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# Strain mapping using high-resolution electron backscatter diffraction technique: The influence of sample preparation

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## Background

When developing a new metallic alloy, one of the critical parameters is how it will resist expected loads without failure. Understanding how materials deform is important when doing research and development in fields such as aerospace, where public safety is the top priority. An emerging strain distribution measurement technique is based on cross-correlation analysis of high-resolution electron backscatter diffraction (HR-EBSD) patterns. The technique measures lattice distortion-related differences between EBSD patterns obtained within the same grain. The HR-EBSD technique was applied to elastic strain, evidence of plastic strain, and geometrically necessary dislocations (GND) measurements [1]. However, aside from intrinsic instrumentation limitations, HR-EBSD is very sensitive to diffraction pattern quality. Therefore, sample preparation factors prominently into the accuracy and precision attained in HR-EBSD strain analyses.

However, it is not easy to distinguish between the residual real strain present in a material and the strain induced by sample preparation. To be able to persistently illustrate the effect of stress on the microstructure during sample preparation, we have chosen the phenomenon of dynamic strain-induced transformation (DSIT) of austenite to surface martensite [2, 3]. The DSIT can occur during sample preparation by mechanical polishing (MP) or while using ion beam-based sample preparation techniques: focused ion beam (FIB) or broad ion beam (BIB) [4, 5]. We prepared a series of austenitic precipitation hardening grade steel samples using MP, BIB, and FIB techniques and exposed an austenitic precipitation hardening grade steel to Ga ions at a variety of beam energies and sample geometries. Energy-dispersive X-ray spectroscopy (EDS), electron backscatter diffraction (EBSD) and high resolution (HR)-EBSD techniques were used to assess structural damage induced by sample preparation.

## Methods

The samples were mechanically polished by colloidal silica on a grinder/polisher tool [MultiPrep™ system, Allied High Tech Products]. Then the samples were prepared by broad ion beam milling [TrionMill, Fischione Instruments]. Finally, the samples were exposed to Ga ions at a variety of beam energies and milling geometries in a FIB system [Scios DualBeam Ga FIB system, Thermo Fisher Scientific].

After each sample preparation technique, the samples were analyzed using:

- a Ga FIB system equipped with an EDS detector [X-Max 150 mm2 EDS detector with Aztec software, Oxford Instruments] and an EBSD detector [e-FlashFS EBSD detector with Esprt software, Bruker Nano Analytics], and
- a scanning transmission microscope [S-4700 SEM, Hitachi] equipped with an EBSD detector [NordlysNano EBSD detector with Aztec software, Oxford Instruments]. EBSD data were processed using HKL Channel 5 software [Oxford instruments]. High HR-EBSD measurements were done using Open X-Y software [Brigham Young University].

## Results and conclusions

Figure 1 shows HR-EBSD average strain measurements and geometrically necessary dislocations (GND) of steel samples after mechanical polishing (Fig. 1a, 1c) and BIB milling at 5 keV, 2 keV, 1 keV, and 3° milling angle (Fig. 1b, 1d). No DIST phase transformation of austenite to martensite is observed in the sample prepared by BIB milling. In the sample prepared by MP, 20 % strain-induced martensite is observed. Figure 1a shows an HR-EBSD average strain map of partially transformed martensite to austenite grains. Strain accumulation of up to 10-2 of austenite grains around martensite can be observed. This strain gradient can be correlated with the high GND densities shown in Fig. 1c. Compared to the sample prepared by a BIB mill, the GND density is higher – up to two orders of magnitude. No strain is observed in the BIB milled sample. The BIB milled sample with no presence of DIST of austenite to martensite was exposed to Ga FIB at different ion beam energies. The area affected by the Ga ion beam had a very poor quality EBSD pattern (Kikuchi bands shift and overlap). The EDS analyses demonstrate strong Ga ion implantation. Figure 2a shows the HR-EBSD average strain map of regions both exposed and not exposed to a 1 keV Ga FIB. In contrast to the MP sample, no strain gradient correlated with GND density is found. Figure 2b shows {100} crystal plane orientation facing the Ga ions bombardment. The part of the grain exposed to the Ga FIB shows high strain accumulation. This suggests that structural damage is dependent on grain crystallographic orientation. Structural damage and strain accumulation result from FIB-induced atom displacement and vacancies, which leads to strain-induced austenite deformation to martensite.

**Graphic:**

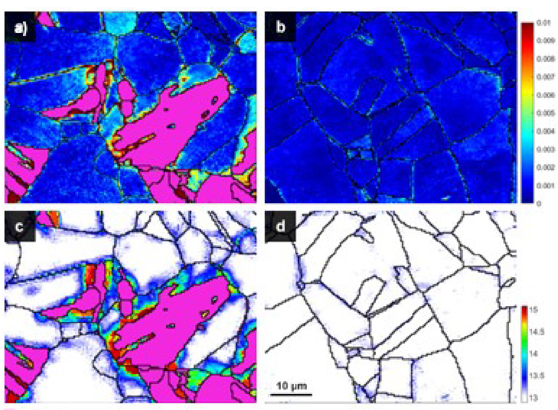


**Figure 1:** HR-EBSD maps showing average strain for mechanically polished sample (a), after argon broad ion beam milling (b), geometrically necessary dislocation density maps for sample after mechanical polishing (c), and broad ion beam milling (d).

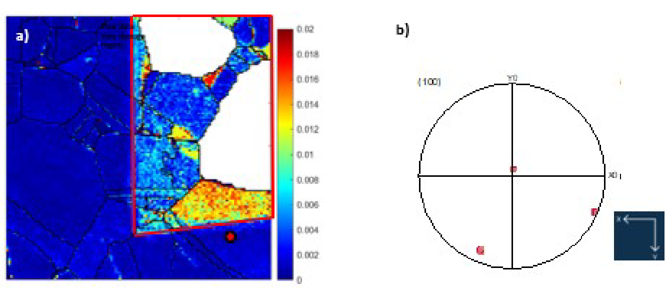


**Figure 2:** HR-EBSD average strain map (a) of regions both exposed and not exposed to a 1 keV Ga FIB (indicated by the red dot). Pole figure (b) showing {100} crystallographic oriented grain (marked with red dot) that was partially exposed to Ga ion bombardment.

**Keywords:**

HR-EBSD, GND, strain, BIB, FIB

**Reference:**

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