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ION PLATING CHROME COATINGS IN TUBES

William F. Henshaw
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October 1982

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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of ion plated films. Plating rates of 1 $\mu\text{m}/\text{min}$ per cm of tube length have been achieved which makes the process feasible for the ion plating of erosion protective coatings inside gun tubes.

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INTRODUCTION

During the firing of a gun, the high temperature, high velocity propellant gases cause a melt and blow-off condition which increases the wear rate of the gun tube. It has been found that a high melting temperature coating can reduce the wear rate considerably. A chromium film deposited in the gun tube by electro-deposition will extend the gun tube life but also will fail unpredictably and catastrophically, resulting in the tube being more damaged than an uncoated one. Other coating techniques such as conventional physical vapor deposition (PVD) and chemical vapor deposition (CVD) have produced similar unpredictable coating lifetimes. Heat treating of the chrome plate has not improved the reliability and in some cases has decreased the durability of the chrome plate.¹

Ion plating is a PVD process with a negative voltage applied to the object being coated (substrate) in the presence of a working gas such as Argon. The negative voltage ionizes the working gas, causing some material to be sputtered off the substrate during deposition. Initially, an intermixing of the deposition material with the substrate material occurs, but later the deposition material mixes only with the previously deposited material. Other important physical processes which occur during ion plating² are: (a) desorption of absorbed impurities from the substrate surface, (b) ion penetration and entrapment in the substrate and coating, and (c) recoil displacement of substrate and coating atoms leading to their intermixing. When the deposition rate is greater than the sputtering rate, a coating is produced which has a graded film-substrate interface instead of the sharp interface characteristic of other plating processes. The graded interface is typical of ion plating³ and is generally thought to be instrumental in the observed excellent adhesion of the film to the substrate. The ion plating process further differs from other forms of PVD in that the process uses pressures in the 10^{-1} Torr range as compared to 10^{-4} Torr for sputtering and 10^{-6} Torr for evaporation. The high pressure used in ion plating, by virtue of the very short mean free paths involved, gives the deposition process a greater throwing power which is the ability to uniformly coat substrate surfaces shadowed from the vapor source. The throwing power is especially important when coating a surface which has lands and grooves. Therefore, the ion plated coating with its graded interface and uniform thickness should be an excellent method of protecting gun tubes against erosive wear.

Conventional ion plating sources such as the electron-beam gun or the evaporation boat work well with geometries which are essentially planar. A large three-dimensional object can be ion plated using multiple sources or a mechanism to rotate the substrate during deposition. These conventional methods

¹R.S. Montgomery, "The Laser Treatment of Chromium Plated Steel," Benet Weapons Laboratory Technical Report ARLCB-TR-80023, June 1980. AD#088901

²G. Carter, D.G. Armour, *The Interaction of Low Energy Ion Beams With Surfaces, Thin Solid Films*, 80 13 (1981).

³D.M. Mattox, *Fundamentals of Ion Plating*, J. Vac. Sci. Tech., Vol. 10 No. 1, Jan/Feb 1973.

fail when trying to apply a uniform ion plated coating on the inside surfaces of tubes having a large length to diameter ratio (l/d). The need for the plating of a high temperature metal vapor in a cylindrical geometry has resulted in the development of a device⁴ which uses the heat developed by a high current hollow cathode discharge to evaporate chromium or other materials having similar properties.

EXPERIMENTAL APPARATUS

The details of the gas fed hollow cathode described in this paper are shown in Figure 1. The cathode assembly consists of a thin walled tantalum tube, two additional concentric, tubular Ta radiation shields and a thick walled copper outer shield, all coaxially mounted in a water-cooled holder. The length of the hollow cathode assembly is determined by the length of the substrate to be ion plated. In these experiments, hollow cathodes up to 30 cm in length have been used. A Ta constriction, placed in the end of the cathode, facilitates establishing the high current discharge at lower gas flow rates and consequently allows lower system pressures. Figure 2 shows the cathode voltage as a function of system pressure for a fixed current of 15 amps. The lower voltage with the constriction (curves numbered 3 and 4) is probably due to a higher cathode operating temperature but whether this is a result of plasma confinement or reduced thermal radiation is not known. This higher temperature hypothesis is supported by the fact that the cathode voltage is lower with a radiation shield than without. At low pressures, the cathode discharge becomes unstable and then self-extinguishes where the cathode potential rises sharply.

At a given gas flow rate, cathode diameter (d), and pressure (p), a pressure gradient is developed along the tube. The cathode wall temperature profiles, as shown in Figure 3, have maxima where the condition $pd = \text{constant}$ is satisfied. The material to be evaporated is centered around the position of maximum temperature for the gas flow rate of operating pressure desired and is removed from the cathode through atomic collisions with the working gas. Care is taken not to place the evaporant too near the orifice as this results in poor coating due to the "spitting" of large drops of the evaporant.

The experimental setup shown in Figure 4, consists of a movable cylindrical substrate, a stationary cathode and anode, and their associated power supplies. The Hollow Cathode Discharge (HCD) is started by using a high gas flow rate which gives a system operating pressure of several hundred millitorr and cathode to anode current of 2 amps provided by a 600V DC power supply. After the discharge is started, the temperature rises, causing a drop in the cathode voltage so that a low voltage high current (50 amp) supply takes over. Typical operating parameters are: (a) cathode to anode current of 15 amps, (b) gas flow rate of 4 ml min^{-1} , which for our particular vacuum system results in a pressure of 20 millitorr, and (c) a negative substrate bias of

⁴W.F. Henshaw, J.R. White and A. Niler, *A Hollow Cathode Discharge Source of Metal Vapor*. ARRADCOM Patent Application Docket # 53-81, Sep 1981.

