Study on the Ionic Bombardment in a Glow Discharge Lamp

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The surface of copper alloys was studied by means of a scanning electron microscope after having been sputtered by a glow discharge source under typical conditions for spectrochemical analysis. The surface topography developed during the process of sputtering gave the following two results: It would appear that the energy spread of the bombarding ions is small and the variation in the angles of incidence of these ions is less than usually accepted.

1. Introduction

The general features and analytical applications of the glow discharge lamp [1] have been the subject of several investigations. A general description of the lamp and its characteristics have been given by various authors [2, 3]. Reports on the glow discharge lamp deal mainly with the dependence of sputtering rates and burn-off curves on various discharge parameters or with general analytical problems such as optimising sputtering conditions for various samples, precision, detection limits and matrix effects [4–7]. Basic studies on various parameters have been made in order to gain insight into the physical properties of the glow discharge [8–10]. This paper deals with a scanning electron microscopy investigation of burn spots in order to infer the energy spread and the angular distribution of the incident ions.

2. Experimental

Mechanically polished copper-based alloys were exposed to sputtering in a glow discharge lamp. Argon was used as carrier gas. The two discharge parameters, current and gas density, as well as sputtering time were varied, while the voltage was kept constant at 1200 volt. The sputtered surfaces of the samples were studied by means of a scanning electron microscope (Jeol JSM-U3) which was equipped with a solid-state detector to record secondary electron images and with a semi conductor-pair detector for the formation of backscattered electron images. The backscattered electron yield depends on the atomic number of the target material and backscattered electron micrographs are therefore representative of the composition of the specimen [11]. A 2×400 channel energy-dispersive X-ray spectrometer (EDAX International Inc.) was added to the scanning electron microscope in order to perform qualitative and quantitative analyses.

3. Results

Figure 1 * is a secondary electron micrograph of a typical sputtered area of a copper alloy containing approximately 85 wt. % copper and 5 wt. % each of zinc, tin and lead (Ounce Metal). The specimen was tilted about the primary electron beam in the scanning electron microscope by 40 degrees. The machined sample surface was exposed to sputtering in a glow discharge lamp for 60 seconds under conditions where $U = 1200$ V, $i = 200$ mA and $p = 1.0$ kPa. The scanning electron microscope examination of the burn spot revealed that two types of sputtered areas (up to a few hundreds of microns in diameter) could be distinguished. One type showed the effect of a relatively uniform sputtering, the other type contained cones set in an area more deeply eroded than the first. The secondary electron micrograph (Fig. 2 a) and the backscattered electron micrograph (Fig. 2 b) taken at normal incidence show part of a deeply eroded area containing cones. The white spots (high atomic number) in Fig. 2 b are lead-rich inclusions. These are present on many of the cones. The size, shape and surface structure of these cones are shown more clearly in Figures 3 a and 3 b. Both truncated and perfectly tapered cones can be found in the same area. The cones normally have faceted surfaces and are surrounded by circular grooves.

The spectra in Figs. 4 a and 4 b are the results of a micro-analysis performed on the top of a cone and of a macro-analysis obtained by scanning an unsput-

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* Figures 1; 2 a, b; 3 a, b; 4 a, b; 5 on Table 1248 a, b.

tered area of approximately 0.5 mm^2, respectively. The latter spectrum is representative of the chemical composition of the alloy whereas that of Fig. 4 a shows pronounced peaks due to lead. The peaks at 2.3, 10.5 and 12.6 keV correspond to Mα-, Lα- and Lβ1-radiation of lead. Spectra obtained from about the base of cones are similar to that of the bulk material and show no enhancement or deficiency in lead concentration.

Figure 5 is a secondary electron micrograph of a sputtered brass sample containing approximately 73 wt. % copper, 27 wt. % zinc and traces of lead (Cartridge Brass) which clearly shows the two types of sputtered areas, including the presence of cones in the deeper eroded areas.

The cone parameters of the specimens were determined by tilting about the primary electron beam in the scanning electron microscope by angles up to 45 degrees. The axes of the cones were found to be in the direction of the normal to the flat sample surface which is also the direction of the incident ion beam. The angles of the cones were similar and varied between 60 and 65 degrees. The contrast in topography became very marked when the tilt angle was increased and the two types of sputtered areas could then be clearly differentiated as in Figures 1 and 5.

It was found that for input-powers of 240, 120 and 85 watts the typical surface topography as described above could only be observed after the sample was sputtered for approximately 30, 100 and 150 seconds, respectively. The size and the angle of the cones were not significantly influenced by the electrical input-power.

