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ScienceDirect

Acta Materialia 63 (2014) 16–29

Acta MATERIALIA

www.elsevier.com/locate/actamat

Concurrent microstructural evolution of ferrite and austenite in a duplex stainless steel processed by high-pressure torsion

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Received 16 August 2013; received in revised form 9 September 2013; accepted 18 September 2013
Available online 7 October 2013

Abstract

A duplex stainless steel with approximately equal volume fractions of ferrite and austenite was processed by high-pressure torsion. Nano-indentation, electron backscatter diffraction and transmission electron microscopy were used to investigate the hardness and microstructure evolutions of the steel. Despite the different strain-hardening rates of individual ferrite and austenite, the microstructures of the two phases evolved concurrently in such a way that the neighbouring two phases always maintained similar hardness. While the plastic deformation and grain refinement of ferrite occurred mainly via dislocation activities, the plastic deformation and grain refinement process of austenite were more complicated and included deformation twinning and de-twinning in coarse grains, grain refinement by twinning and dislocation–twin interactions, de-twinning in ultrafine grains and twin boundary subdivision.

Keywords: Steel; High-pressure torsion; Twinning; De-twinning; Grain refinement

1. Introduction

Bulk ultrafine-grained (UFG, grain size < 1 μm) and nanocrystalline (nc, grain size < 100 nm) materials have been extensively investigated because of their superior properties, including a combination of high strength and reasonably good ductility and improved corrosion resistance [1–5]. Among the many methods that have been developed to produce bulk UFG and nc materials [4,6–12], coarse grain refinement via severe plastic deformation (SPD) has been the most effective [7–12]. SPD-induced grain refinement processes usually involve complicated microstructure evolutions [7–9] that have significant impact on the mechanical behaviour of SPD-processed materials. In order to produce UFG and nc materials with desirable microstructures and properties, it is crucial to understand the SPD-induced microstructural evolution processes.

Extensive investigations have been carried out to understand SPD-induced microstructural evolutions of materials with a single phase or with minor amounts of secondary phases/precipitates [13–17]. There are basically two types of SPD-induced microstructural evolution and grain refinement. The first type primarily involves dislocation activities that include dislocation accumulation, interaction, tangling and spatial rearrangement that subdivide a large grain into many smaller ones with the formation of dislocation cells and the evolution of cell boundaries into low-angle and then high-angle grain boundaries (GBs) [14–17]. This type of SPD-induced grain refinement has been observed in body-centred cubic (bcc) materials such as ferritic steel [16] and face-centred

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1359-6454/$36.00
http://dx.doi.org/10.1016/j.actamat.2013.09.030

cubic (fcc) materials with high stacking fault (SF) energies such as nickel [18] and aluminum alloys [19]. The second type of SPD-induced grain refinement is dominated by deformation twinning or twin boundary (TB) subdivision and TB–dislocation interactions and this has been widely reported in fcc materials with low SF energies, e.g. twinning-induced plasticity (TWIP) steels [20,21], Cu–Zn alloys [22] and Cu–Al alloys [23,24]. Wang et al. [22] reported that, for the second type of grain refinement, primary and secondary twinning subdivides large fcc grains into small ones and the interactions between dislocations and TBs transform the TBs into conventional high-angle GBs. The minimum achievable grain sizes are determined by the minimum TB spacing of the materials [22]. It has been known that the average TB spacing in a severely deformed material is a function of the SF energy of the material [25]. It is possible to produce TB spacings of smaller than 2 nm in a material with a very low SF energy [26]. However, there has been no report of SPD-induced grain refinement down to smaller than 10 nm, indicating that the grain refinement process in fcc materials with very low SF energies must be more complicated than reported by Wang et al. for a Cu–Zn alloy [22].

It is expected that different single-phase materials have different strengths and strain-hardening behaviours due to factors that include the differences in crystal structure [27,28] and the elemental content [27,29]. For a dual-phase material, there must be a mechanism to coordinate the SPD-induced grain refinement processes of the two phases due to their mutual constraint. Our previous research of high-pressure torsion (HPT) processing of a duplex stainless steel [30], which comprises roughly the same volume fractions of bcc ferrite and fcc austenite, showed that the two phases had concurrent hardness evolutions so that the hardness difference between the two phases was maintained at a minimum at every stage of HPT deformation although the strain-hardening rates of ferrite and austenite are very different [27,31,32]. Previous investigations of other dual-phase materials including a Zn–22% Al eutectoid alloy [33], a Fe–27% Cr–9% Ni steel [34], and a Cu–Ag alloy [35] demonstrated that the microstructures of different phases evolve in different ways to accommodate the concurrent hardness evolutions. However, the mechanism for achieving these concurrent microstructural evolutions is not yet established.

Accordingly, this study reports a systematic investigation of the evolution of the microstructures and hardnesses of the two phases in a duplex stainless steel during HPT processing. This steel has equal volume fractions of the ferrite and austenite phases and the austenite phase deforms with extensive twinning under HPT processing [36], giving deformation behaviour which is similar to TWIP steels [20,37]. The present study provides information on the concurrent evolution of the two phases in a dual-phase material, it describes the method of achieving the final grain size in fcc materials with extremely small TB and it also provides an insight into the TWIP behaviour occurring during SPD processing.

2. Experimental procedures

The material used in this investigation was a commercial DP3W duplex stainless steel in the form of rectangular plates. The material had approximately equal volume fractions of the bcc ferrite (α) phase and the fcc austenite (γ) phase. A summary of the compositions of the material and the two phases in the material is listed in Table 1. The overall composition of the material was provided by the supplier and the compositions of the two phases in the material were measured using energy-dispersive X-ray spectroscopy (EDX) in a scanning electron microscope (SEM). Because of the inaccuracy in the quantitative measurement of light elements using EDX and because of the small amounts of these light elements, the measured compositions of light elements are not included in the table.

The steel plates were firstly cut into disks with a diameter of ~9.8 mm and a thickness of ~1.7 mm. The disks were then ground using 800 and 1200 grit sand papers sequentially to obtain a smooth surface and a uniform thickness of ~0.8 mm for HPT processing. The processing was conducted under quasi-constrained conditions [38,39] using an applied pressure of 6.0 GPa and with the disks processed through ¼, 1, 2 and 5 revolutions. Slippage was not detected during the HPT processing.

Samples for SEM characterization and nano-indentation testing were mechanically polished using 1200 grit sand papers, 6 μm and 1 μm diamond papers and 0.4 μm colloidal silica suspensions sequentially to acquire a flat and smooth surface. Electro-polishing was conducted to remove the strained layer caused by mechanical polishing using a Struers LectroPol-5 unit and an electrolyte of 23% perchloric acid and 77% acetic acid under the voltage of 20 V at a temperature of ~20 °C for 50 s. Samples for transmission electron microscopy (TEM) characterization were prepared by grinding HPT disks to a thickness of ~60 μm, cutting the disks into small strip-pieces of 2 mm × 3 mm with the width of the strips along a radius direction of the disks, and then electropolishing using a Struers TenuPol-5 jet electropolishing unit and a solution of 23% perchloric acid and 77% acetic acid under an operating voltage of 20 V and at a temperature of ~20 °C until a small hole, surrounded by thin areas, was formed on the strips. Each strip was then glued to a TEM grid with a diameter of 3 mm. The location of each strip relative to the disk centre was recorded in order to estimate the HPT strain value.

An IBIS nanoindentation system was used to obtain the hardness values of samples at different strains using a Berkovich indenter tip under the force control mode with a maximum load of 28 mN. For each disk sample, nanoindentation was conducted along two mutually perpendicu-