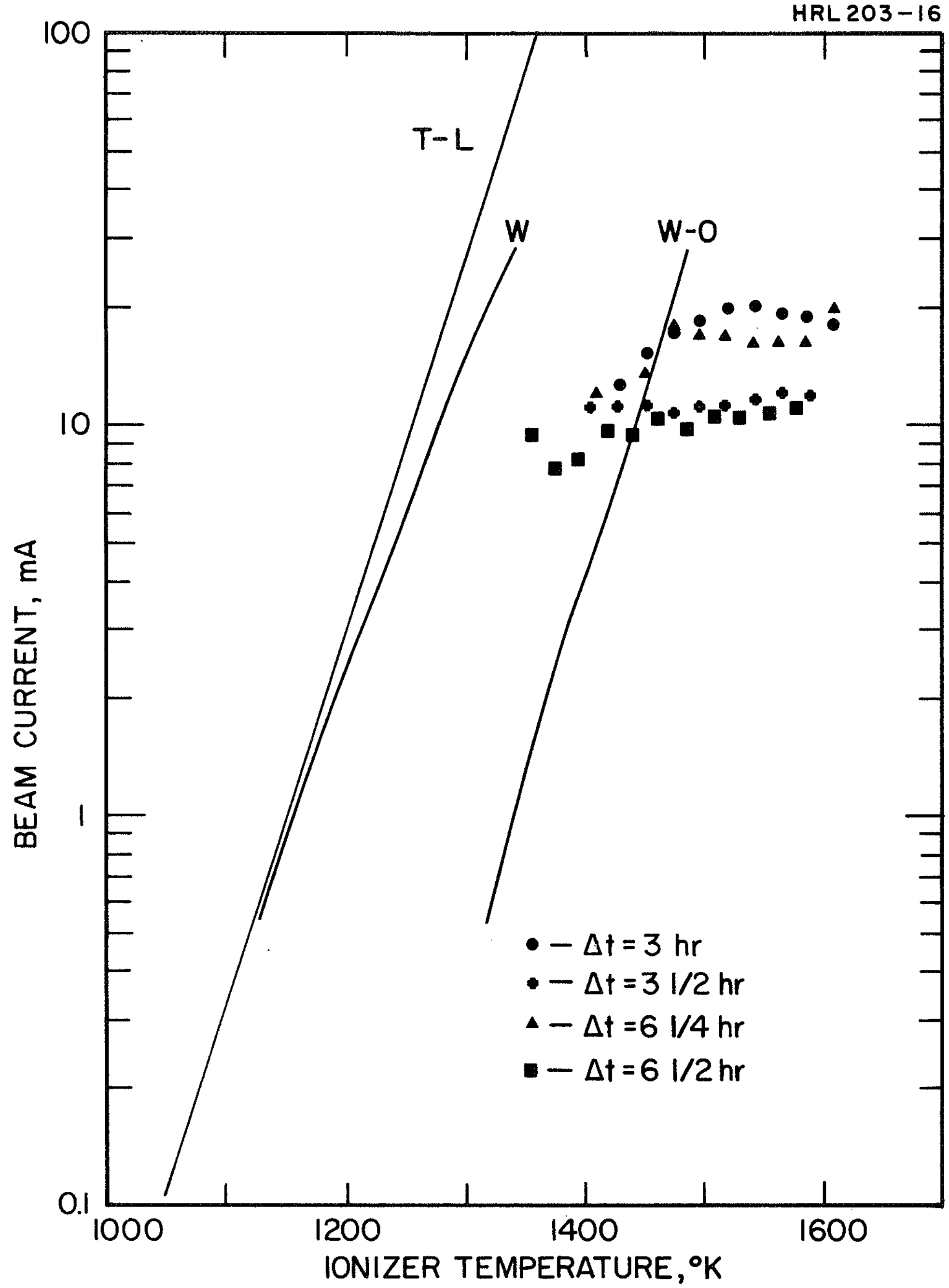

Fig. V-19. Critical temperature for HRL 3.9 μm material, following sputtering, exposure in laboratory for 54 hours, and silver evaporation.

