Type of presentation: Poster

IT-13-P-2741 Ultra-Fast Three Dimensional Microanalysis Using the Scanning Electron Microscope Equipped with Xenon Plasma Focused Ion Beam

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The three dimensional microanalysis became a widely accepted analytical method during the past few years, gaining from the ability to describe the materials structure and composition as it exists in real components.

A high resolution scanning electron microscope (SEM) combined with a focused ion beam (FIB) is used for a high precision tomographical method based on FIB slicing and SEM observation of the slice. The FIB-SEM can be further equipped by analytical methods like energy-dispersive X-ray spectrometer (EDS) for elemental composition or electron backscattered diffraction analyzer (EBSD) for crystal orientation mapping, resulting in 3D microanalysis, i.e. 3D EDS and 3D EBSD [1].

However, the main limitation of this tomographic method so far has been the speed of data acquisition. This influences also the volume which can be analyzed in reasonable time of several hours. A novel solution for rapid 3D microanalysis is introduced in this paper using a high performance Xe^+ plasma focused ion beam. Such a system allows FIB-SEM tomography on objects with dimensions of hundreds of microns easily within few hours [2, 3], newly combined also with high speed EDS and EBSD. Utilizing the recently developed “static position” approach [4], the speed of 3D EDS and 3D EBSD acquisition can be maximized.

The conventional Ga^+ FIB systems have a limitation of maximum beam current of about 60 nA. For practical FIB-SEM tomography, the volume is limited to units or maximum several tens of microns. Contrary to that, the Xe^+ plasma source FIB incorporated in the TESCAN’s FERA3 FIB-SEM allows ion beam currents up to 2 µA [5]. Together with the higher sputtering yield of accelerated Xe ions it reaches about 50 times higher milling rates than Ga^+ based FIB.

Examples of 3D EDS and 3D EBSD obtained using the Xe^+ plasma FIB-SEM are shown. The 3D EBSD was acquired on a Cu wire commonly used in microelectronics, see Fig. 1. A total volume of 100×100×30 µm was analyzed in about 2.5 hours. Data acquisition time was about 1 min for FIB slicing at 30 keV beam energy with 1 µA beam current and 4 min for 200×200 points EBSD map acquisition for each of the 30 slices.

The 3D EDS example in Fig. 2 shows the volume of 100×70×45 µm of Sn60Pb40 solder processed in about 2.3 hours. Acquisition of 45 slices was done at SEM beam energy 20 kV with lateral resolution 0.5 µm. EDS maps were stored with full spectra at each point. Elemental ROI maps using Sn Lα, and Pb Mα peaks were used for 3D visualization.

References:

[1] S Zaefferer, Book of abstracts EMAS Workshop (2009) p 123.

[2] T Hrnčíř et al, Microsc.Microanal. 19 (Suppl 2) (2013) p. 860.

[3] T Hrnčíř et al, 39th ISTFA Proceedings (2013) p. 27.

[4] Patent CZ 301692 (2009).

[5] T Hrnčíř et al, 38th ISTFA Proceedings (2012) p. 26.

Acknowledgement: The research leading to these results has received funding from the European Union Seventh Framework Program [FP7/2007-2013] under grant agreement No. 280566, project UnivSEM.

