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ZEISS Crossbeam – Advancing Capabilities in High Throughput 3D Analysis and Sample Preparation

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On the one hand, research in Materials Science often calls for most precise sample machining on the micro- or nanometer scale, while on the other hand the volumes of interest that have to be analyzed are constantly growing. A scanning electron microscope equipped with a focused ion beam (FIB-SEM) is ideally suited to address both of these needs. In this contribution, we highlight new features of the ZEISS Crossbeam family that enable advanced sample preparation and high throughput 3D analysis.

Depending on the intended experiment, there are different aspects of importance when it comes to sample preparation. For example, it may be necessary to expose nanometer scaled patterns over micrometer large areas. This requires precise scanning of the ion beam and finest beam shapes. Excellent long-term stability of the beam is needed as well, since such exposures can take long time in the order of tens of hours. The FIB column on the Crossbeam has been designed and is constantly improved to enable these applications. Automated routines reduce the amount of user interaction and alignment needed, and easily provide continuous operation times of longer than 72 hours.

Crossbeam features the largest FIB currents available using a liquid metal gallium source, up to 100 nA while maintaining smallest spot sizes. This assures highest throughput and shortest milling times during e.g. deep cross-section or large lamella preparations. However, to speed up the milling process even more, ZEISS has recently developed a scanning strategy that allows up to 40% faster material ablation. Figure 1 shows two cross-sections milled to the same target depth in silicon using a 65 nA probe. The left cross-section was executed using conventional line scanning and took almost 11 minutes, while the cross-section on the right used the new-patented scanning strategy and finished in less than 7.5 minutes.

Above-mentioned long-term stability is also important in the context of 3D investigations, like FIB tomography, where large volumes of interest have to be imaged and analyzed. In order to achieve high resolution in 3D with isotropic voxel sizes it is necessary to use low electron energies in the range of a few kV. However, this has drawbacks if analytic methods like energy dispersive x-ray spectroscopy (EDS) are to be carried out simultaneously. Depending on the sample material, the low electron energies may not be sufficient to excite characteristic x-ray lines at higher energies necessary for the analysis.

The latest release of ZEISS software for advanced FIB tomography, Atlas 5, does now not only include 3D EDS, it also features automated switching of the electron energy to provide perfect conditions in both situations, electron imaging and analytics. An exemplary slice out of a 3D dataset acquired on a lead free solder sample is shown in Figure 1. The electron imaging was done with 1.6 kV and 10 nm isotropic voxel size using the Inlens SE detector, while the EDS maps were acquired with 6 kV and a pixel size of 40 nm every tenth slice. The switching of the electron energy in this experiment made it possible to easily distinguish the silver and copper precipitates inside the tin matrix without compromising the electron imaging resolution in 3D.

