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Evolution of twins and sub-boundaries at the early stage of dynamic recrystallization in a Ni-30%Fe austenitic model alloy

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ABSTRACT

Evolution of twins and sub-boundaries at the early stage of dynamic recrystallization (DRX) is investigated in a Ni-30%Fe austenitic alloy. It is found that the primary nucleation of DRX is accomplished by formation of annealing twins along the bulged pre-existing grain boundaries and at the triple junctions at the early stage of deformation. With the strain increasing, the DRX nucleation due to the formation and evolution of sub-boundaries also plays a significant role. The results also indicate that the Σ9 twin boundaries are generated by mutual interactions between the encountered Σ3 twin boundaries at the initial stage. The pre-existing twin boundaries can gradually convert to general boundaries or twin steps owing to the interaction of twin boundaries with dislocations. Continuous DRX (CDRX) mechanism associated with the sub-boundaries evolution is found to be an assistant mechanism at the early stage of DRX. Besides, formation of sub-boundaries inside the growing DRX grains can stimulate a new round DRX nucleation by impeding migration of grain boundaries and making them serrated.

1. Introduction

Dynamic recrystallization (DRX) is one of the most important restoration mechanisms, which occurs during hot working in steels. It can not only reduce the load of hot working, but also reconstruct the microstructure of materials by introducing new grains with high angle grain boundaries (HAGBs) [1–13]. Since DRX is an efficient way to optimize the grain structures and thus the mechanical properties of the final products, it has always been an important issue to provide a detailed understanding on microstructural behaviors of DRX during the hot deformation. The essence of DRX is to rebuild the microstructures by replacing the deformed grains with new recrystallized grains. In general, two main mechanisms of DRX are widely accepted in metal materials, namely the discontinuous DRX (DDRX) [2,8,9,14,15] and the continuous DRX (CDRX) [7,8,16–19]. Typically, DDRX is excepted to take place in materials with low stacking fault energy (SFE), which is characterized by nucleation in areas that contain large strain gradients and high stored energies. Afterwards, the new DRX nuclei grow up through migrations of the grain boundaries driven by stored energy gradients on both sides of the migratory boundaries. In contrast, CDRX is generally approved to operate in materials with high SFE, during which new DRX grains are formed by integrating dislocations into sub-boundaries and then transforming into general grain boundaries.

DRX in austenite is usually considered to take place through the DDRX mechanism in view of the sluggish motion of dislocations due to the low SFE. In the course of hot deformation, some sub-boundaries are expected to form inside the deformed grains before the occurrence of the DRX nucleation. Their formation is supposed to influence the grain structures and local accumulations of dislocations during further deformations, which thereof affects the subsequent DRX behaviors, especially at the early stage of DRX [1,20]. However, this issue has attracted little attention in prior studies. In addition, it has been confirmed that a large portion of twin boundaries is formed after annealing in most austenitic alloys. These twin boundaries have a positive contribution to material properties [21–24]. However, there are few studies focused on the extensive evolution of pre-existing twin boundaries which could prominently affect the deformation and dislocations accumulations in the early stage of hot deformation, and inevitably influence the DRX behaviors. Besides, the new twins that can easily form in the materials with low SFE are believed to play a crucial role during the DRX [5,20,25,26].

The purpose of this study is to make a detailed understanding on the roles of twins and sub-boundaries evolution in the DRX process in austenitic steels by closely inspecting the characteristics of these structures, especially, at the early stage of DRX.

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2. Experimental procedure

A Ni-30%Fe austenitic model alloy with chemical composition of Ni-30.5Fe-0.02C-0.04Mn (wt%) was used in present work, which has approximately the same SFE as austenite in low carbon steels [27,28]. This alloy offers a facility to observe the microstructure of austenite in hot deformation because it does not undergo any transformation in the course of quenching to room temperature [29,30]. Cast materials were forged followed by hot rolling at 1000 °C to produce an initial grain structure with a mean grain size of about 12 µm.

Hot compression tests were performed on a Gleeble-3500 thermo-mechanical simulator. Cylindrical samples for compression tests with a height of 15 mm and a diameter of 8 mm were made. The specimens were firstly heated to 1000 °C, held for 120 s, and then deformed at a strain rate of 0.01 s^{-1} to different strains. The samples were water quenched immediately after the deformation to preserve the microstructure formed during the hot deformations.

The microstructures were analyzed by the electron backscattered diffraction (EBSD) and the transmission electron microscope (TEM). The deformed samples were cut in parallel to the compression axis. The microstructural characterization was carried out in the central region of the tangential sections. Samples for EBSD were ground with SiC paper, and then mechanical polished with diamond paste and finished with Ion milling to remove the surface deformation layer. EBSD examinations were carried out using a FEI Nova NanoSEM 430 field-emission scanning electron microscope equipped with a fully automatic *HKL* Technology EBSD attachment operated at 20 kV. The *HKL* Channel 5 software was used for the EBSD data acquisition and post-processing. EBSD maps were acquired using step sizes of 0.2–0.5 µm to analyze the twins and substructures. In this study, the observed boundaries were classified by various extents of misorientations, i.e. (i) low angle grain boundaries (LAGBs) with the misorientation angles between 2° and 5°, (ii) medium angle grain boundaries (MAGBs) with the misorientation angles between 5° and 15°, and (iii) HAGBs, with misorientation angles larger than 15°, respectively.