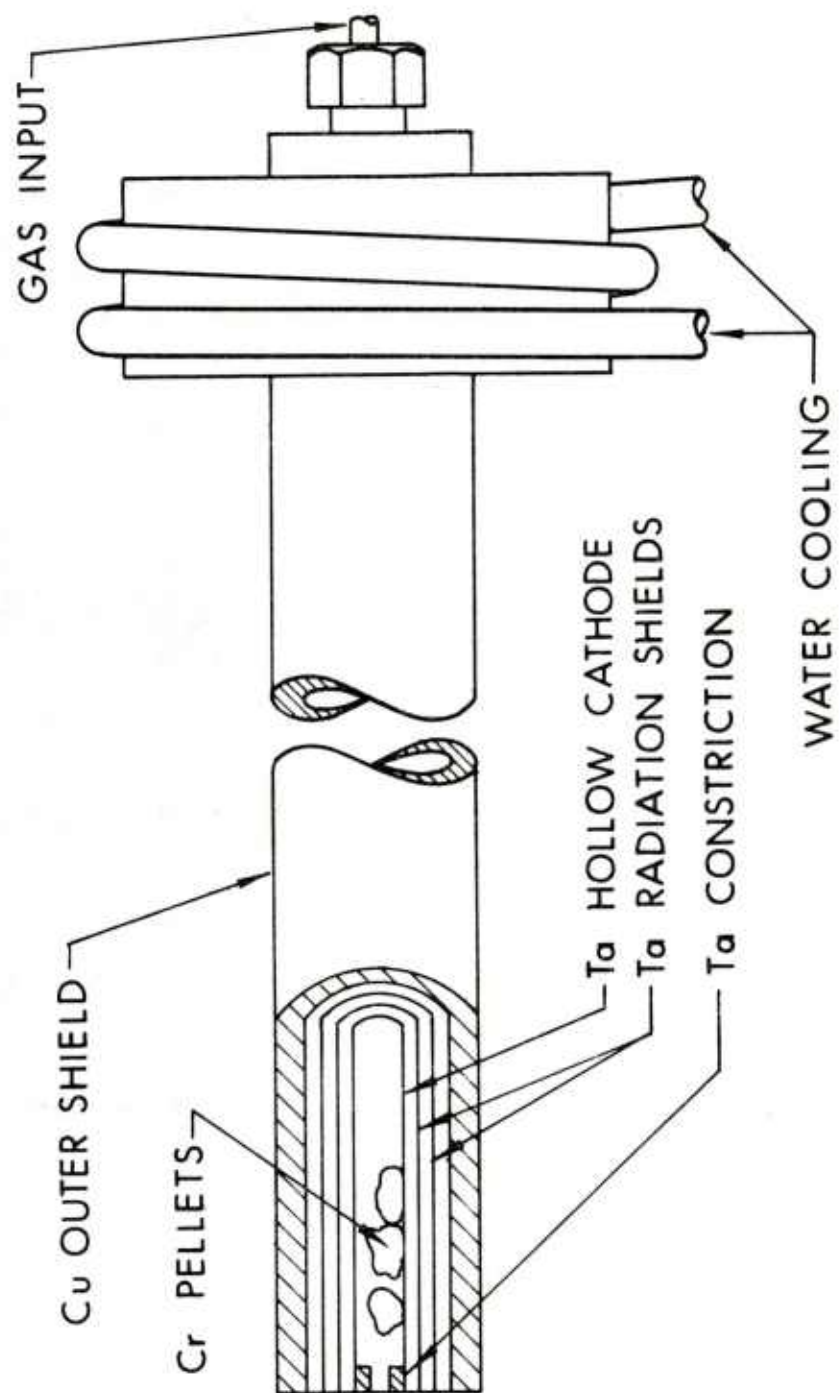


Figure 1. Hollow Cathode Metal Vapor Source.

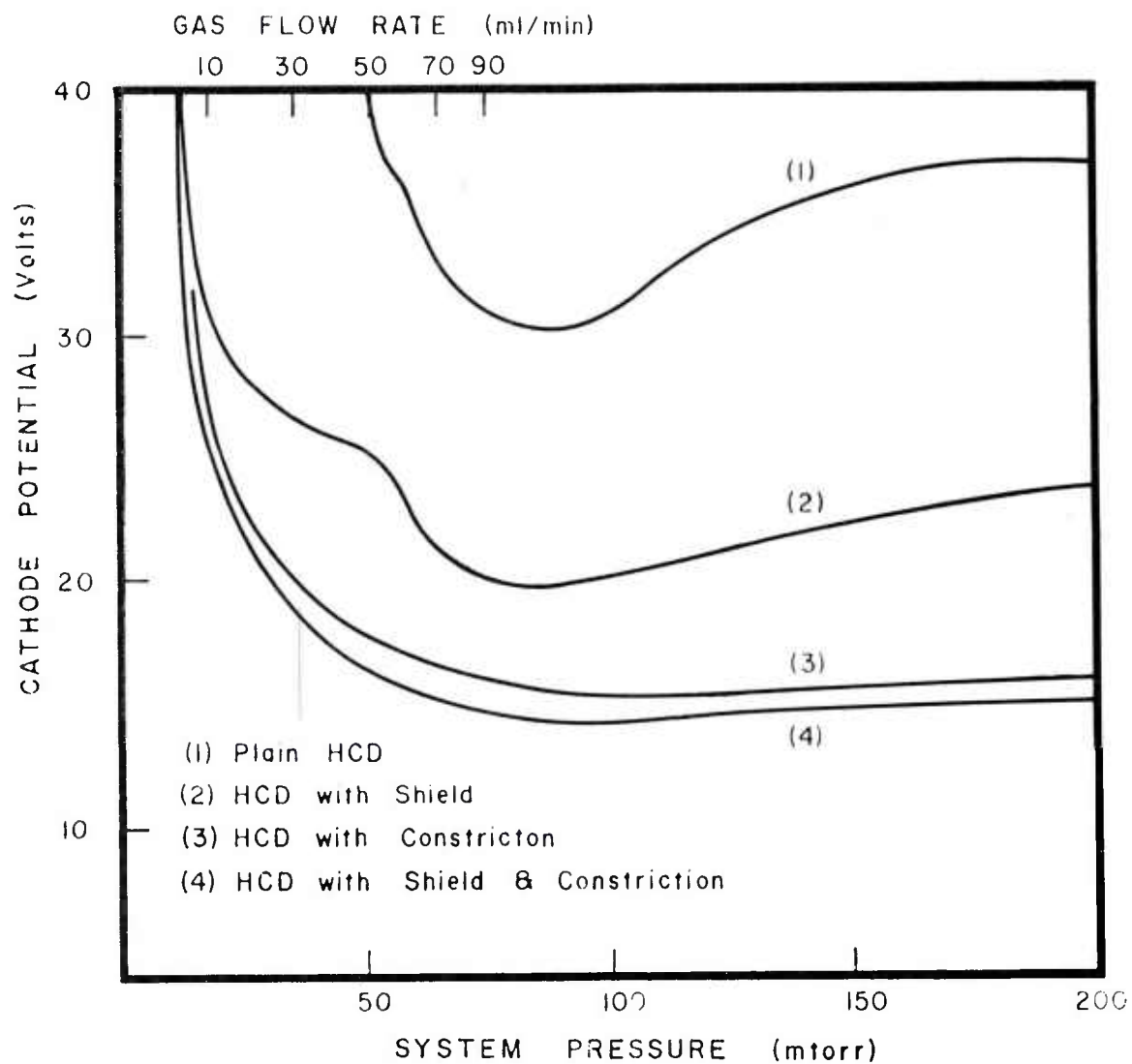


Figure 2. Voltage-Pressure Characteristic of the Hollow Cathodes Showing the Effects of the Construction and Radiation Shields. $I = 15$ amp.