4. Discussion

Stewart and Thompson [12] have described formation of cones resulting from the ion bombardment of mechanically polished samples. This theory has since been supported by Wilson and Kidd [13] who investigated cone development on gold during bombardment with argon and xenon ions. Monoenergetic ions up to 20 keV were used in these studies. The angle of incidence of the ions was found to be important and related to the formation of the cones.

On the basis of the abovementioned theory one can conclude that the cones described were formed in the glow discharge lamp under the impact of a rather monoenergetic ion beam with a small angular distribution. The cones were formed in such a way that the angle between the incident ion beam and the normal to the cone surface was the angle for optimum sputtering as predicted by theory [12].

The collision rate of the argon ions under the present experimental conditions indicates that the bombarding ions can be expected to be rather monoenergetic. Side-on intensity measurement in the glow discharge lamp [2, 8] taken together with the mean free path length of the argon ions (approximately 0.06 mm for the present experiment) show that for example only 3 to 5 collisions can occur before the ions strike the sample.

This finding and the present results contradict the description of the bombarding ions given by Boumans [3] who stated that in the glow discharge lamp multiple collisions between the gas particles give rise to a large energy spread among the bombarding ions and to an undetermined angle of incidence.

The erosion of the sample surface in a glow discharge lamp and the subsequent formation of two types of sputtered areas as illustrated in Figs. 1 and 5 are presumably the result of a strong dependence of the sputtering rate on crystallographic orientations. The alloy containing approximately 85 wt. % copper and 5 wt. % each of zinc, tin and lead was chemically etched and differently orientated grains of only one phase (α-brass) could be distinguished. Sputtering is reduced when the close-packed directions are parallel to the incident ions [14]. Optimum sputtering occurs for $\langle 111 \rangle$ orientations for copper and there is reduced sputtering for the $\langle 100 \rangle$ and $\langle 110 \rangle$ orientations. This difference in sputtering is sufficiently pronounced for grains with a normal close to $\langle 111 \rangle$ orientations to be recessed below the general surface area after bombardment. Defects within grains such as mechanical twins and microdendrites as well as inclusions (lead) can be considered to be grains with a surface normal not necessarily parallel to $\langle 111 \rangle$ parent grains. These would thus erode at a speed comparable to the general surface and so appear as prominences in the $\langle 111 \rangle$ normal surface grains.

Machining of the samples causes damage in the subsurface. This damaged surface layer had thus to be removed before the dependence of the sputtering rate on orientations became apparent.

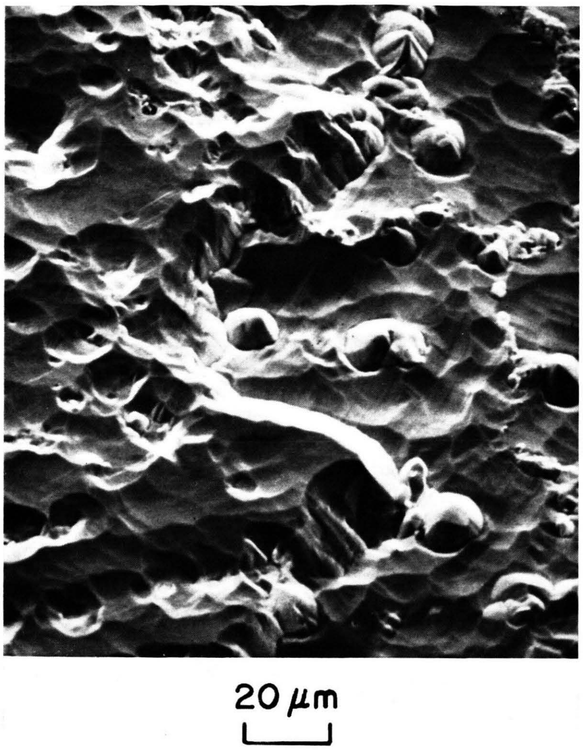

Fig. 1. Scanning electron micrograph of tilted surface of copper alloy containing 85 wt. % copper and 5 wt. % each of zinc, tin and lead.

Fig. 5. Secondary electron micrograph of tilted surface of sputtered copper alloy containing 73 wt. % copper and 27 wt. % zinc (plus traces of lead).

