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EBSD and reconstruction of pre-transformation microstructures, examples and complexities in steels

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ABSTRACT

Electron backscattered diffraction has provided a quantitative tool to study micro/nano-structures in large scales. A recent application of electron backscattered diffraction is the reconstruction of pre-transformed phases in polymorphic systems, especially when there is no retained pre-transformed phase at room temperature. This capability has been demonstrated by various researchers utilizing different approaches towards grain structure and orientation recovery. However, parameters affecting reconstruction have not been investigated systematically. Factors such as post-transformed microstructures (morphology and crystallography), lattice strain (deformation), pattern and sample quality are among the affecting factors. Two-dimensional datasets of different steels have been reconstructed along with a limited 3-dimensional dataset in the current paper. Preliminary results intended for large-scale automatic reconstructions have been presented. They indicate that the successfulness of reconstruction is strongly dependent on the post-transformed microstructure. Factors such as morphology, grain size, variant selection, and deformation play roles. Few examples of reconstruction complexity at prior austenite boundaries leading to uncertain results are presented. Lastly, reconstructions are discussed in terms of meaningfulness and if they correctly represent pre-transformed grains and orientations.

1. Introduction

Solid state phase transformations have been observed since early days of crystallography. They have been investigated using different theoretical and experimental methods. Stability of phases was calculated by thermodynamic laws and their crystallographic structures were determined via X-ray diffraction, electron microscopy, and other characterization techniques [1–5]. Orientation relationships (OR) were determined for common phase transformations (i.e. FCC to BCC) which are still being used to-date [6–12]. Although actual transformation systems may deviate from these exact ORs as reported in the case of near-equilibrium cooled iron meteorites, they are still accurate within a certain tolerance to be implemented in phase transformation studies [13].

Allotropic phase transformations are a key component of various alloy systems and applications. Metals (steels, Ti, Zr, and other less common elements), binary systems (NiTi, TiAl),

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ternary systems such as $Zn_xAu_yCu_z$, as well as non-metals (ZrO_2) could be named. In order to better understand these systems, large-scale quantitative studies of phase transformations and the resulting microstructures are necessary. Electron Backscattered Diffraction (EBSD) and Orientation Imaging Microscopy (OIM) are capable tools that provide large-scale quantitative datasets to study phase transformations [14–19].

EBSD's capability in reconstructing pre-transformed phases has been demonstrated by various researchers utilizing different approaches towards grain structure and orientation recovery [20–40]. These approaches use pattern recognition in pole figures, reverse transformations, and misorientation comparisons (parent–parent, daughter–daughter, and parent–daughter) to reconstruct pre-transformed structures from that of the post-transformed.

Reconstructed data has been used to investigate variant selection, cracking behavior, transformation toughening, deformation and micro-texture. As a result, areas such as alloy development (steels, Ti, TiAl, NiTi, Zr, ZrO_2 etc.), geology (ice and minerals), astrophysics (iron meteorites), ternary alloys ($Zn_xAu_yCu_z$) for medical sensors and actuators will benefit [14,22,23,41–80].

Complexities such as grain boundary variant selection, coinciding variants, and parent grain size effect have to be addressed for accurate automated reconstructions [23]. It has been reported that reconstruction becomes complex when post-transformation microstructures are highly deformed (Friction Stir Welded) or highly polygonal ferritic (Fe–Si electric steels) [20–23,41–44].

Prior austenite (PA) $\Sigma3$ boundaries which compose high fractions of the overall austenite boundaries strongly influence variant selection. Selection of "coinciding" ferrite variants which maintain near ideal OR with both neighboring PA grains leads to low ferrite–ferrite misorientations along these boundaries [23]. This selection regime causes difficulties in reconstruction, especially in high $\Sigma3$ fractions [20,23]. In order to achieve accurate reconstructions, such difficulties have to be investigated, understood and overcome.