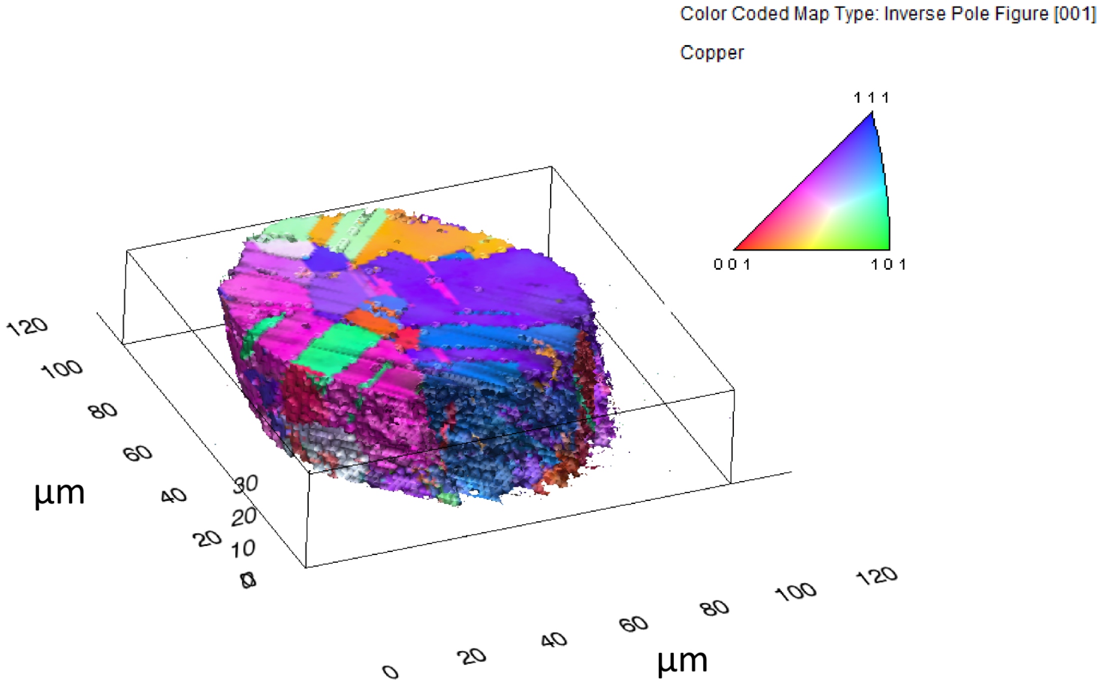

Fig. 1: 3D EBSD reconstruction of a copper wire used in microelectronic. The volume 100x100x30µm was analyzed and all data were acquired within 2.5 hours using FERA3 Xe^{+} Plasma FIB-SEM equipped with EBSD by EDAX . Crystal orientation was mapped using a color coded inverse pole figure (IPF-Z).

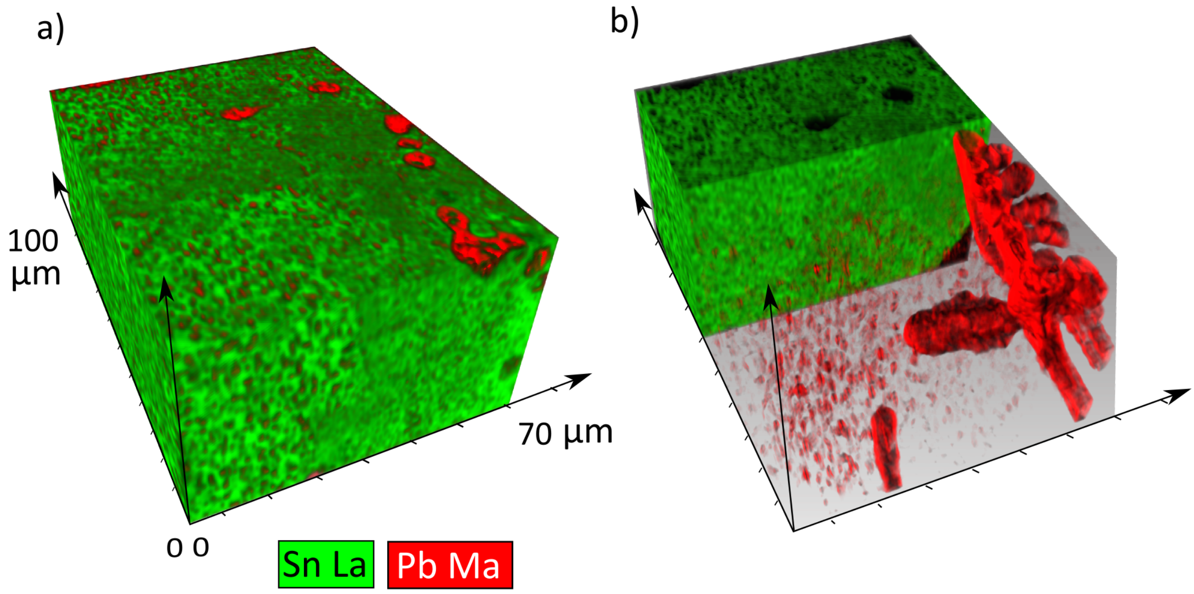

Fig. 2: 3D EDS reconstruction of a Sn60Pb40 solder the FERA3 Xe^{+} Plasma FIB-SEM. Volume of 100x70x45µm was analyzed and all data were acquired in about 2.3 hours. a) Reconstruction of a composite 3D elemental map using Sn Lα, and Pb Mα peaks. b) Separation of Pb phase reveals formation of dendritic structure.