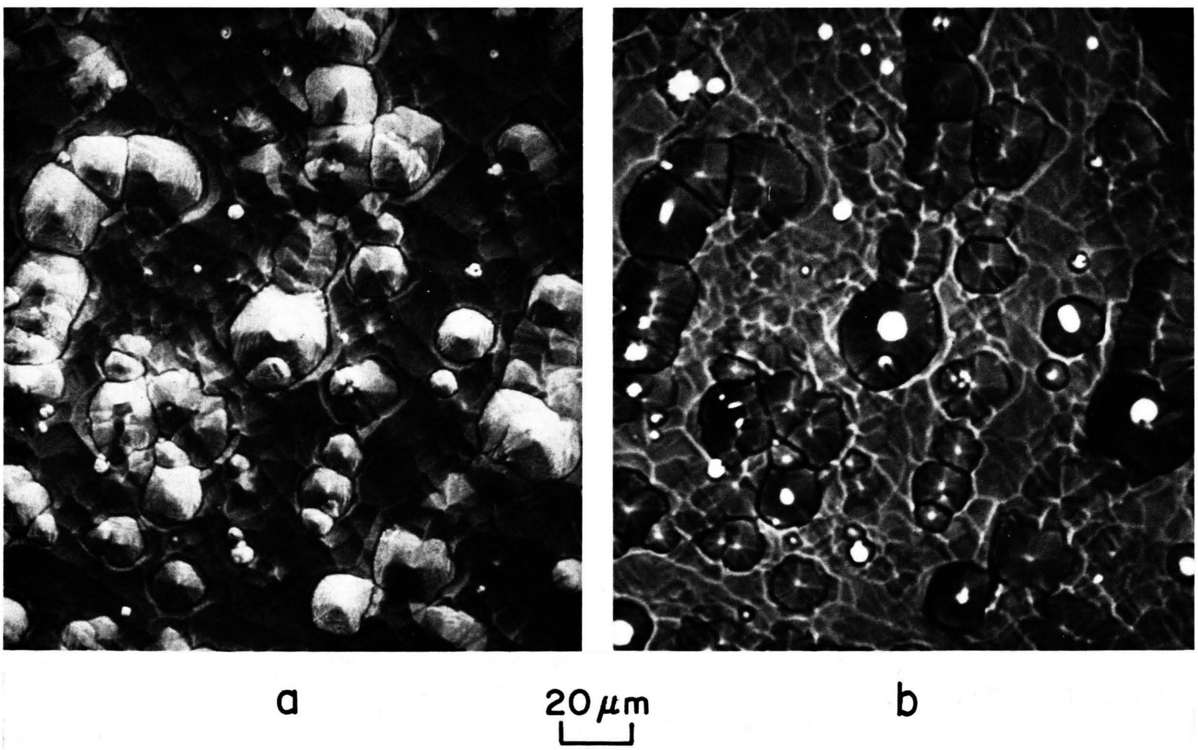

Fig. 2. Secondary electron (a) and backscattered electron (b) micrographs of cones found in deeper eroded area taken under normal incidence.

