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STEM-in-SEM highly deformed structure investigation with focus on electron-transparent specimen preparation

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In recent years, scanning electron microscopy (SEM) techniques have undergone significant development and expansion in academia and industry. SEM is used for a wide range of applications in the materials science, engineering, and earth science fields. The technique can gather data from large areas (to millimeter scale) and spatial resolution to nanometric scale. However, when using some analytical techniques, such as electron backscatter diffraction (EBSD) or energy dispersive X-ray spectroscopy (EDS), the SEM spatial resolution can be limited to ~100 nm (depending on several parameters, including the accelerating voltage of the microscope and the atomic number of the material) [1]. The spatial resolution limitation is the result of the electron beam volume interaction created in the analyzed material. Putting an electron-transparent specimen into a SEM and conducting analytical scanning transmission electron microscopy (STEM) observation overcomes this problem; the STEM-in-SEM technique can offer improved sub-nanometric spatial resolution. Following the development of the STEM-in-SEM approach, analytical techniques typically associated with SEM have been adapted to electron-transparent specimens. One example is transmission Kikuchi diffraction (TKD) [2-5]. The TKD technique can offer improved spatial resolution of ~3 to 10 nm, depending on specimen thickness [6], within maximum fields of view in the ~101 to 102 µm2 range [7]. The development of on-axis detectors specific for TKD [5, 8] offer not only crystallographic orientation mapping of an electron-transparent specimens, but also bright field (BF) and dark field (DF) forward-scattered electron imaging possibilities.

Many factors affect the outcome and effective spatial resolution of STEM-in-SEM analyses, such as the material density, material atomic number, electron beam voltage, detector type, and specimen preparation (especially specimen thickness and uniformity). The latter of these, specimen preparation, appears to be one of the most critical factors. Specimen preparation can greatly affect the resolution and maximum field of view attainable. For the accurate study of material microstructures, specimen preparation artifacts (e.g., contamination, lattice damage, and additional plastic deformation) must be minimized or avoided.

Standard transmission electron microscopy (TEM) specimen preparation techniques have been adapted for STEM-in-SEM observation. Ga focused ion beam (FIB) systems are a popular tool for TEM specimen preparation. However, Ga FIB milling may damage the specimen surface through amorphization and Ga ion implantation; Ga ion implantation can cause strain induction, which leads to microstructure damage [9, 10].

Controlling the specimen thickness is critical for successful STEM observation. Precise control of FIB lamella thickness necessitates the application of low ion beam energy and current. This degree of precision can require a highly experienced FIB operator and can be time consuming. An alternative specimen preparation workflow that is accurate, reproducible, and expeditious is preferable.

In this work, we demonstrate that adding low-energy concentrated Ar ion beam milling to the workflow effectively and reliably removes Ga FIB damage and results in high quality electron-transparent specimens for STEM-in-SEM observation.

Specimens from highly deformed Ni alloy (cold-rolled sheet reduced by 95% in thickness) were prepared in a Ga FIB system [Scios Dualbeam, Thermo Fisher Scientific] by conventional in situ lift-out technique after

sample thinning using 30 keV ion beam energy; final specimen thickness was approximately 300 nm. The specimens were then thinned and polished (milling steps ranged from 900 eV to 100 eV) by low energy, concentrated Ar ion beam milling [Model 1040 NanoMill® TEM specimen preparation system, Fischione Instruments]. This technique allows uniform control of specimen thickness down to 10 nm.

The results of 500 eV concentrated ion beam milling of lamellae prepared by Ga FIB milling are shown in Figs. 1-2. Figure 1 presents an inverse pole figure (IPF) TKD map with high (>10° and low (>2°) angle boundaries overlaid on a band contrast TKD map. The specimen thickness is uniform across the entire lamella and high quality TKD patterns were obtained, as shown in Fig. 2a. Figure 2b presents a DF image (from the area marked by the black rectangle on Fig. 1) on which dislocation domains can be observed (yellow arrows). Figure 2 presents corresponding IPF (Fig. 2c) and local misorientation (Fig. 2d) TKD maps, respectively. The TKD data were collected at 30 kV acceleration voltage and 1.6 nm beam current, with 3.3 nm step size. Changes in color shades within the grains on the IPF maps (Figs. 1 and 2c) indicate deformation accumulation by lattice rotation. Presence of deformation defects observed in the DF image (Fig. 2b) is confirmed by local changes in crystal orientation within a grain (Fig. 2d).

Aside from the critical aspects of sample preparation on accuracy of STEM-in-SEM data, STEM contrast (diffraction, DF, BF) dependence on specimen thickness on will be discussed.