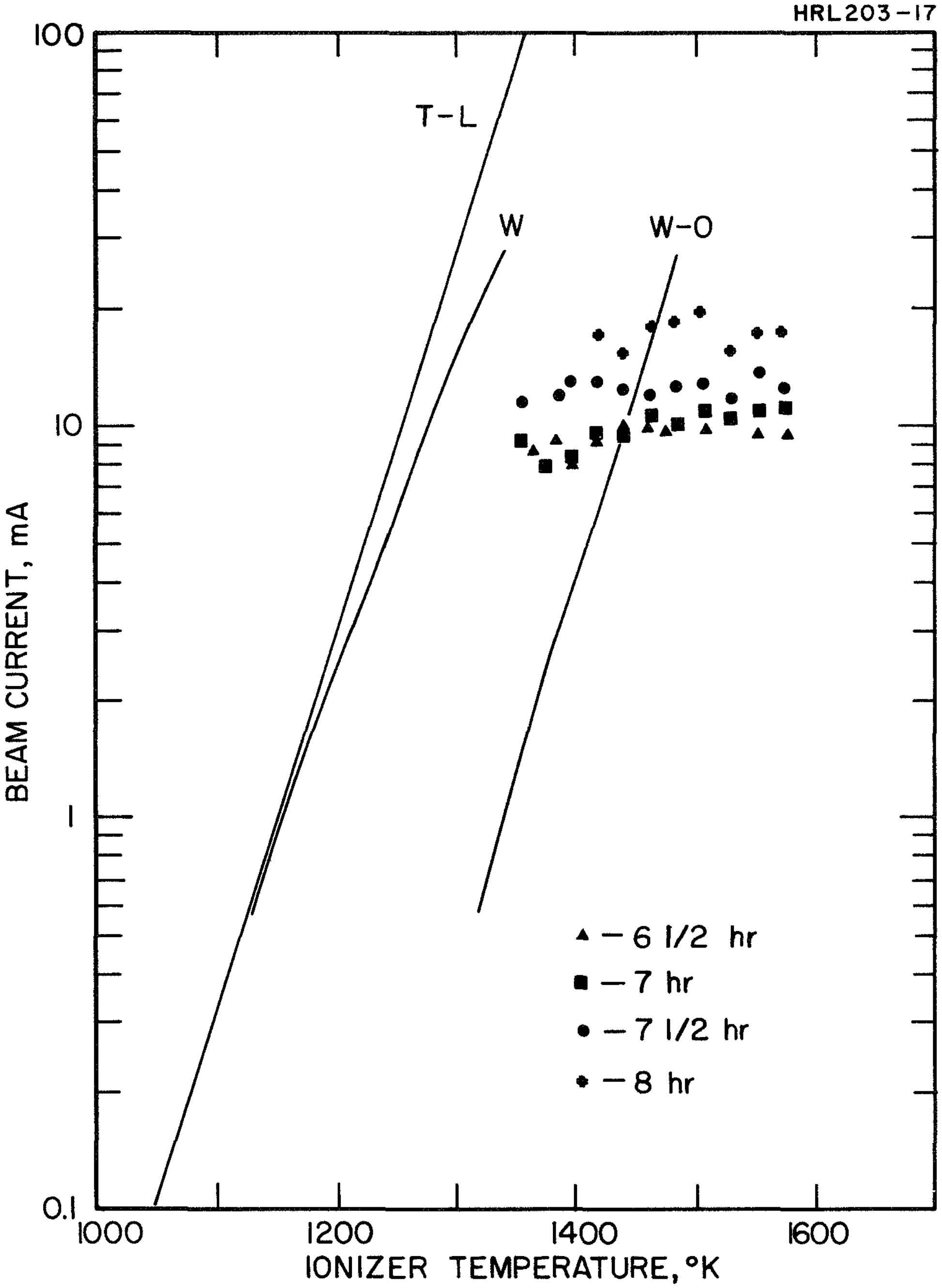

Fig. V-20. Critical temperature for HRL 3.9 μm material, following sputtering, exposure in laboratory for 54 hours, and silver evaporation.

