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## **The Role of Neutral Atoms in Ion Plating**

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## NOTE

# The Role of Neutral Atoms in Ion Plating

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## INTRODUCTION

Ion plating is essentially vapour deposition on to a clean substrate in a glow discharge. Usually, the substrate is the cathode of the glow discharge and is cleaned by the sputtering action of the discharge. The adhesion between coating and substrate is excellent, even in those cases where the materials do not alloy.<sup>1, 2</sup> The requirements for good adhesion have been discussed by Mattox<sup>3</sup> and in brief are a clean substrate surface and the formation of a graded interface, causing not only a good metallurgical bond, but dispersing stresses due to differences in properties, e.g. thermal expansion coefficients, between coating and substrate. Deep graded interfaces have been reported in ion plated specimens where alloying is possible,<sup>4</sup> but not for incompatible materials. One of the main reasons given in the past for the excellent adhesion of ion plated films has been penetration of the substrate lattice by the ionised depositing vapour atoms, under the action of the substrate bias voltage, i.e. ion implantation. However, this view can be criticised on three counts. Even if the ions had the full energy of the discharge, i.e. 5 keV for a 5 kV discharge the depth of penetration would not be more than about  $50 \text{ \AA}$ <sup>5</sup> rather than the depths of several microns reported,<sup>4</sup> also the ions must lose energy in collisions with neutrals and finally, only a small percentage of the atoms are ionised.<sup>6, 7</sup> Davis and Vanderslice<sup>8</sup> have presented a theory of the energy distributions of ions in a glow discharge and Teer<sup>7</sup> has extended this to obtain an approximate expression for the number of energetic neutrals produced

together with the distribution of energy. The conclusion reached was that ion plating was the deposition of a small number of ions and a large number of neutrals, the mean energies of both ions and neutrals being of the order of 100 eV. There is little direct evidence with which to test this conclusion, but Komiya and Tsuruoka<sup>9</sup> found that the major contribution to heat input to the substrate in ion plating experiments using a hollow cathode source and low substrate bias voltages came from energetic neutrals. In order to test the importance of energetic neutrals, under more conventional bias voltages and gas pressures, the experiment described below has been performed.

## EXPERIMENTAL

The apparatus was as shown in Figure 1. Instead of a specimen, a fine wire mesh has been attached to the H.T. electrode. The specimen is at earth potential and is placed a few millimeters (less than the cathode dark space for

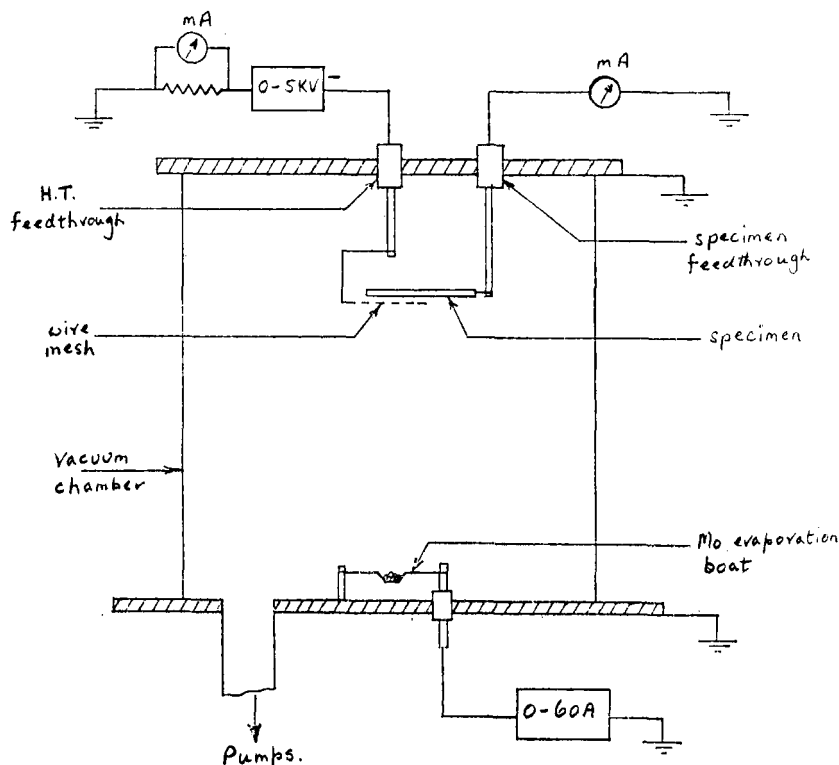


FIGURE 1 Schematic of apparatus.

the conditions used), above the mesh, part of the specimen being directly behind the mesh and part unobscured by the mesh. The experimental conditions were mesh potential  $-4$  kV, specimen potential zero, and argon gas pressure  $10 \mu$ . The specimens were of copper and steel. The discharge between remote earthed parts of the apparatus and the mesh was struck and was left on for 50 minutes, the current to both mesh and specimen being measured. The specimen was then examined under an optical microscope. New specimens of both copper and steel were then placed behind the mesh, the discharge was struck for 50 minutes, and then silver was evaporated from a molybdenum foil source, into the discharge. The specimens were coated in both the regions behind and remote from the mesh. Scratch tests using a modified microhardness tester were performed on the coatings on both regions of the specimens.

