1. Introduction

Thin layers of indium tin oxide are applied as electrical contacts in flat displays since they are conductive and optically transparent [1]. Targets are coated with ITO by reactive magnetron sputtering. A partially reduced ITO-sputtering target is eroded by a Ar/O plasma. Sputtered In and Sn atoms are oxidized within the plasma and redeposited on the substrate. During the sputtering process small nodules with diameters up to 100μm grow on the surface of the sputtering target. These nodules cause small arcs that destabilize the sputtering process. In addition small particles are eroded from the surface and contaminate the substrate.

It is well known that inhomogeneously distributed impurities with a lower sputtering yield than the matrix may cause such nodule growth [2].

In this study we have used secondary ion mass spectrometry to determine: (1) whether or not such inhomogeneously distributed impurities can be found in the ITO-sputtering targets, (2) the compositions of any impurities found, and (3) the influence impurities have on the sputtering behavior. SIMS is one of the few analytical methods being capable of acquiring 3D images of elemental distributions [3] and is, therefore, particularly suited for this type of investigation. In addition, the influence of impurities on sputtering behavior should be directly seen in the count rates of secondary ions.

2. 3D-Imaging & Processing

During magnetron sputtering macroscopic amounts of the sputtering target are eroded away (areas up to several cm² and several mm deep). In order to analyze a sample volume of similar size high beam currents are required. However, based on the average size of the nodules on the sputtering target, the impurities we were looking for were expected to be
rather small (several μm). Therefore, measurements (using a Cameca IMS 5f) were carried out using an O$_2^+$ primary beam since this offers the best compromise of high sputtering rate at relatively low beam diameters.

Measurements were made with a 1μA primary ion beam current at 12.5kV accelerating voltage and a raster size of 500μm; a single measurement took 12 hours. Several elements with count rates higher than 100cps were selected for analysis. These elements are C, Mg, Al, Si, Ti, Cu, and Zr.

Relatively few impurities were found, so statistical evidence is limited, but some trends can be discerned. Cu is distributed homogeneously within the sample. All other impurities are distributed inhomogeneously, most of them consist of C or Al.

The influence of the impurities on sputtering behavior was estimated by calculating depth profiles from the 3D elemental distribution images of the impurities. In Fig. 1 part of a 3D carbon image can be seen. This is the largest impurity detected in this study (diameter: approx. 50μm). The figure shows the layer in which the impurity has its maximum width and the cuboid through which the depth profile was calculated, by adding up the counts in all the layers.

Fig.1: One plane and a cuboid through an impurity from the 3D carbon image. The grayscale represents the count rates: from black (few counts) to white (many counts). The graph shows the volume eroded and analyzed by SIMS. The crater is 500μm x 500μm wide and 150μm deep. The topmost layer represents the surface of the sample.
Calculating similar depth profiles for several elements yields the profile shown in Fig. 2, in which the influence of this C impurity on the sputtering behavior can clearly be seen: as the C count rate increases, the count rates of indium, oxygen, and of the primary ions decrease. The implantation signal (\(^{16}\text{O}^+\)) originates from the ions implanted into the sample by the \(^{16}\text{O}_2^+\) primary beam. In contrast \(^{18}\text{O}\) was chosen to measure oxygen originating from the sample. The fact that all secondary ion signals - except C - decrease uniformly in the depth profile at the location of the impurity may be seen as an indication of lower sputtering rate. A major change in composition would lead to a different decrease of the implantation signal and the matrix signals, since the implantation signal should not be influenced by the sample composition. A change in the ionization yields should lead to a different decrease of the In and the \(^{18}\text{O}\) signal; it is unlikely that a difference of the matrix has the same influence on the In and the O signal. In addition a small nodule (height approx. 8\(\mu\)m) was found at the bottom of the sputter crater after the measurement, suggesting an influence of this impurity on sputtering behavior as well.

![Graph](image)

**Fig.2**: Depth profile calculated through a C impurity. Layer 0 represents the count rates on the sample surface, layer 150 at the bottom of the sputter crater. Carbon seen on top of the cuboid in Fig. 1 is part of a homogeneous surface contamination and is, therefore, not shown in this diagram. The maxima of all curves in this depth profile are normalized to one. The implantation signal originates from ions implanted in the sample by the \(^{16}\text{O}_2^+\) primary beam. Changes in the implantation signal are indices for changes in the sputter rate. Note that the count rate is normalized to 1 and the scale is linear!
Such depth profiles were calculated through several other inhomogeneities, but no influence on sputtering behavior could be observed. However, these inhomogeneities were significantly smaller (average diameter 13μm) than the one shown above.

3. Analyses of the Nodules

Only the analysis of a significant number of nodules can yield information about what type of impurities are responsible for the growth of nodules on the ITO sputtering targets. About 100 nodules were examined for traces of the most important impurities (C, Mg, Al, Si, Ti, and Zr). These nodules came into existence during magnetron sputtering.

On top of nearly all of the nodules the carbon count rate is much higher compared to blank measurements carried out between the nodules. The high carbon count rate is not a topological effect since the count rate of the matrix (In) is not increased on top of the nodules. There were only three of the nodules on which no impurity was detected. Therefore it can be concluded that C impurities play the most important role in nodule growth, although it is possible that other impurities cause nodule growth as well.

4. Conclusions

3D-SIMS analyses show heterogeneously distributed impurities, especially of C and Al, but also of Mg, Si, Ti, and Zr. Most of these impurities are not large enough to influence the sputtering behavior significantly, but in one case it was possible to demonstrate the influence of a C impurity in detail.

Analyses of individual nodules suggest that C impurities are the main reason for nodule growth on these samples. The influence of other impurities is negligible compared to the influence of C impurities.

5. References

[1] Latz R., Michael K. und Scherer M.


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