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Dislocation density measurement by electron channeling contrast imaging in a scanning electron microscope

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We have measured the average dislocation density by electron channeling contrast imaging (ECCI) in a scanning electron microscope under controlled diffraction conditions in a Fe–3 wt.% Si alloy tensile deformed to a macroscopic stress of 500 MPa. Under optimal diffraction conditions, ECCI provides an average dislocation density close to that obtained by bright-field transmission electron microscopy. This result confirms that ECCI is a powerful technique for determining dislocation densities in deformed bulk metals.

Keywords: Dislocations; Low-temperature deformation; Electron diffraction; Scanning electron microscopy (SEM); Ferritic steels

The storage of dislocations during deformation of metals plays a key role in most metallurgical phenomena such as strain hardening, damage, creep, fatigue, athermal phase transformations, recrystallization and strain-induced grain boundary migration. In the former cases, it determines the mechanical properties such as strength and ductility. In the latter cases, it plays an important role in the mechanisms acting during annealing and transformation of deformed microstructures. In many cases, the average dislocation density is even linearly related to characteristic phenomena such as strengthening, creep rate, recovery and primary recrystallization. For this reason, the determination of the average dislocation density is important to better understand such phenomena. Dislocation density is commonly measured by direct methods such as transmission electron microscopy (TEM) [1–3] and indirect methods such as X-ray diffraction (XRD) [4,5]. TEM provides a highly accurate determination of the dislocation density provided that the dislocations can be clearly distinguished, i.e. it can be applied with high accuracy below a certain dislocation density ($\sim$5–10 $\times$ 10^{-14} m^{-2}). However, the determination of average dislocation density values in heterogeneous microstructures by TEM is time consuming owing to the demanding sample preparation technique involved. On the other hand, XRD provides an average dislocation density of the bulk deformed material in a shorter time but with a limited spatial resolution. In addition, XRD analysis of defect structures requires the use of a well-justified underlying model that connects a certain dislocation density and distribution with a total displacement gradient field.

An alternative microscopy technique for characterizing deformed microstructures is electron channeling contrast imaging (ECCI) [6–9]. ECCI is a scanning electron microscopy (SEM) technique that makes use of the fact that the backscattered electron intensity is strongly dependent on the orientation of the crystal lattice planes with respect to the incident electron beam due to the electron channeling mechanism. Slight local distortions in the crystal lattice due to dislocations cause a modulation of the backscattered electron intensity, allowing the defect to be imaged. The ECCI technique has been used to image dislocation structures in metals deformed during fatigue loading [10] or in the vicinity of cracks [11], and even stacking faults [12]. In particular, we have recently characterized complex mixed dislocation and twin substructures, as well as their individual contributions to strain hardening, on a highly deformed Fe–22 wt.% Mn–0.6 wt.% C alloy [13,14] by ECCI. For this purpose we used a novel ECCI set-up [9] which makes use of combined electron backscatter diffraction (EBSD) to image dislocations at enhanced contrast. In this paper, we demonstrate that the ECCI technique allows the determination of the average dislocation density of a deformed metal. The aim of the work is to establish ECCI

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as a very powerful, versatile, fast and experimentally robust method for determining dislocation defects densities and arrangements that involves a relatively simple preparation process. For this reason we used ECCI to measure the average dislocation density in a Fe–3 wt.% Si alloy tensile deformed to a macroscopic stress of 500 MPa. Under optimal diffraction conditions, the ECCI technique provides an average dislocation density close to that obtained by bright-field TEM.

The material selected was a Fe–3 wt.% Si alloy sheet 260 μm thick supplied by ThyssenKrupp Electrical Steel GmbH. The material has a large grain size in the centimeter range and a strong Goss texture {1 1 0}<0 0 1>. This microstructure makes the sample suited for the measurement of the dislocation density. Tensile tests were performed at room temperature at a strain rate of $2 \times 10^{-3}\ \mathrm{s}^{-1}$ to a macroscopic stress of 500 MPa. The mechanical tests were carried out using test instrument of Kammrath & Weiss GmbH (44141 Dortmund, Germany) equipped with a digital image correlation (DIC) system (ARAMIS system, GOM-Gesellschaft für Optische Messtechnik mbH, 38106 Braunschweig, Germany) for measuring the local strain. Details of this set-up are described in Ref. [15]. The surface pattern required for DIC was obtained as explained in Ref. [13]. Dislocation densities were measured in areas with a local strain of 0.05 by means of ECCI. A new recently reported set-up for ECCI [9] was used in this study to obtain ECCI images under controlled diffraction conditions which produced an enhanced dislocation contrast. This ECCI set-up has been successfully used in the imaging of dislocation substructures in Fe-based alloys [13,14]. The set-up uses EBSD patterns for calculating the optimal orientation of the crystal inspected through a specific combination of tilts and rotations. These are determined from the corresponding calculated diffraction pattern using the algorithm developed in Ref. [16]. ECCI observations were carried out in a Zeiss Crossbeam instrument (XB 1540, Carl Zeiss SMT AG, Germany) consisting of a Gemini-type field emission gun (FEG) electron column and a focused ion beam (FIB) device (Orsay Physics). ECCI was performed at 10 kV acceleration voltage and a working distance of 6 mm, using a solid-state four-quadrant backscattered electron detector. ECCI images were obtained with the sample normal aligned parallel to the incident electron beam.

