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ScienceDirect

Acta Materialia 73 (2014) 12–18

Acta MATERIALIA

www.elsevier.com/locate/actamat

Mechanical behavior of a microsized pillar fabricated from ultrafine-grained ferrite evaluated by a microcompression test

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Received 2 September 2013; received in revised form 14 January 2014; accepted 23 March 2014
Available online 24 April 2014

Abstract

This paper studied the local mechanical properties of ferrite processed by high-pressure torsion (HPT). The evaluation was conducted by a microcompression test using a non-tapered microsized pillar as the specimen. Electron backscattering diffraction analysis was conducted to investigate the structural changes with different amounts of HPT strain. While the crystal orientation and grain size did not change with increasing strain in the HPT process beyond the structural saturation point, the grain boundary misorientation angle distribution tended towards a random distribution, which indicates dynamic recrystallization. The microcompression test revealed site-specific mechanical properties of ultrafine-grained ferrite. The strength was increased with decreasing grain size and reached 1.5 GPa, accompanied by a decrease in elongation. Further increasing the amount of HPT strain did not change the strength, but the elongation increased slightly. These improvements in the mechanical properties are discussed based on the nature of the grain boundary.

Keywords: Microcompression; Ultrafine-grained materials; Severe plastic deformation; Electron backscatter diffraction; Grain boundaries

1. Introduction

In recent years, fabrication of ultrafine-grained (UFG) metals and alloys using severe plastic deformation (SPD) processes such as high-pressure torsion (HPT) [1,2], equal-channel angular pressing [1,3] and accumulative roll bonding [4] have been attractive research topics. The unusual and extraordinary mechanical properties of UFG materials have attracted considerable interest from many researchers. These features are related to the grain boundaries, owing to the larger volume fraction of the grain boundary area and the interaction with mobile dislocations in the small grains of UFG materials. UFG materials fabricated by an SPD process have grain boundaries that contain an extensive number of defects, such as dislocations and vacancies, and are termed non-equilibrium grain boundaries [1,5–7]. The grain boundary characteristics (e.g. equilibrium, non-equilibrium, coincidence site lattice, misorientation angle) play an important role in deformation behavior [1,8,9].

In tensile tests of UFG materials, the yield drop phenomenon, Luder's band formation and shear banding, which are not observed in their conventional grain size counterparts, are observed [10]. Elongation decreases abruptly as the grain size decreases to 1 μm. In this grain size regime, necking can occur, accompanied by localized shear. Outside of the necked section, surface observation does not show any sign of deformation. This sudden drop in elongation is also observed in UFG interstitial-free steel and aluminum [11]. The lack of dislocation storage due to decreasing grain size is responsible for the notable decrease in work hardening capability. When the grain size becomes smaller than 1 μm, work hardening does not occur to maintain the shape

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http://dx.doi.org/10.1016/j.actamat.2014.03.048
1359-6454/

of deformed area, resulting in early failure. Because of this early failure, work hardening behavior cannot be observed in tensile tests. Therefore, compression testing is needed to investigate the deformation behavior across a wide range of strain. Observation of the deformation behavior in UFG materials with a wide range of strain is the main subject of this paper.

In this work, we used HPT to refine the grains of ultralow-carbon steel. The HPT process introduces a large amount of torsional strain to the periphery of disk-shaped materials. Materials subjected to such non-homogeneous deformation with a large strain or a large strain gradient have different crystallographic or structural features within the specimen [12]. Bulk size testing, which is widely used as a mechanical testing method, normalizes the effects of such differences on the mechanical properties. In contrast, the site-specific response of mechanical properties can be obtained using microsized test specimens fabricated from the area of interest using a focused ion beam (FIB). As is widely accepted, the strengths of materials are increased with decreasing dimensions to the micro- or submicroscale. Uchic et al. [13] reported that microcompression of 10 μm diameter samples displayed a mechanical response that matched the behavior of the bulk tension test. The present microcompression test was with 20 μm pillars, and was expected to show comparable mechanical properties to bulk counterparts. Micropillars fabricated from HPT-processed disks were evaluated by the compression test to investigate the site-specific mechanical properties of microstructures observed by electron backscattered diffraction pattern (EBSD) analysis.

