Application note

Laser PFIB for large-volume 3D EBSD: a correlative microscopy perspective

Authors

Bartłomiej Winiarski, Ph.D.,
Thermo Fisher Scientific.

The Thermo Scientific™ Helios™ Laser Hydra System features our most recent advances in multi-beam instrumentation, combining a femtosecond laser with a rapidly switchable multi-ion species plasma focused ion beam (PFIB) and a field-emission gun scanning electron microscope (FEG-SEM). This combination expands the boundaries of traditional Thermo Scientific™ DualBeam™ Technology to deliver high-quality macroscale cross-sections for rapid, large-volume analysis of challenging materials at nanometer resolution.

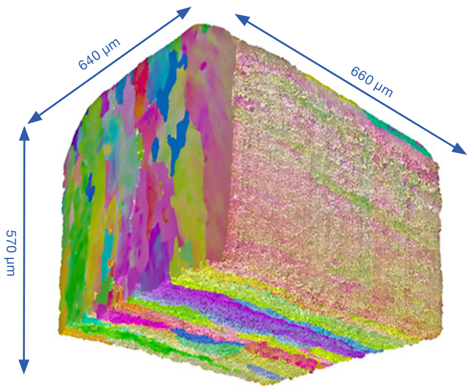

Figure 1. 3D volume of an aluminum alloy obtained with laser serial sectioning.[2] Images were rendered in Dream 3D, freeware software for 3D EBSD post-processing and visualization.

Introduction

The advent of commercial FIB-SEM in the 2000s enabled automated serial-sectioning tomography, 3D-energy-dispersive spectroscopy (3D-EDS), and 3D electron-backscatter diffraction (3D-EBSD) of material volumes smaller than 40×40×40 μm^3. Since 2015, Xe^+ plasma FIB-SEM (PFIB-SEM) has expanded these techniques to volumes of ~250×250×250 μm^3, with voxels approximately dozens of nanometers in size. Since 2019, the addition of femto-second laser ablation to Xe^+ PFIB-SEM pushed these 3D techniques further to millimeter-scale volumes,[1-3] raising the standard for multi-modal data collection from nanometers to millimeters. In 2021, fast-switching multi-ion PFIB (Xe^+, O^+, Ar^+, or N^+) further extended the applications of laser PFIB.

These "DualBeam" and "TriBeam" platforms fit seamlessly in multi-scale and multi-modal correlative microscopy (CM) methodologies. CM workflows coordinate the characterization of materials across a range of length scales to solve scientific problems in 2D and 3D, including time-resolved experiments.

Correlative microscopy in materials and life sciences can use a number of potential 3D methods such as micro/nano X-ray computed tomography, optical microscopy, (P)FIB-SEM, transmission electron microscopy (TEM), as well as nano-computed tomography in TEM and SEM.[4,5] Combining these methods for the same region of interest at different length scales allows 2D/3D spatial and temporal registration of many imaging modalities, i.e., visible light, cathodoluminescence, and electron imaging, ion microscopy, EBSD, WDS and EDS analytics, X-ray tomography, magnetic resonance imaging, Raman, atomic force microscopy, etc.

Figure 2 demonstrates various 3D data collection techniques used in material science and the length scales they can access.[6] Figure 3 shows the serial sectioning techniques used for 3D EBSD.[1]

This application note discusses current developments in 3D EBSD for correlative microscopy, using the laser multi-ion PFIB-SEM. A study from The University of Manchester is highlighted as a practical example, where the CM workflow is applied to the large-volume analysis of environmentally assisted cracks in 7xxx alloys.[2]

Methods

3D correlative microscopy/tomography workflow

The basic workflow (Figure 4a) uses the Helios Laser Hydra System together with a micro X-ray computed tomography (X-ray µCT) scanner, a cross-platform holder/adapter kit (Figure 4b), and Thermo Scientific™ Avizo™ 3D Software. The workflow can be extended to 3D nanoscale investigations using TEM and/or atom probe tomography (APT) (Figure 2).

