Materials Science & Engineering A 631 (2015) 181–188

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Micromechanical behavior of eutectoid steel quantified by an analytical model calibrated by in situ synchrotron-based X-ray diffraction

Chengsi Zheng [a], Longfei Li [a,b,*], Yandong Wang [a,b], Wangyue Yang [c], Zuqing Sun [a]

[a] State Key Laboratory for Advanced Metals and Materials, University of Science & Technology Beijing, Beijing 100083, China
[b] The Collaborative Innovation Center of Steel Technology (CICST), University of Science and Technology Beijing, Beijing 100083, China
[c] School of Materials Science and Engineering, University of Science & Technology Beijing, Beijing 100083, China

ARTICLE INFO

Article history:
Received 15 August 2014
Received in revised form
5 February 2015
Accepted 5 February 2015
Available online 13 February 2015

Keywords:
Eutectoid steel with $\alpha+\theta$ structure
Dislocation density
Analytical modeling
Micromechanical behavior
Work-hardening

ABSTRACT

A eutectoid steel with three types of ferrite (α)+cementite particle (θ) microstructures, i.e., a coarse-grained $\alpha+\theta$ structure, a fine-grained $\alpha+\theta$ structure and an ultrafine-grained $\alpha+\theta$ structure, was fabricated to explore the effects of the microstructural features on the micromechanical behavior of hard particle-strengthened two-phase alloys. An analytical model based on the Kocks–Mecking model was established to elucidate the evolution of the geometrically necessary dislocations (GNDs) and statistically stored dislocations (SSDs) in the hard particle-strengthened alloys and, hence, to predict the stress partitioning for each phase and the enhancement in the work hardening during uniform plastic deformation. In situ synchrotron-based X-ray diffraction was used to verify the stress partitioning and the important material parameters predicted by our analytical model. Our results showed that a decrease in the geometric slip distance leads to an appreciable increase in the GND density, whereas an increase in the grain size of the ferrite causes an increase in the SSD density under uniform plastic deformation for eutectoid steel with an $\alpha+\theta$ structure. Both the stresses for the individual phase and the difference in stress between the two phases for eutectoid steel with various $\alpha+\theta$ structures were closely related to the change in the GND density near the phase interfaces. The GND density also played an important role in determining the work-hardening rate for eutectoid steel with various $\alpha+\theta$ structures.

1. Introduction

One of the important metallurgical strategies for simultaneously enhancing both the strength and plasticity of metals is controlling the volume and distribution of precipitated hard particles in a soft matrix. In the steel industry, various thermomechanical treatments have been used to fabricate a variety of ultrafine-grained microstructures consisting of an ultrafine-grained ferrite (bcc-α) matrix and ceramic particles (θ-Fe_3C, cementite) [1–10], which provide an effective way of improving the work-hardening capability and the strength plasticity of ultrafine-grained ferritic steel [11–13]. Compared with those of single-phase ferritic steel with a grain size of ≈ 1 μm, the ultimate strength and uniform elongation of ultrafine-grained $\alpha+\theta$ steel with a similar grain size can be improved from approximately 540–735 MPa and from 1% to 11%, respectively, and the yield-to-ultimate stress ratio can be reduced from approximately 1 to 0.86 [4,12]. Moreover, ultrafine-grained $\alpha+\theta$ steels with relatively simple chemical compositions [1–10] exhibit higher strengths and lower ductile-to-brittle transition temperatures (DBTTs) than does conventional steel, indicating their potential as advanced structural materials for replacing high-strength low-alloyed steels (HSLAs) [10]. Recently, this strategy was also implemented by generating nanoscale precipitates distributed homogeneously in nanostructured or ultrafine-grained metals, such as nanostructured aluminum alloys [14] and ultrafine-grained molybdenum alloys [15], to realize an enhancement in plasticity and ductility by increasing dislocation accumulation and ultimately to overcome the intrinsic brittleness of these high-strength alloys. Although the two mechanisms for precipitation hardening, i.e., dislocation cutting and Orowan bowing, are completely described by two well-known equations [16,17], a quantitative understanding of the enhancement of plasticity is still lacking, particularly how the volume, size, and distribution of hard particles affect the work-hardening rate and the homogeneous deformation strain. In addition, to date, there is no open literature on the effect of the combination of particle strengthening and variation in the grain size of the matrix on the strength and plasticity of structured materials, which should be particularly important for designing next-generation, advanced, high-strength steels with multi-scale microstructures.

