Comparison of Different Methods for Large Volume 3D EBSD Analysis of Dual-Phase Steel

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Dual-phase (DP) steels are characterized by a combination of high strength and formability. Their outstanding properties are due to their soft ferritic matrix and hard martensitic islands which results in continuous yielding behaviour. As martensite plays an important role for the mechanical properties of these steels, it is important to locate and distinguish it from ferrite. Being able to characterize large numbers of grains allows better statistics on grain size and other properties. Two methods are used to visualize the martensite fraction. Image Quality (IQ) estimates the quality of the EBSD pattern -- lower quality areas are associated with martensite because of higher dislocation density. Kernel Average Misorientation (KAM)[1] compares misorientation between neighboring pixels. The supersaturated and distorted microstructure of martensite induces a misorientation inside the grains, which results in higher local misorientations in martensitic grains.

Serial-sectioning (SS) by mechanical polishing has been employed to collect a large reconstructed volume representing the grain structure of the DP steel sample[2]. The sample shown in Figure 1 was mechanically polished using oxide polishing suspension (OPS) to obtain 1 micron thick slices. This method allowed for greater analysis area than feasible with serial-sectioning by focused ion beam tomography (SSFIB)[3]. Coarse alignment was done by observing micro-indentations placed at the sample edges. Fine alignment was fulfilled by comparing misorientation between every pixel in the current slice and previous slice. The previous is considered fixed and the current slice is translated, stretched, and rotated until the average misorientation is minimal. This procedure is repeated for every slice. Unfortunately, since KAM analysis uses misorientation, it is possible that during the alignment procedure the natural gradient inside the grains is misunderstood as measurement error and has been eliminated by the alignment algorithm and quantification of martensite would be underestimated.

Broad-ion-beam milling (BIB) can efficiently acquire tomographic data of several mm^2 areas because of its intense and large-diameter beam size. The low keV ions employed with BIB milling results in lower surface damage and permits excellent imaging and EBSD analysis. Automated SSBIB[3] tomography automates the sample exchange between the ion mill and SEM/EBSD[4]. The sample receiving dock on the SEM stage keeps sample rotation and shift minimal between successive exchanges by design. Autofocus algorithms are used prior to EBSD acquisition to determine the thickness of the previous slice and keep the current sample surface in focus. Drift and residual sub-micron misalignment between slices is compensated for by translating each slice in x and y. Recently advances in SSBIB development have improved understanding and control over the repeatability and uniformity of increments in polishing depth.

A similar sized volume of dual-phase steel was acquired using automated SSBIB as shown in Figure 2. These results show that automated SSBIB can be used to acquire similarly large volumes of high quality 3D EBSD data as manual methods but with significantly less user labor. Furthermore, since automated SSBIB does not rely on stretching and rotating slices for alignment, KAM analysis is expected to give a more realistic estimate of the martensite fraction.

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[2] Garrido Sorribes, À, 2017, '3D Orientation Contrast Microscopy on Dual-Phase steel', MSc Thesis, Universiteit Gent, Belgium.

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[4] T. Hosman et al., Microsc. Microanal. 23:1, (2017), p16-17.

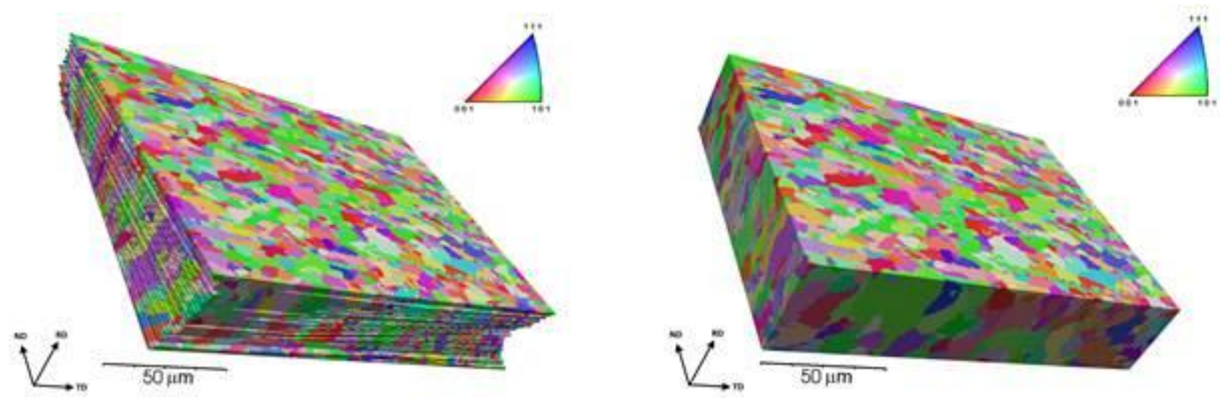

Figure 1. Left: 3D volume reconstructed from aligning 40 consecutive mechanically polished slices of a dual-phase steel. Right: Cropped to the volume common in all slices (185x135x40 um^3), losing 17% of the data.

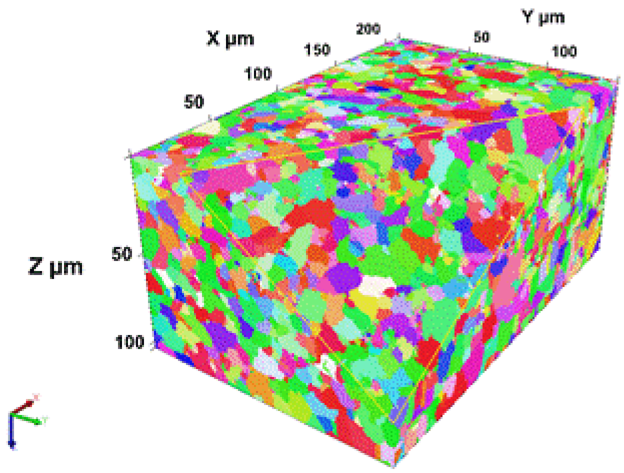

Figure 2. 3D reconstruction of EBSD x IPF data acquired using automated SSBIB tomography. The volume is 213x141x 103 um^3 with 0.5x0.5x0.7 um^3 voxels. Milling used 8 keV Argon ions at 5° incidence for 7 minutes per slice, followed by EBSD acquisition of 20 minutes per slice.

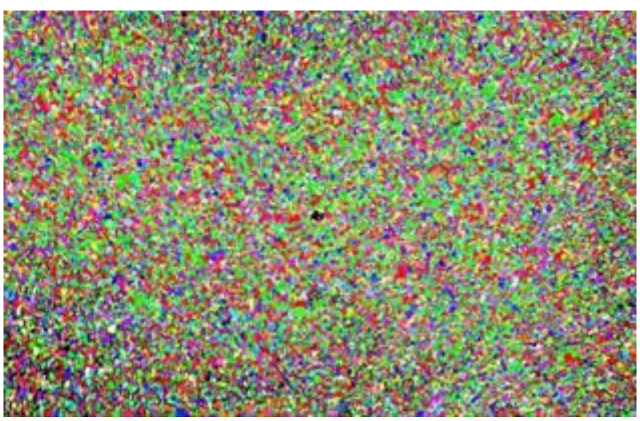

Figure 3. BIB produces polished surfaces much larger than 1 mm^2. A 2.0x1.3 mm^2 EBSD x IPF map of the top surface of the same sample is shown. The pixel size is 0.54x0.54 um^2. The edges of sample areas this large are difficult to keep in perfect focus, which results in an increase in unindexed (dark) pixels.