

Recent progress in automation in SEM based microscopy – from 2D to 3D and in-situ deformation and heating

Ali Gholinia, Zhening Yang, Albert Smith, Jack Donoghue, Michael Preuss

¹*Henry Royce Institute and Department of Materials, The University of Manchester,
Manchester, M13 9PL, UK*

Email: ali.gholinia@manchester.ac.uk, zhening.yang@manchester.ac.uk,

albert.smith@manchester.ac.uk, jack.donoghue@manchester.ac.uk,

Michael.preuss@manchester.ac.uk

*In-situ heating and
deformation*

Tri-beam fs-laser for 3D

Automation in SEM

Abstract

Three dimensional (3D) data unlocks the hidden information in two dimensional (2D) analysis, such as the investigation of crack paths, grain boundary planes and much more [1]. Automation in scanning electron microscopy (SEM) has become more reliable for 3D serial sectioning techniques (SST) to acquire large volume 3D datasets over long periods without interruption. There are various ion species and photon available to cater for different applications in high resolution (in nanometers) with Ar⁺ broad ion beam (BIB) and Ga⁺ focused ion beam (FIB) and more rapid and large volumes investigation by milling using Xe⁺ plasma-PFIB and by ablation using femto second (fs) laser (~1mm³). Figure 1 shows the respective volumes and resolutions possible with the various 3D SST techniques, where the large volumes achievable with fs-laser overlap with X-ray computed tomography (XCT) techniques for practical correlative microscopy investigations [2]. The in-situ tribeam fs-laser PFIB enables simultaneous multi-modal investigation using electron back scatter diffraction (EBSD) and energy dispersive spectroscopy (EDS) with flexibility to use both ablation and milling.

In recent years automation in SEM has also helped to develop new in-situ techniques for deformation and heating too. In-situ deformation and heating experiments present a new insight into phenomena such as dislocation activity during deformation, phase transformation, recrystallization and grain growth during heating. The conventional post-mortem, ex-situ techniques are limited to a snap shot of the events, which limit our full understanding of these events. The conventional manual in-situ techniques are limited to few data points and relocation of the area of interest is not precise between data points. We have developed an automated in-situ deformation and heating integrated system, which can run autonomously over a long time

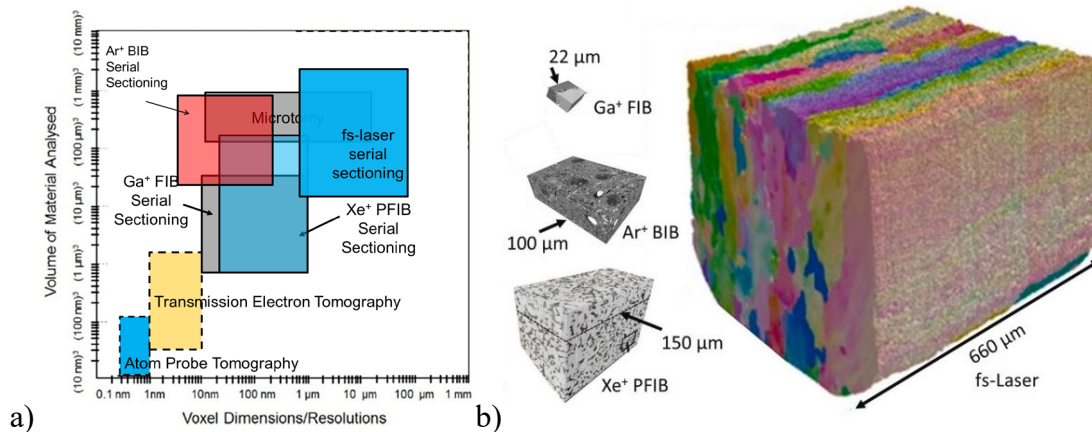


Figure 1- a) Comparison of resolution and volume between 3D techniques, b) representative volumes analysed during a similar time scales by various 3D techniques.

to acquire data with exceptionally high temporal resolution and high stability in automated site location and focus using a fiducial. The SEM, EBSD and High Resolution Digital Image Correlation (HR-DIC), have improved dramatically with the development of a fully integrated systems enabling fully automated scans over long durations. Figure 2 shows the high GND density and high local strain near a twin boundary for a Ni Inconel 690 alloy after 10% deformation, which are calculated from EBSD and HR-DIC maps, respectively.

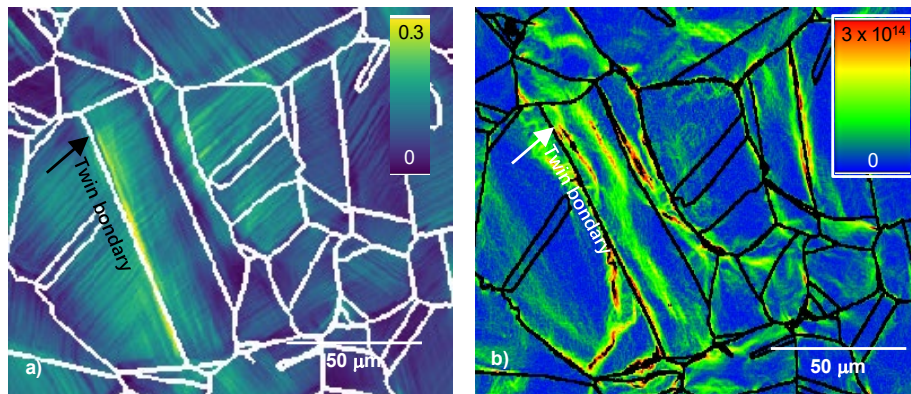


Figure 2- a) Effective shear strain from HR-DIC and b) GND density per m^2 from EBSD in IN690 deformed to 10% tensile strain at room temperature.

References

- [1] A. Gholinia, J. Donoghue, A. Garner, M. Curd, M.J. Lawson, B. Winiarski, R. Geurts, P.J. Withers, T.L. Burnett, Exploration of fs-laser ablation parameter space for 2D/3D imaging of soft and hard materials by tri-beam microscopy, *Ultramicroscopy*, 257 (2024) 113903.
- [2] T.L. Burnett, R. Kelley, B. Winiarski, L. Contreras, M. Daly, A. Gholinia, M.G. Burke, P.J. Withers, Large volume serial section tomography by Xe Plasma FIB dual beam microscopy, *Ultramicroscopy*, 161 (2016) 119-129.