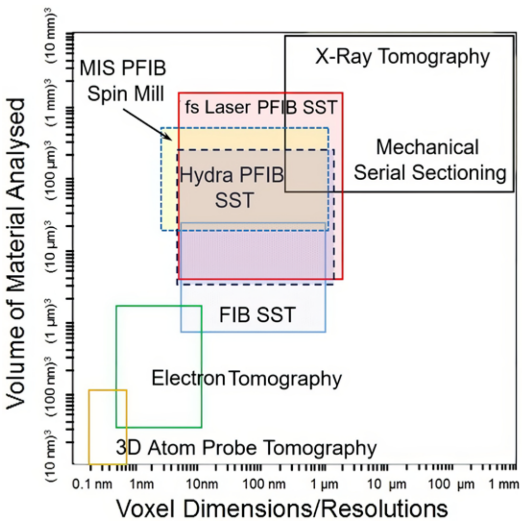

Figure 2. 3D imaging methods for materials science. SST = serial sectioning tomography.[6]

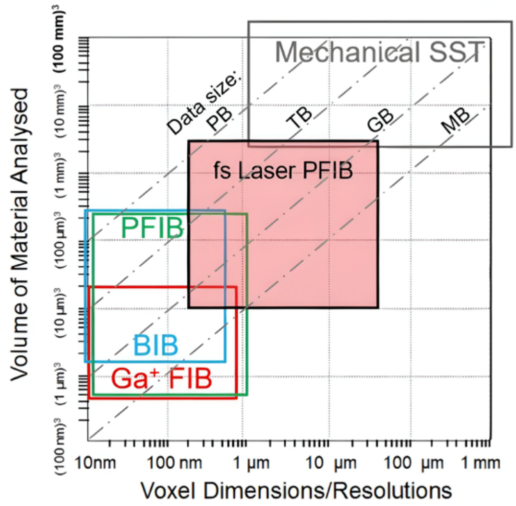

Figure 3. Serial sectioning techniques for 3D EBSD, including the size of data collected, based on Reference 1. BIB = broad ion beam.

Femtosecond laser PFIB-SEM

The Helios Laser Hydra System is built on the fifth generation of the Thermo Scientific™ Helios Hydra™ DualBeam platform (Figure 5) and includes a fully enclosed femtosecond laser optical path. The laser beam, electron, and multi-ion columns are positioned on a plane and oriented toward a single point. The fs-laser beam supports two wavelengths: 1030 nm (IR) and 515 nm (green). Fs-laser ablation is an athermal process that enables material removal without thermal melting. In general terms, thermal diffusion is slower than the Coulombic explosion in femtosecond laser ablation, resulting in only a nanometer-size heat-affected zone (HAZ). Material removal by the fs-laser, meanwhile, is an order of magnitude higher than what is possible with a Xe^+ PFIB system.

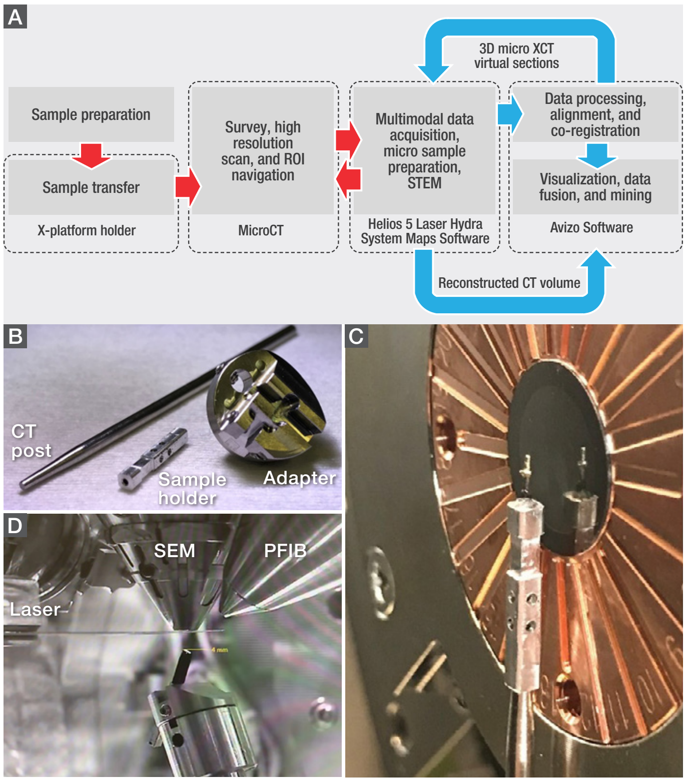

Figure 4. a) The 3D correlative microscopy/tomography workflow. Red arrows indicate sample transfer, blue arrows mark data transfer. b) Cross-platform holder/adapter kit. c) Sample and holder in the µCT. d) Sample and holder in the Helios Laser Hydra System.