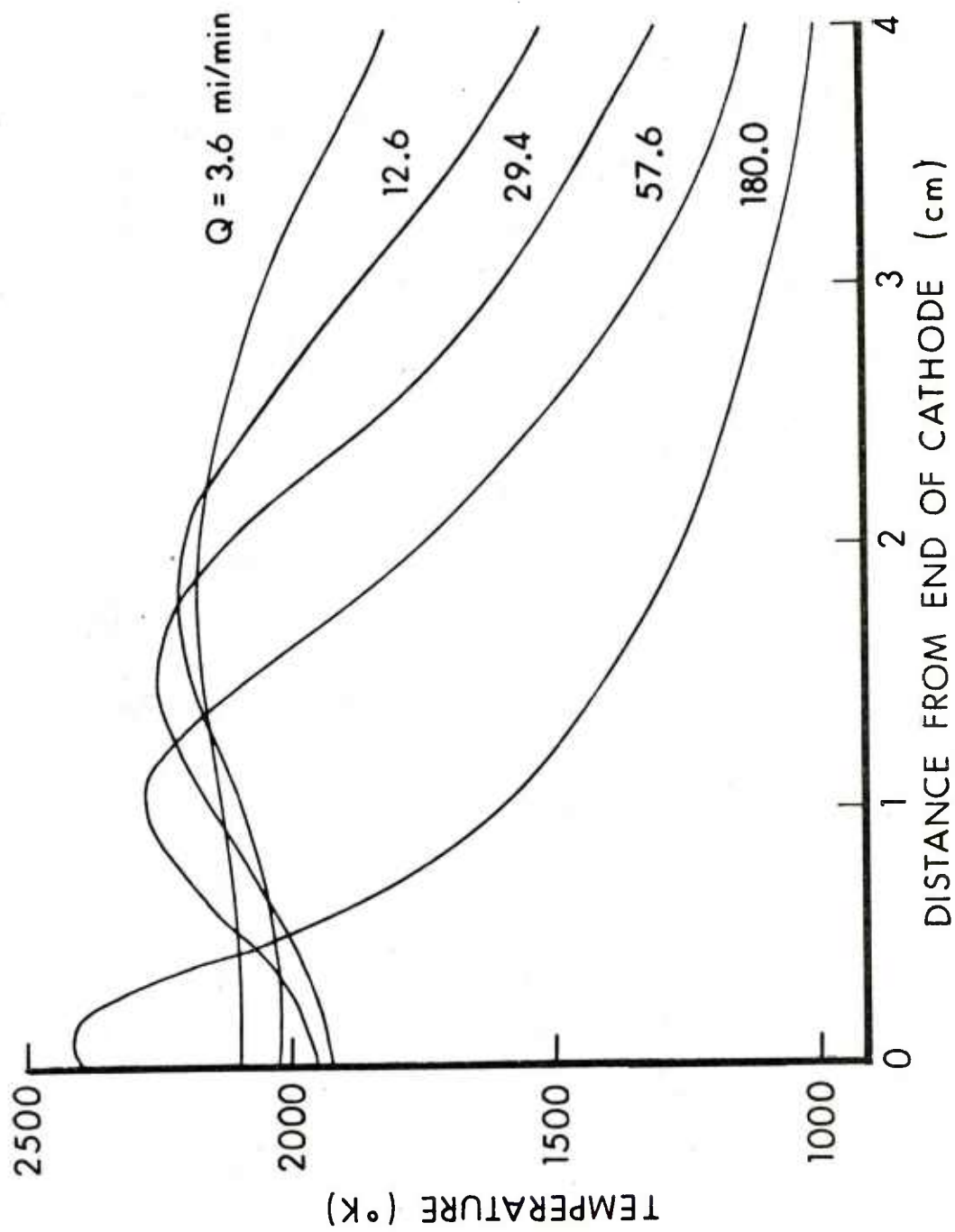


Figure 3. Temperature Profiles of the Hollow Cathode at Various Gas Flow Rates.

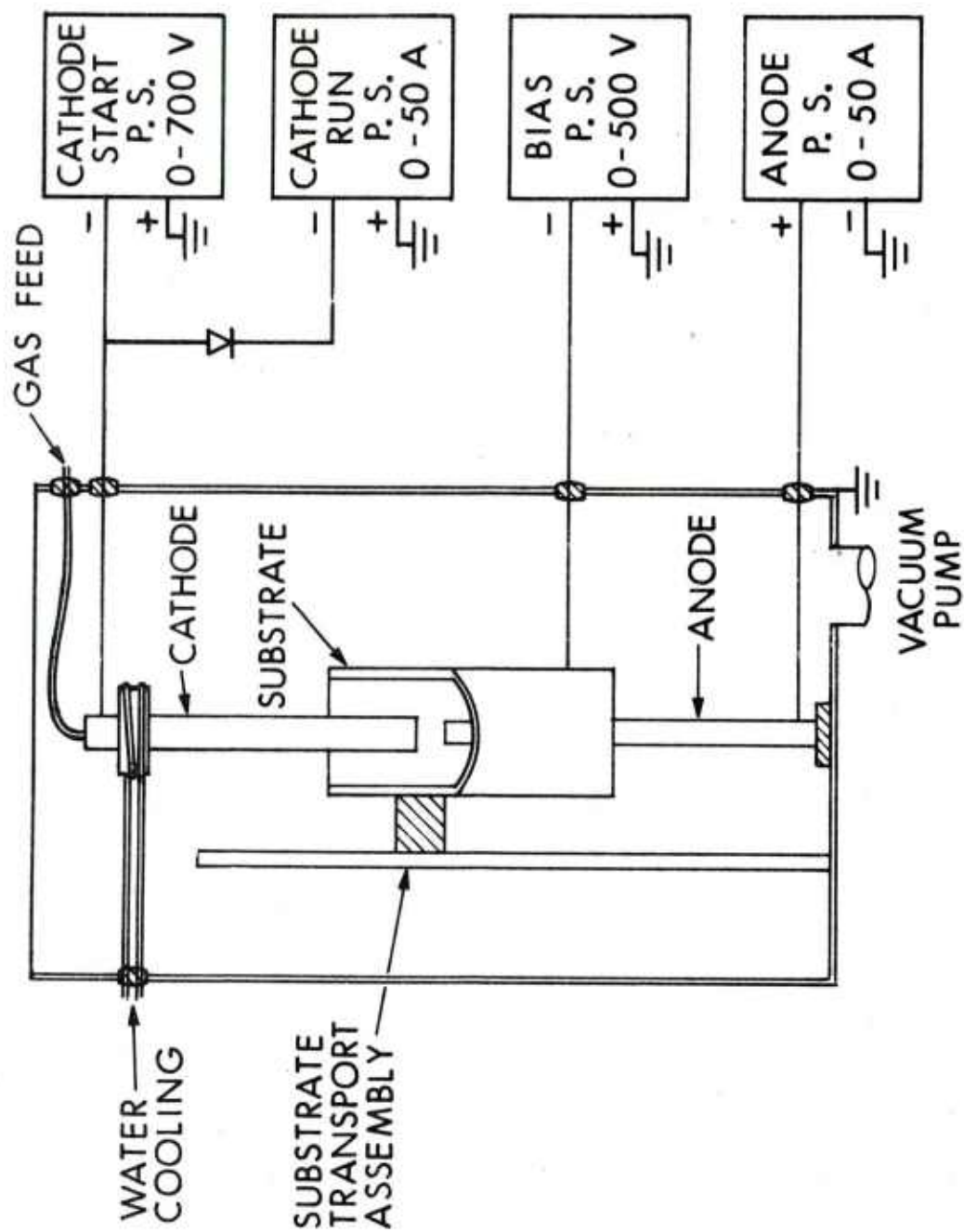


Figure 4. Schematic of the Ion Plating Apparatus.

250 volts. The low substrate bias is a major point of departure from the bias of several kilovolts used in conventional ion plating. In the conventional high pressure systems, the average energy of the atoms striking the substrate is approximately five percent of the bias voltage^{5,6} due to the short mean free path. The average energy of the atoms at the lower pressures used in this experiment is closer to the bias voltage due to the long mean free path and absence of a normal glow discharge around the substrate. Another major point of departure from conventional ion plating is the low operating pressure. This is reasonable since planar geometries require pressures an order of magnitude higher in order to cause some of the atoms sputtered from the substrate to be returned to it through atomic collisions and thus forming the graded film-substrate interface. The confined nature of the cylindrical geometry gives a high probability of a sputtered atom returning to the substrate, forming the graded interface desired.

It is desirable to have the ion plating uniform throughout the length of the substrate tube. In order to achieve this condition, the pumping speed through the tube should be much greater than the system pumping speed. The effective pumping speed⁷ of a tube is $10 d^3/\ell_{\text{eff}}$ liters/sec cm². For a tube with an internal gas source, ℓ_{eff} is equal to or less than $\ell_{\text{actual}}/4$; therefore, the effective pumping speed of the tube is $40 d^3/\ell_{\text{actual}}$ liters/sec cm². A typical tube used in our experiments was 5 cm in diameter by 25 cm in length and had an effective pumping speed of 200 liters/sec. The system had an effective pumping speed of 12 liters/sec and thus there was no noticeable pressure change as the plating region moved from the center to the end of the tube. It can be shown that the actual plating pressure in the tube varies by less than 10% over the length of the plating limits and, as a consequence, uniform plating would be achieved. Typical plating rates observed were 1 $\mu\text{m}/\text{min}$ per centimeter tube length at 20 millitorr of pressure. This decreased at higher pressures due to increased sputtering.