## RESULTS

Optical microscopy of those regions of the specimen behind the mesh indicated the specimens had been sputter etched, as the grain structure of the previously polished surfaces was clearly visible. The current to the mesh dropped to a steady value of 6 mA during the argon ion bombardment, while

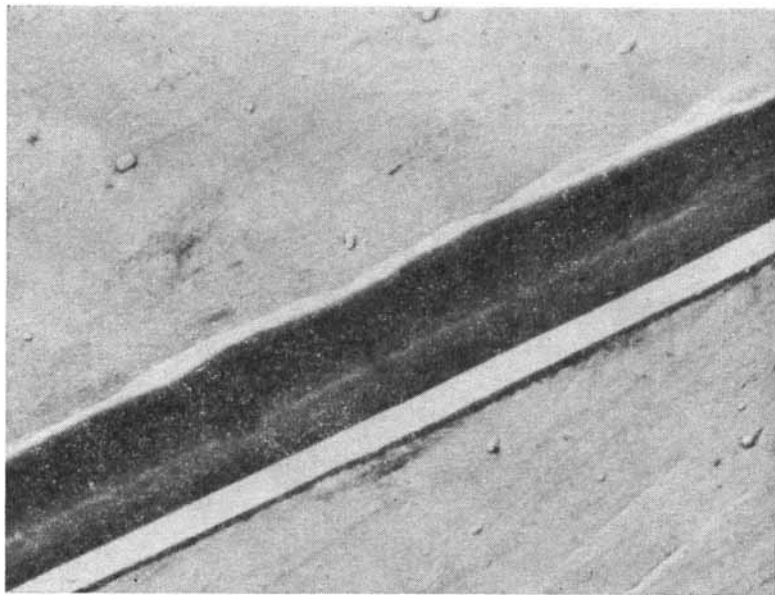


FIGURE 2 Ag/Cu, with mesh in front.

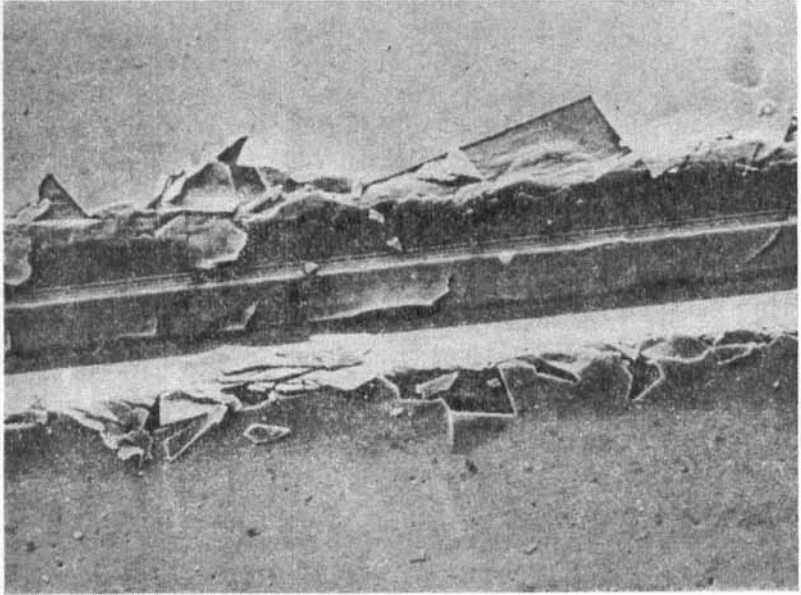


FIGURE 3 Ag/Cu, without mesh.