Comparison of 3D EBSD parameters for various serial sectioning techniques

	Ga FIB	Xe PFIB	Laser PFIB	Xe PFIB SM*	Broad IB	Mech. Polish.
Slice thickness (nm)	5-50	25-500	250-10,000	1-1,000	10-10,000	200-10,000
Slice rate ($\mu m^3/s$)	20	400	40,000	400	33	100,000-50,000
Max sample size (µm)	50x50	400x400	2,000x2,000	800x800	300x300	50,000x50,000
Damage depth (nm) Si amorphisation	3-22	2.5-13	20-50	2.5-13	<30	35-60

* Spin milling

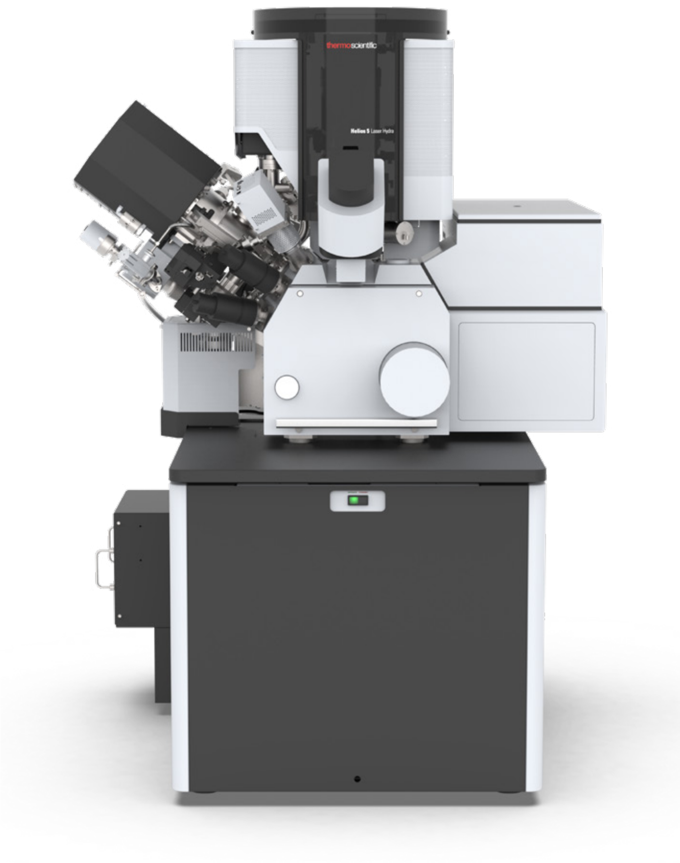

Figure 5. The Thermo Scientific Helios Laser Hydra System.

3D EBSD workflows

3D EBSD workflows are controlled and customized in the laser UI (Figure 6a) or via Python scripting application programing interface (API). These channels control acquisition hardware (Figure 6b) and software. During an automated run, various 2D datasets are sequentially collected (Figure 6c) and assembled in 3D with Avizo 3D Software as well as any other EBSD evaluation packages.

Laser PFIB-SEM stage positions for 3D EBSD data collection (shown in Figure 7) are automatically cycled through as the data is collected in sequence. Some materials require additional surface polishing of the laser-ablated cross-section to remove laser-induced periodic surface structures (LIPSS), as shown in Figure 7e and 7f. These periodic structures can be suppressed by fine tuning of laser parameters such as wavelength, repetition rate, pulse energy, etc.[7,8]

Figure 6. 3D EBSD with Helios Laser Hydra PFIB-SEM. A) Automation is enabled with the laser UI or the Python scripting API.. B) Available EBSD (top) and EDS (bottom) data acquisition hardware. C) Resulting multimodal serial sections data.

Customer case study

Figure 8 shows correlative microscopy results from The University of Manchester on stress corrosion cracking in an AA7050 aluminum alloy, combining X-ray analysis with large-volume 3D EBSD.[2]

Correlative microscopy was used to precisely locate regions of interest and conduct multiple studies in order to better understand the drastically different environmentally assisted cracking (EAC) behaviors of the aviation and aerospace alloys AA7050 and AA7085. For more experimental and scientific details see Reference 2.

