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Enabling Science through European Electron Microscopy

Protocol of sample preparation

Protocols and softwares

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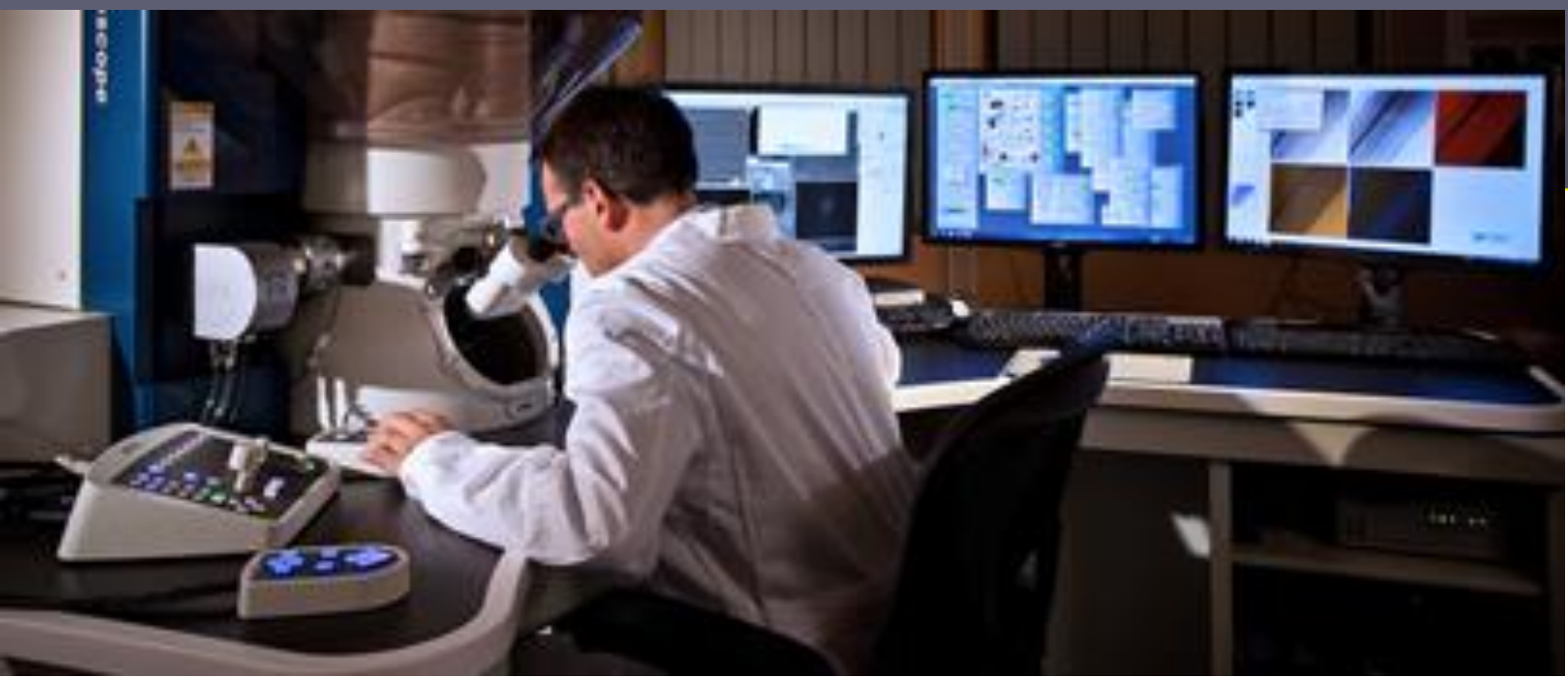

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Protocol of sample preparation

Correlative multiscale electron microscopy, 3D FIB-SEM tomography with detailed TEM investigation - targeted sample removal from analysed volume of ATI 718Plus nickel-based superalloy.

A novel methodology for combining FIB-SEM 3D tomography with the preparation of cross-section lamellae has been developed.

FIB-SEM tomography is an imaging technique that combines a focused ion beam (FIB) with a scanning electron microscope (SEM) to obtain information on the internal structure of specimens. In a typical FIB-SEM tomography experiment, the specimen is situated at the coincident point of the FIB and SEM beams. After finding an area of interest (AOI) in the microstructure and depositing platinum protective layer (IBID - ion-induced deposition) a trench is first carved on the upper surface of the sample with the FIB (Fig. 1). Line marks are made for further stack alignment and slice thickness calibration.

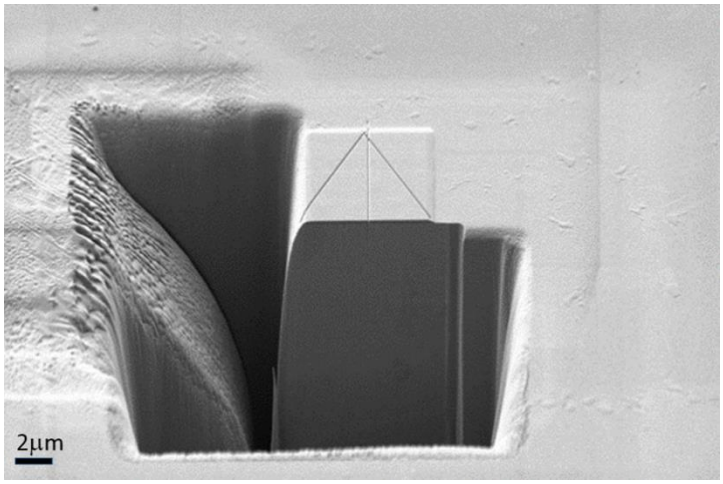

Fig. 1. Platinum layer deposited IBID method with the carved trench.

