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The Challenges of High-Resolution Analytic FIB-SEM Tomography and Their Solution

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A FIB-SEM offers access to elemental and structural site-specific information at the nano-scale by the means of FIB-SEM tomography. However, as valuable as the obtainable data is, as much care has to be taken during the acquisition to assure integrity and quality. With this contribution, we want to raise awareness for the different challenges of FIB-SEM tomography but also share the knowledge on optimal utilization of this technique.

When it comes to FIB-SEM tomography, one first has to think about it as an imaging technique. A trench is milled into the sample surface and the created cross-section is imaged with the SEM. Then, a slice of material is polished off the cross-section face by FIB, before another image is acquired by the SEM. This alternating process continues and results in an image stack of cross-section images of the sample. These images are used to reconstruct the scanned volume and generate a 3D representation. For this purpose, it is essential that no information from deeper lying regions be captured during the acquisition of a slice. Otherwise, this would cause artefacts and lead to an ambiguous 3D reconstruction. To avoid this the interaction volume of the imaging electrons has to be kept small enough so that it does not extend further than one slice thickness. This can be a challenge when slice thicknesses of below 10 nm are targeted. Lowering the electron energy may then not be sufficient to reduce the interaction volume, but detectors capable of filtering the detected electrons so that only those closest to the position of the primary beam contribute to the image, may become necessary.

Reducing the electron energy in order to get unambiguous image data, however, is in contrast to the need for higher electron energies, when it comes to combining FIB-SEM tomography with analytics. The analytical information is often needed to interpret the image data correctly. However, acquiring the corresponding maps together with the tomography leads to certain requirements. First, the electron energy has to be high enough to excite the corresponding x-ray lines of interest in the case of EDS, or form the corresponding diffraction patterns in the case of EBSD. Second, in many cases the beam current has to be increased to improve the signal-to-noise ratio on the EDS detector or EBSD camera, so that the analytic map can be acquired in reasonable time. Fulfilling these requirements means to sacrifice image data integrity because of mixture of information given the increased interaction volume and sacrifice image resolution because of the increased beam current. Of course, the continuous improvement of analytical detectors helps to overcome this drawback, but to avoid it completely in the first place, it would be necessary to be able to switch beam conditions during the tomography acquisition. Most recent acquisition software developments offer exactly this. Two sets of beam conditions can be stored, which are automatically switched during the run, depending on the task at hand, imaging or analytic mapping. This way, the beam is optimally tuned for each purpose when it needs to be.

A third aspect that has to be considered especially when it comes to nanometer resolution is the sample drift and milling relocation. FIB-SEM tomographies typically run over several tens of hours and under tilted conditions. Obviously, the sample will not be perfectly stable under these conditions for such long time but drift. This becomes even worse if stage moves are involved in the process like for 3D EBSD. On the other hand, a stable execution and constant slice thicknesses are important for a high-resolution and high-quality dataset. Means to track the current slice thickness and sample drift are therefore essential. The system should be able to detect in which direction the sample is drifting (e.g. towards or away from the FIB beam) and adapt the milling progress accordingly. Recent versions of acquisition software offer corresponding solutions and enable FIB-SEM tomographies with constant slice thicknesses in the range of only a few nanometers.

Application examples illustrating the before mentioned challenges of high-resolution analytic FIB-SEM tomography will be presented within this contribution as well as detailed information about the proposed solution, proving the unique capabilities of FIB-SEMs as nanoscale analytic laboratories.