It is well known that optimum dislocation contrast in ECCI is obtained by orienting the crystal to the exact Bragg angle, i.e. $s = 0$ where s is the deviation vector, in a two-beam condition [8,17]. Dislocation contrast becomes weaker under diffraction conditions deviating from the optimal, i.e. when $s \neq 0$. However, the latter, although not optimal, can be useful in the determination of the average dislocation density, as we show below. In the present work, we have evaluated the influence of diffraction condition in the determination of the average dislocation density. Dislocation densities were determined from ECC images that were taken under two different diffraction conditions, namely two-beam conditions with one set of *hkl* planes at the Bragg angle, and, alternatively, three-beam cases with two sets of *hkl* planes in an out-of-Bragg condition. For the first diffraction condition, dislocations appear as sharp bright lines over a dark background, whereas in the second diffraction condition, dislocations are visible as bright and dark sharp lines over a brighter background.

Figure 1 shows examples of ECC images of dislocation arrangements at the same area under different diffraction conditions with the corresponding calculated diffraction patterns. The ECC image of Figure 1a was obtained after orienting the crystal into Bragg condition using a high-intensity reflection of (1 1 0)-type. The calculated diffraction pattern is shown in Figure 1b. Under this diffraction condition, the crystal matrix appears dark and dislocations appear as sharp bright lines. The ECC image presented in Figure 1c was obtained after orienting the crystal out of the Bragg condition with $s < 0$. The calculated diffraction pattern is shown in Figure 1d. In this orientation, the crystal matrix appears brighter than in Bragg condition due to electron channeling mechanism [8]. In the first case, electrons are more effectively channeled into the lattice and the backscattering yield is low, leading to a dark appearance of the crystal. In the second case, the backscattering yield is enhanced and the crystal appears bright. In Figure 1c, we can identify dislocations appearing as white and black lines with uniform and sharp contrast. According to the dislocation contrast theory developed for TEM, the dislocation contrast exhibits an oscillatory black–white color with a periodicity of ξ_g, where ξ_g is the extinction distance [3]. Spencer et al. [17] showed that in ECCI, similar to TEM, the oscillatory behavior disappears for a dislocation that is located deeper than $2\xi_g$ from the sample surface, due to anomalous absorption phenomena related with inelastic scattering processes. According to the dislocation contrast profiles calculated by the authors [11], in a two-beam condition with one set of *hkl* planes at or close to Bragg orientation, dislocations exhibit a uniform white contrast. As in ECCI the $g.b = 0$ invisibility criterion holds [8,18,19], we can ascribe the bright sharp dislocations to those fulfilling $g.b \neq 0$ with g: 110, where b is the Burgers vector and g the diffraction vector. The origin of the

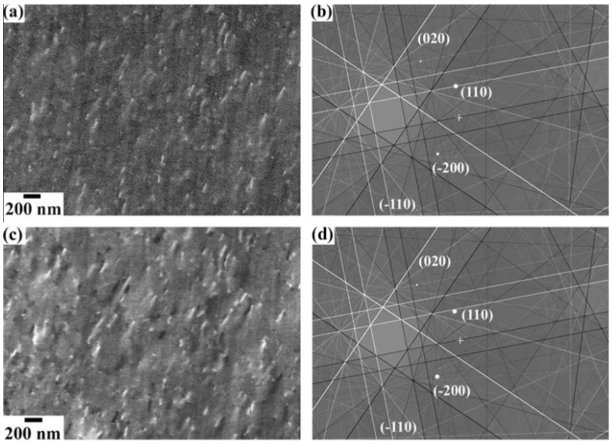

Figure 1. ECC images of dislocation arrangements of the same area in a Fe–3 wt.% Si alloy tensile deformed to a macroscopic stress of 500 MPa under two different diffraction conditions with the corresponding calculated diffraction pattern: (a and b) two-beam condition with g: 110 at Bragg orientation; (c and d) three-beam condition with g: 110 and g: −200 out-of-Bragg (g is the diffraction vector).