Then dozens or hundreds of cross-sections of the internal structure of the specimen are made with the FIB and consecutively imaged with the SEM-BSE. The outcome is a stack of SEM images of every single cross-section, which after alignment and further image processing serves to reconstruct the investigated volume in three dimensions. During the slicing process, it is possible to live image every cross-section (Fig. 2).

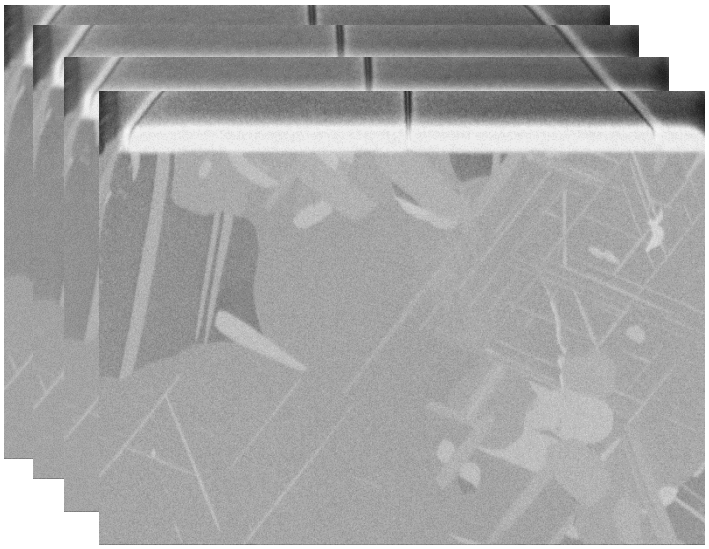

Fig. 2. Stack of SEM-BSE ATI 718 plus microstructure images

When we spot an extraordinary part of microstructure, which can't be visible on the surface of our material, we can stop the slicing process and start FIB lamellae preparation for further TEM microstructure investigation. Preparing a FIB lamella for TEM was started with the deposition of a thicker protective layer at the top of existing Pt layer (Fig. 3).

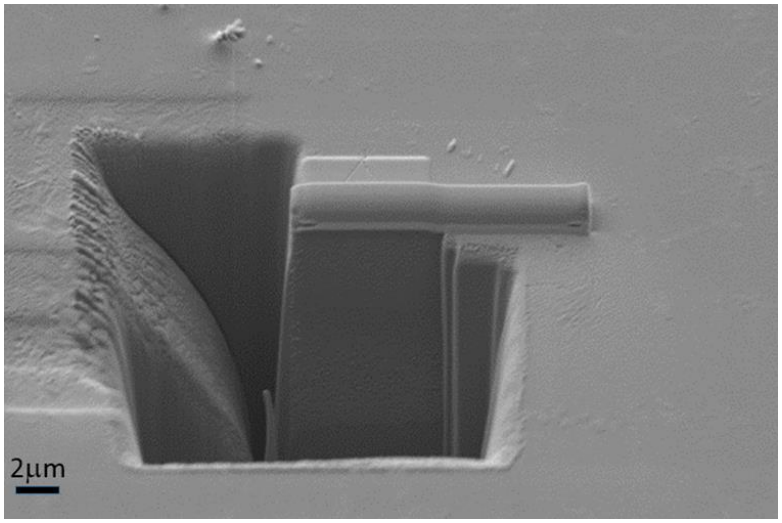

Fig. 3. Platinum layer deposited IBID method fort FIB lamellae preparation.

The next step was made by cutting a second trench with Ga+ focused ions. The accelerating voltage with a value of 30kV and beam current of 10nA were used. Lamella was thinned with an energy beam 30kV 200pA and prepared for transfer to omniprobe TEM holder (Fig. 4).