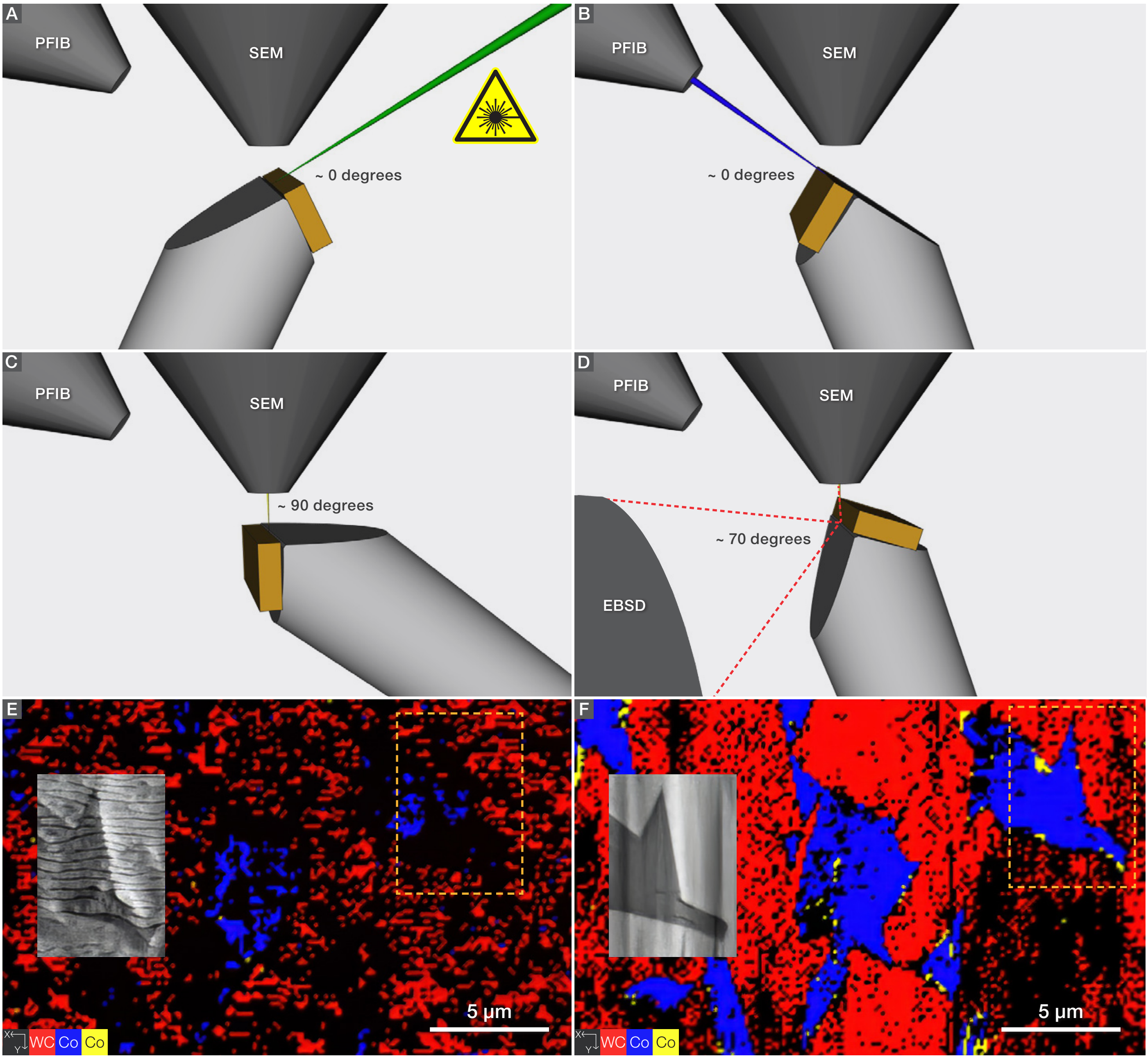

Figure 7. Laser PFIB-SEM stage positions for 3D EBSD data collection. a) Layer removal with laser. b) Optional surface polishing with PFIB. c) SEM (EDS) data mapping. d) EBSD (EDS) data collection. e) EBSD phase map of WC-11 wt% Co after laser ablation. SEM-SE inset shows the LIPSS pattern. f) EBSD phase map after additional PFIB cleaning. SEM-SE inset shows PFIB polished surface.

X-ray CT survey scan | X-ray CT high-resolution scan | 3D-EBSD using femtosecond laser plasma FIB-SEM

