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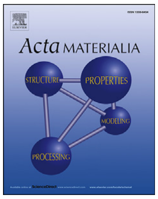

Full length article

Evolution of dislocations and twins in a strong and ductile nanotwinned steel

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A twinning-induced plasticity (TWIP) steel was subjected to a simple processing route (i.e. cold rolling followed by a recovery heat treatment) suitable for large-scale industrial production, resulting in the production of a strong and ductile nanotwinned steel. This nanotwinned steel combines high yield strength (1450 MPa), high ultimate tensile strength (1600 MPa) and good ductility (25% total elongation). Detailed transmission electron microscopy observation reveals that the twin volume fraction of the nanotwinned steel remains constant during tensile deformation. This is different to the deformation behaviour of recrystallized TWIP steels whose twin volume fraction increase continuously with strain during tensile deformation. The constant twin volume fraction indicates that a maximum twin volume fraction has been reached during the cold rolling process. In contrast, the dislocation density of the nanotwinned steel increases with strain as measured by the synchrotron X-ray diffraction experiments. In other words, the plastic deformation of the nanotwinned steel is mainly accommodated by glide and multiplication of dislocations. Based on the experimental results, an analytical model was developed to capture the respective effects of dislocations and twins on the strength and ductility of the present nanotwinned steel. The modelling results indicate that the strength is contributed by both twins and dislocations while the ductility is mainly attributed to dislocation multiplication.

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1. Introduction

The strength of a metallic material increases with the decrease of grain size as the grain boundary is a barrier for dislocation glide. The dependence of strength on grain size can be described by the well-known Hall–Petch law [1,2]. As a result, reducing grain size to the nanometre scale has recently received great attention. For instance, severe plastic deformation (SPD) such as high pressure torsion (HPT), equal-channel angular pressing (ECAP) and accumulated roll-bonding (ARB) have all been applied recently to produce bulk nanostructured metals with grain sizes ranging from tens of nanometres to sub-micrometre, resulting in ultrahigh strength [3–9]. Besides grain boundaries, coherent twin boundaries can also be effective obstacles to dislocation motion. Thus, this can be employed as complementary strengthening mechanism for metals. Lu and co-workers [10–13] recently demonstrated that Cu containing intensive nanoscale coherent twins, namely nanotwinned Cu, exhibits very high strength. The strength of nanotwinned Cu increases with the decrease of twin spacing, following a similar Hall-Petch relationship. Besides ultrahigh strength, nanotwinned Cu also possesses high ductility.

Besides nanotwinned Cu, it is interesting to introduce nanotwins in other engineering alloys with the aim to achieve simultaneously high strength and high ductility for structural application. For instance, nanotwins were introduced in a twinning-induced plasticity (TWIP) steel by torsion so that the yield strength of the TWIP steel was doubled at no reduction in ductility [14]. Nanotwins can also be introduced in steels by other methods such as surface mechanical grinding treatment [15] and dynamic plastic deformation [16]. More interestingly, a simple method (i.e. cold rolling followed by a recovery heat treatment) has been proven to be an effective way to introduce intensive nanotwins in steels and it was applied to twinning-induced plasticity (TWIP) and 316 L stainless steels [17–21]. Bouaziz et al. [17,20] demonstrated that introducing nanotwins in these two alloys can significantly increase their yield stresses while retaining substantial tensile uniform elongation. It is worth noting

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that cold rolling followed by a recovery heat treatment is well suitable for large-scale industrial production in the current steel industry, making the nanotwinned steel a realistic structural material for automotive application [21].

The optimization of the process including cold rolling and annealing has been obtained by some studies [18,22], which demonstrate that an excellent combination of high yield strength and good ductility in the nanotwinned steel can be achieved [22]. Nevertheless, the deformation mechanism of the nanotwinned steel produced by cold rolling followed by a recovery treatment, especially the evolution of dislocations and twins during plastic deformation, has not yet been investigated. In the present work, the evolution of twins and dislocations was investigated by detailed transmission electron microscopy (TEM) and synchrotron X-ray diffraction (XRD) experiments. The evolution of dislocations and twins and their respective effects on the strength and ductility were further quantitatively evaluated with a physically-based model. The systematic experimental and modelling works provide a better fundamental understanding in the deformation mechanism of the nanotwinned steel.

