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Microstructure and mechanical response of single-crystalline high-manganese austenitic steels under high-pressure torsion: The effect of stacking-fault energy

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ABSTRACT

We investigate the kinetics of the structural deformation and hardening of single-crystalline austenitic Fe–13Mn–1.3C (Hadfield steel), Fe–13Mn–2.7Al–1.3C, and Fe–28Mn–2.7Al–1.3C (in wt%) steels with different stacking-fault energies after cold high-pressure torsion. Independently of the stacking-fault energy, mechanical twinning was found to be the basic deformation mechanism responsible for the rapid generation of an ultrafine-grained microstructure with a high volume fraction of twin boundaries. Under high-pressure torsion, the spacing between twin boundaries increases, and the dislocation density and microhardness decrease as the stacking-fault energy increases. The formation of a twin net from the beginning of plastic flow in Fe–13Mn–1.3C steel provides a homogeneous distribution of microhardness values across the discs independent of strain under torsion. Lower hardness values in the disk centers compared to the periphery were observed for the two other steels, Fe–13Mn–2.7Al–1.3C and Fe–28Mn–2.7Al–1.3C, with higher stacking-fault energies due to changes in the densities of the twin boundaries. An additional increase in the dislocation density for the Fe–13Mn–1.3C steel was detected compared with the Fe–13Mn–2.7Al–1.3C and Fe–28Mn–2.7Al–1.3C steels, which was a result of torsion in the temperature range of dynamic strain aging. The appearance of small fractions of ε and α′ phases in the structures of the Fe–13Mn–1.3C, Fe–13Mn–2.7Al–1.3C, and Fe–28Mn–2.7Al–1.3C steels is discussed.

1. Introduction

In recent decades, the physical studies of strength and plasticity have been directed at developing methods for achieving high-strength states in metals by refinement of their structures under severe plastic deformation (SPD) [1]. Progress in this area is not only associated with the creation of new deformation schemes but also, to a large extent, with the modification and combination of known methods, the optimization of the composition and structure of alloys prior to SPD and with grain-boundary engineering.

High-pressure torsion (HPT) refers to the processing technique in which samples in the form of thin disks are subjected to simultaneous high compressive stress and torsion [1,2]. Using this method, nanocrystalline structural states are typically formed in various metals and alloys [2]. The obvious advantage of this SPD method is the ability to realize unlimited strains, which are often not achievable in conventional static tests, while maintaining the integrity of the workpiece and its shape (volume). The principles of the HPT method are described in detail in [2].

The HPT method has successfully been used to deform Armco iron [3,4] and steels [5–12]. HPT of twinning-assisted austenitic steels was not studied in detail [5,6,12]. However, mechanical twinning can be one of the factors that contribute to the rapid formation of ultrafine-grained structures with low-energy, high-angle special boundaries $\Sigma 3^n$ [13]. At temperatures that do not allow recrystallization, severe plastic deformation typically causes degradation of the dislocation structure by forming bands of localized deformation. The development of fine deformation twins in steels with a high concentration of interstitial atoms hinders this process because the deformation twins are more resistant to "disintegrating" compared with general boundaries.

Some authors found deformation twins in nanocrystalline face-centered cubic (fcc) metals and alloys (with grain sizes of a few tens of nanometers), such as Cu, Al, Ni, and Cu–Zn, due to the emission of partial dislocations from the grain boundaries, overlapping of stacking-fault ribbons, grain boundary splitting, etc. [14,15]. The volume fraction of twin boundaries formed in

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nanostructured materials is not considerable; it contributes but does not define the hardening of such materials.

Forming a structural state with the maximum possible fraction of special $\Sigma 3^n$ boundaries is not a trivial task because twinning in fcc alloys is largely complementary to the slip deformation mechanism and is generally observed in the low-temperature deformation regime [16]. In contrast, SPD is often conducted at elevated temperatures. Therefore, the deformation of alloys prone to high-temperature twinning offers the potential to create high-strength nanostructured materials with low-energy high-angle misorientations between the structural elements and that are resistant to degradation during annealing or subsequent deformation [17].

For AISI 304L stainless steel processed by equal channel angular pressing in the temperature range of 500–900 °C, Huang and coauthors [18] observed deformation twins in addition to deformation bands and subgrains (many bundles of deformation twins was observed at temperatures below 700 °C). Karaman et al. [19] reported the development of high-temperature twinning (up to 800 °C) for some difficult-to-work alloys, including 316L austenitic steel. They concluded that deformation twinning could be one of the main deformation modes in many metallic alloys over a wide temperature range when a high strength level is reached, irrespective of the way it is achieved [19]. As promising materials for the development of SPD methods, high-manganese austenitic steels with low stacking-fault energies (Hadfield steel-based) can be used. The high strength levels are reached in these steels due to solid solution hardening (high carbon content). Twinning in single- and polycrystalline Fe–(11–13)Mn–(1.0–1.3)C and Fe–13Mn–(1.3–2.7)Al–(1.3–1.75)C steels develops under uniaxial tension/compression at room temperature [20–25]. Under torsional deformation and high applied pressure, twinning had a significant contribution to the refinement and strengthening of the Hadfield steels Fe–13Mn–1.0C [6] and Fe–13Mn–1.2C [5].

