SIMS 3-D IMAGING OF PARTICLES AND OTHER SAMPLES WITH A HETEROGENEOUS MATRIX

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1. Introduction

In recent years it has been shown that 3-D imaging is a powerful new application of secondary ion mass spectrometry [e.g. 1, 2]. This analytical method combines the surface imaging capabilities of SIMS with depth profiling, creating layer-by-layer images of elemental distributions as the primary ion beam sputters deeper and deeper into the sample. The amount of data produced during a typical measurement of this type can easily reach several hundred megabytes, but with suitable imaging software it is possible to convert this information into easy to understand three-dimensional visualizations of elemental distributions within a given sample volume near the original surface. When the images are created by rastering the primary beam, the achievable lateral resolution is mostly limited by the beam diameter which can be significantly smaller than 1 μm and the depth resolution is only limited by the thickness of the ion beam mixing layer.

2. Limitations of the 3-D SIMS technique

In many ways this method appears to be the ideal tool in surface and near-surface analysis, by giving direct insight into a sample's internal structure. However, it has been found that SIMS 3-D imaging can easily be used in only a few particularly well suited samples. One of the problems is due to changes in the local sputter rates, resulting in uneven bottoms of the sputtered craters. This, in turn, leads to distortions of the acquired images which then no longer represent the elemental distributions at a well-defined depth. Therefore, the typical ideal samples for 3-D SIMS analyses are trace impurities in homogeneous matrices with constant sputter rates.
3. 3-D SIMS in samples with a heterogeneous matrix

In some cases it is possible to correct the acquired data for the artificial distortions mentioned above and therefore to extend the applicability of 3-D SIMS to a whole range of new samples. Examples for simple cases where this can be done are shown in Figs. 1 and 2. In the first case, the assumption is made that the sample consists of layers with different sputter rates. These differences can lead to significant misrepresentations of the layer's thicknesses, as indicated by the "raw data" in Fig. 1. However, it is possible to correct the raw data so that the original distribution of elemental abundances within the sputtered volume can be visualized. This information may be the sputter rates in individual phases or the geometric dimensions of certain features in the sputtered area. Thus, the correction involves converting the z-axis from representing sputter time to actual sample depth.

Figure 1: Schematic of a 3-D SIMS measurement of a sample consisting of layers with different sputter rates. By means of data processing it is possible to convert the raw data into a realistic representation of the three-dimensional composition of the sputtered sample volume.

A different example is shown in Fig. 2. Here, phases with different sputter rates are located next to each other at the sample surface. After the measurement, the sputter crater does not have a flat bottom, but shows a step as indicated in the figure. Obviously, the acquired data does not represent the sputtered volume in a geometrically correct way. Here too, a correction can be applied if the sputter rates in the different phases are known. In the example shown, it is easy to determine the two sputter rates from the depths of the crater bottom at the locations of the phases.
In many real-world situations, the samples may contain some combination of the examples shown in Figs. 1 and 2, but data processing can still be used in many cases to correct the acquired 3-D images. The results will be better, when the sample is less complex.

![Figure 2: Schematic of a 3-D SIMS measurement similar to the one shown in Fig. 1, however, here two phases with different sputter rates are located next to each other.](image)

### 4. 3-D SIMS in particles

In general, particles represent one of the least suited categories of samples for analysis by 3-D SIMS. Not only do particles often have a heterogeneous composition, leading to all of the aforementioned artifacts, but their morphologies often make meaningful 3-D SIMS impossible. One of the minimum requirements of this measurement technique – a flat sample surface at the beginning of an analysis – is generally not given for particle analysis.

However, in a few cases the 3-D SIMS technique can successfully be applied to particle analysis. One example is shown in Fig. 3. Here we have analyzed a mica platelet with a diameter of approx. 150 μm that is covered with Ti and Cr oxide layers on both sides and that is used as a coloring pigment. A cut-out 3-D view shows the O distribution inside the particle. Clearly visible are the two disk-like areas where O is enriched – the oxide coating of the particle. The mica platelet itself is between the top and bottom oxide areas. Since the 3-D measurement was done for a total of 10 elements, it was possible to study the particle's internal structure in detail, to verify the lateral homogeneity of a layer's composition and to check the uniformity of the coating thickness. The oxide layers in this 3-D image appear thicker than the mica platelet due to their lower sputter rate. Data processing could be used to correct for this effect as described above.
Figure 3: 3-D view of the oxygen distribution in an oxide-coated mica particle. The full block represents a volume of approximately $200 \times 200 \times 10 \ \mu m^3$. For this visualization of the raw data no special data processing has been applied.

5. Summary

We have shown that it is possible to extend the use of SIMS 3-D imaging to certain samples with heterogeneous matrices by means of post-measurement data processing. As long as some other information about the sputtered area is given – such as sputter rates in individual phases, geometric dimensions of features in the sputtered area, or the exact morphology of the crater bottom – it can be possible to transform the acquired data into a realistic representation of the three-dimensional distribution of elemental abundances. In this way SIMS 3-D imaging can be applied to the analysis of multi-phase samples and even particles.

References