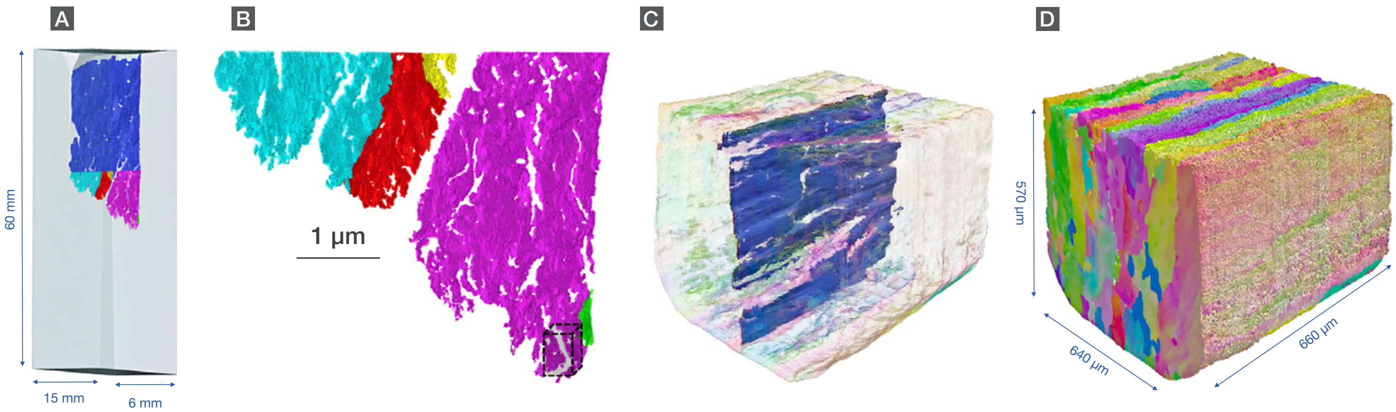

Figure 8. Correlation of X-ray CT datasets and laser serial sectioning for an AA7050 alloy.[2] a) Initial survey XCT scans with overlaid high-resolution ROI scans of the crack tip region. The metal is rendered transparent, and the cracks are shown in dark blue. b) High-resolution ROI XCT scan with each isolated crack feature colored differently (i.e., the crack front is split into separate fingers) within the volume. Approximate positions of the volumes extracted for serial sectioning analysis are also shown. c) 3D reconstruction of the crack path (shown in dark blue). D) The entire 3D volume of the laser serial sectioned specimen. Images were rendered in Dream 3D, freeware software for 3D EBSD post-processing and visualization. *Data courtesy of The University of Manchester, UK*

Summary

Correlative microscopy, combining µCT for site targeting with laser PFIB-SEM for serial sectioning, allowed for precise access to a large volume of material containing EAC cracks. This high-resolution SEM study has provided new insight into the interactions between microstructure and EAC cracks in aluminum alloys, particularly in relation to the size and morphology of the grains, as well as deviations in crack direction. A novel, optimized lift-out procedure for large-scale serial sectioning was also developed by researchers from The University of Manchester, which can be used for similar scientific and industrial research problems.

The Thermo Scientific Helios Laser Hydra System offers researchers the unique opportunity to perform 3D analysis of beam-sensitive and difficult-to-mill microstructures with sizes ranging from nanometers to millimeters. This range of scales helps bridge the gap between conventional Ga^+ FIB and 3D X-ray tomography. The Helios Laser Hydra System is ideally suited for the correlative tomography workflow by coupling micro X-ray CT with serial sectioning tomography while also enabling multiple types of data (structural, crystallographic, chemical, etc.) to be brought into registry for the same region.

Acknowledgements

We would like to thank Prof. Tim L. Burnett and Prof. Philip J. Withers of The University of Manchester for allowing us to highlight their work in this application note.

References

1. Echlin, MP, et al. Recent Developments in Femtosecond Laser-Enabled TriBeam Systems. JOM 73, 4258–4269 (2021)
2. Garner, A, et al. Large-scale serial sectioning of environmentally assisted cracks in 7xxx Al alloys using femtosecond laser-PFIB. Mater Charact 188:111890 (2022)
3. Winiarski, B and Geurts, R. Laser Xe+ Plasma FIB-SEM: Correlative microscopy of 3D microstructures from nanometers to millimeters. Wiley Analytical Sci. Magazine (2020)
4. Winiarski, B, at al. Correlative Tomography for Additive Manufacturing of Biomedical Implants. Microscopy and Microanalysis 31(6), S4-S9 (2017)
5. Burnett, LT, et al. Correlative Tomography. Scientific Reports 4:4711 (2014)
6. Burnett, LT, et al. Large volume serial section tomography by Xe Plasma FIB dual beam microscopy. Ultramicroscopy 161, 119-129 (2016)
7. Winiarski, B, and Geurts, R. Proceedings of the 6th International Congress on 3D Materials Science, Washington, DC. (2022)
8. Jelinek, A, at al. A Perspective to Control Laser-Induced Periodic Surface Structure Formation at Glancing-Incident Femtosecond Laser-Processed Surfaces. JOM 73, 4248–4257 (2021)