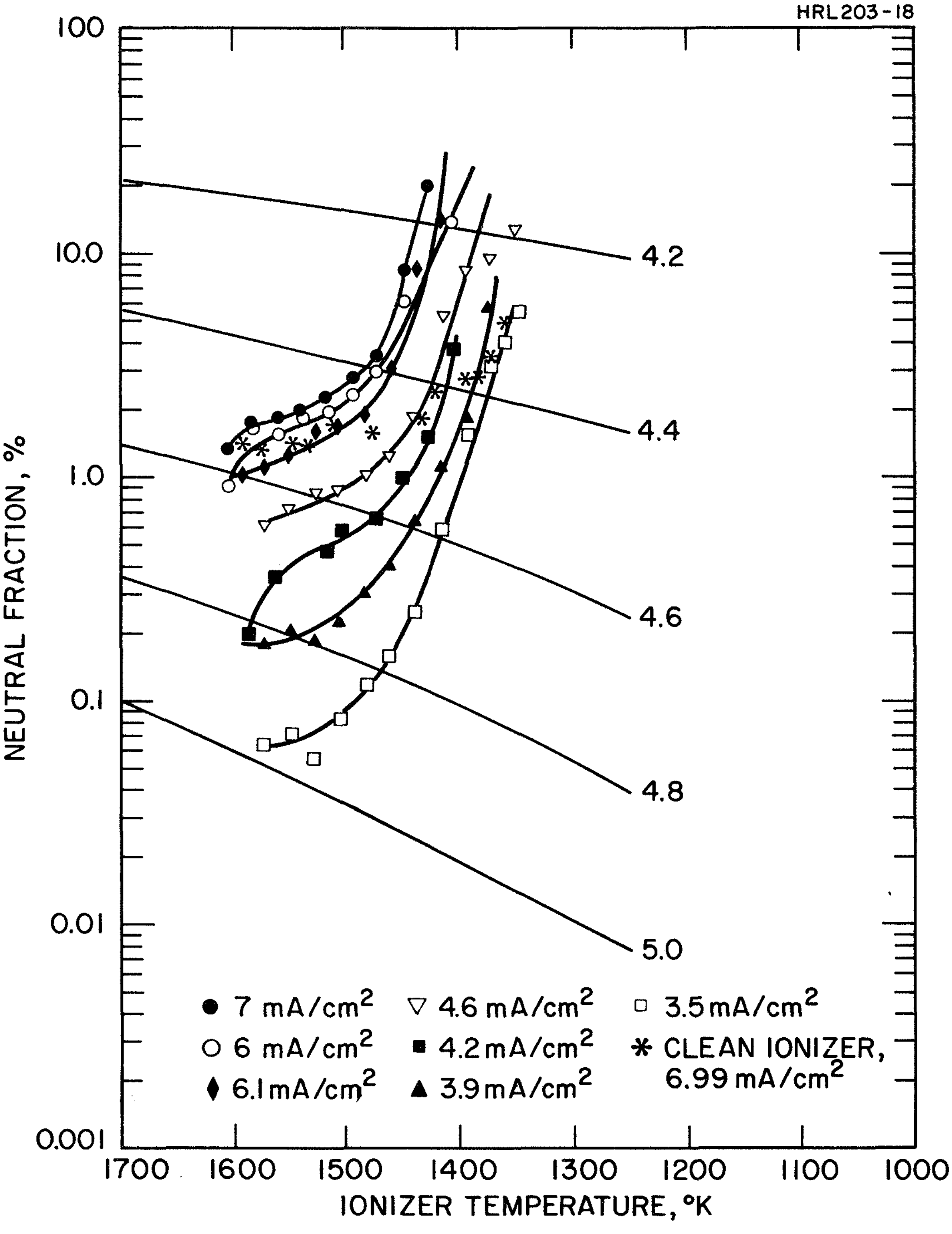

Fig. V-21. Neutral fractions for HRL 3.9 μm material.

SECTION VI

CONCLUSION

Under Task I of this program a technique was successfully developed for applying a metallic coating to the surface of a porous tungsten ionizer. It was shown that an ionizer could virtually be sealed with a 20 μm thick silver coating. Furthermore, once the silver was removed the gas flow characteristics of the ionizer were the same as those prior to the coating. Task II extended the results of Task I to an actual ion thruster. A method (and, subsequently, a system) was developed by which an ionizer of an operational thruster could be coated while the thruster was still in the vacuum chamber.

The coating system is completely operational and can be used for detailed studies of the effectiveness of the coating as a protection of an ionizer surface. During the present program, the system was used to evaluate the contamination protection capabilities of a 100 Å silver coating on an ionizer surface. While it was found that the 100 Å coating itself did not contaminate the ionizer, it did not protect the ionizer from the laboratory environment. The 100 Å coatings were considered initially because if this thickness was adequate for protecting the ionizer, the possible shorting out of the high voltage insulators would be minimized. These initial results, however, suggest that the other parameters, such as greater coating thickness, should be investigated. Greater coating thicknesses are probably necessary because the 100 Å silver coating does not appear to penetrate into the pores of the ionizer surface. Thus, it is quite possible that the important ion generating pores receive no coating. Unfortunately, it is virtually impossible to determine the penetration of the 100 Å coatings into the pores because these are in the range of monolayer thicknesses.

Thicker coatings can be seen easily, however. As an example from a previous program, a cross section of an ionizer with a chemical coating is shown in Fig. VI-1. Similar photographs of sputtered material could furnish valuable coverage information. If it is found that the pores are not filled (and the present results appear to support this view), the process can be modified further. One very promising modification is to heat the ionizer during the sputtering; the temperature would have to be relatively low so that there would be adequate surface diffusion but limited evaporation. Uniform and complete coverage should be obtainable in this manner.

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Fig. VI-1. Cross section of ionizer coated with iridium.