Microsc. Microanal. 28 (Suppl 1), 2022
doi:10.1017/S143192762200099X

Applications of a Triple Beam Microscope in Materials Science

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Focused ion beam (FIB) is a typical tool for the preparation of electron transparent films for the investigation of microstructure with transmission electron microscopy (TEM). For scanning transmission electron microscopy (STEM) at 30 keV however, thicknesses well below 100 nm are necessary. The addition of a triple beam with noble gas source (Ar) permits low-energy broad ion milling and further thinning of the films with the mitigation of the amorphous layer induced by the FIB [1]. Figure 1 shows the potential of such apparatus on a Si lamella thinned to 100 nm using Ga ions, then further thinned to 40 nm and less with Ar where the lattice spacing of 0,19 nm of the (022) planes is clearly visible at 30 keV with the ultra-high-resolution FE-SEM/STEM SU9000 from Hitachi.

FIB is also a critical tool for the characterization of various types of specimens in material sciences when combined with scanning electron microscopy (SEM). It can deliver crucial, site specific information from materials with minimal sample preparation prior to analysis in the FIB-SEM. In this presentation, FIB was used to produce a ‘thick’ lamella of a single splat of cold sprayed Ti, using the typical lift-out technique [2], but on a 45µm x5µm slab. Regular cross-sectioning for this specimen, with cutting, grinding, and polishing, only resulted in detachment of the cold sprayed particles and/or separation from the bulk. Contrarily to the TEM thinning procedure, only one face of interest of the lamella was milled with the FIB to a fine polish to get rid of curtaining artefacts. The specimen was later brought to a SEM for ex-situ electron channeling contrast imaging (ECCI) where both the microstructure of the splat and the interface with the substrate was studied (Figure 2a-c).

FIB-SEM cross-sectioning can also prove advantageous to avoid the typical downsides of mechanical polishing techniques. Use of lubricants for cutting the specimen as well as abrasive media for polishing such as diamond and silica leave residues that can masks the features of interest. Grinding and polishing are also detrimental to observations, especially when dealing with thin coatings on a substrate, causing rounding of edges and unwanted scratch marks. Embedding can be a solution to this problem, but often leads to the separation of fragile coatings or surfaces layers due to air gaps at the interface when drying. FIB-SEM is therefore indicated for the preparation of specimens that would otherwise be damaged with the conventional methods. Here, a comparison with the traditional embedding and mechanical polishing and preparation with FIB-SEM cross-sectioning was performed on the surface oxide layer of a Ni-based coating following wear sliding tests (Figure 2d-f). A trench of 30µm of the fragile oxide surface layers was prepared in less than an hour with the FIB-SEM and the observation of the microstructure of the intact wear layer free of artifacts was possible with the FE-SEM column of the FIB, avoiding further oxidation of the fresh cut surface.

References:

[1] H Suzuki, Hitachi Scientific Instrument News, **11** (2018) 110302.
[2] L Giannuzzi et al., MRS Online Proceedings Library, **480** (1997), 19–27. doi:10.1557/PROC-480-19

[3] FIB results were collected on Hitachi Ethos NX5000 FIB-SEM located at the Facility for Electron Microscopy Research at McGill University.

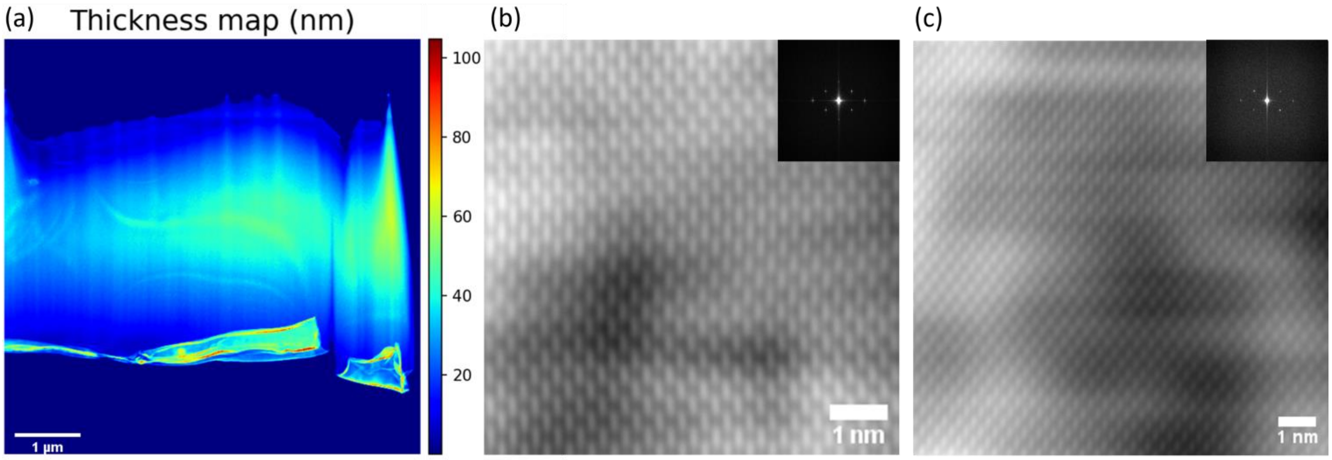

Figure 1. Si thin film. (a) EELS thickness map showing a maximum thickness at 40nm and thinner parts, (b) BF and (c) HAADF in Hitachi SU9000 STEM at 30keV with FFT in inset.

