Materials Science and Engineering A 390 (2005) 159–165

MATERIALS SCIENCE & ENGINEERING A

www.elsevier.com/locate/msea

# Grain boundary statistics in nano-structured iron produced by high pressure torsion

Yu. Ivanisenko[a,*], R.Z. Valiev[b], H.-J. Fecht[a,c]

[a] *Forschungszentrum Karlsruhe in der Helmholtzgesellschaft, Institut für Nanotechnologie, Postfach 3640, 76021 Karlsruhe, Germany*
[b] *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K.Marks Str. 12, 450000 Ufa, Russia*
[c] *Division of Materials, Ulm University, Albert Einstein Allee 47, 89081 Ulm, Germany*

Received 21 April 2004; received in revised form 30 July 2004; accepted 11 August 2004

**Abstract**

The microstructure and the spectrum of grain boundary misorientations were studied in Armco iron, following high pressure torsion (HPT) deformation, by means of transmission electron microscopy (TEM) and orientation imaging microscopy (OIM). It was found that HPT deformation results in the formation of an equiaxed grain structure with a mean grain size of 270 and 130 nm using a shear strain of $\gamma = 210$ and 420, respectively. The misorientation spectra in HPT iron have a bimodal character with maxima in low (at 1–2°) as well as in high misorientation angle ranges. A marked increase in the fraction of special boundaries ($\Sigma3$–$\Sigma45$) was revealed as a result of HPT. The microstructural changes due to HPT are discussed and compared with those obtained during conventional deformation modes.


*Keywords:* Armco iron; High pressure torsion; Orientation imaging microscopy; Boundary misorientations

## 1. Introduction

Severe plastic deformation (SPD) is widely used now as a method to obtain an ultrafine grained (nano- and submicrocrystalline) structure in metals and alloys [1,2]. It was proposed [3,4] that storage and rearrangement of dislocations in cell walls can lead to their transformation to high angle grain boundaries (HAB) and therefore to the formation of the ultrafine grained structure. As a rule, the conclusion about the presence of high misorientations in the SDP-processed materials is based on the inspection of selected area electron diffraction (SAED) patterns. SAED patterns having many spots randomly distributed along the Debay–Scherrer rings are regarded as evidence for the formation of an ultrafine grained structure. Limitations of such a qualitative approach have been considered in [5,6] and it was suggested that quantitative investigations of the misorientation spectra are required. However, reliable estimates of grain boundary misorientations in severely deformed materials with a very small grain size by means of TEM are difficult and only a limited number of reports have been published on this subject [7–10].

The development of orientation imaging microscopy on the basis of modern FEG-SEM microscopes equipped with an electron backscattered diffraction (EBSD) setup allows for the inspection of the misorientations of large ensembles of grain boundaries (GBs). Recently, this technique was applied successfully to Al [11,12], Al-alloys [13,14], and Ni [15] processed by equal channel angular pressing (ECAP). For all materials investigated the HAB fraction was found to be between 60 and 70%. To study the misorientation spectra in materials subjected to high pressure torsion deformation is of special interest, because refinement of the microstructure in this case is usually much stronger as compared to ECAP and it is important to know whether the formed structure is granular, i.e. with high misorientations between the grains. Recently, it was confirmed in a study on HPT in deformed Ni

* Corresponding author. Tel.: +49 7247 82 6961; fax: +49 7247 82 5080.
*E-mail address:* ivanissenko@int.fzk.de (Yu. Ivanisenko).

0921-5093/$ – see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.msea.2004.08.071

[15], that the fraction of HAB is 85%. Obviously, such investigations should be continued for other materials. In this paper we report about OIM investigations of the grain boundary statistics in commercial purity iron after high pressure torsion deformation. This research can shed additional light on the question about the mechanisms of grain structure formation during severe plastic deformation.