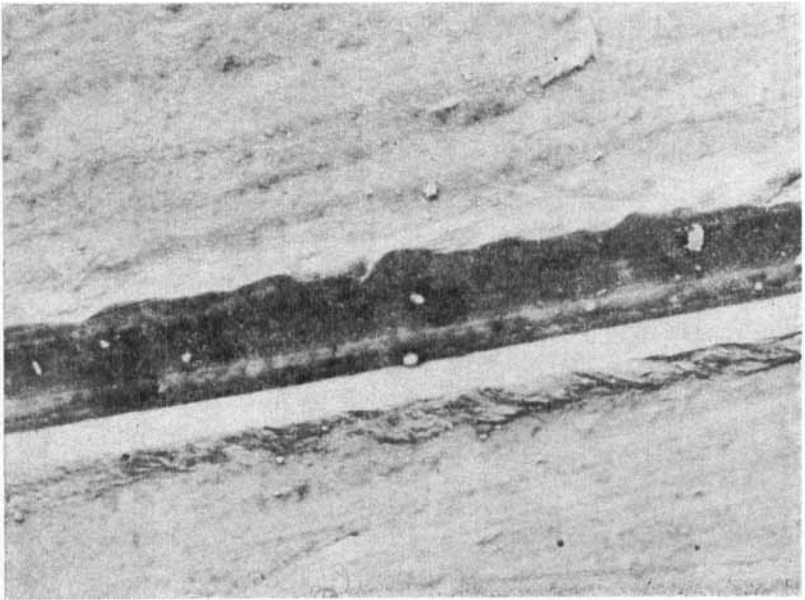


FIGURE 4 Ag/Steel, with mesh in front.

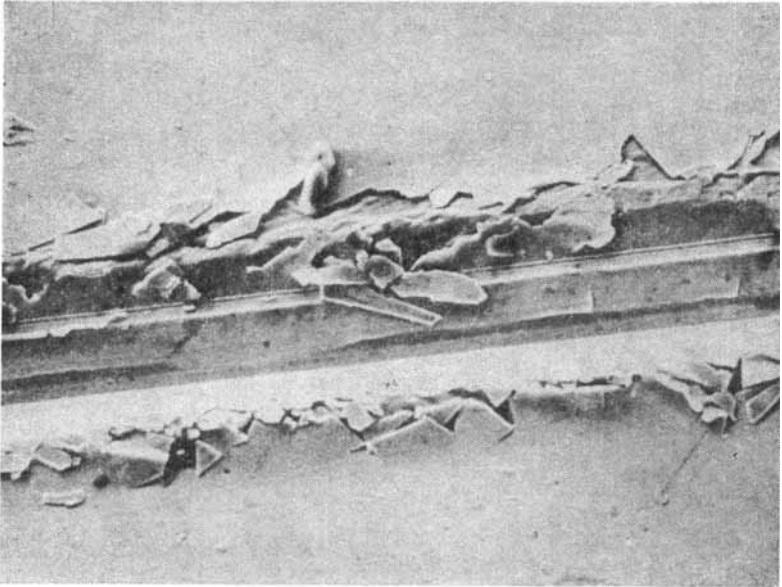


FIGURE 5 Ag/Steel, without mesh.

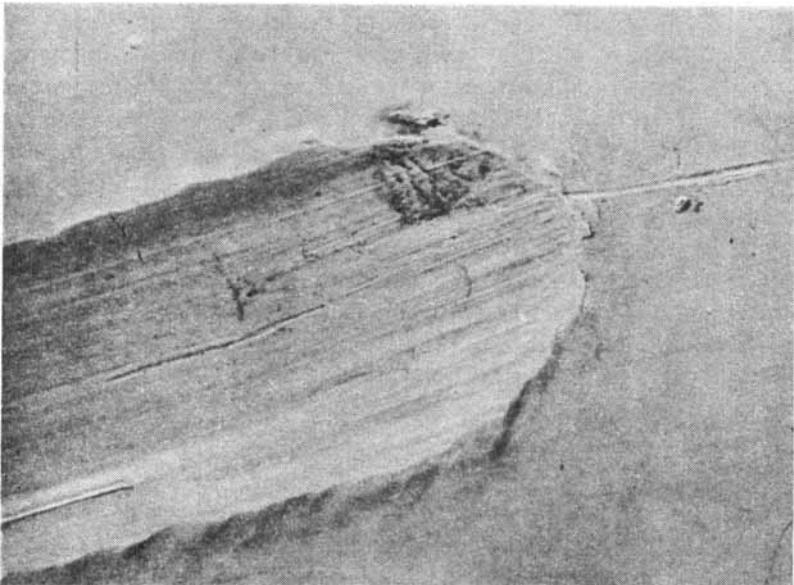


FIGURE 6 Ag/Steel, with mesh. Hemispheric indenter.