EXPERIMENTAL RESULTS

Ion plating with the first version of the hollow cathode resulted in the substrate becoming hot enough to melt aluminum on several occasions. Therefore, an array of thermocouples was attached to a 5 cm diameter stainless steel substrate to measure the temperature profiles shown in Figure 5. The fact that the temperature peak is skewed toward the anode indicates that anode radiation is a major source of heat. This is supported by observations by Lidsky⁸ et. al. that the power dissipated by the anode is approximately one-

⁵W.D. Davis, T.A. Vanderslice, *Ion Energies at the Cathode of a Glow Discharge*, Phys. Rev. 131, No. 1, 219 (1963).

⁶D.G. Teer, *Adhesion of Ion Plated Films and Energies of Deposition*, J. Adhesion, 8, 289 (1977).

⁷H.A. Steinherz, *Handbook of High Vacuum Engineering*, Reinhold, p. 18, (1963).

⁸L.M. Lidsky, S.D. Rothleder, D.J. Rose and S. Yoshikawa, *Highly Ionized Hollow Cathode Discharge*, J. Appl. Phys. 33, 2490 (1962).

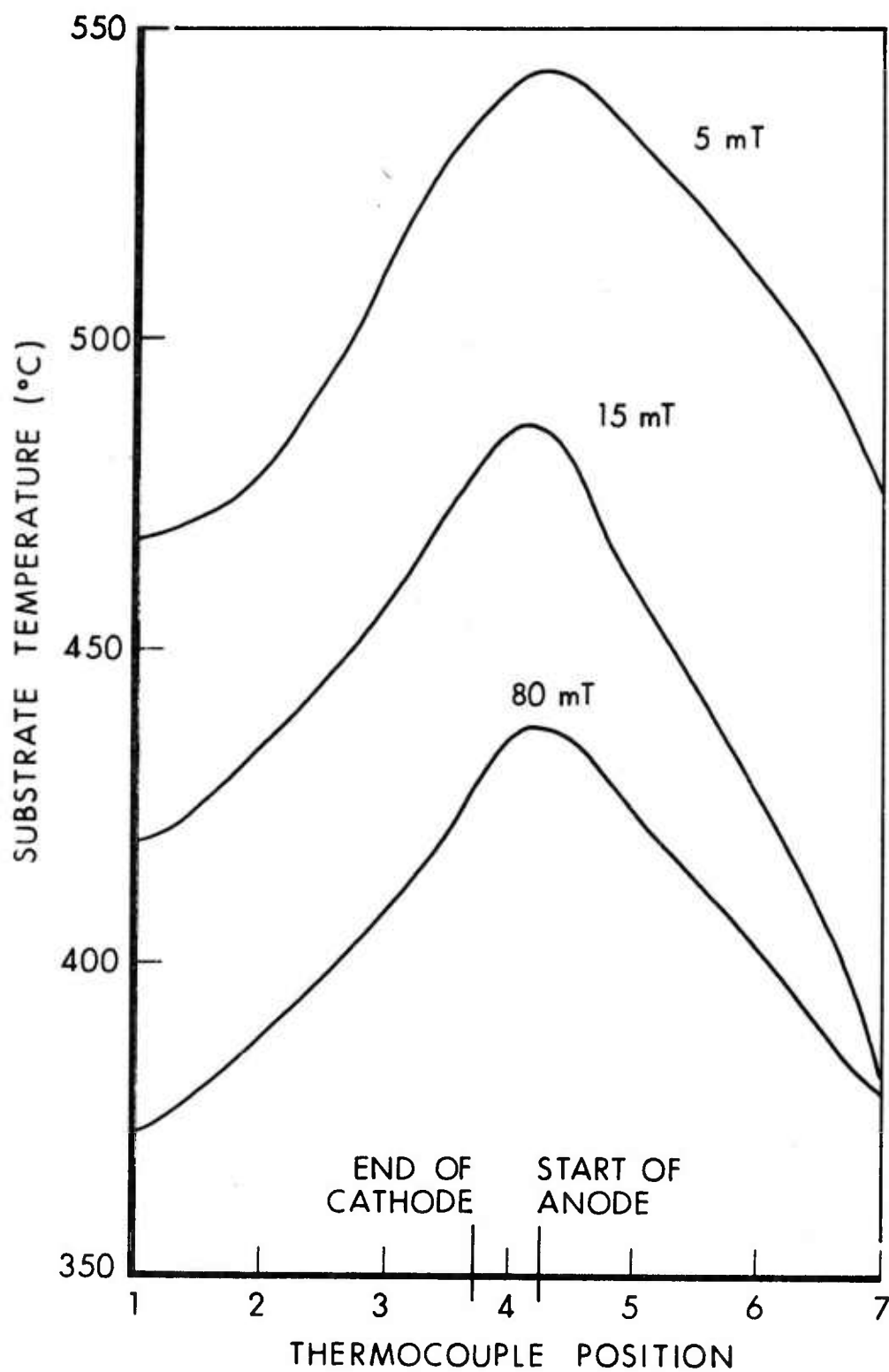


Figure 5. Substrate Temperature Profiles. Thermocouples Were Placed at One Inch Intervals Along a Stainless Steel Tube.

half that of the cathode. The temperature increase with decreasing gas flow rate (lower pressure) is a result of cathode thermal radiation. Although the substrate temperature could have been lowered by water cooling, a temperature gradient may thereby have been established between the inner and outer surfaces of a gun tube. This could have resulted in the temperature rising high enough so as to destroy the gun tube tempering. Therefore, the approach used to reduce the substrate temperature was to improve the control of the cathode-anode thermal radiation. In this approach, the hollow cathode vapor source was assembled by (1) using screw threads rather than light pressure fits in connecting water-cooled copper parts, (2) using indium washers for improved thermal conduction and (3) water-cooling the anode. The substrate temperature profiles shown in Figure 6 resulted from operating this revised version of the HCD. The fact that the curves were no longer slewed toward the anode are the result of the improved anode cooling scheme. The increase in the substrate temperatures from 15 to 80 millitorr was the result of increased ion current heating of the substrate and is an indication that nearly optimal cathode-anode cooling has been achieved.

A series of experiments were conducted to determine the pressure dependence for ion plating using the HCD metal vapor source. The properties measured were: (a) plating efficiency, (b) throwing power and (c) substrate power. A subjective evaluation of the plating uniformity was also made.

The plating efficiency was determined by measuring the film thickness and mass of Cr evaporated. The ratio of the film thickness to the mass of Cr evaporated was then plotted as a normalized thickness. The throwing power was determined by using a 20mm x 40mm microscope cover-slide facing the Cr vapor source. After being coated, the Cr thicknesses were measured on the front and rear surfaces, their ratio being reported as the throwing power. As would be expected, the Cr coating on the rear of the cover glass was not uniform but still provided information about the pressure range for operation of the vapor source. The power dissipated in the substrate was determined from the measurement of the ion current (I) and the substrate bias voltage (V). The substrate power (I-V) is a relative measure of the average temperature of the substrate even though the substrate bias voltage was not an exact measure of the ion energies since the ions lose some energy in gas collisions.

The results of these tests, as shown in Figure 7, indicate that the pressure range of 25 to 50 millitorr is best for ion plating with the HCD vapor source. These pressures are approximately a factor of three lower than are normally used in planar ion plating geometries. This fact can be explained as follows.

