Problems Occurring in Depth Concentration Profiling

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Results of the depth profiling of a laser mirror are reported and discussed. The sample was bombarded with $^{40}Ar^+$, $^{32}O_2^+$ and $^{16}O^-$ in order to establish optimum conditions. It was found that the bombardement by means of $^{16}O^-$ results in the best depth concentration profile.

Depth concentration profiling is regarded as one of the greatest capabilities offered by secondary ion mass spectrometry. Many publications [e. g.[1–3]] give examples of the successful application of this technique. Problems, however, still occur, especially when an unknown layered sample has to be profiled. In this paper results of the depth profiling of a laser mirror are reported and discussed.

The sandwich structure, illustrated in Fig. 1 a, was investigated by means of an ARL-ion microprobe mass analyser (IMMA). The sample was bombarded with $^{40}Ar^+$, $^{32}O_2^+$ and $^{16}O^-$ in order to establish optimum conditions for depth concentration profiling. In addition the sample was coated with carbon to prevent a possible surface charge. Apart from the bombarding species, all the other primary conditions were kept as constant as possible. Two different acceleration voltages (10 and 20 keV) were used in the experiments to investigate their influence on the depth concentration profiling. A computer program was developed to enable simultaneous recording of the intensities in logarithmic units of all the elements in question (up to 20) using one burnspot only.

The secondary positive ions (C^+, Zn^+ and Th^+) and the negative ions (C^-, S^- and F^-) were recorded for the three different bombarding conditions. Similar depth concentration profiles were obtained when these secondary positive and negative ions were recorded, only the intensities differed. The intensity profiles obtained by recording positive ions are given in Figures 1 b, c and d. The intensity of the negative sulphur ion is included in Fig. 1 b to illustrate the similarity of the depth profiles obtained from positive and negative ions. The change in the acceleration voltage from 10 to 20 keV did not influence the profiles significantly.

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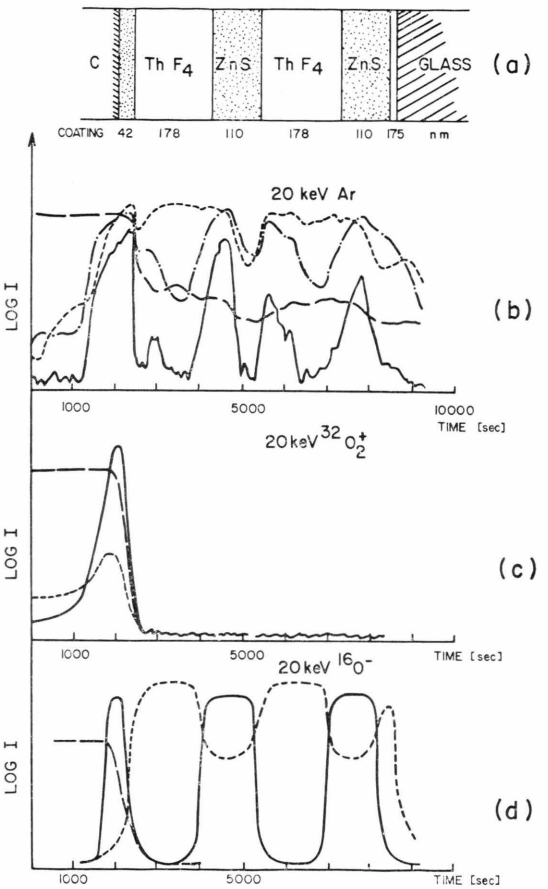

Fig. 1. A schematic illustration of the laser mirror and the resulting depth concentration profiles using $^{40}Ar^+$, $^{32}O_2^+$ and $^{16}O^-$ as bombarding species. A rastered beam of about 1 nA, a diameter of about 5 μm, a raster size 200×200 μm, an electronic appertur 100×100 μm and a pressure of $7 \cdot 10^{-5}$ Pa in the sample chamber were used. — — C^+, —— Zn^+, · · · · Th^+, — · — S^-.

From Figure 1 c it is immediately evident that $^{32}O_2^+$ completely failed to give a correct depth concentration profile, while $^{16}O^-$ results in a satisfactory profile. The explanation for the failure of $^{32}O_2^+$ is probably the presence of the second layer (ThF_4).