The aim of this study is to characterize the microstructure and strength properties (microhardness) after the high-pressure torsion of single crystalline twinning-assisted Fe–13Mn–1.3C (Hadfield steel), Fe–13Mn–2.7Al–1.3C, and Fe–28Mn–2.7Al–1.3C steels. The choice of such steel compositions allows one to change the stacking-fault energies by alloying Hadfield steel with manganese and aluminum while maintaining a high level of solid solution hardening. The use of single crystals allows for a detailed study of the strain hardening mechanisms and avoids the added complexity of the contribution from grain boundaries.

2. Experimental

The high-Mn steels used in this study had the following chemical compositions: Fe–13Mn–1.0C, Fe–13Mn–1.3C (Hadfield steel), Fe–13Mn–2.7Al–1.3C, and Fe–28Mn–2.7Al–1.3C (in wt%). Single crystals of the high-manganese austenitic steels were grown using the Bridgman technique under an inert gas atmosphere. All crystals were homogenized in an argon atmosphere at 1373 K for 24 h, solution-treated and water-quenched from 1373 K after 1 h. Electro-discharge machining was utilized to cut flat disks of 10 mm (diameter) × 0.6 mm (height) for high-pressure torsion. The surfaces of the disks were cut parallel to the {001}-type planes for the Fe–13Mn–1.0C, Fe–13Mn–1.3C, and Fe–13Mn–2.7Al–1.3C steels and parallel to the {111}-type planes for the Fe–28Mn–2.7Al–1.3C steel. The stacking-fault energies (SFE) for the investigated steels were assumed to be 25–30 mJ/m^2 for Fe–13Mn–1.0C and Fe–13Mn–1.3C, 45 mJ/m^2 for Fe–13Mn–2.7Al–1.3C, and 60 mJ/m^2 for Fe–28Mn–2.7Al–1.3C according to [26].

The unconstrained high-pressure torsion was conducted at room temperature (p=5–6 GPa) for one to five revolutions at a rotation speed of 1 rpm. The true logarithmic strain was calculated using the equation $\varepsilon=\ln(\upsilon r/h)$ [2], where r is the radius, υ is the rotation angle, and h is the thickness of the disk after high-pressure torsion.

Mechanical grinding and a final electrochemical polishing (50 g of CrO_3 in 200 ml of H_3PO_4) were employed to remove all preparation-induced surface artifacts. The evolution of the microstructure was studied by repolishing and etching the specimens that were strained to different degrees. Chemical etching of the polished disk surfaces was conducted in a solution of 1 ml of HCl in 99 ml of H_2O. Optical microscopy (OM) was performed using an Olympus GX-71 microscope.

Electron-transparent foils were prepared by conventional electropolishing in a solution of 50 g CrO_3+200 ml H_3PO_4 at an applied potential of 30 V. For microstructural analysis, transmission electron microscopy (TEM) was performed using a Philips CM200 transmission electron microscope. The foils for TEM were cut with the observation area located at half of the radius of the disks after high-pressure torsion. The average sizes of the microstructural elements (subgrains, twins) were determined using dark-field TEM images [27]. Selected area electron diffraction (SAED) patterns from areas of 0.5 and 12.6 μm^2 were evaluated to identify the different phases. The density of twin boundaries was estimated as $\rho_{tw}=q/S$, where q is the number of twin boundaries and S is the area of the TEM image.

A Shimadzu XRD-6000 X-ray diffractometer with Cu-K_α radiation was utilized for the X-ray diffraction studies. The scalar dislocation density was determined by X-ray diffraction (XRD) using the method described in [28].

The microhardness was determined at room temperature using a Duramin 5 instrument with a load of 200 g.

3. Results

3.1. Microstructural observations

High-pressure torsion leads to the fragmentation of single crystals of Fe–13Mn–1.3C, Fe–13Mn–2.7Al–1.3C, and Fe–28Mn–2.7Al–1.3C mainly due to the formation of a high dislocation density, twins and localized deformation bands, which were detected by optical metallography (Fig. 1) and under TEM studies (Figs. 2–4). Analysis of the crystal surface after repolishing and etching shows that there are several twinning systems and that the twins and shear bands are internally twinned (Fig. 1). The tendency for plastic flow to localize increases as the stacking-fault energy increases (Fig. 1). In the Fe–13Mn–1.3C steel, the localization processes are suppressed because, from the beginning of deformation, a twin net forms with a spacing between the boundaries of a few nanometers. In single crystals of the Fe–28Mn–2.7Al–1.3C steel, localized shear bands were formed prior to or simultaneously with the development of deformation twinning (Fig. 1c).

In the Fe–13Mn–1.3C steel at N=1 revolutions, there is a formation of twin packs with a micron width and shear bands (SB) (Fig. 1a). Using TEM, we observed twins in which the thicknesses of the plates were tens of nanometers (Fig. 2a, b). The effective size of the structural fragments is determined from the thickness of the deformation twins and the distance between the twin boundaries, which is 5–15 nm. With an increase in strain (N=3) in the Fe–13Mn–1.3C steel, the twin net remains; however, in the TEM and optical images, deformation and a large number of microbands were detected (Figs. 1d and 2c, d). Twins and shear bands with the fragment widths of ~0.5 mm are filled with deformation twins with a thickness of 5–10 nm (Fig. 2c). Fragments within such a twin net have a high density of slip