# Sample Preparation Methods

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## Diamond knife versus Gallium Ions

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The functionality of materials obviously depends on a precise control of the size, shape, crystal structure and composition of the material being synthesized. In order to characterize solids in an appropriate way many sophisticated analysing methods were established and combined. The demand for these highly developed investigation techniques arise because micro- and nanotechnology present many exciting opportunities.

At the beginnings of 3D-FIB EDXS the acquisition time for a three dimensional reconstruction of a certain volume was restricted to a treatable number of counts per second depending on the EDXS-system being used [1, 2, 3]. Today nearly all established X-ray detecting systems are able to handle 100000 counts and more per second. Therefore the time for gathering morphological and chemical information is nearly diminished to the cutting procedure. Gatan, Inc. (Pleasanton, CA, U.S.A.) provided an automated slice and view device for 3D microscopy based on an ultramicrotome developed by Denk et al. [4] that can be used inside the sample chamber of an environmental scanning electron microscope (ESEM). The method is called serial block face scanning electron microscopy (SBFSEM) [5] and was originally developed for studying biological samples, which offer a good cutting performance. However the maximum size of the reconstructed sample volume is often limited by the used microscopic method. Slice and viewing volumes lager than 10 µm³ using FIB techniques is generally a time consuming process and may cause drift problems due to the long slicing processes [6, 7].

This work presents results from two different approaches concerning applications in the field of microscopic serial sectioning. An aluminum-copper alloy (EN AW-2024 T351 by AMAG, Ranshofen, Austria) was used to compare the results acquired by SBFSEM and 3D-FIB-EDXS.

For the ESEM (FEI ESEM Quanta 600) experiment an X-Max silicon drift detector (SSD) from Oxford Instruments Analytical Ltd., UK was used for fast recording of the elemental maps. The total reconstructed volume is about 42.7 µm x 34.5 µm x 20.0 µm at a voxel size of 100 nm x 100 nm x 100 nm. The recording time for the 200 elemental maps (slices) was 22 hours. This comparatively short recording time was rendered possible by the combination of high detector count rates (75 kcps - kilo counts per second) and the fast and automated slicing process of the *in situ* ultramicrotome.

For the FIB (FEI Nanolab Nova200) experiment a Bruker-AXS system (Berlin, Germany, 10 mm2 SDD, Quantax400) was used with a total acquisition time of 53 hours for 200 elemental maps (sample volume: 40 µm x 30 µm x 20.0 µm) (“Figure 1, 2, 3”). Final data visualization was performed using the Amira 3.1 software (Mercury Computer Systems SA) (“Figure 4”) .

1. B. J. Inkson et al., Journal of Microscopy, Vol. 201, Pt 2, (2001), p. 256.
2. L. Holzer et al., Journal of Microscopy, Vol. 216, Pt 1, (2004), p. 84.
3. P.G. Kotula et al., Microsc. Microanal. 12 (2006), p. 36.
4. W.Denk, H.Horstmann PLoS Biol 2(11) (2004), p.329.
5. H. Reingruber, A. Zankel, C. Mayrhofer, P. Poelt, J. Membr. Sci. 372 (2011) p. 66.
6. M. Schaffer et al., Ultramicroscopy 107 (2007), 587.
7. M. Schaffer et al., Michrochimica Acta and X-ray Microanalysis, Plenum, New York, (1992).