The high electron current densities from the HCD source produce a much higher ionization of the working gas than is possible with conventional sources. The larger ion concentrations result in greater sputtering rates of the substrate and thus require the reduced pressures to achieve acceptable sputtering rates. In addition, the lowered pressure produces a higher chromium vaporization rate, increasing the chromium ion concentrations. The resulting higher chromium concentrations and lower substrate sputtering produce an acceptable plating rate.

The subjective aspect of these tests was performed by ion plating 3/4" x 14 TPI brass nuts at various pressures. The plated nuts were sectioned and a

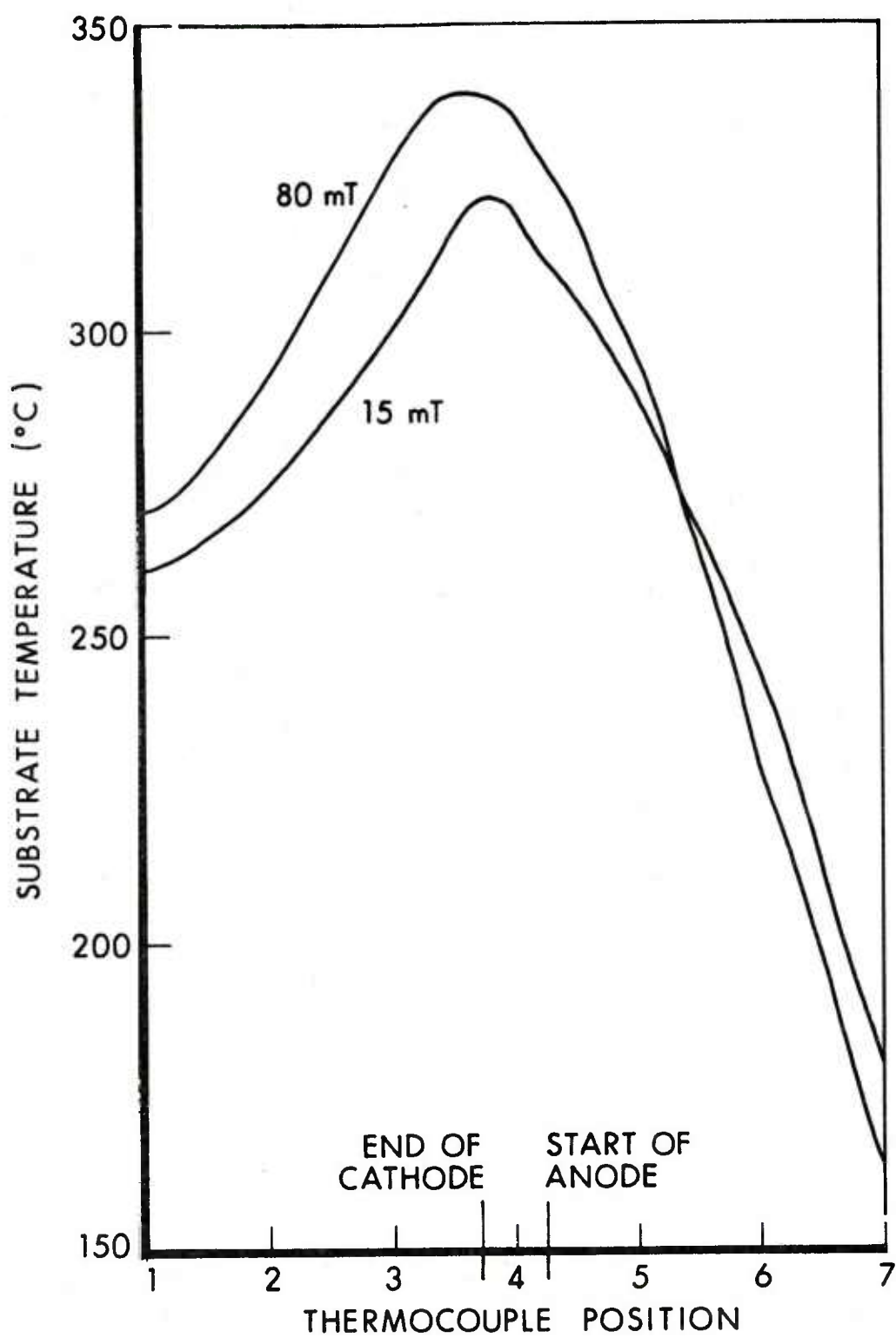


Figure 6. Substrate Temperature Profiles with Improved Cathode-Anode Cooling.

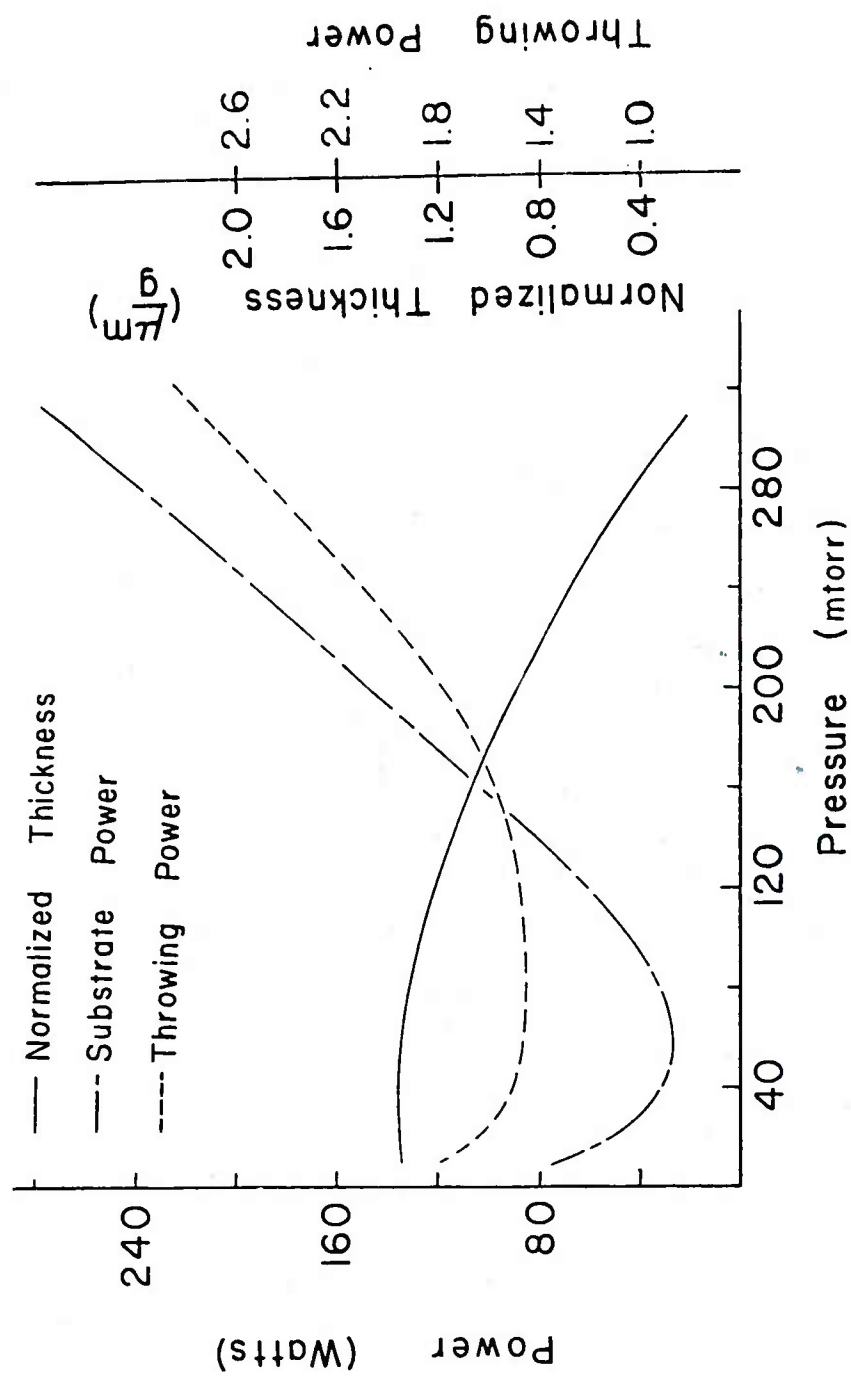


Figure 7. Working Gas Pressure Effects for Ion Plating.

visual appraisal of the coatings was performed whereby plating thickness and throwing power were noted. These evaluations were in good agreement with the above, more quantitative measurements and indicated best results in the 25 to 50 millitorr pressure range.