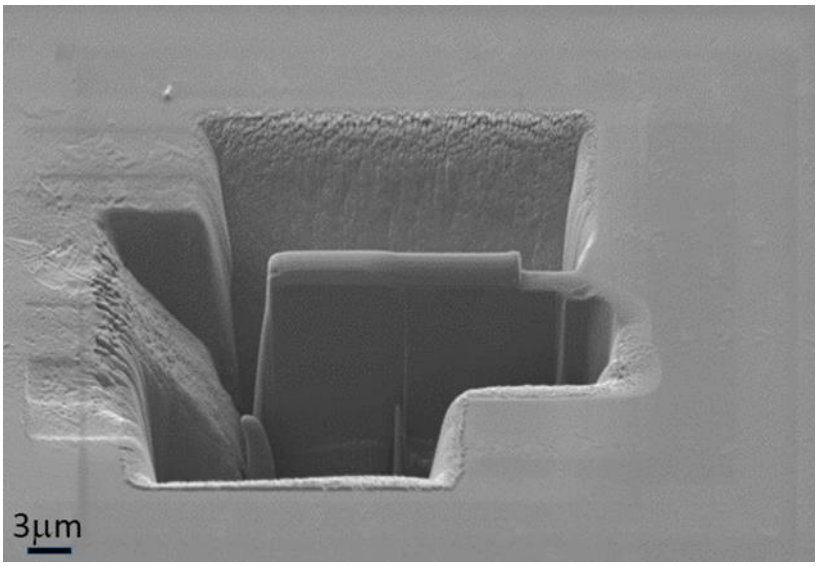

Fig. 4. Lamellae prepared for omniprobe TEM holder.

Transferred lamella was thinned with energy beam 30kV 50pA and polished with 4kV 10pA. The polishing process is complete when the lamellae start to be transparent for electrons, observed in the SEM with 5kV of voltage. (Fig. 5.)

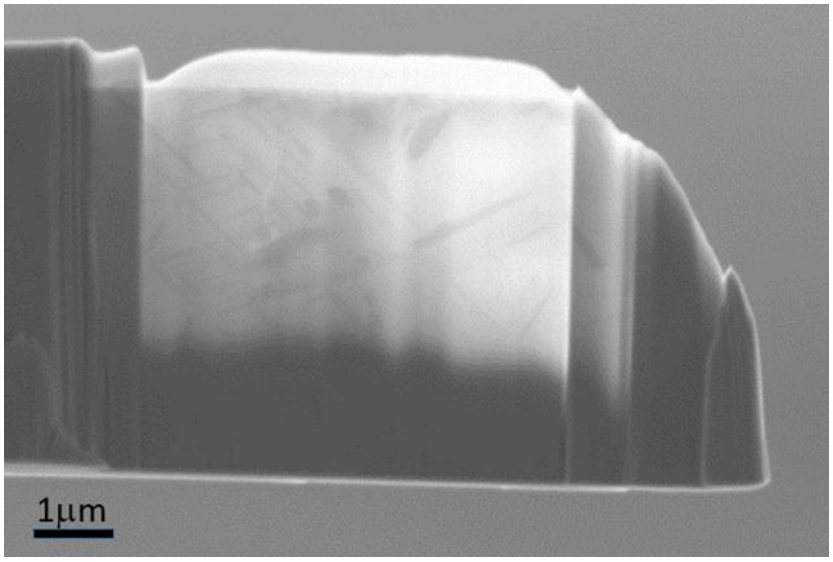

Fig. 5. Polished FIB lamellae of Inconel 718 plus.

Figure 6 presents results of the investigations of the sample a) 3D visualization and b) results of STEM-EDS elemental map.

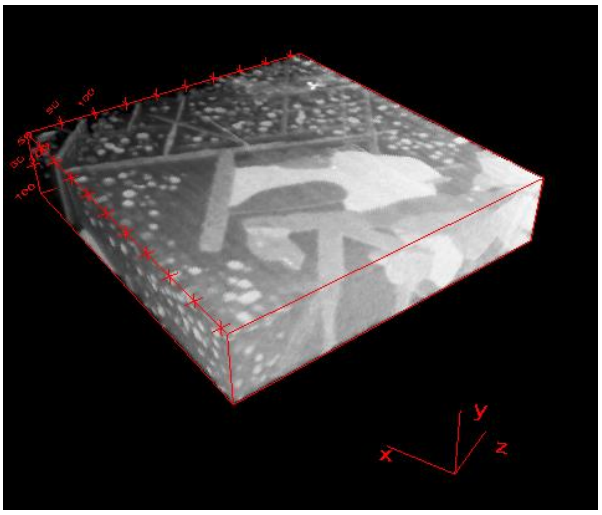

Fig 6a. 3D visualization of the investigated volume. Result from FIB-SEM tomography.

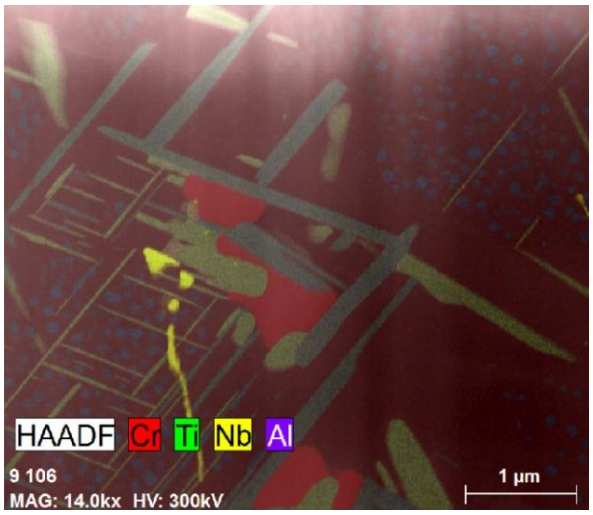

Fig. 6b. Superposition of selected STEM-EDS elemental maps. TEM lamella.