To date, experimental studies generating tensile stress–strain (S–S) curves have demonstrated that steel with a fine-grained ferrite matrix and refined cementite particles, i.e., fine-grained $\alpha+\theta$ steel, possesses a better balance between work-hardening capability and

* Corresponding author. Tel.: +86 10 62334862; fax: +86 10 62333447.
E-mail address: lilf@skl.ustb.edu.cn (L. Li).

http://dx.doi.org/10.1016/j.msea.2015.02.003
0921-5093/

strength plasticity than ultrafine-grained $\alpha+\theta$ steel does [4]. The mechanical behavior of ultrafine- or fine-grained $\alpha+\theta$ steel may be directly correlated with the evolution of the dislocation density inside the ferrite matrix and near the ferrite–cementite interfaces [18]. The role of accumulated dislocations on the mechanical properties of the steel may also be understood based on the mechanism associated with the alteration in stress partitioning between ferrite and cementite. Therefore, an investigation into the evolution of the local stress for ferrite and cementite is beneficial in further understanding the mechanical behavior of ultrafine- or fine-grained $\alpha+\theta$ steel. The stress of ferrite and cementite in bulk $\alpha+\theta$ steel can be reliably measured using synchrotron-based X-ray diffraction due to the method's high penetration depth and reasonable single-peak fitting of the cementite phase [18–24]. Additionally, this technique can help further explain the micromechanical behavior of ultrafine- or fine-grained $\alpha+\theta$ steel from other points of view by establishing a model based on the evolution of the dislocation density. To date, the Kocks–Mecking model [25] and the modified Kocks–Mecking model [26] have been established, which are based on the evolution of the SSD density and the GND density, respectively. Because the dislocation density within $\alpha+\theta$ steel consists of the SSD density and the GND density [7,27], it is clearly unsuitable to apply the Kocks–Mecking model or the modified Kocks–Mecking model to discuss the mechanical behavior of ultrafine- or fine-grained $\alpha+\theta$ steel. Therefore, a hybrid model [26] was established by considering the roles of both the SSDs and GNDs; the model has been widely used by researchers [28–31]. However, no analytical solution for the strain–stress relationship can be obtained from the hybrid model, which leads to a failure in predicting the phase stress between hard particles and the metal matrix.

In the present work, a eutectoid steel with an ultrafine- or fine-grained $\alpha+\theta$ structure was prepared by a thermomechanical process based on the dynamic transformation of undercooled austenite and subsequent annealing for a short period, as described in previous works [4,32]; a coarse-grained $\alpha+\theta$ structure was also prepared for comparison. Transmission electron microscopy (TEM) and in situ synchrotron-based high-energy X-ray diffraction (HE-XRD) were used to monitor the evolution of the dislocation substructures and the stress partitioning during tensile testing of the eutectoid steels with various $\alpha+\theta$ structures, respectively. Based on this quantitative characterization, an analytical model considering the evolution of the GND density and the SSD density, particularly with certain material parameters calibrated with HE-XRD experiments, was established in an attempt to predict the micromechanical behavior focused on the work hardening of eutectoid steel with an $\alpha+\theta$ structure, which should be easily extended to other hard particle-strengthened alloys.