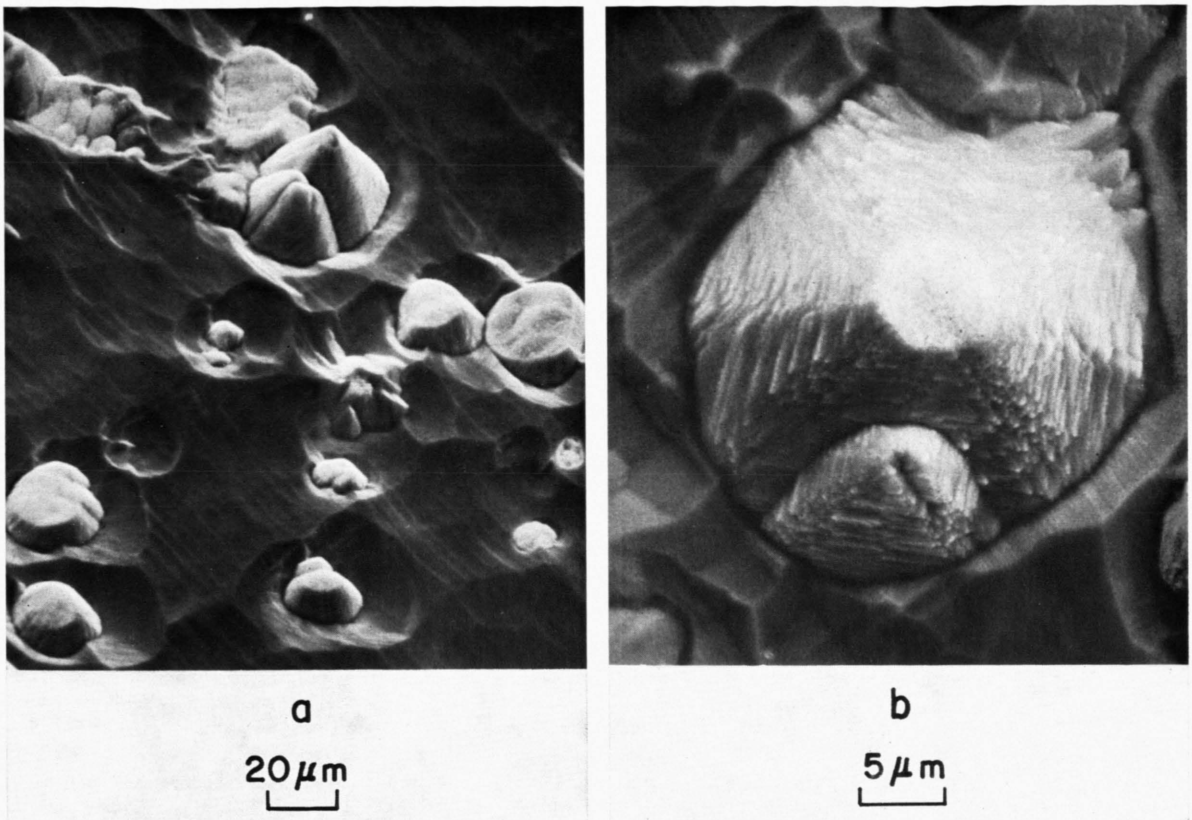

Fig. 3. Secondary electron micrographs of tilted sample surface revealing cones in deeper eroded area (a) and of cone taken under normal incidence (b).

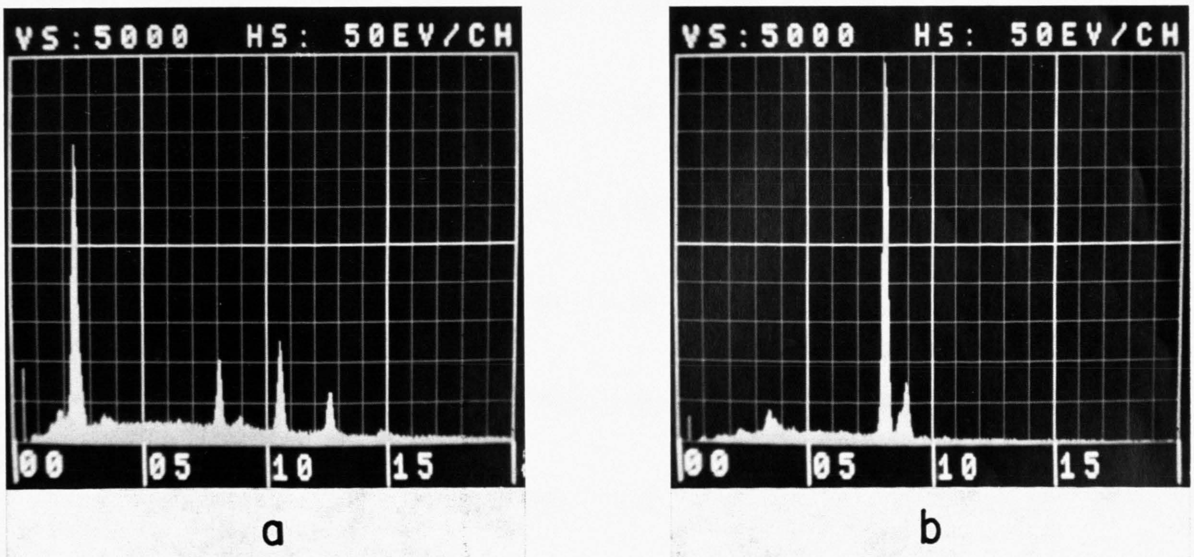

Fig. 4. X-ray spectra as a result of microanalysis of the top of a cone (a) and macroanalysis of unsputtered area (b). VS: Total counts along vertical scale. HS: X-ray energy (keV) along horizontal scale calibrated in channels of 50 eV per channel.

5. Conclusion

The surface topography developed on brass samples after being exposed to sputtering in the glow discharge lamp were found to be similar to the formation of cones and surface steps resulting from the bombardment of solids by a monoenergetic ion beam with a defined angle of incidence. The results of these experiments provide evidence that the energy and the angle of incidence of the bombarding ions seem to be better defined in the glow discharge lamp than are usually described.

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