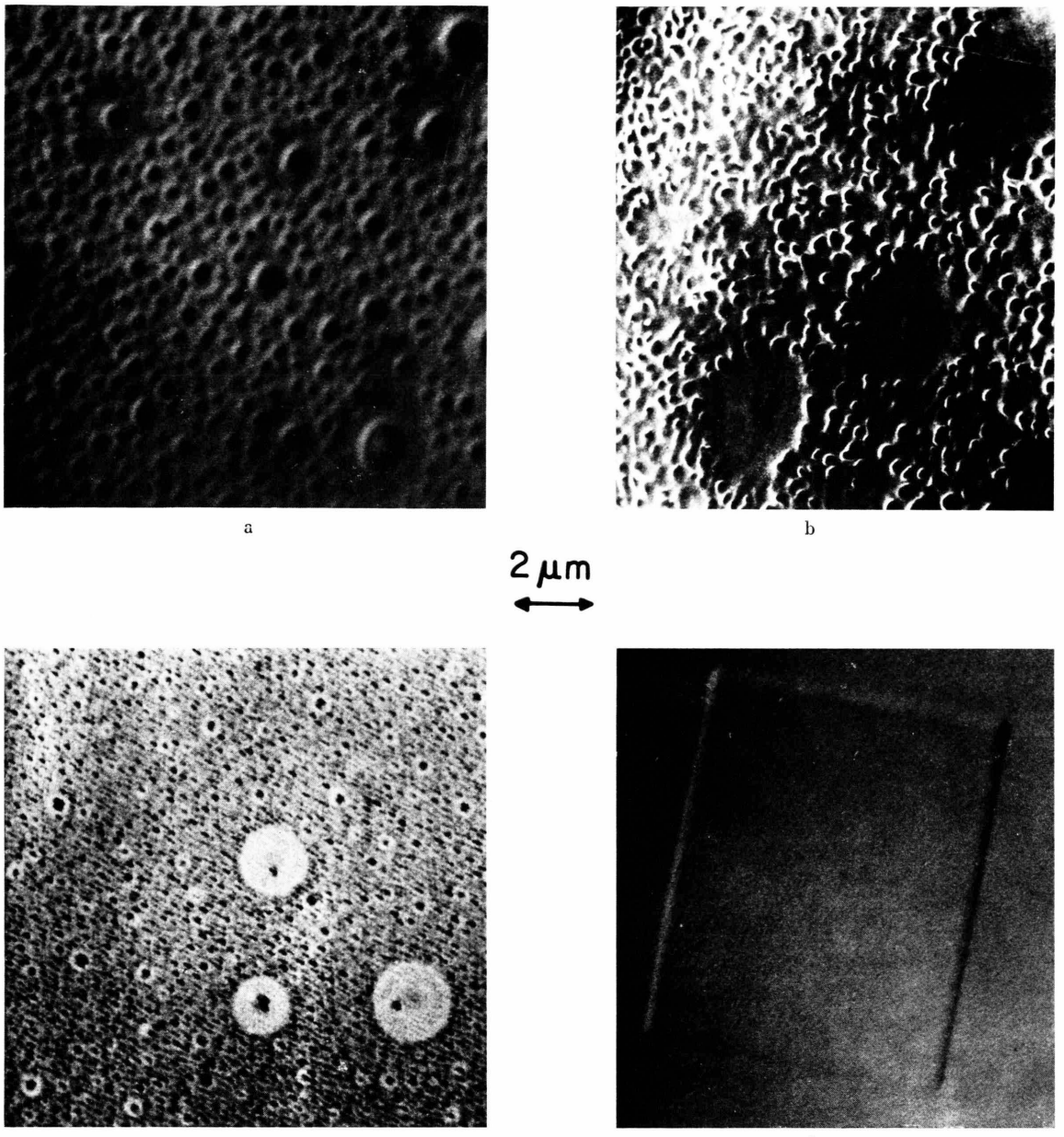

Fig. 2. Scanning electron micrographs of a rastered burnspot on a layered sample (tilt angle 40°) when using a, b, c: argon as bombarding species, d: oxygen as bombarding species.

Although the sample is coated with carbon and the first layer (ZnS) is conductive, the signals of all three elements are cut off completely when the first layer is sputtered away. The use of $^{40}Ar^{+}$ as bombarding species results in an intensity pattern which would correspond to a series of layers not properly defined. Four to five layers of ZnS could be identified. At the border of such a ZnS layer the Th^{+}-signal always seems to disappear only to recover suddenly. An explanation for this rather strange behaviour can be deduced from scanning electron micrographs.

The rastered burnspot, as illustrated in Fig. 2, was examined by means of a scanning electron

microscope. Pictures a, b and c show the secondary electron micrographs of the surface of the profiled sandwich structure when using argon, while picture d illustrates the surface of a raster where oxygen was chosen as bombarding species. In the case of oxygen the surface was so uniformly sputtered that no difference could be found between the unsputtered sample and the burnspot. Therefore, in order to show that the surface was really sputtered, a much smaller area than was used in the real experiments was eroded, as shown in the micrograph 2 d. In micrograph 2 a the first layer (ZnS) only is sputtered away, while micrograph 2 b depicts the condition of the surface after the first thorium fluoride layer has been removed. Micrograph 2 c, which was recorded by means of backscattered electrons, reveals differences in the chemical composition of the sputtered surface. From these pictures it can be seen that the surface is not homogeneously eroded under the $^{40}Ar^+$ conditions. A breakthrough to the next layer occurs randomly, which results in a formation of discs and hillocks spread over the whole bombarded surface. Conductive parts of the sample seem to be present under these conditions and sputtering as well as secondary ion emission are therefore not inhibited. The signals of the secondary ions can therefore be recorded as indicated by the results. The presence of these discs and hillocks can lead to a completely wrong interpretation of the profiled sandwich structure.

These results indicate the desirability of using $^{16}O^-$ ions intead of $^{40}Ar^+$ and $^{32}O_2^+$ as bombarding species when profiling an unknown layered sample.

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