Focused ion beam nano-tomography using different detectors

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FIB-Tomography combines FIB milling with the imaging capabilities and the variety of different detection modes (SE, low kV BSE, EDX, EBSD) of a modern SEM in order to analyze the structure of the sample in 3 dimensions. In the interdisciplinary Center for Electron Microscopy (CIME) at EPFL FIB-Nanotomography has become an indispensible tool for the analysis of nanoscale structures in 3 dimensions.

Through fully automated slicing and imaging it is possible to generate series of images with rate of 40-60 images (slices) per hour. These stacks (typically 1500-2500 images) can then be used to reconstruct the 3 dimensional microstructure of the sample. With a typical voxel size of 10x10x10 nm (3x3x3 nm or even smaller is possible) and accessible volumes of up to 20x20x20 micrometer FIB tomography closes nicely the gap between X-ray tomography and TEM tilt series tomography. Imaging parameters like high-tension, beam current and detector have to be chosen carefully in order to obtain a high acquisition rate, a high signal-to-noise ratio and the desired image contrast that allows a correct segmentation and analysis of the obtained image stack [1].

Modern electron microscopes like the ZEISS NVision40 allow recording the signal of more than one detector in one scan. Series of images with complementary information can be obtained in one scan. The following example shows how the use of the in-Lens secondary electron detector and the energy selective backscattered (EsB) electron detector has allowed segmenting the 3 chemically different phases of a Pb-free solder (Au, Ag, Cu). The FIB stack consists of 2000 slices (10nm slice thickness, 10x10nm image pixel size). In Fig. 1a the image was obtained at 1.8 keV detecting the backscattered electrons (EsB). We observe a grain orientation (channelling) contrast in the matrix phase and a strong material contrast in the precipitates (Cu rich). In Fig. 1b obtained with the in-Lens secondary electron detector the precipitates are bright while the channelling contrast in the matrix is still visible. Careful inspection shows that there are more bright grains in Fig. 1b than dark ones in Fig. 1a. EDX analysis reveals that the bright grains in Fig. 1b consist of 2 different phases: one rich in Ag and the other one rich in Cu. The darker ones in Fig. 1a however are the Cu-rich phase only. Using both images it is possible to segment the 3D data (Fig 2) based on the differences in the secondary (in-lens SE) and backscattered (EsB) electron yield.

The recent technological progress in energy dispersive X-ray microanalysis with the introduction of large surface silicon drift detectors (SDD) has made it possible to acquire elemental maps within a few minutes. Fig. 3 shows a reconstructed volume of a laser solder joint between NiTi and stainless steel. The segmentation of the complex microstructure was based on 44 elemental maps and 360 secondary electron images acquired automatically over 14 hours. The acquisition of each elemental map took 6 minutes with an image pixel size of 100nm and 100nm spacing in the z direction. The secondary electron images were recorded at 12.5nm image pixel size and 12.5nm slice thickness (isometric voxel dimensions). The secondary electron images were used to refine at higher resolution the segmentation done through the analysis of the elemental distribution maps.

References


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Figure 1  Left: EsB signal: Cu-rich precipitates appear dark, Right: In-Lens secondary electron signal showing similar bright contrast for the Cu-rich precipitates as well as the Ag-rich precipitates.

Figure 2  Reconstructed volume of the two different precipitates (matrix transparent). For the segmentation both images (In-Lens SE and EsB) were required to properly identify the different phases.

Figure 3  Reconstructed volume of the complex microstructure of a NiTi-stainless steel solder joint. The segmentation and reconstruction is based on EDX elemental maps and secondary electron images.