2. Experiments

A fully recrystallized TWIP steel with an average grain size of 4 μm was employed to produce the nanotwinned steel in the present study. The chemical composition was listed in Table 1. The TWIP steel was firstly subjected to 50% rolling reduction at room temperature followed by a recovery heat treatment at 773 K for 15 min. Intensive nanotwins were induced by the cold rolling. During the recovery heat treatment, nanotwins were thermally stable [23,24] and only dislocation density was reduced by the recovery treatment [17,20,25]. The nanotwinned steel was therefore produced by such cold rolling and recovery heat treatment in the present study. The tensile specimens in dog-bone shape were fabricated from the nanotwinned steel sheet along the rolling direction with gauge dimensions of $12 \times 5 \times 2$ mm^3. The uniaxial tensile tests were performed on a universal testing machine at room temperature with a nominal strain rate of 10^{-3} s^{-1}. Interrupted tensile tests were conducted in order to study the evolution of dislocations and twins. The samples for nanoindentation tests were mechanically ground and polished down to 1 μm. The surface was finally vibration polished (solution: colloidal silica polishing suspension with a particle size of 0.06 μm produced by Buehler). The nanoindentation tests were carried out in an Agilent Nano indenter G200 equipped with a Berkovich indenter at ambient temperature. A peak load of 5 mN was applied with a loading time of 10 s. Three matrices of indentations (each contains 6×6 indents with a spacing of 5 μm) were performed on each sample. Microstructural characterization was carried out by XRD and electron microscopies. Normal XRD was carried out with a Cu Kα radiation with a wavelength of 1.5405(6) Å for phase analysis. Synchrotron XRD experiments were carried out in reflection mode at the beamline BL14B1 in the Shanghai Synchrotron Radiation Facility. The energy of the monochromatic X-ray beam was 18 keV, corresponding to a wavelength of 0.68879 Å. The beam size is 0.5×1 mm^2. The diffraction pattern of each sample was recorded with a step size of 0.01° and a dwell time of 0.5 s. In order to minimize the effect of strain layer, the surface of the sample for XRD experiment was finally vibration polished. The electron backscatter diffraction (EBSD) mappings were performed in a Leo 1530 scanning electron microscope (SEM) operated at 20 kV. In order to increase the resolution of EBSD for characterizing the nanoscale twins, the transmission EBSD (t-EBSD) technique [26,27] was applied and the corresponding TEM samples were used. TEM specimens were prepared from the gauge parts of the tensile specimens. After mechanically grinding down to a thickness of ~100 μm, disks with a diameter of 3 mm were punched out from the thin foils. The disks were then subjected to a twin-jet electrochemical polishing in a solution of 5% perchloric acid +95% ethanol at 30 V and a temperature of around 243 K [28,29]. TEM observation was carried out in an FEI Tecnai G2 20 S-TWIN scanning transmission electron microscopy operated at 200 kV.

Table 1
The chemical composition of the present nanotwinned steel.

Elements	Mn	C	Al	Si	S	P	Fe
wt.%	17.59	0.75	1.7	0.52	<0.005	0.014	Bal.

3. Experimental results

Fig. 1 displays the microstructure of the nanotwinned steel at different magnification. As shown by the SEM image in Fig. 1a, numerous bundles of lamellae in bright contrast can be observed within the elongated grains in a large area. Some boundaries of these lamellae can be identified by EBSD as the Σ3 twin boundaries as delineated by the red lines in the band contrast image in Fig. 1b. Therefore, the numerous lamellae observed in Fig. 1a should be bundles of deformation twins, yet, EBSD cannot index all of them due to the resolution limit. TEM observation was carried out in order to investigate the details of the nanotwin structure. As shown by the bright field image in Fig. 1c, a large number of nanotwins confirmed by the selected area diffraction pattern can be found with a thickness generally smaller than 10 nm. These nanoscale twins group into parallel bundles, which traverse across the entire grains. Careful TEM observation reveals that single twinning system is favoured within a single grain after cold rolling consistent with the study of K. Renard et al. [30].

The engineering stress–strain curve of the nanotwinned steel is shown in Fig. 2a. The yield strength (YS) is as high as 1450 MPa, and the ultimate tensile strength (UTS) is 1600 MPa. The nanotwinned steel also exhibits considerable ductility with a total elongation of 25%. As displayed in Fig. 2b, the work-hardening rate ($d\sigma/d\varepsilon$, where σ and ε are the true stress and true strain, respectively) is high at the beginning of plastic deformation, however, decreases steeply. Necking took place at a true strain of ~13.5% when the work-hardening rate was equal to the true stress.

Depending on the chemical composition and deformation temperature, there are three possible deformation mechanisms including martensitic transformation, deformation twinning and dislocation glide in the present TWIP steel. In Fig. 3, the XRD patterns show that the nanotwinned steel remains a single austenite phase before and after the tensile test. The TEM bright field images of the nanotwinned steel before and after the tensile test are displayed in Fig. 4. Numerous deformation twins can be observed in the nanotwinned steel before and after tensile test as shown in Fig. 4a and b, respectively. No substantial difference in the twin volume fraction is discernible between the initial and fractured nanotwinned steels. High density of dislocations can also be found in the microstructure before and after tensile test as shown in Fig. 4. However, no obvious difference in dislocation density can be distinguished by TEM. Based on the results in Fig. 4, direct measurement of dislocation density by TEM observation is not applicable because it is very difficult to count the number of dislocations in a certain area in the TEM when the dislocation density is very high.

In order to quantitatively evaluate the evolution of twin volume fraction during plastic deformation following prior cold rolling and recovery annealing, detailed TEM observation was carried out to

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