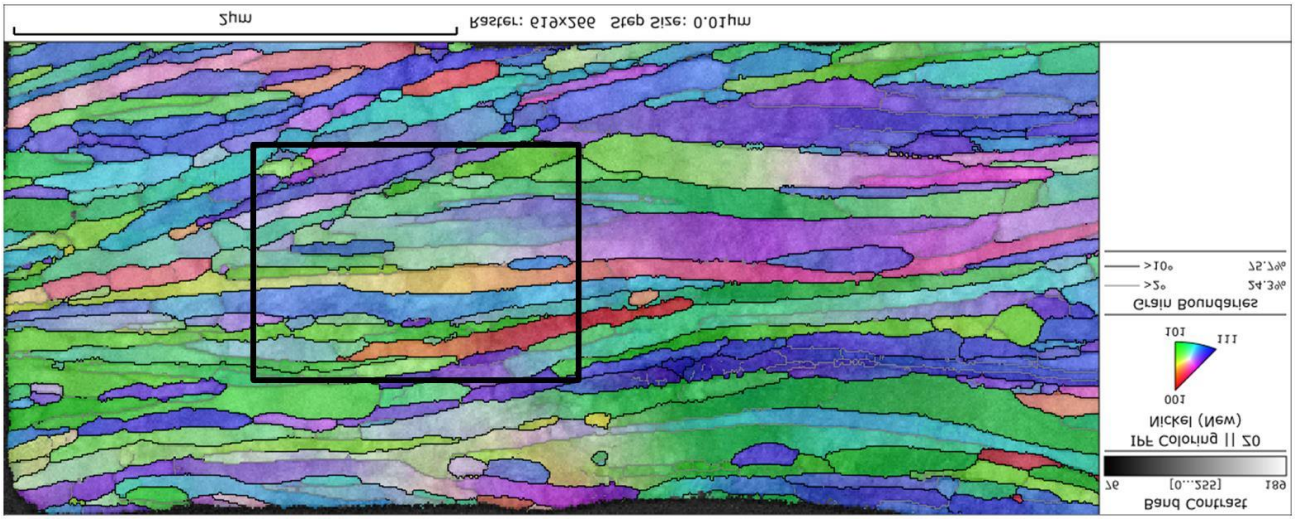

Figure 1. Figure 1. TKD data acquired from entire FIB lamella after ultralow 500 eV energy ion beam thinning using Model 1040 NanoMill® [Fischione Instruments], showing inverse pole figure color-coded map with grain boundary network overlay on band contrast map. Data acquired at 30 kV acceleration voltage (1.6 nA beam current) from the entire FIB lamella with 10 nm step size.

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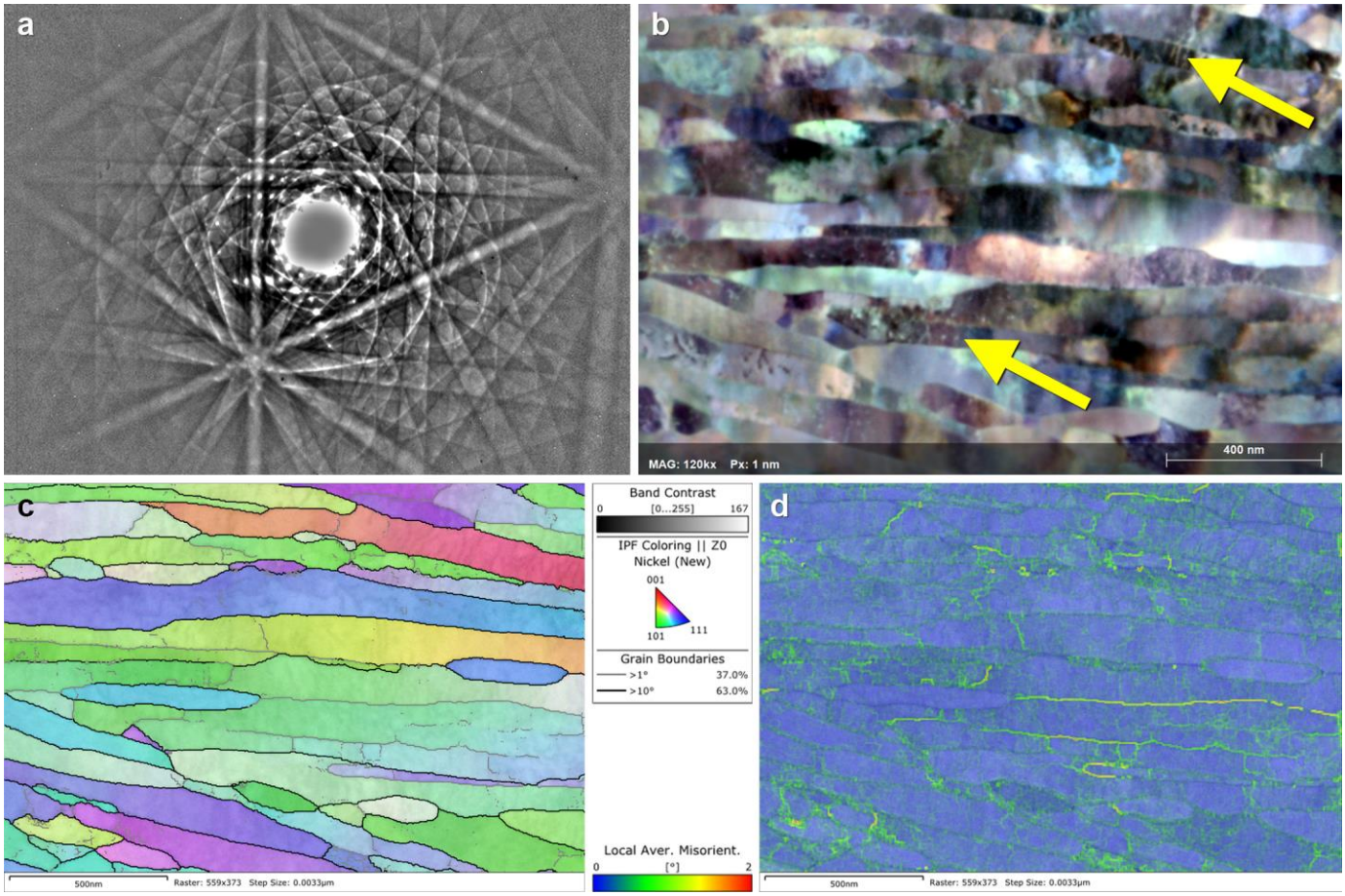