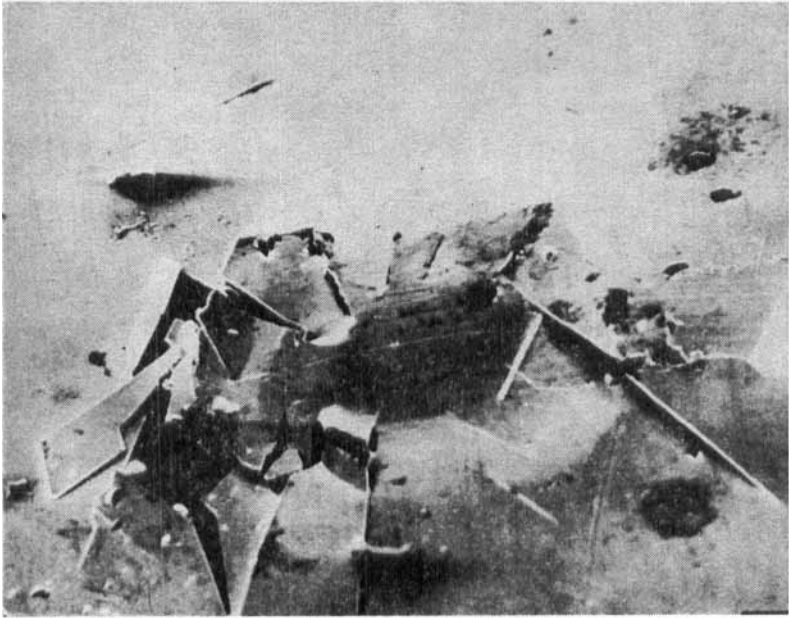


FIGURE 7 Ag/Steel, without mesh. Hemispheric indenter.

the corresponding steady value of current to the specimen was  $-0.4$  mA (the minus sign indicating a net flow of electrons to the specimen). The currents during the silver deposition were 1 mA to the mesh and no current could be detected to the specimen. Scanning electron micrographs of the scratch tests are shown in Figures 2–7. Figure 2 shows the good adhesion typical of conventional ion plating obtained for silver on copper in the region behind the mesh, while Figure 3 is a scratch in the silver on copper deposited on that part of the specimen away from the mesh. The adhesion is poor, and similar micrographs have been obtained previously for vacuum evaporated films. Figures 4 and 5 are for silver on steel, Figure 4 is of the region behind the mesh and Figure 5, the region away from the mesh. Figures 6 and 7 are comparable but a hemispherically ended steel indenter was used rather than the diamond used in the previous tests.

## DISCUSSION

The adhesion characteristics of the coatings produced behind and away from the mesh are quite different. Those behind the mesh appear to be comparable to conventional ion plating and those away from the mesh have poor adhesion. This poor adhesion is to be expected and will not be discussed further.

Consider now the coatings produced behind the mesh. It might be argued that in the case of the silver on copper the adhesion was improved by diffusion aided by heat radiated and convected from the mesh, as silver and copper can alloy. However, such diffusion will be inhibited unless oxide films on the copper are removed. In fact, it appears that such films are removed during the 50 minute discharge period, which indicates that the copper specimen was subject to bombardment by energetic particles, in which case it appears probable that the deposition was by energetic particles.

In the case of the silver on steel, alloying and diffusion are not possible, but again the adhesion appears to be comparable to that produced by ion plating. The most likely explanation for this result is that the depositing particles were of high energy. The measurements of current to the specimen and mesh indicate that the depositing particles were not positive ions. It is possible that some of the particles were negatively charged ions, accelerated across the field between mesh and specimen, but if so the numbers involved must have been very small, and therefore it is unlikely that they could affect the overall adhesion. It is more likely that the current to the specimen during the ion bombardment of the mesh was due to electrons, emitted by the mesh under the ion bombardment, and accelerated to the specimen. Again it is unlikely that the electron bombardment could markedly influence the adhesion.

Remembering the two facts, that the specimen behind the mesh was etched during the discharge, and that the adhesion of the coatings was comparable to that of ion plated specimens, the most probable explanation is that the specimen is bombarded by energetic argon neutrals, and that the deposition is by energetic silver neutrals. If this is correct, then it adds some confirmation to the views presented by Teer<sup>7</sup> on the energies of particles involved in ion plating and the reasons for the high adhesion obtained.

## SUMMARY AND CONCLUSIONS

Coatings have been deposited on earthed specimens, placed close behind a mesh held at  $-4$  kV. The adhesion was comparable to that obtained by ion plating. The most probable reason for the adhesion is that deposition in this case is by high energy neutrals. In the case of conventional ion plating it is probable that deposition is by a large number of energetic neutrals and a small number of energetic ions.

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