FILM ANALYSIS

Several chromium films and a few copper films have been deposited by the HCD process. Copper is not a suitable plating material since it forms a low melting temperature alloy with the Ta cathode causing its destruction. Analysis of some of the chromium films was done with a technique developed by Niiler et al.⁹ This analysis consists of placing the plated sample in a beam of 1 MeV deuterons and observing the Rutherford Backscattering (RBS) and Nuclear Reaction (NR) spectra shown in Figure 8. The nuclear reaction peaks are analyzed to give the relative concentrations of the light elements, carbon, oxygen and nitrogen. Curve-fitting the RBS distribution in conjunction with nuclear reaction analysis yields a unique solution for the concentrations of all the major elements in the film. The concentration depth profiles are shown in Figure 9 for a chromium film on an iron substrate. It is observed from this figure that a graded film-substrate interface is present, in agreement with conventional ion plating results. Since chromium diffuses readily into iron at high temperatures, it is difficult to distinguish atomic mixing from thermal broadening. The remainder of the experiments involved ion plating chrome onto the inner surfaces of copper tubes since this film-substrate combination has very little thermal broadening of the film-substrate interface. Figure 10 is the concentration depth profiles of chrome on a 38mm x 25 cm tube. The plating parameters were: (a) a substrate bias of 250 volts, (b) a working gas pressure to 50 millitorr and (c) the tube sweeping past the plating region in three sweeps at four minutes per sweep. The figure shows the effect of the three sweeps by the chrome peaks and illustrates the fact that a continuation of the sweep-plating process would have resulted in building a thicker, pure chromium coating. We believe the broad interface is the result of the confined nature of the ion plating plasma in the tube. As the tube is being moved the sputtered substrate atoms are deposited on a portion of the tube which has already been plated. This process is repeated until all the substrate atoms are covered or escape through the ends of the tube. The depth of the film-substrate intermixing can easily be varied by changing the ion plating parameters of bias voltage and/or working gas pressure. The horizontal scales for Figures 9 and 10 show depth in units of mg/cm^2 . These units can be translated into μm only if the surface densities are exactly known. In the case of Cr and Fe, $1 \mu\text{g}/\text{cm}^2$ translates to approximately $1.3 \mu\text{m}$.

The next phase of the experiment was the investigation of plating uniformity over lands and grooves as would be found in gun tubes. To this end, grooves were cut into a 30mm x 15 cm copper tube. The plating parameters were: (a) substrate bias of 250 volts, (b) working gas pressure of 40 millitorr and the tube being swept past the plating region 10 times in 30 minutes.

⁹ A. Niiler, R. Birkmire and J. Gerrits, "PROFILE: A General Code for Fitting Ion Beam Analysis Spectra," Ballistic Research Laboratory Technical Report ARBRL-TR-02233, April 1980. (AD A084984)

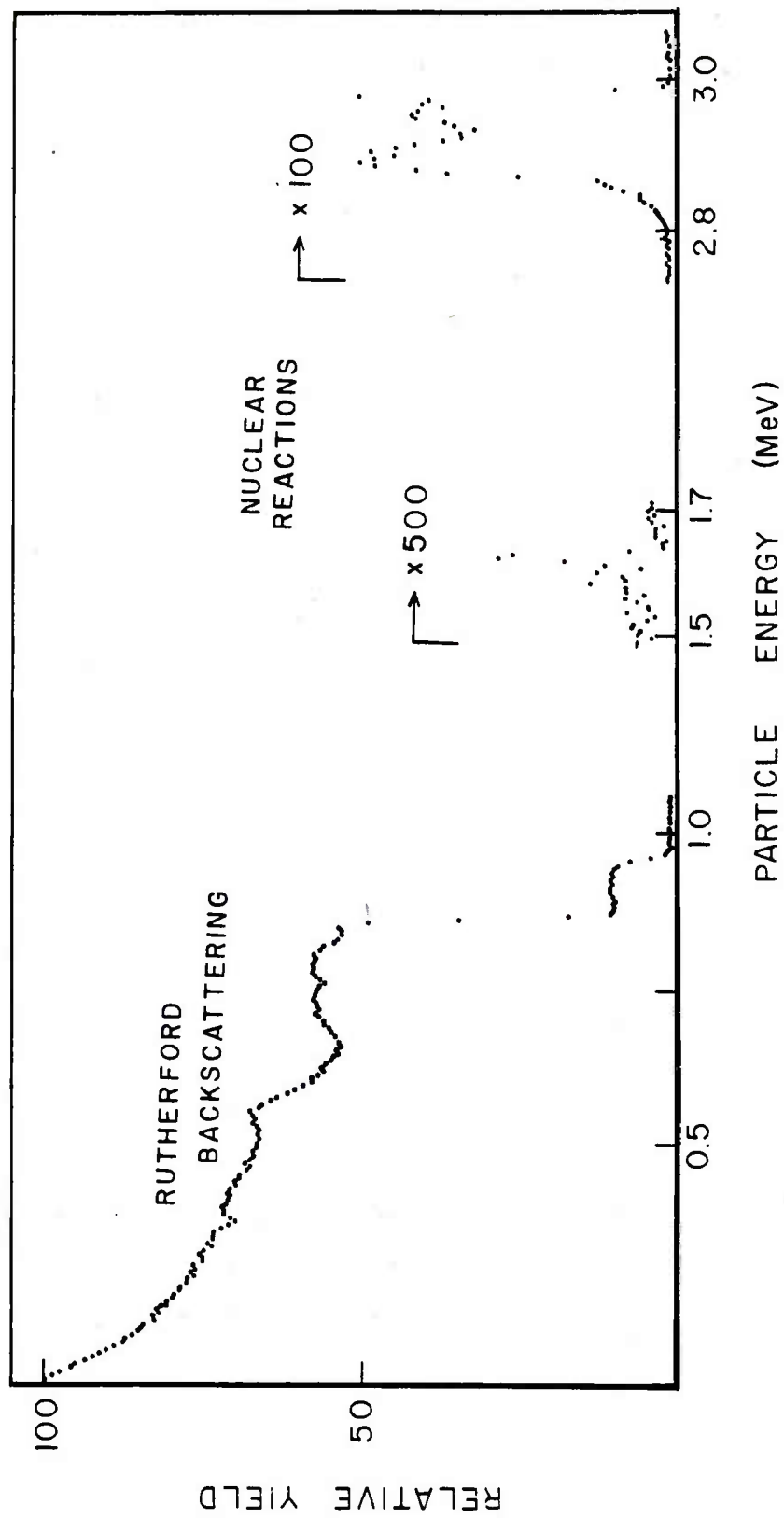


Figure 8. RBS and NRA Spectra for a Chrome Film on an Iron Substrate.

