Redeposition of Sputtered Material in a Glow-Discharge Lamp Measured by Means of an Ion Microprobe Mass Analyser

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The redeposition of sputtered material on the target in a Grimm-type glow-discharge lamp was studied by means of an ion microprobe mass analyser (IMMA) using $^{16}O_2^+$ ions as bombarding species. The target was an aluminium disc with a cylindrical copper insertion, one mm in diameter. The lamp was operated at currents of 50 mA and 100 mA and a voltage of 1200 V. It is estimated that 17% of the copper atoms sputtered are redeposited and may be resputtered.

1. Introduction

Sputtering rates are normally estimated by weighing a sample before and after the sputtering process [1, 2], or by measuring the depth of the sputtered area [3]. These methods will only render true sputtering rates if no redeposition of sputtered material occurs. To measure the redeposition rate, the redeposited layer and sample must be of different compositions. This was achieved by using an aluminium disc with a copper insert for use as the tracer metal. Redeposition of the tracer during sputtering in a Grimm-type glow-discharge lamp was then measured by means of an ion microprobe mass analyser.

Redeposition of sputtered material in the Grimm-type glow-discharge lamp [4] may be caused by

1. Backscattering by carrier gas: The pressure in the lamp is between 200 and 800 Pa, and at room temperature the mean free path length of the argon atoms is in the range of 8–30 μm. By collisions, a portion of the sputtered atoms will be reflected back onto the sample.
2. Ionisation: The sputtered atoms will be partly ionised in the plasma of the negative glow by the Penning ionisation process [5] and will be attracted towards the sample as a result of the electric field.
3. Carrier gas flow: The gas flows at rates of 1–2 Pa litres/s in the general direction of the sample, carrying some of the sputtered particles towards the anode-cathode gap region.
4. Diffusion: After a sufficient number of collisions the sputtered particles leave the plasma by diffusion in all directions [6, 7].

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2. Experimental

Sample discs were prepared as follows: A hole, 1 mm in diameter, was drilled into the flat surface of an aluminium disc, 20 mm in diameter, and a copper rod of correct diameter was inserted. These samples were then exposed to sputtering in a glow-discharge lamp with a burnspot of 8 mm in diameter, thus making it possible to study the redeposition of the core material on the matrix material.

Discharge currents of 50 and 100 mA and exposure times of 60 and 120 s were used. The voltage was kept constant at 1200 V.

The sputtered surfaces were studied by means of an ion microprobe mass analyser (IMMA, ARL type). The area available for investigation was 5.2 mm in diameter. Oxygen ions $^{16}O_2^+$ with an energy of 20 keV were used as bombarding species. A rather soft beam of about 1 nA and a beam diameter of 2 μm was selected to prevent too rapid removal of the thin layer of redeposited material.

Apart from taking ion micrographs, the sample was driven mechanically by means of a motor to establish a long line scan across the whole sample. A low speed of about 2 μm per second was used.

A scanning width of 2 μm and a measuring interval of half a second give an effective covered area of 2 μm² per recorded signal. The transmission of the instrument was assumed to be 10%, therefore, each measurement represents 10% of the sputtered ions from an area of 2 μm².

3. Results

Figures 1a, b and c depict ion micrographs of ^{63}Cu ions taken from the core and its immediate vicinity. Figure 1a represents an image of the virgin surface and Figs. 1b and 1c are taken after 30 and 120 s, respectively. Figure 1d is a ^{63}Cu ion