Figure 2. Figure 2. FIB lamella prepared at 30 keV and post-FIB processed at 500 eV using concentrated ion beam milling. TKD pattern acquired at 30 kV acceleration voltage (a); DF forward-scatter electron color-coded image from area marked by a black rectangle in Fig. 1 – dislocation domains are indicated by yellow arrows (image collected with 1 nm step size at 30 kV acceleration voltage) (b); corresponding inverse pole figure TKD maps (c); and a local misorientation TKD map collected from the area marked by the black square in Fig. 1 (d). TKD data were collected at 30 kV acceleration voltage and 1.6 nA beam current with 3.3 nm step size.

References

1. Tripathi, A., & Zaefferer, S. (2019). On the resolution of EBSD across atomic density and accelerating voltage with a particular focus on the light metal magnesium. *Ultramicroscopy*, *207*, 112828.
2. Keller, R., & Geiss, R. (2011). Transmission EBSD from 10 nm domains in a scanning electron microscope. *Journal of Microscopy*, *245*(3), 245-251.
3. Trimby, P. W. (2012). Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope. *Ultramicroscopy*, *120*, 16-24.
4. Liu, J., Lozano-Perez, S., Wilkinson, A. J., & Grovenor, C. R. (2019). On the depth resolution of transmission Kikuchi diffraction (TKD) analysis. *Ultramicroscopy*, *205*, 5-12.
5. Brodu, E., Bouzy, E., Fundenberger, J., Guyon, J., Guitton, A., & Zhang, Y. (2017). On-axis TKD for orientation mapping of nanocrystalline materials in SEM. *Materials Characterization*, *130*, 92-96.
6. Van Bremen, R., Ribas Gomes, D., De Jeer, L., Ocelík, V., & De Hosson, J. (2016). On the optimum resolution of transmission-electron backscattered diffraction (t-EBSD). *Ultramicroscopy*, *160*, 256-264.

https://doi.org/10.1017/S1431927621005900 Published online by Cambridge University Press

7. Nowakowski, P., Wiezorek, J., Bathula, V., Mielo, S., Khanal, S., Bonifacio, C., & Fischione, P. (2018). SEM and TEM characterization of plastic deformation structures in aluminum by EBSD, TKD, and PED-based orientation imaging techniques. *Microscopy and Microanalysis*, *24*(S1), 2182-2183.
8. Fundenberger, J., Bouzy, E., Goran, D., Guyon, J., Yuan, H., & Morawiec, A. (2016). Orientation mapping by transmission-SEM with an on-axis detector. *Ultramicroscopy*, *161*, 17-22.
9. Kato, N. I. (2004). Reducing focused ion beam damage to transmission electron microscopy samples. *Journal of Electron Microscopy*, *53*(5), 451-458.
10. Mayer, J., Giannuzzi, L. A., Kamino, T., & Michael, J. (2007). TEM sample preparation and FIB-induced damage. *MRS Bulletin*, *32*(5), 400-407.

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