2. Experimental procedures

The starting material of this work was ultralow-carbon steel (11C, <30Si, 20P, <3S <2B 8N, 14O, 300Al, <20Ti, <30Cr, <30Cu, in mass ppm). Homogenization treatment was conducted at 1273 K for 1 h in pure Ar atmosphere. After homogenization, material was cut and polished into disks of 10 mm diameter and 0.85 mm thickness, to subject them to the HPT process. Holding the disk with two anvils, materials were subjected to torsional strain for one or five rotations at a rotation speed of 0.2 rpm under a hydrostatic pressure of 5 GPa. The recorded processing temperature of the anvil increased by only a few degrees during the HPT process. Details of the HPT process have been described elsewhere [14,15].

For fabrication of the microsized compression pillar and investigation of the microstructures, HPT-processed disks were cut and polished mechanically, followed by electrolytic polishing using a solution of 900 ml of CH_3COOH + 100 ml of $HClO_4$. This etching process removed the deformation layer by cutting and mechanical polishing, and improved the qualities for subsequent orientation mapping. The samples were mounted on holders for ease of handling and to ensure alignment for the uniaxial compression test.

The microstructures and grain boundary characteristics of the electrolytically polished surface normal to the torsional axis were examined with a scanning electron microscope (SEM; S-4500SE, Hitachi) equipped with an electron backscatter diffraction pattern detector (INCA Crystal software, Oxford Instruments). The scanning step size, corresponding to the size of the pixels in the orientation map, was 30 nm. Compression pillars were fabricated by FIB from the region analyzed by the EBSD technique. Sequences of pillar fabrication are illustrated in Fig. 1, along with the corresponding scanning ion microscope (SIM) images. In the course of fabrication, we first made a pillar with the thickness of a thin plate (∼100 μm), as shown in Fig. 1a. Using irradiation at a 45° angle to the thin plate, we made a small pillar from the bigger one, as shown in Fig. 1b. Focusing an ion beam from the side of the specimen enabled us to fabricate a pillar with uniform dimensions (non-tapered, non-filleted). Finally, we milled each side of the pillar at a tilt angle of ±2.3° with a 400 pA ion beam to minimize the damage from ion bombardment. Ion irradiation from a grazing angle with an acceleration voltage of 30 kV causes a 100 nm layer of damage on the surface of a fabricated pillar [16]. However, the effect on the mechanical properties of pillars with a sample size of 20 μm fabricated from heavily strained metals will be negligible. The pillar had a square cross-section, 20 μm per side, and a height of 30 μm. The sample this had an aspect ratio of 1:1.5.

Compression tests were carried out at room temperature using a testing machine specifically designed for microsized specimens and assembled in our laboratory, the details of which are described in a previous paper [17]. This testing machine enables us to demonstrate single stroke compression of up to 32 μm displacement with a resolution of 0.2 nm, whereas other nanoindentation modules utilized in microcompression tests usually require multiple cycles of loading/unloading. A flat-ended diamond tip, 50 μm in diameter, attached to a piezoelectric loading device displaces the specimen. During compression, displacement of the tip was controlled at a constant rate of 0.1 $\mu m\ s^{-1}$, which result in a strain rate of 0.003 s^{-1}. The load–displacement data were recorded at a rate of 30 data points per second, with load resolution of <0.3% of the maximum load recorded in compression.

3. Results and discussions

3.1. EBSD analysis

The crystal orientations of the materials were determined using scanning electron microscopy/EBSD. Fig. 2 shows the orientation map of UFG ferrite obtained by EBSD analysis. The grains are slightly elongated in the shear direction in the upper part of the images, as indicated by the black arrow. Grain size is defined as the diameter of an equivalent circle having the same area surrounded by the grain boundary with a misorientation larger than 15°