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Metrology of Sample Preparation for Electron Microscopy: Application to Strain Measurements

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Many techniques have been developed for strain measurements, such as X-ray diffraction [1], electron back-scattered diffraction (EBSD) [2], electron channeling contrast imaging (ECCI) [3], and digital image correlation (DIC) [4]. All the techniques can be complex and require specific calibration routines.

EBSD is a scanning electron microscope-based technique widely used for investigating the microstructure of crystalline materials. EBSD strain measurement techniques rely on recording changes in EBSD pattern quality or local changes in crystal orientation within a grain. The accuracy of those methods, however, depends on various factors, such as:

- detector characteristics (high-resolution image sensor, low-distortion optic)
- measurement of crystallographic orientation (calibration, band detection, indexing algorithm).

Aside from intrinsic instrumentation limitations, sample preparation factors prominently into the accuracy and precision attained in EBSD strain analyses. The goal of sample preparation is to get a representative sample that reflects the native state of the material. However, sample preparation itself can change the sample structure and, thus, have a dramatic impact on the investigation and interpretation of observed phenomenon. This is especially true when considering surface analytical techniques, such as EBSD analyses. For example stainless steels (e.g., 17-4 SS, 300 series, or Fe-30Ni cryogenic steel) have a metastable austenite phase that transforms very easily to martensite. This transformation phenomenon is known as dynamic strain-induced transformation (DSIT); the product of that transformation is surface martensite (SM) [5, 6]. The DSIT can occur during sample preparation by mechanical polishing [7] and focused ion beam (FIB) techniques [8]. Therefore, accurate sample preparation metrology, which is applicable to engineering and manufacturing sectors, is critical for determining appropriate protocols (such as heat treatment) and process to establish the desired material structure-properties relationships.

We propose a sample preparation method that produces artifact-free samples for electron microscopies and microanalysis. In the presented work, stainless steel is studied in relation to its sensitivity to strain-induced structural changes. The microstructural changes caused by sample preparation techniques are revealed by EBSD analyses. Figure 1 shows 300 series stainless steel after conventional mechanical polishing (MP) (Fig. 1a) and after a proposed sample preparation methodology using broad argon ion beam (BIB) milling (Fig. 1b). In the case of MP, 50% SM is observed. In comparison, the proposed sample preparation method does not introduce any strain to the material and exposes the native microstructure of the studied steel. Strain accumulation due to MP can be visualized easily by local misorientation (Fig. 2a), in contrast to the sample prepared by BIB milling (Fig. 2b). A high resolution EBSD (HR-EBSD) strain measurement comparative study is presented of different sample preparation techniques – MP, FIB milling, and BIB milling. To illustrate the universalism of the presented sample preparation technique, data are collected from two different SEM/EBSD systems.

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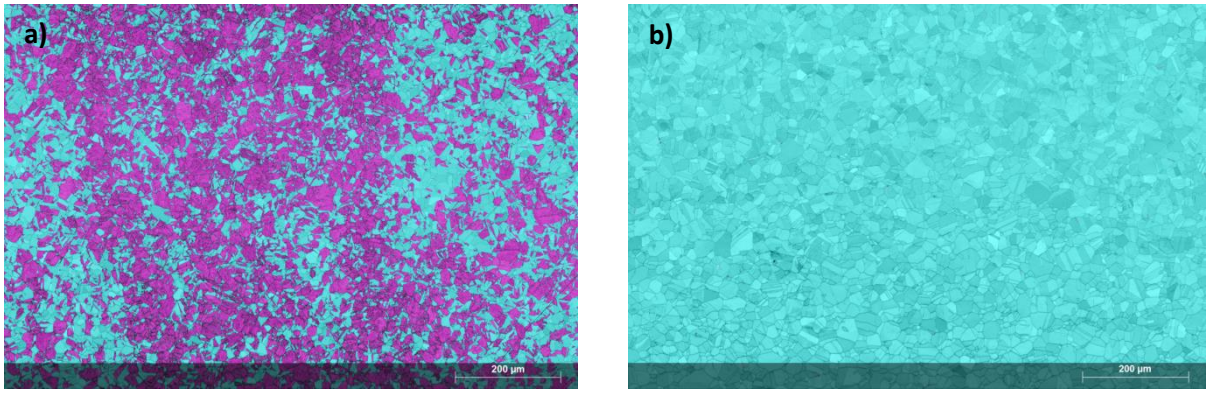
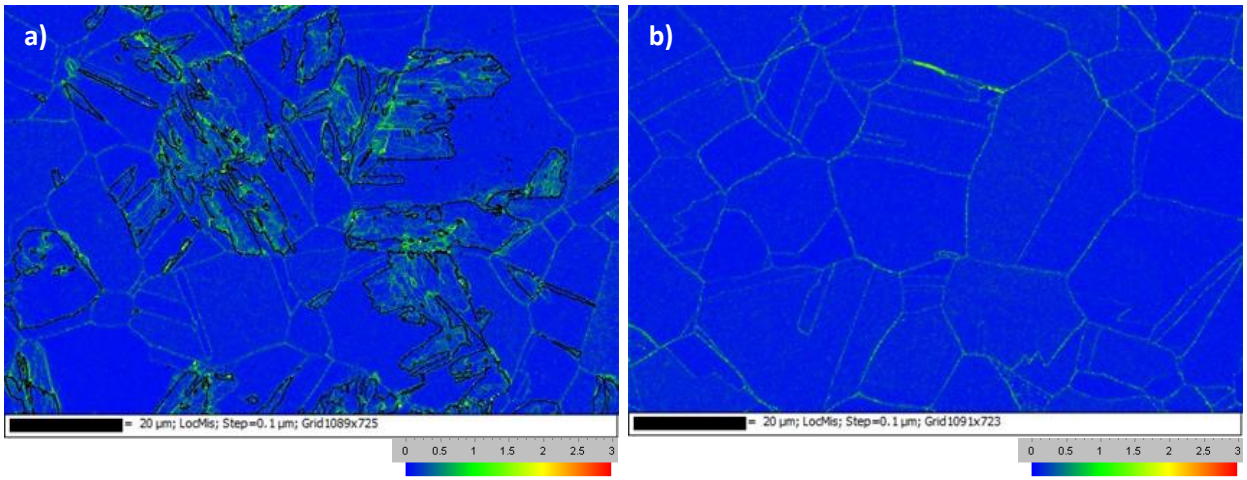


a. Mechanical polishing preparation technique. **b.** Broad Ar ion milling preparation technique.

Figure 1: Austenitic stainless steel 300 series. Low-magnification EBSD measurements (step size: 200 µm). EBSD phase distribution maps (blue: austenite; pink: martensite).



a. Mechanical polishing preparation technique. **b.** Broad Ar ion milling preparation technique.

Figure 2: Austenitic stainless steel 300 series. High magnification EBSD measurements (step size: 100 nm). EBSD local misorientation maps (martensite/austenite phase boundary is in black).

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