Miyamoto et al. [33] improved the detection of austenite twins ($\Sigma3$) by using an average measured OR between retained austenite and ferrite. Germain et al. [28] calculated "in-common" variants in order to improve reconstruction by detecting austenite twins. Bernier et al. [35] developed an approach to combine OR refinement, pixel-to-pixel (misorientation) analysis, and nuclei identification/spreading strategy to achieve accurate reconstructions. Cayron [81] used average geometric ORs such as Greninger–Troiano (GT) to increase the reconstruction accuracy over the initial reconstructions which utilized Kurdjumov–Sachs (KS), Nishiyama–Wassermann (NW) and Pitsch relationships [26]. Abbasi et al. [20,23] proposed the combination of Bain zone detection in <001> pole figures, austenite orientation recovery, misorientation comparisons, as well as Image Quality (IQ) evaluations for accurate PA boundary detection.

Despite the improvements, there are still ambiguities regarding the nature of difficulties. The purpose of the current paper is to present some of these ambiguities and factors that affect reconstruction results. Reconstructions in different steels are presented along with details that will improve future automatic reconstructions.

2. Experimental procedure

Steel samples with different compositions, heat treatment, and deformation history were selected. For full reconstructions, high strength low alloy (HSLA) steel samples were used. The studied steel grades were: API X65, HSLA-65, API X80, and API L80. These samples were processed via friction stir processing [20]. Poly-crystalline cubic boron nitride (PCBN) tool was used [23]. Room temperature microstructures were examined via optical microscopy and EBSD.

There was no detectable retained austenite at room temperature to assist the reconstruction. Samples were mechanically polished to 1 μm surface roughness. Final polishing was carried out via 50 nm colloidal silica (nano-chemical–mechanical Buehler-VibroMet® polishing) and electropolishing (10% perchloric + 90% ethyl alcohol, 20 V, 30 s). Commercially available systems such as EDAX-TSL, Oxford-HKL, and Bruker-QUANTAX were used to collect room temperature EBSD datasets. Scans were converted to a text format for analysis with TSL-OIM software (version 7.0.1).

Analysis of successive layers was performed by removing layers with the thickness of 7 ± 1 μm from the surface via nano-chemical–mechanical polishing. A mixture of 50 nm colloidal silica and alumina with a 10 to 1 ratio was used.

In order to investigate the effect of strain, surface deformation was applied to polished and scanned surfaces. Mounted samples were placed on a polishing pad immersed in distilled water with no polishing agents. Vibratory–circular motion was applied with a gravitational pressure of 3.5 kN/m^2 on each sample for 6 hours. Rotational motion of nearly 1 rpm was recorded as a result of vibration. Micro-deformed surfaces were scanned with no further polishing.

Prior austenite reconstructions were performed via Bain zone detection in <001> ferrite pole figures [20,23]. Small areas containing few Bain zones were cropped and the zones were detected. Detected Bain zones were assigned to prior austenite grains. Austenite orientations were recovered for the reconstructed grains via a series of matrix operations [20]. KS OR was used for orientation recovery. However, the approach is able to utilize other ORs or their combinations. Image Quality (IQ) evaluations were performed to accurately detect PA boundaries.

3. Results and discussion

Reconstruction solely based on pixel-to-pixel ferrite–ferrite misorientation comparison would not provide accurate results. Distribution of ferrite–ferrite misorientation angles for ideally transformed <001> austenite orientation is plotted in Fig. 1. Two <001> austenite orientations were transformed to ferrite using KS OR. Misorientation between each resulting ferrite variant from one austenite was calculated relative to the 24 variants from the other austenite. Resulting 576 (24 × 24) misorientations cover angles as low as 0° (for similar variants) to 60°. More than 50% of the misorientations fall in the 45°–65° range.

Two ferrite orientations that do not belong to the same prior austenite grain (PAG) may well fall near one of the misorientations shown in Fig. 1. This will lead to dismissing