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The five-parameter grain boundary character and energy distributions of a fully austenitic high-manganese steel using three dimensional data

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Abstract

The three-dimensional interfacial grain boundary network in a fully austenitic high-manganese steel was studied as a function of all five macroscopic crystallographic parameters (i.e. lattice misorientation and grain boundary plane normal) using electron backscattering diffraction mapping in conjunction with focused ion beam serial sectioning. The relative grain boundary area and energy distributions were strongly influenced by both the grain boundary plane orientation and the lattice misorientation. Grain boundaries terminated by (111) plane orientations revealed relatively higher populations and lower energies compared with other boundaries. The most frequently observed grain boundaries were $\{111\}$ symmetric twist boundaries with the $\Sigma 3$ misorientation, which also had the lowest energy. On average, the relative areas of different grain boundary types were inversely correlated to their energies. A comparison between the current result and previously reported observations (e.g. high-purity Ni) revealed that polycrystals with the same atomic structure (e.g. face-centered cubic) have very similar grain boundary character and energy distributions. © 2014 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Grain boundary energy; Microstructure; TWIP steel; Electron backscattering diffraction; Focused ion beam

1. Introduction

A new generation of high-manganese steels has recently received significant attention among research groups worldwide as these steels offer an outstanding mechanical property balance (e.g. a high strength of ~ 1 GPa and high ductility of $\sim 60\%$ [1]). The unique combination of properties was attributed to the formation of nanosized (i.e. 20–50 nm) mechanical twins during deformation, which retards the onset of necking and consequently enhances both strength and ductility. This phenomenon is referred to as twinning-induced plasticity (TWIP). Mechanical twin

* Corresponding author. E-mail address: hossein.beladi@deakin.edu.au (H. Beladi). formation is common in austenitic materials with low stacking fault energy (SFE) in the range 20–50 mJ m $^{-2}$ [2].

Similar to other phase transition phenomena, the formation of mechanical twinning consists of nucleation and growth processes, which are both controlled by the dislocation substructure characteristics [3,4]. Kamaran et al. [5] argued that the activation of multiple slip systems and the existence of dislocation pile-ups are prerequisites for mechanical twin formation. Interestingly, mechanical twinning mostly nucleates at grain boundaries and subsequently propagates across the grain [1]. This is not surprising as multiple slip mostly takes place near grain boundaries to maintain the strain compatibility between adjacent grains [6,7]. It should be noted that grain boundary properties are anisotropic, depending upon the lattice

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misorientation and grain boundary plane orientation [8-12]. This suggests that the grain boundary character and energy distributions may contribute somewhat to the extent of mechanical twin formation in low-SFE austenitic materials.

To quantitatively characterize the grain boundary plane distribution and energy, five independent macroscopic crystallographic parameters are required: three for the lattice misorientation and two for the orientation of the boundary plane [12]. The latter needs advanced equipment to accurately resolve the three-dimensional internal microstructures of materials. Despite the complexity of the existing three-dimensional techniques, recent developments in automated microscopy, such as the dual-beam focused ion beam scanning electron microscope (i.e. serial sectioning), have made these measurements possible. Using this technique, the geometries of grain boundaries meeting at triple junctions can be accurately resolved [8-11]. This makes it possible to measure both the grain boundary character distribution (GBCD) and the grain boundary energy distribution (GBED) as a function of all five macroscopic crystallographic parameters [8-11]. This approach was recently employed to systematically analyze the relative area and energy of grain boundaries in a limited number of polycrystalline materials with different crystal structures such as MgO [8], yttria [9], Ni [10] and ferritic steel [11]. These measurements revealed that there is a strong inverse relationship between the relative population and energy of grain boundaries for all of these polycrystalline materials [8–11]. However, the crystal structure significantly influenced the extent and characteristics of the anisotropy observed in the GBCD and GBED [8-11].

In spite of the industrial significance of austenitic TWIP steels, there is hardly any information available regarding the relative population and energy of grain boundaries for this class of materials. Although the extent of research performed on the grain boundary energy in face-centered cubic (fcc) metals is comparatively greater than other crystal structures, these measurements were performed either over a limited range of the crystallographic parameters [13–27] or on high-SFE metals (e.g. Ni [10], ~128 mJ m⁻² [28]). The objective of the current paper is to present a comprehensive description of the grain boundary character and energy distributions in a fully austenitic high-manganese TWIP steel through the five-parameter grain boundary analysis approach using three-dimensional data obtained from serial sectioning combined with EBSD data.

2. Experimental procedure

2.1. Material

The steel composition in the current study was 0.6C-18Mn-1.5Al (wt.%). The SFE of this steel was reported elsewhere [29] to be ~ 25 mJ m⁻² at room temperature [2]. This ensures that mechanical twinning takes place during deformation. Therefore, the current composition

falls in the TWIP steel class. The experimental material was in the form of a sheet product with a thickness of ~1.5 mm supplied by POSCO Research Laboratory, Gwangyang, South Korea. The sheet was produced through ~60% cold rolling followed by a 1 min anneal at 800 °C, resulting in a fully recrystallized microstructure with an average grain size of ~2.5 μ m.

2.2. Three-dimensional EBSD measurement

A specimen $5 \text{ mm} \times 4 \text{ mm}$ was first cut from the sheet. It was then mechanically ground from both sides to produce a thin strip $\sim 150 \,\mu m$ thick with parallel surfaces. 3-D EBSD measurements and serial sectioning were performed on the rolling direction-normal direction (RD-ND) plane using a FEGSEM Quanta 3D FEI scanning electron microscope. The 3-D EBSD procedure and setup were discussed in detail elsewhere [11]. In brief, 200 nm of material was removed at each serial sectioning step using a 30 kV, 3 nA Ga⁺ ion beam. The ion-milling condition was carefully selected to minimize any surface damage (e.g. mechanical twinning and/or martensite phase transformation) that might occur through serial sectioning, specifically for low-SFE austenitic materials. EBSD mapping was performed using an electron beam with a voltage of 20 kV and a current of 4 nA. The in-plane point spacing (i.e. step size) of the EBSD scans was 150 nm. The current results were collected from one 3-D EBSD run consisting of 100 slices. The total volume of the 3-D EBSD run was $65 \,\mu\text{m} \times 40 \,\mu\text{m} \times 20 \,\mu\text{m}$, covering approximately 6300 grains.

2.3. Data processing

EBSD data were initially processed using functions in the TSL software to extract boundary line traces/segments, which were then employed to construct the triple lines, where triple points on adjacent layers were made up of crystals with the same orientation. Afterwards, the triple lines were used to calculate the relative grain boundary area and energy distributions using a five-parameter approach described in detail elsewhere [9]. The grain boundary energy calculation was performed through the capillarity vector reconstruction method demonstrated by Morawiec [30]. The current data set yielded $\sim 67,100$ triple lines. The grain boundary normal vector (i.e. grain boundary plane orientation) was calculated through the cross-product of the triple line connecting adjoining layers and the corresponding grain boundary line trace. This calculation yielded \sim 402,600 normal vectors.

The GBCD and GBED were analyzed discretely, with 9 bins per 90°, which offers 10° resolution. For this case, 97% of the bins contained 10 or more observations. The energy reconstruction was performed on the 67,100 triple junctions. An iterative approach was employed to reconstruct the capillarity vector, as described in Ref. [30]. In the present study, 300 iterations were used and the relaxation

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