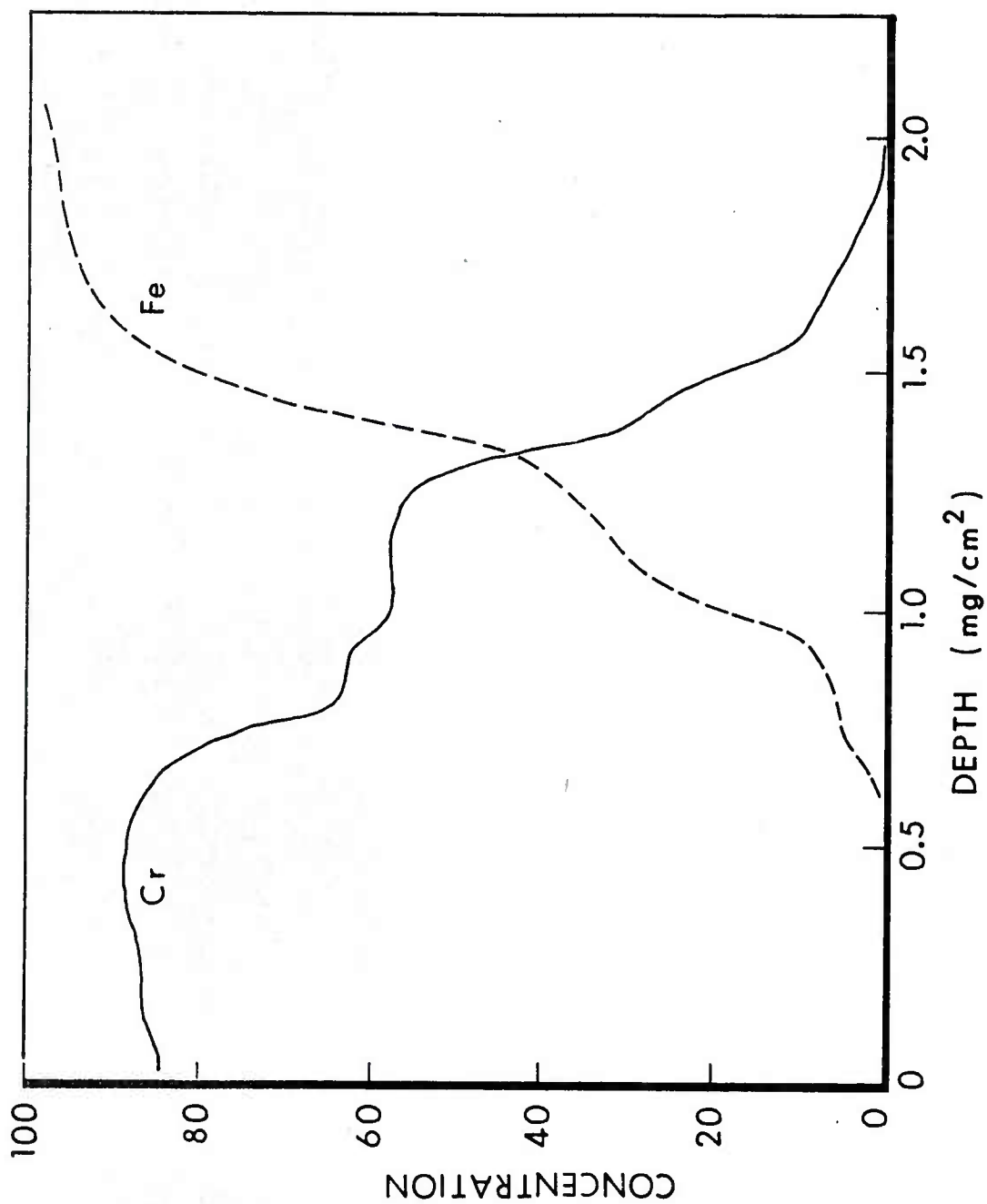


Figure 9. Concentrations of the Chrome Film on Iron Substrate Versus Depth Found by Computer Analysis of the RBS and NRA Spectra.

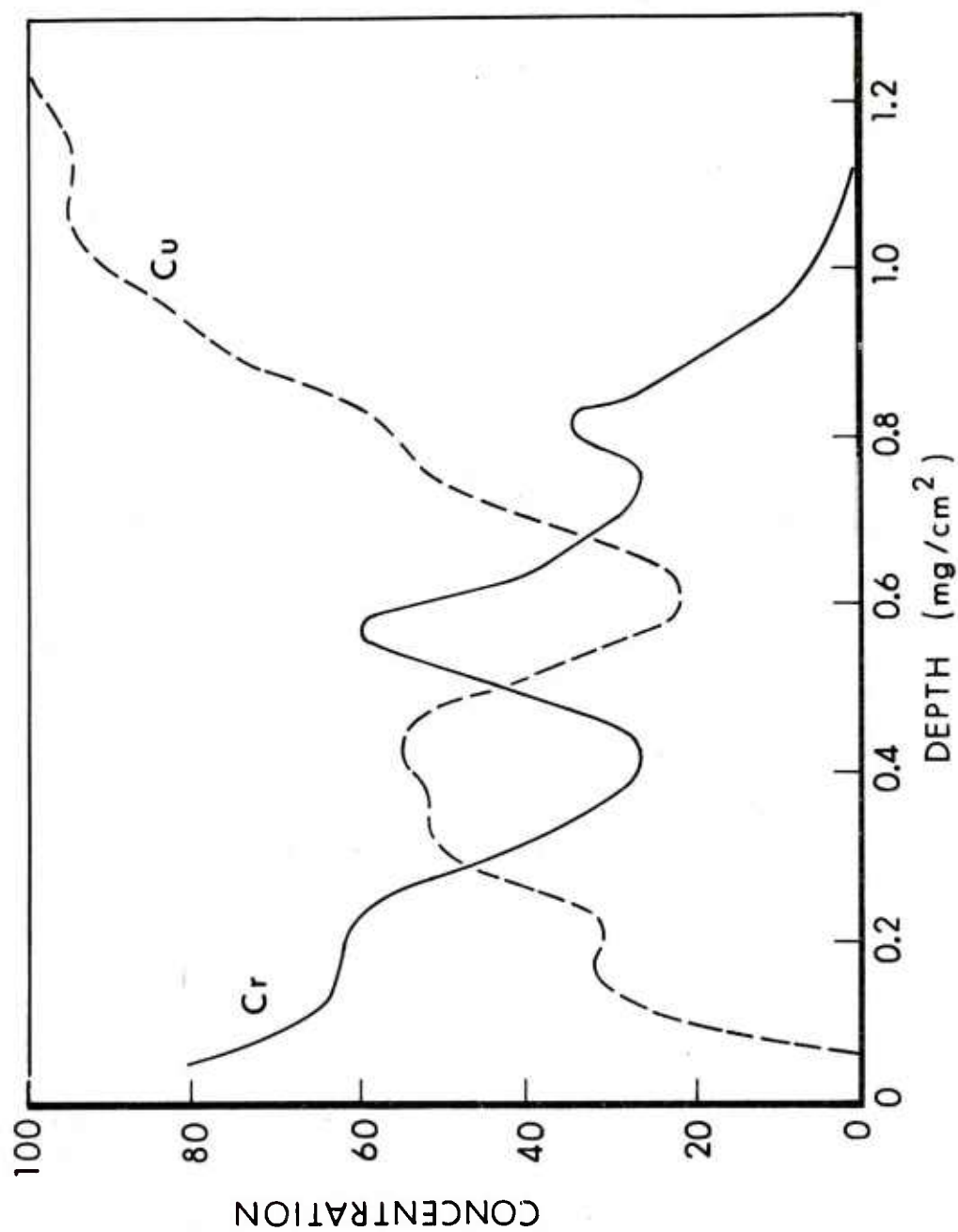


Figure 10. Concentrations of a Chrome Film on Copper Substrate Versus Depth.