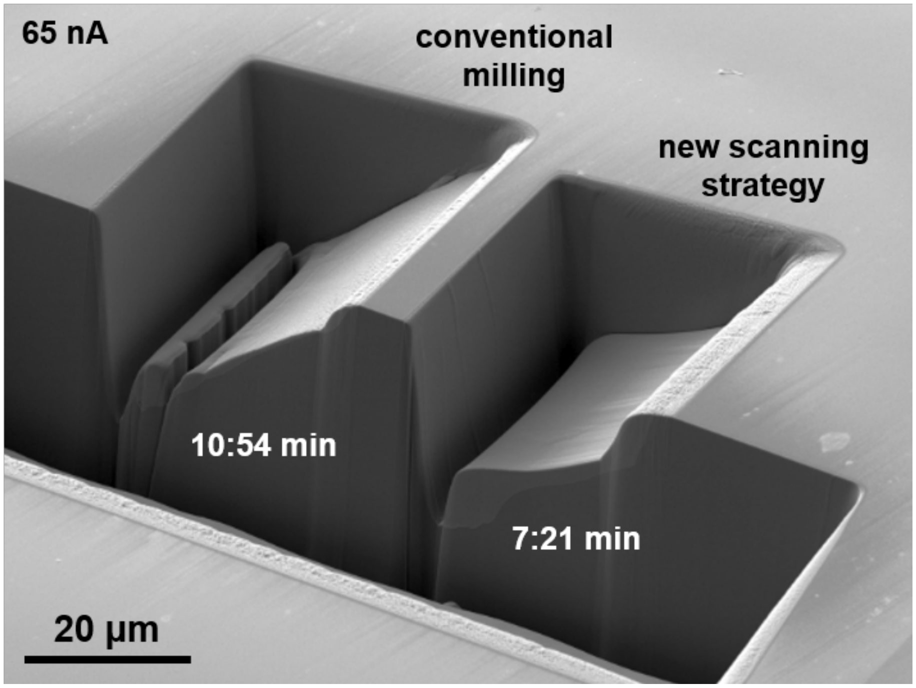

Figure 1. SE image of two FIB milled cross-sections in a silicon sample using 65 nA FIB current but different scanning modes.

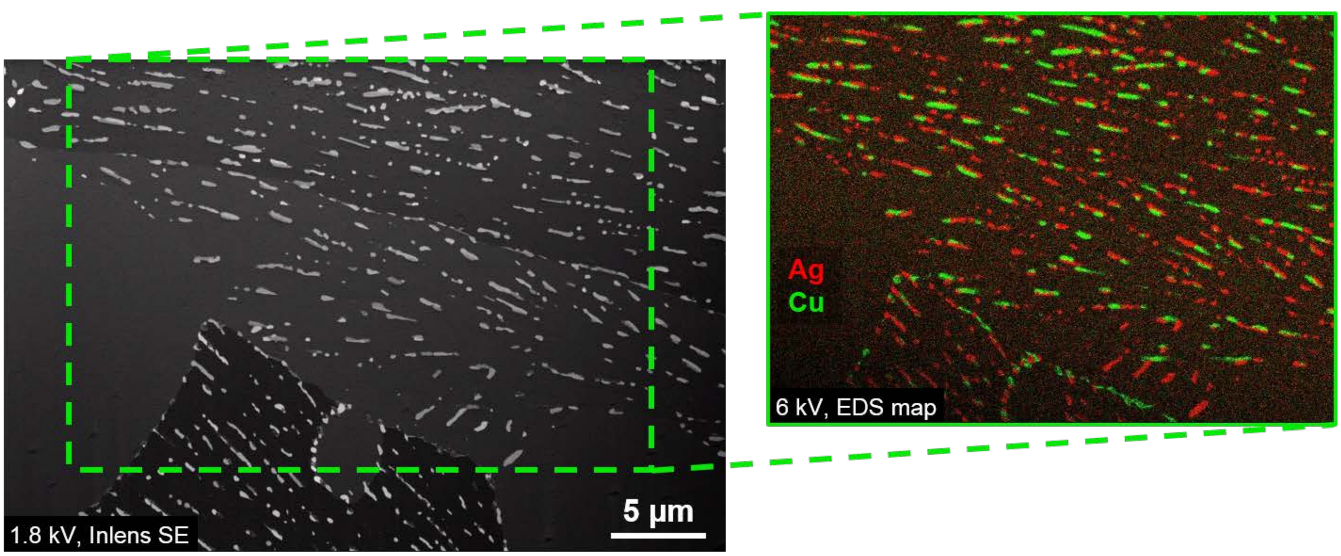

Figure 2. Exemplary slice out of a FIB tomography dataset acquired using Atlas 5 on a lead free solder sample, showing the imaging at 1.8 kV using the Inlens SE detector at a voxel size of (10 x 10 x 10 nm^3) and EDS maps every tenth slice at 6 kV with a voxel size of (40 x 40 x 100 nm^3). The switching of the electron energy during the acquisition is fully automated.

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