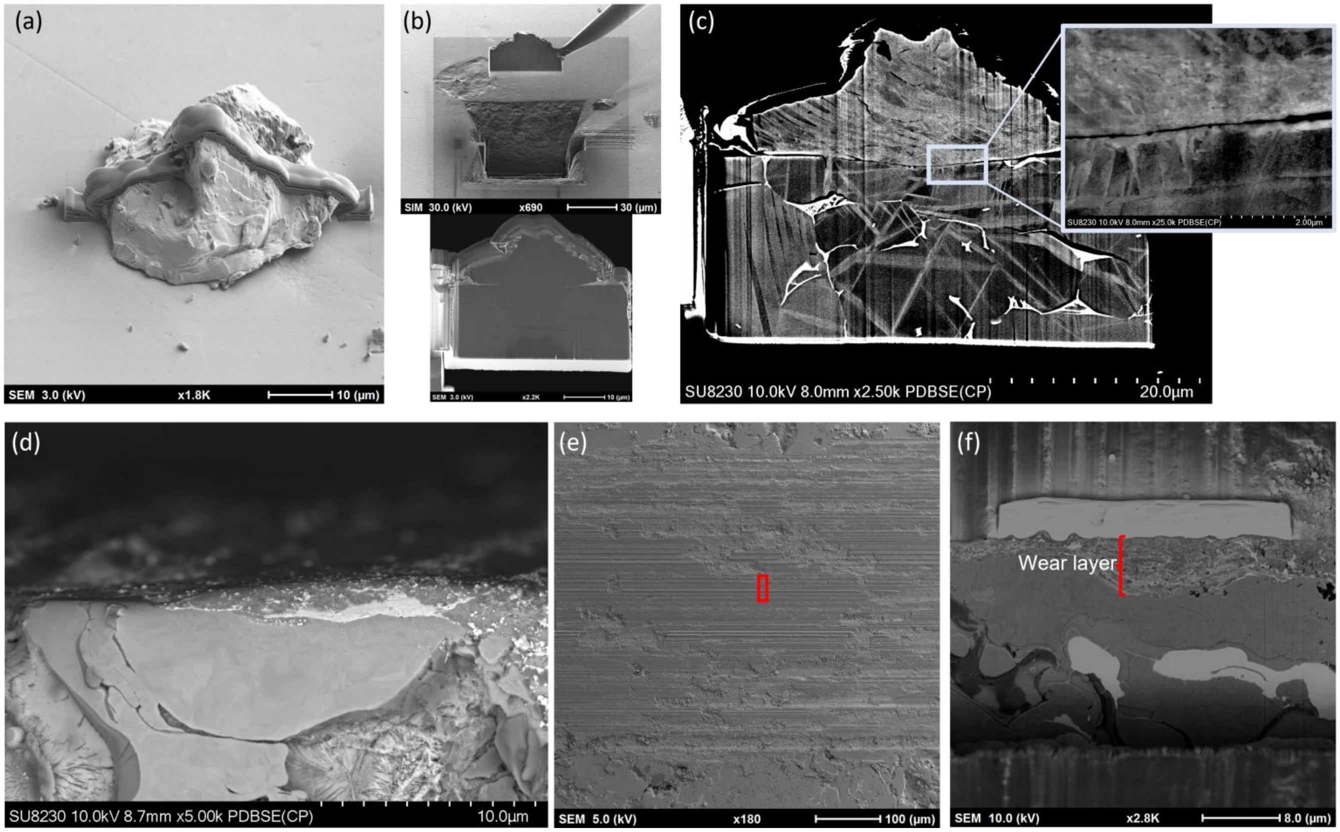

Figure 2. Local characterization with FIB-SEM of sensitive specimens. (a) Single Ti splat on substrate with carbon ion deposited protective layer, (b) Lift out of the thick lamella and thinning of the face of interest at the FIB prior to transfer to SEM, (c) ECCI imaging in the SEM at 10keV. (d) Traditional cross-section preparation with mechanical polishing on Ni-based coating showing rounding of the worn layer and therefore inadequate characterization, (e) Surface view of a wear track in FIB-SEM with region of interest for the FIB cross-section indicated, (f) FIB trench showing cross-section with intact worn layer.