2. Materials and experimental procedure

2.1. Sample preparation

The material studied was a commercial eutectoid steel with the chemical composition (by mass percent) 0.81 C, 0.28 Mn, 0.20 Si, 0.016 P, and 0.014 S. Wing-shaped specimens [4] for a hot-compression test were machined from a hot-forged and air-cooled ingot. The forging temperature ranged from 1100 to 850 °C. The hot-compression tests were performed using a Gleeble 1500 hot simulator. After austenitizing at 850 °C for 10 min, the specimens were cooled at 20 °C/s to 650 °C and deformed to a strain of 1.61 at a strain rate of 6 s^{-1}; the specimens were then water-quenched, and an additional annealing step was performed in a muffle furnace at 650 °C for 30 min to obtain a fine-grained $\alpha+\theta$ structure. An ultrafine-grained $\alpha+\theta$ structure was also obtained by a similar process but using a strain rate of 1 s^{-1}. The microstructural evolution of a eutectoid steel with an ultrafine- or fine-grained $\alpha+\theta$ structure was reported in detail in our previous work [4,32]. For comparison, a coarse-grained $\alpha+\theta$ structure was also prepared by divorced eutectoid transformation (DET). That is, the specimens were heated at 20 °C/s to 650 °C and deformed to a strain of 1.61 at a strain rate of 0.01 s^{-1} and then air-cooled; the deformed specimens were then reheated to 750 °C for seven minutes and subsequently furnace-cooled to 710 °C for three hours, after which they were air-cooled.

2.2. Conventional tensile tests

Room-temperature (RT) conventional tensile tests were performed in a Reger 3010 tensile tester at a strain rate of 1×10^{-3} s^{-1}. Dog-bone-shaped specimens were cut from the middle of the thermo-mechanically processed specimens with a gage length of 4 mm × 12 mm and a thickness of 1.8 mm [4]. Interrupted tensile tests – with engineering strains of 0.5%, 4%, 8% and 16% for the coarse-grained $\alpha+\theta$ structure, engineering strains of 0.7%, 4%, 7% and 10% for the ultrafine-grained $\alpha+\theta$ structure, and engineering strains of 4%, 6% and 10% for the fine-grained $\alpha+\theta$ structure – were performed to investigate the evolution of the dislocation substructures.

2.3. Microstructural characterization

Microstructural observation was conducted using scanning electronic microscopy (SEM) in a Zeiss SUPRA55 field-emission scanning electron microscope. The SEM micrographs were captured parallel to the directions of hot deformation, and the plane of observation was detailed in our previous work [4]. The specimens for SEM analysis were electropolished by the standard method using an electrolyte composed of 20% $HClO_4$ + 10% glycerol + 70% C_2H_5OH under a voltage of 15 V at room temperature, and they were etched with 4% Nital. The average sizes of the ferrite grains and cementite particles were measured using Image-Pro Plus 6.0 (produced by Media Cybernetics Company, USA) image-analysis software by means of linear intercepts taken in the SEM micrographs. The dislocations within the ferrite matrix were observed by TEM (JEM 2010, operated at 200 kV), and thin foils were prepared by twin-jet electropolishing, using a solution of 5% $HClO_4$ + 95% CH_3COOH under a voltage of 75 V at −20 °C to −30 °C. The crystallographic defects within the cementite particles were observed by another TEM (Tecnai G^2 F30, operated at 300 kV), and thin foils were first prepared by the twin-jet electropolishing mentioned above and then by ion-milling with appropriate incident angles. The TEM micrographs were captured parallel to the direction of the tensile tests.

2.4. Synchrotron-based X-ray diffraction experiments and analysis

In situ synchrotron-based X-ray diffraction experiments were performed at the 11-ID-C beamline of the Advanced Photon Source (APS), Argonne National Laboratory [20]. A monochromatic X-ray beam with a high energy of 115 keV (with a wavelength 0.108 Å) penetrated through the dog-bone-shaped specimen with a gage length of 3.2 mm × 10 mm and thickness of 0.5 mm during in situ tensile loading at a strain rate of 1×10^{-3} s^{-1}. Then, the diffraction patterns, i.e., the Debye rings, were collected by a two-dimensional (2D) detector placed approximately 1.0 m from the specimen. The distance between the specimen and the 2D detector was accurately determined by processing CeO_2 (standard sample) diffraction patterns using the FIT2D software [33], which was important for the subsequent analysis to maintain the interplanar spacing within a relative error of $\approx 2\times10^{-4}$. The Debye rings were transformed into diffraction peaks by the FIT2D software [33], and then the interplanar spacings were obtained via single-peak fitting using a Gaussian function and Bragg's law.

The lattice plane strain (ε_{hkl}) can be obtained by calculating the change in the interplanar spacing between the loading state and