doi:10.1017/S1431927611005289

Microsc. Microanal. 17 (Suppl 2), 2011

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 are 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.

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). In Fig. 1a the image was obtained at 1.8 keV detecting the backscattered electrons (EsB). We observe a grain orientation (channeling) 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 channeling 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 energy filtering capability of the EsB detector of the ZEISS NVision 40 can be further used to increase the z-resolution in 3D block-face imaging on biological samples like brain tissue [1]. Low-loss electrons that have travelled only a short distance (scatter range) within the sample provide a signal that is surface near. This limited escape depth allows acquiring 3D stacks with 3x3x3nm voxels size and high z resolution.

References

[1] M. Cantoni et al., *Microsc. & Anal.* 24(4): 13-16 (2010).
[2] We thank M. Maleki (EPFL) for the sample.

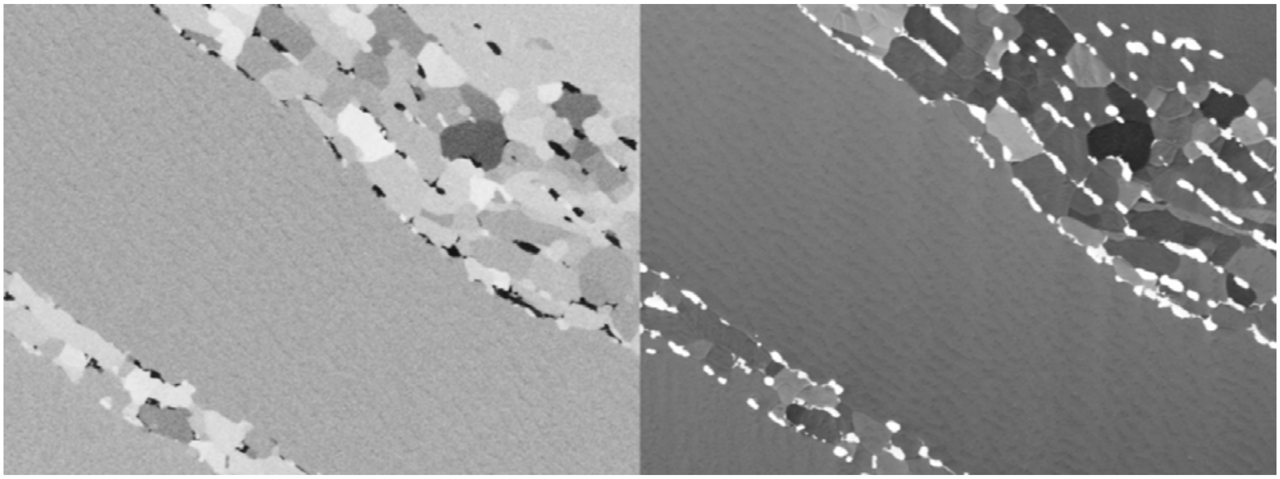

FIG 1: Pb-free solder with 3 different phases: 10x10nm image pixel size (20 micrometer horizontal width), part of a FIB 3D stack with 2000 slices (10nm slice thickness).

a) Left: Backscattered electron (EsB) signal: Cu-rich precipitates appear as dark grains
b) Right: In-Lens secondary electron signal showing similar bright contrast for the Cu-rich precipitates and Ag-rich precipitates.

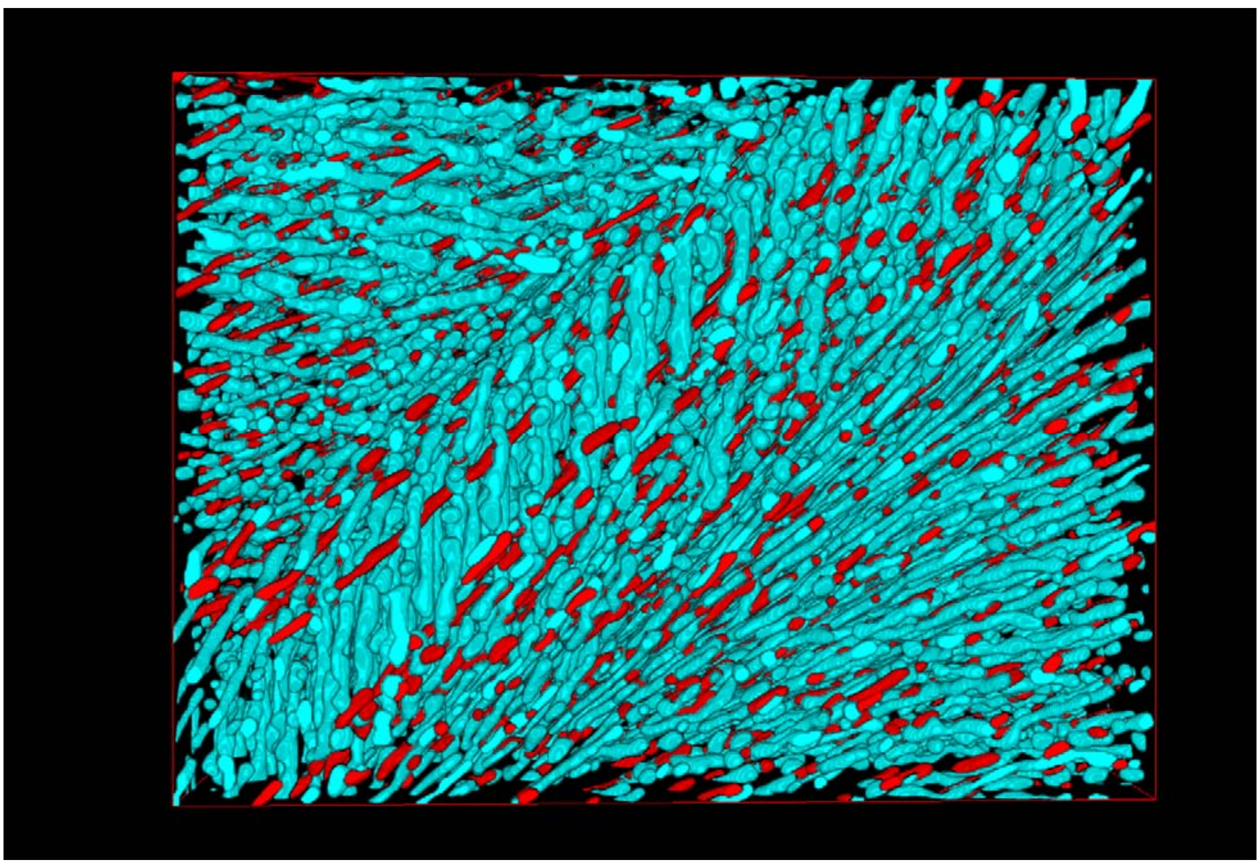

FIG 2: Reconstructed volume of the two different precipitates (matrix transparent). For the segmentation both images (In-Lens SE and EsB) were required to properly identify the different phases.

https://doi.org/10.1017/S1431927611005289