To produce the specimen for TEM, foils of 3 mm in diameter were prepared from the central region of the sections parallel to the compression axis. They were ground firstly to a thickness of about 60 µm by hand, and then twin-jet electro-polished with a solution of 10% perchloric acid in ethanol at the temperature of about − 30 °C and a voltage of 15 V. The TEM observation was performed on a FEI Tecnai G2 F20 field-emission transmission electron microscope at 200 kV.

3. Results

3.1. The overall flow stress of DRX and the microstructure evolutions

The stress-strain curve obtained by the hot compression test is presented in Fig. 1. It can be seen that the flow stress increases quickly at the early stage of deformation to a peak at a strain of about 0.18 and then decreases gradually to a constant stress level. The flow stress behavior observed here is a typically feature of occurrence of DRX during the hot deformation. Stress oscillation with multiple peaks is also observed under the given thermo-mechanical condition, which indicates that multi-cycle of the DRX does occur in the course of hot deformation. The inserted figure in Fig. 1 describes the stress dependence of the experimental working hardening rate ($\theta = d\sigma/d\varepsilon$). The deviation of the θ-σ curve from the fitting one occurs at 83 MPa, where the strain is about 0.1, signifying the onset of DRX.

The microstructures of the Ni-30%Fe austenitic alloy specimens deformed by hot compression at various strains under the strain rate of 0.01 s^{-1} and temperature of 1000 °C are shown as orientation imaging microscopy (OIM) maps in Fig. 2. In these figures, the LAGBs, MAGBs, HAGBs, Σ3 and Σ9 twin boundaries are represented by silver, green, black, red and magenta lines respectively. It can be seen that the initial microstructure absence of deformation consists of equiaxed grains with

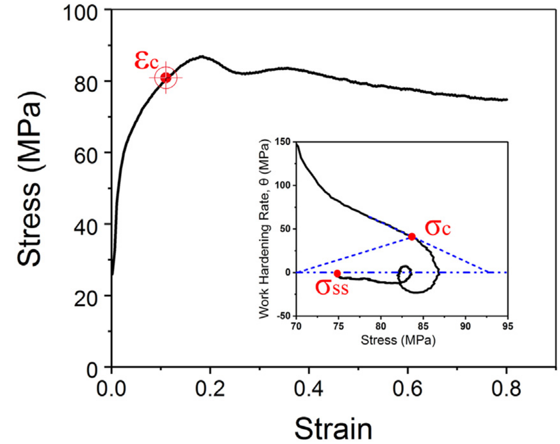

Fig. 1. Stress-strain curve and corresponding work hardening rate curve of the Ni-30%Fe austenitic alloy deformed at the temperature of 1000 °C and strain rate of 0.01 s^{-1}. ε_c and σ_c are the critical strain and stress for the onset of DRX, and σ_{ss} is the critical stress for the steady-state of DRX.

large amounts of Σ3 twin boundaries as shown in Fig. 2a. At the strain of 0.1, bulging of pre-existing grain boundaries is observed frequently as indicated by the white arrows in Fig. 2c, which is generally considered as a result of the strain-induced boundary migration (SIBM) during deformation [31,32]. The DRX nuclei were preferentially formed at the triple junctions as well as on the pre-existing grain boundaries [25,26,33,34]. And almost all of nuclei appeared to be twin-related (Σ3) to the matrix as showed in the circles in Fig. 2c. With increasing of the deformation strain, the sub-boundaries are readily generated as a result of rearrangement of dislocations just behind the bulged area of the pre-existing grain boundaries and triple junctions. Quite a few of the DRX nuclei are thereof formed at the triple junctions as well as on the bulged pre-existing grain boundaries as indicated by the rectangles in Fig. 2d. Evolution of the statistical fraction of boundaries within various scopes of misorientation angles during the deformation is shown in Fig. 3. It can be seen that the amount of the LAGBs and MAGBs both increase significantly at the early stage of deformation, which implies that new sub-boundaries are continuously formed following with gradually increasing of their misorientations during subsequent hot deformations.

The result in Fig. 1 has indicated that the steady-state of the DRX has already been achieved when the strain reaches 0.8. The corresponding microstructural observation also shows quite different characteristic in grain structures in comparison with the initial state (Fig. 2a). It is seen that there exist an abundance of dislocation walls and sub-boundaries inside the growing DRX grains as shown in Fig. 2e. The grain boundaries are serrated and seriously bulging into the adjacent grains. Some small grains with a few of sub-structures are also observed on the grain boundary, which implies a new round DRX nucleation during the hot deformation.

3.2. Evolution of twin boundaries

According to Brandon's criterion, the first order Σ3 twin boundary hereof is characterized by misorientation of 60° < 111 > , the second order Σ9 twin boundary is characterized by misorientation of 38.9° < 110 > , and the limited angle deviation is permitted [35]. It can be seen from Fig. 2a that the initial grains contain a large quantity of Σ3 twin boundaries and a few of Σ9 twin boundaries. At the early stage of the deformation, however, some of the twin boundaries are found distorted and deviated from their unique misorientation (Fig. 4). It is seen that partial segments of the general HAGBs are formed mixed up with the Σ9 twin boundaries at a low strain of 0.1, as marked by the white arrows in Fig. 4a, which indicates that the partial Σ9 twin