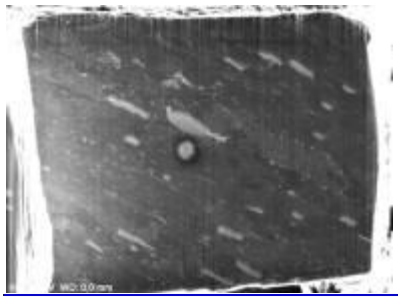

**Figure 1.** SE image of the Aluminium-alloy sample

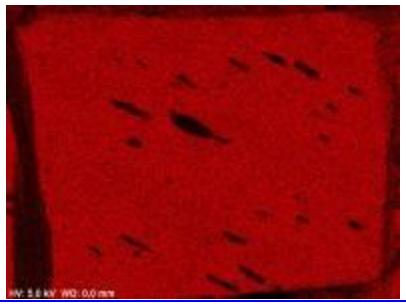

**Figure 2.** Aluminium distribution

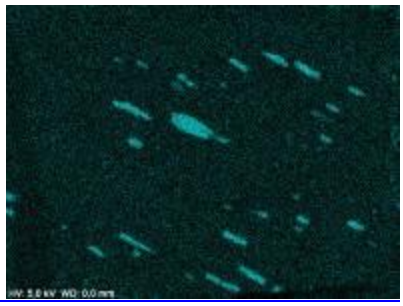

**Figure 3.** Magnesium distribution

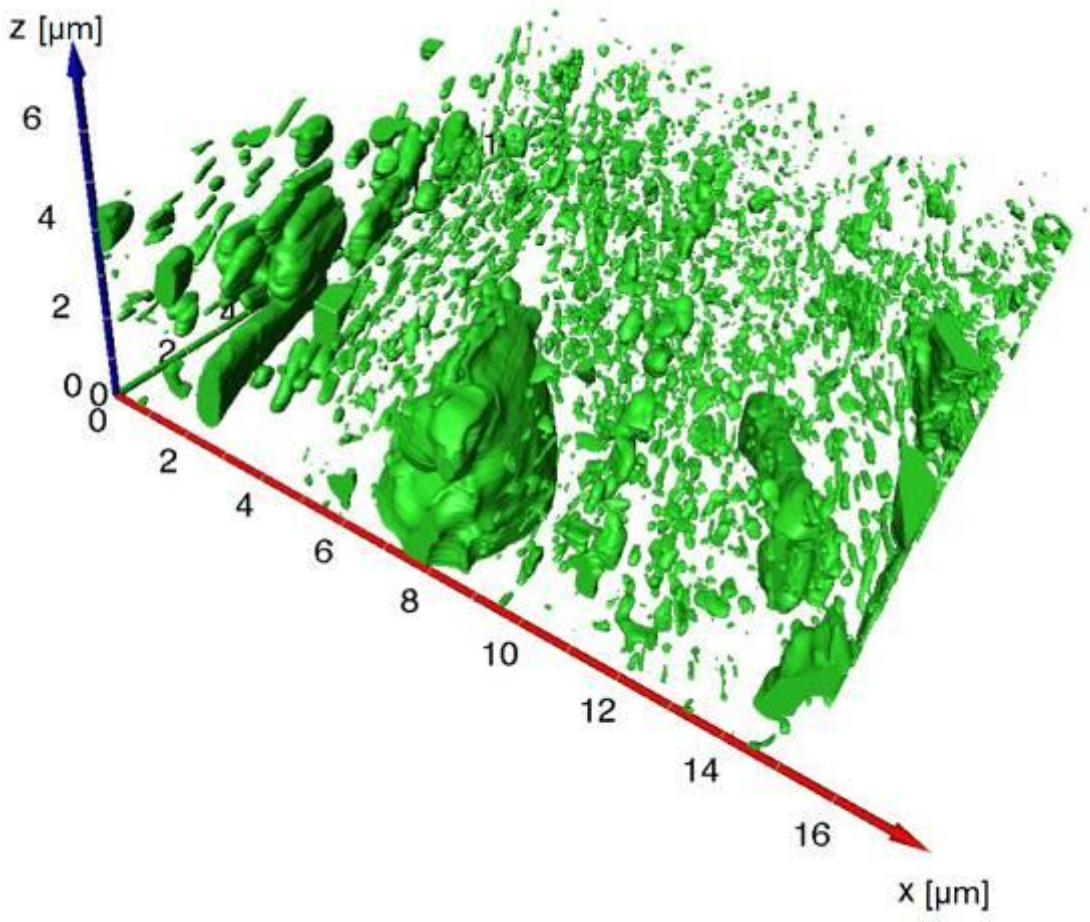


**Figure 4.** 3D-reconstruction of the $Al_2CuMg$ phase (SBFSEM)