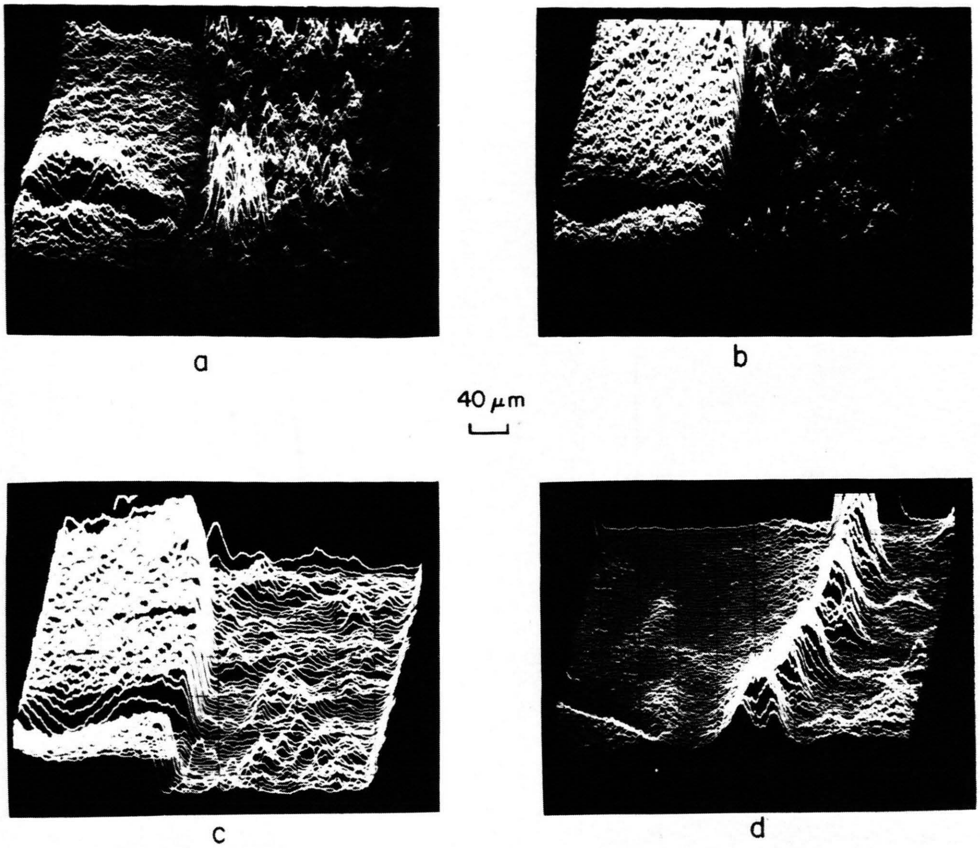

Fig. 1. Ion micrographs of $^{63}Cu^+$ ions. Copper from the insertion is redeposited on aluminium when exposed to a glow discharge. Bombarding species $^{16}O_2^+$ ions, current about 1 nA, beam diameter about 2 µm. a) Initial scan; b) After 30 s; c) After 120 s; d) Ring in anode-cathode gap region.

micrograph of the anode-cathode gap region. From the figures it is seen that copper is unevenly distributed near the insertion and also accumulated near the anode-cathode gap.

Although a very soft beam of about 1 nA was used, the signal from the redeposited copper disappeared after a sputtering period of 120 s (cf. Figure 1c). The redeposited copper layer could not be seen on a scanning microscope (20 kV), which says that it must be thinner than 10 nm. Longer exposure to sputtering in the glow discharge did not influence the signal nor the thickness of the layer considerably. An equilibrium between redeposition and resputtering of copper appears to be reached.

The results obtained using the line scan across the sample are plotted in Fig. 2, which shows the measured counts per half second vs. the distance from the copper insertion. The line scan was repeated three times along the same line. The accumulation of copper atoms on the anode-cathode gap region can again be seen.

4. Evaluation of Results

The following assumptions are made to enable quantitative statements:

1. An equilibrium exists between the redeposited and the resputtered copper.
2. No resputtered copper is redeposited on the sputtered area.
3. The redeposition around the copper core is radial symmetric and the surface density of the copper atoms is uniform within a concentric ring 1 µm in width.
4. The ion yields for copper from the copper insertion and from the deposited alunimium-copper layer on the aluminium are the same.

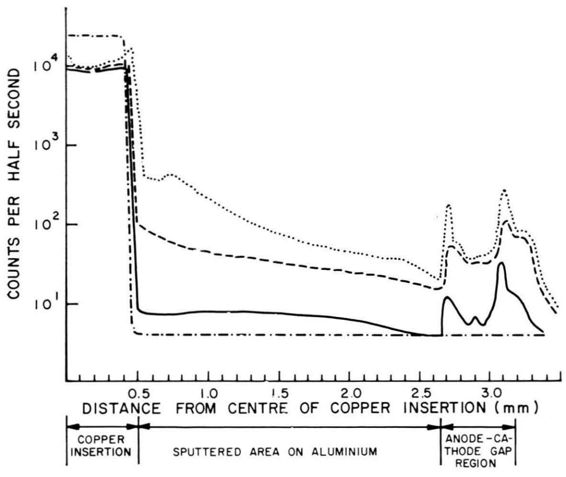

Fig. 2. Counts of $^{63}Cu^+$ ions as obtained from a repeatedly performed line scan over an aluminium disc with copper insertion which was exposed to a glow discharge. –·–·–·– Cu before sputtering in glow discharge; ······· first scan; ----- second scan; ——— third scan.