## 2. Experimental procedures

Iron of 99.7 wt.% purity (Armco iron) was used for this investigation. The initial samples had a polyhedral grain structure with a mean size of 30 μm. Specimens were subjected to a HPT deformation under a pressure of 5 GPa as described elsewhere [4]. The shear strain $\gamma$ in the certain point of the sample is a function of the distance of this point from the sample center, $R$ as given by

$$\gamma = \frac{2\pi NR}{h}, \tag{1}$$

where $N = 5$ is the number of turns and $h = 0.3$ mm is the thickness of the sample. The microstructure of the HPT specimen formed after $N = 5$ turns at the radius of 2 and 4 mm was studied in the present investigation. The corresponding shear strain $\gamma$ estimated by Eq. (1) was 210 and 420, respectively. The structure of the HPT-processed specimen was studied in a FEI Tecnai F20 ST TEM operated at an accelerating voltage of 200 kV. The mean grain size was determined from bright and dark field images obtained with 50,000× magnification by a linear intercept method [16]. Samples were investigated by orientation imaging microscopy in a scanning electron microscope (FEG-SEM) LEO 1550 operated at 20 kV and INCA Crystal software (Oxford Instruments) was used for indexing the electron back scattering diffraction patterns. The scanning step size corresponds to a pixel size, which was 14 nm × 14 nm. The error in the determination of the crystallographic orientation of each grain was less than 2°. The analysis was performed for approximately 500 grains mapped in 3–5 OIM scans for each sample. Boundaries with misorientation angle less than 1° were neglected. The as-obtained OIM maps were subjected to a "cleaning" procedure, where single unsolved pixels were filled with the value from its nearest neighbours. A group of unsolved pixels is not changed in this procedure. This routine also changes a solved single pixel to the value of the majority of its neighbours if the pixel orientation is more than 3° (this takes care of a mis-solved pixel which is clearly within a grain of correct solved pixels). The average grain size (i.e. equivalent diameter) and grain size distribution were estimated from the "cleaned" grain maps, from which the amount of pixels corresponding to the areas with the same crystallographic orientation and surrounded by grain boundaries with a threshold value of 15° was counted.

## 3. Results

### 3.1. TEM observation

The microstructure formed at the lower shear strain level ($\gamma = 210$) is very inhomogeneous with respect to the grain morphology, grain size and dislocation distribution. Typically, areas with equiaxed grains (Fig. 1a) are observed; however, elongated structures are present as well (Fig. 1b). The elongated structures consist of grains arranged in conglomerates showing similar diffraction contrast. Usually, such conglomerates are just one grain in the width (Fig. 1b). Boundaries delineating grains are relatively wide that indicates a high dislocation density next to the boundary; however, often narrow boundaries can be seen as well. Narrow boundaries surround equiaxed grains. The mean distance between the boundaries was measured as 320 nm (Table 1) and in areas with elongated grains the mean grain width was about 270 nm and their length was 510 nm. Some larger grains (1–2 μm in diameter) are also present in the structure (Fig. 1a). Moreover, the distribution of dislocations in the grain interior is not uniform. There are grains with interior regions that contain several individual dislocations, and other grains with a developed dislocation substructure in the form of dislocation subboundaries, arrays and tangles (Fig. 1a and b).

Increasing the shear strain up to $\gamma = 420$ leads to the formation of a more uniform structure (Fig. 2a). Because of the complex diffraction contrast, due to the high level of internal stresses, it is difficult to reveal individual grains. On the dark field images (Fig. 2b) equiaxed grains with a mean grain size of about 140 nm can be seen (Table 1); however, some larger grains with sizes of about 200 nm are also present. The SAED patterns taken from 0.5 μm$^2$ areas (Fig. 2a) display rings that are composed of many individual spots.

### 3.2. Orientation imaging microscopy

The orientation imaging analysis was conducted for both specimen conditions ($\gamma = 210$ and 420) and the corresponding OIM maps are presented in Figs. 1c and 2c. Areas with different crystallographic orientation have different colours corresponding to the projection of the normal direction (ND) in the inverse pole figure (Fig. 1c). A comparison of the corresponding TEM and OIM images of the structures after a shear strain of $\gamma = 210$ (Fig. 1a–c), reveals that the conglomerates of grains observed in TEM (Fig. 1a) to be elongated in the shear direction correspond to the elongated areas that have a close crystallographic orientation (similar colouring in inverse pole

Table 1
Mean grain size in Armco iron after HPT

| Shear strain ($\gamma$) | Mean grain size (nm) | |
|---|---|---|
| | TEM | OIM |
| 210 | 140 ± 15 | 130 ± 15 |
| 420 | 320 ± 30 | 270 ± 30 |