Influence of Sample Preparation on 3D Microanalysis with EDX in a Dual-Beam FIB

P. Burdet[1], C. Hébert[1], and M. Cantoni[1]

1. Interdisciplinary Centre for Electron Microscopy (CIME), Federal institute of technology (EPFL), station 12, CH-1015 Lausanne, Switzerland.

pierre.burdet@epfl.ch
Keywords: Focused Ion Beam, Energy dispersive X-ray spectroscopy, sample preparation

Gathering information from 2D view of a sample has always been a limitation for traditional microscopy. Many materials properties depend on microstructure, more precisely on the 3D shape of the different phases. Thus microscopy is developing more and more towards 3D. In the past, life science and medicine was the driving motor of 3D microscopy. With the advent of nanotechnology, the importance of 3D microscopy in materials science is growing and many analytical 3D microscopy techniques have been developed.

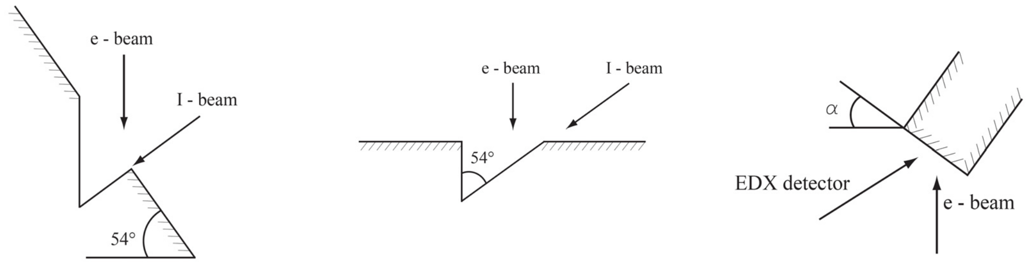
A dual-beam focused ion beam (DB-FIB) microscope is an ideal tool for 3D microscopy. The focused ion beam allows specimen manipulation depositing or removing materials and the scanning electron microscope (SEM) permits to analyze the specimen at micro- and nano-scale. For 3D microscopy, the focused ion beam is used to remove material from the sample and the scanned electron beam is used to analyse the freshly created surfaces. Then these two operations are sequentially repeated. This yield to a stack of images acquired equidistantly through the volume. Modern dualbeam FIB have several detectors such as conventional imaging SEM detectors (secondary electron or backscatter electron), energy dispersive X-ray spectrometer (EDXS) and electron-backscatterdiffraction (EBSD). Each of them has been used to produce 3D models, [1,2,3] for each detecting technique respectively.

Three dimensional EDX (EDX-3D) in a dual beam FIB has been fully automated [4] and some applications have been studied [5], but this technique is not widely spread given the long time acquisition. Arriving of a new, faster detector, the silicon drift detector (SDD), makes it more interesting. This technique gives raise to rich information. The acquisition rate can be greatly improved and quantification becomes possible within reasonable time intervals. But background Xray noise produced by BSE hitting the wall around the sample can induce errors. Changing sample preparation, this problem can be reduced. One method permits to totaly avoid this problem, block lift-out sample preparation [6]. But it’s time consuming. Other sample preparations could reduce this drawback without increasing too much the preparation time in the machine before acquisition.

Using a two phase Al/Zn alloy in which grain size varies from some tenth of micrometer to some micrometers, different sample preparations method were studied, as shown in Figure 1. Platinum deposition on the hole around the freshly cut section is used to highlight background contribution. Figure 2 shows aluminium particles 3D reconstruction out of 269 EDX maps. The tilted surface geometry (c.f. 1(a)) has been used for this acquisition.

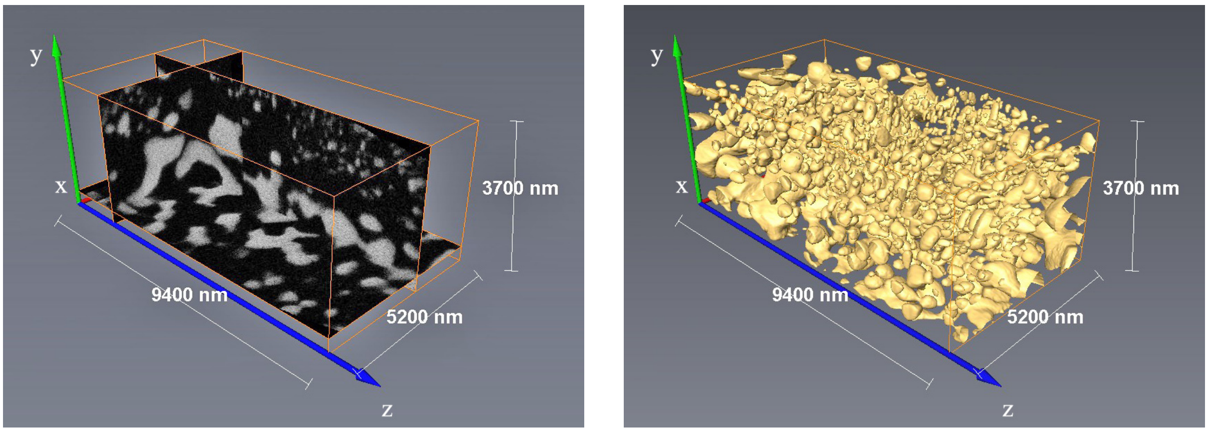
1. L. Holzer et al., Journal of Microscopy 216 (2004) p84.
2. P.G. Kotula et al., Microscopy and Microanalysis 12 (2006) p36.
5. M.A. Groeber et al., Materials Characterization 57 (2006) p259.
4. M. Schaffer et al., Microchimica Acta 161 (2008) p421.
6. F. Lasagni et al., Adv. Eng. Mat. 10 p62.

7. M. Schaffer et al., Ultramicroscopy 107 (2007) p587.
8. This research is supported by Carl Zeiss SMT.



(a) Tilted sample surface. (b) Horizontal sample surface. (c) Change azimuth

Figure 1: Schema of the studied sample preparation method. (a) and (b) are from the side, (c) from the I-beam position.



(a) Al EDX maps. (b) Reconstruction of Al particles surface.

Figure 2: EDX-3D of an Al/Zn alloy acquired in Zeiss cross-beam Nvision40. 269 EDX maps (along z) with a resolution of 256 X 192 (x and y). Pixel size in x and y of 20 nm. Slice thickness (pixel size in z) of 40 nm.

G. Kothleitner, M. Leisch (Eds.): MC2009, Vol. 1: Instrumentation and Methodology,
DOI: 10.3217/978-3-85125-062-6-118, © Verlag der TU Graz 2009