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X-ray Fluorescence Analysis in an Electron Microscope: Improved Spotsize of Polycapillary Focusing Optics at the IfG Modular X-ray Source (iMOXS/2®)

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The IfG modular X-ray source iMOXS/2® can be attached to any scanning electron microscope (SEM) with a special adapter flange. Combining the iMOXS/2® with the existing energy dispersive X-ray detection system enables X-ray fluorescence analysis (XRF). This allows taking advantage of the high elemental sensitivity of XRF compared to electron probe microanalysis (EPMA) [1].

Guiding and focusing the X-rays from the X-ray tube within the iMOXS/2® to the sample is accomplished by using polycapillary optics. A multitude of glass capillaries with a diameter in the range of a few micrometers utilizes multiple total reflections to focus the X-rays [2]. This is a challenge, especially to achieve small spotsizes combined with a high photon flux, large output focal distance and a variety of source to sample distances (up to 500 mm). Therefore, IfG constantly works on improving their polycapillaries.

In this work, an iMOXS/2® system attached to a Zeiss Evo 40 electron microscope was equipped with new generation of polycapillary optics. Test structures of Cu on a Si-wafer were examined with this setup. The test structure is moved with the X-Y-Z table of the electron microscope, while the iMOXS/2® illuminates a fixed, but adjustable, position. Figure 1 shows a test structure of 50 µm Cu stripes on a Si-wafer with increasing spacing in three steps from left to right of 50, 100 and 150 µm. An XRF linescan over that structure demonstrates, that the intensity of the Cu Kα as well as the opposed Si Kα signal can clearly resolve the structure. This is true for all three different distances between the stripes.

While Figure 1 demonstrates the potential of the iMOXS/2® for linescans, Figure 2 presents the result of an XRF map. This is particularly interesting, since it demonstrates the X and Y dimension of the spot. Again the test structure is Cu on a wafer, this time 50 x 50 µm squares of Cu with 50 µm spacing. The far right squares are reduced to 50 x 25 µm. A map scan over the whole structure, enabled by the detector software, shows the squares are distinguishable from each other. This improved spatial resolution requires fine adjustment of the iMOXS/2® instrument, especially of the focus on the sample, and was only achieved with this new generation of polycapillary optics.

The improved focal spotsize of the polycapillary optics leads to an improved spatial resolution of the XRF analysis enabled by the iMOXS/2® instrument. This is particularly interesting if several small structures are to be analysed with trace amounts of heavy elements. Due to the high background using electron excitation caused by bremsstrahlung, detection limits of EPMA are usually in the range of 1-0.1 mass%. XRF analysis improves this detection limit down to the range of 100 ppm or even less.

References:

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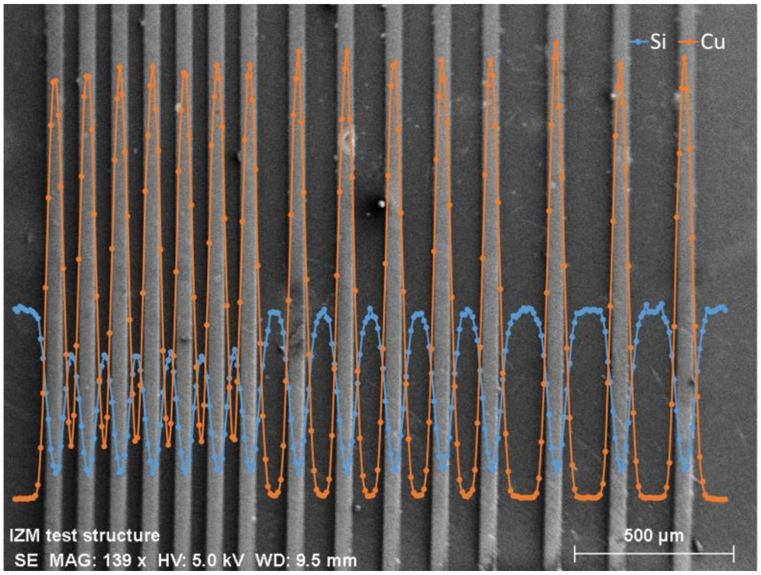

Figure 1. Results of an XRF linescan performed by moving the sample over a Cu structure on a Si-wafer shown beneath (secondary electron picture).

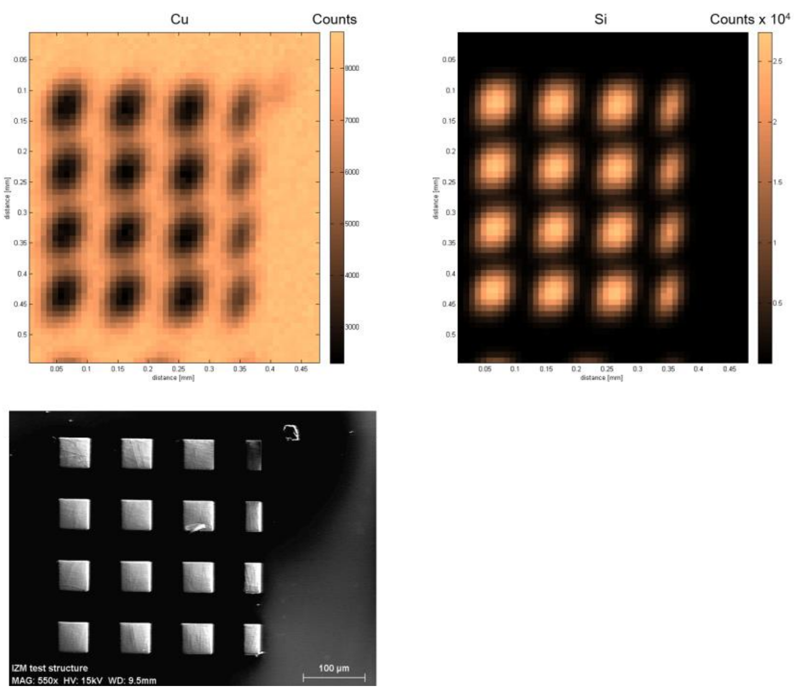

Figure 2. XRF Maps of Cu Ka and Si Ka (top left and top right), compared to the picture generated by the detection of secondary electrons.