Based on these assumptions two quantities can be derived from the data in Figure 2:

1. The ratio β of redeposited copper atoms to copper atoms sputtered from the insertion.

The average copper content of the mixed aluminium-copper layer is smaller than 1%, therefore the contribution of copper in the sputtering process from this layer will be minimal and the sputtering yield can be assumed to be that of aluminium.

The assumption is made that a 20 keV $^{16}O_2^+$ ion has the same effect as two 10 keV $^{16}O^+$ ions, although Anderson reported higher ionic yields from molecules than from atoms at the same energy per atom [8]. The calculated atomic sputtering yields S for 10 keV $^{16}O^+$ ion bombardment are 4.2 atoms/ion from aluminium and 6.8 atoms/ion from copper, and in the glow discharge, the ratio between the aluminium and copper sputtering yields is calculated as 0.75. The calculations of sputtering yields are based on Sigmund's proposals [9].

When the data in Fig. 2 for the copper ionic sputtering rate from the insertion under 10 keV $^{16}O^+$ ion bombardment are accepted, the copper sputtering rate from the layer is underestimated and must be corrected for by a factor equal to the ratio between the sputtering yields of copper and aluminium.

The ratio between the corrected number of ^{63}Cu ions detected from the mixed aluminium-copper layer during the first scan and the maximum number of ^{63}Cu ions from the copper insertion (Fig. 2) gives a value of the surface coverage of copper atoms within each of the concentric rings with radii between 0.5 and 2.6 mm. The average surface coverage of copper for the area between these limits is calculated as 0.87%.

The ratio β, as calculated from the respective areas, the average surface coverage of copper and the ratio between the aluminium and copper sputtering yields, turns out to be $\beta = 17\%$.

2. The number N_d of copper atoms accumulated by redeposition on the aluminium.

It can be seen in Fig. 2 that most of the copper atoms are removed by the third scan. The total number of ions sputtered by oxygen per μm^2 at a given distance from the insertion is the sum of the three scans. Integration over all concentric rings with radii between 0.5 and 2.6 mm (see Fig. 2) gives the total number of sputtered copper ions $N_i = 2 \times 10^{10}$ ions.

The ionic sputtering yield S_{Ar}^+ from clean copper for 3 keV Ar^+ ions was measured by Benninghoven [10, 11] as

$$S_{Ar}^+ = 0.00013 .$$

From this a value for S^+ of about 0.00017 for 10 keV $^{16}O^+$ was calculated.

The ratio R_i of the ionic sputtering yield S^+ to the calculated atomic sputtering yield S for 10 keV $^{16}O^+$ ions on copper is given by

$$R_i = S^+/S = 0.00017/6.8 = 2.5 \times 10^{-5} .$$

This value of R_i is of the same order as the number 8.2×10^{-5} given by Schroeer et al. [12].

The ratio R_i is equivalent to the ratio of sputtered ions to sputtered atoms. The number of redeposited atoms N_d in the layer after 60 s exposure in the glow discharge is then estimated as

$$N_d = \frac{2 \times 10^{10}}{2.5 \times 10^{-5}} = 8 \times 10^{14} \text{ atoms.}$$

If an equilibrium state is assumed between copper atoms redeposited and resputtered, N_d will be the number of copper atoms in the redeposited layer.

5. Discussion

When evaluating the deposition ratio β and the number of copper atoms in the redeposited layer, the following must also be considered.

The sputtered copper and aluminium atoms were deposited simultaneously on the exposed area during glow-discharge sputtering and the sputtering yield from this binary system will differ from the sputtering yields from pure metals.

There is an uneven distribution of copper on the surface from which the signal on a line scan of 2 µm width is used in the estimation of the deposited atoms. Data taken from Fig. 2 will therefore only give an average atom distribution over the whole surface.

In the estimate of N_d no account is taken of the fact that copper was redeposited on aluminium in the glow discharge and then exposed to air or that oxygen is used as bombarding species in the IMMA. The possibility therefore exists that the surface atoms are oxidised to some extent. There is a significant difference between the sputtering yields from clean metals and metallic oxides, when bombarded with high energy ions. However, to simplify the calculation, the value from clean copper is used as a first approximation.

6. Conclusion

The estimated ratio $\beta = 17\%$ of sputtered atoms redeposited on the area exposed to Ar^+ ion sputtering in the Grimm-type glow-discharge lamp, shows that a significant part of the sputtered atoms are redeposited and therefore resputtered. This resputtering of atoms must be taken into account when the true sputtering rates are estimated.

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