Micrographs, Figure 11, show the plating is continuous with approximately a factor of two difference between the thickest and thinnest portions of the film, with the thin sections lying in regions on the wall shadowed from the HCD source as would be expected. The factor of two difference between the thick and thin sections of the coating can be decreased by plating at a higher pressure with the only disadvantage being a slightly slower plating rate.

FUTURE WORK

Several areas of investigation are necessary for determining the optimum plating for the protection of gun tubes against erosion. A blow-out chamber designed at the Ballistic Research Laboratory for the testing of nozzles^{10,11} has shown ion plated films have a higher resistance to propellant erosion than conventional coatings, although the sample numbers were too small to give definitive figures of merit.

The optimum plating parameters such as substrate bias voltage and working gas pressure as well as coating thickness need to be determined by ion plating nozzles and testing them in a blow-out chamber. Researchers at Harwell, UK,¹² have observed that as deposited thick films crack under thermal shock, the cracks propagate into the substrate to a depth of about one-half the film thickness. The crack propagation is then parallel to the film surface causing the film to flake off, removing a considerable amount of the substrate with it. This is what may be happening to the thick chromium deposits produced by electro-deposition which has unpredictable, catastrophic failure. The criteria to prevent these cracks from forming can be found from the following analysis. The elastic energy U_0 stored in a tensile film is:

$$U_0 = Y \cdot d \cdot (\Delta\alpha)^2 \cdot (\Delta T)^2 \cdot (1-\nu)^{-1} \quad (1)$$

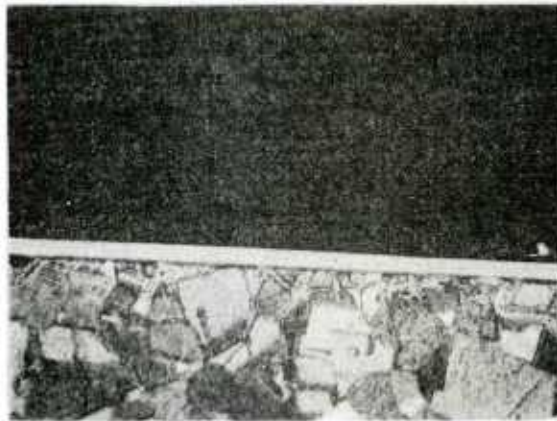
where Y is the Young's modulus, d is the film thickness, $\Delta\alpha$ is the difference in coefficients of thermal expansion of the film and substrate, ΔT is the difference between the deposition temperature and the temperature of the thermal shock and ν is Poisson's ratio. When the internal energy is less than the energy dissipated per unit area of crack surface γ , flaking of the coating can be avoided. I.e., if

$$d < 2\gamma \cdot (1 - \nu) \cdot [\gamma \cdot (\Delta\alpha)^2 \cdot (\Delta T)^2]^{-1} \quad (2)$$

¹⁰ A. Niler, R. Birkmire and S.E. Caldwell, "The Effects of Propellant Burn on The Surface Composition of Gun Steel," Ballistic Research Laboratory Technical Report ARBRL-TR-02380, November 1981. (AD A108292)

¹¹ I.C. Stobie, R.P. Kaste, B.D. Bensinger, T.L. Brosseau, J.R. Ward, J.R. Mullaly, P.A. Allard, "Screening Gun Barrel Coating's Response to Combustion Gases," Ballistic Research Laboratory Technical Report ARBRL-TR-02396, March 1982. (AD A113519)

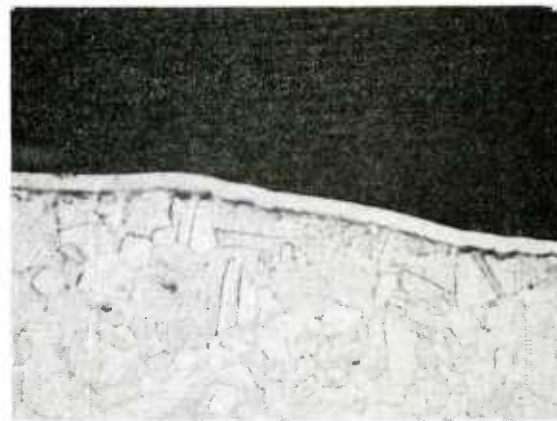
¹² J.F. Coad, R.A. Dugdale and L.P. Martindale, "Ion Cleaning in Relation to Coating Adhesion," Harwell Memorandum Report AERE-R-10256, July 1981.



LAND



WALL



GROOVE

Figure 11. Photomicrograph of a Chrome Coated Copper Substrate With Simulated Rifling.

Typical values for chromium on iron are: $\nu = .25$, $\gamma = 23 \times 10^{10} \text{ Nm}^{-2}$, $\Delta\alpha \text{ Cr-Fe} = 5.5 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$, and $\Delta T = 1000^{\circ}\text{C}$. Gamma (γ) is not known for chromium but from work done on glasses, values between $10^1 - 10^2 \text{ J/m}^2$ would be expected. Using these values, we find the critical coating thickness is between $2 \text{ }\mu\text{m}$ and $20 \text{ }\mu\text{m}$. Therefore, a thin film of about $10 \text{ }\mu\text{m}$ may offer better protection against gun tube erosion than the thick films 150 to $200 \text{ }\mu\text{m}$ which are now used. This latter point of view is also a conclusion of L.H. Russell.¹³

The possibility also exists that a single chrome coating is not the ideal material for erosion protection. The thermal conductivity of chrome is greater than that of the steel substrate so the temperature at the chrome-steel interface may increase to the point that the steel would melt causing the chrome to flake off. Consequently, a better coating might be a thermal insulator such as chrome oxide. The difficulty with chrome oxide is its brittleness which necessitate using it in a laminar structure with Cr. It is well known that coatings thinner than a μm begin to assume the mechanical properties of the substrate. Therefore, an alternating laminate of chrome oxide/chrome built up to a thickness of approximately $20 \text{ }\mu\text{m}$ with each coating less than $1 \text{ }\mu\text{m}$ in thickness, deposited by ion plating, may well have the ideal properties. It would be a thermal insulator, have a high melting temperature and still have the mechanical properties of the substrate.

A third area which needs investigation is the possibility of using ion plating in conjunction with other plating processes. Although it has been extensively documented that a graded film-substrate interface is instrumental in achieving good film adhesion to the substrate, the benefits of mixing the deposition atoms with the film atoms after the interface is formed has not been investigated. Therefore, a chrome film a few μm thick produced by ion plating could then be covered with electrodeposited chrome to bring the film to the desired thickness. The electrodeposited chrome should adhere much better to a chrome substrate than to a steel substrate.

CONCLUSIONS

We have developed a method which is capable of ion plating the inside surfaces of gun tubes with little restriction on their length. The films produced by this process have large graded film-substrate interfaces even for materials such as chrome and copper in which thermal diffusion is negligible. The extended interface is a result of utilizing the confining geometrical nature of a tube to contain and intermix the deposition and substrate atoms more than in conventional planar ion plating technology. The present experiments have also shown that the plating is uniform over simulated lands and grooves. These results show that the process is a feasible method of protecting gun tubes against erosion.

¹³L.H. Russell, "Simplified Analysis of the Bore Surface Heat Transfer Reduction in Gun Barrels as Achieved by Using Wear-Reduction Additives," Naval Surface Weapons Center Technical Report NSWC-TR-3378, October, 1975.

ACKNOWLEDGEMENTS

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