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Realizing Ultrafine Grained Steel by Simple Hot Deformation Using Dynamic Transformation and Subsequent Dynamic Recrystallization Mechanisms

L Zhao^{1,2}, N Park^{1,3}, Y Tian^{1,4}, A Shibata¹ and N Tsuji¹

¹ Kyoto University, Kyoto, Japan

² Colorado School of Mines, Golden CO, U.S.

³ Yeungnam University, Gyeongsan Gyeongsangbuk-do, Korea

⁴ Institute of Metal Research, CAS, Shengyang, China

Email: zhao@mines.edu, nobuhiro-tsuji@mtl.kyoto-u.ac.jp

Abstract. We have found a new strategy for ultra grain refinement without high strain deformation by combining dynamic transformation (DT) and dynamic recrystallization (DRX) mechanisms. Through simple thermomechanical processes using a total plastic strain of 0.92 at elevated temperatures, ultrafine grained microstructures having mean grain sizes down to 0.35 microns could be obtained in a 10Ni-0.1C steel. DRX phenomenon occurring in the dynamically transformed ferrite significantly reduced the strain necessary for the formation of ultrafine grains. The DRX of DT ferrite showed an unconventional temperature dependence, which suggested an optimal condition for grain refinement. The obtained UFG steel exhibited superior mechanical properties, for example, the tensile strength of 970 MPa and the total elongation over 20% at the same time.

1. Introduction

Ultrafine-grained steels having simple chemical compositions (e.g. plain carbon steels) and mean grain sizes smaller than 1 μm have large potential and benefits to replace some high-cost alloy steels with similar mechanical properties. Thermomechanically controlled processes (TMCP) can realize ultrafine ferrite (UFF) through various kinds of solid-solid reactions, i.e., recrystallization and phase transformation, occurring during the high-temperature deformation processes [1-3]. Among the metallurgical mechanisms in TMCP, dynamic transformation (DT), which is the transformation from austenite to ferrite occurring during deformation of austenite, can achieve UFF of 1~3 μm [4-6]. However, the high strain required for fabricating UFF is the main obstacle to the practical application of the TMCP including DT. To obtain a homogeneous UFF structure throughout the thickness of materials, a large accumulated strain over 2 is usually required [4]. As a result, so far, UFF has been only achieved in the surface layers of plates [5]. Recently, some reports have revealed that in the TMCP where DT occurs, DT may not be the only mechanism for ferrite grain refinement [7,8]. Dynamic recrystallization (DRX) of DT ferrite may occur, which suggests a possibility of further development in ferrite grain refinement.

In this work, we investigated refinement mechanisms of ferrite grains formed during dynamic austenite to ferrite transformation using a 10Ni-0.1C steel [8,9]. Occurrence of DRX was confirmed and it was suggested that DRX was the essentially important mechanism for the grain refinement of ferrite. We found an unconventional temperature dependence of the DRX (of DT ferrite) behavior, which indicated an optimal condition for grain refinement, leading to a new strategy proposed in this



work which could make it possible to produce UFG steels without high-strain deformation in industrial processes.

2. Experiment and Methods

The material used in this study is a 10Ni-0.1C steel (C: 0.111, Ni: 10.08, Mn: 0.01, P: 0.001, Si: 0.006, Al: 0.33, S: 0.0017, Fe: bal. (wt.%)). Cylindrical specimens with a height of 12 mm and a diameter of 8 mm were machined from the homogenized plate heat treated at 1100 °C for 172,800 s in vacuum and then installed in a thermomechanical processing simulator (Thermecmastor-Z). The specimens were austenitized at 1000 °C for 300 s, cooled to a temperature of 520 °C (austenite + ferrite region) at a rate of 30 °C s⁻¹, held for 60 s, and then the specimens with single austenite phase were uniaxially compressed at 520 °C to various strains at strain rates ranging from 10⁻¹ s⁻¹ to 10⁻³ s⁻¹ followed by water-quenching.

Microstructures at the center of the specimens on the sections parallel to the compression axis were characterized by a field-emission type scanning electron microscope (FEG-SEM) equipped with an electron back-scattering diffraction (EBSD) and also by transmission electron microscopy (TEM, Philips CM200FEG). The specimens for the EBSD analysis were mechanically polished and then electrically polished in a solution of a 10% HClO₄ and 90% CH₃COOH at 20 °C. The thin-foil TEM specimens were prepared by twin-jet electropolishing using the same solution as that for EBSD. Mechanical properties after the TMCP were examined by an uniaxial tensile test at room temperature at an initial strain rate of 8.3 × 10⁻⁴ s⁻¹ [10,11].

3. Results and Discussion

3.1. Occurrence of DRX

Figure 1a-1d shows microstructure evolution of ferrite during deformation. Martensite transformed from austenite during quenching could be distinguished from ferrite due to higher misorientations [8-11], and is painted in black in Fig.1. At a strain of 0.11 (Fig. 1a), very fine and nearly equiaxed ferrite grains are formed along a prior austenite grain boundary (PAGB). With increasing the strain to 0.36 (Fig. 1b), the ferrite grains grow into prior austenite grains and exhibit an irregular morphology. Some low-angle boundaries (LABs) are observed within the coarse ferrite grains, indicating that a deformed structure is introduced during the growth. The fraction of LABs increases with increasing the strain to 0.60 (Fig. 1c), at which equiaxed UFF grains surrounded by high-angle boundaries (HABs) form along the grain boundaries of coarse ferrite (as marked by blue arrows in Fig. 1c). The fraction of the equiaxed UFF increases with increasing the strain to 1.39 (Fig. 1d). A typical microstructure of the equiaxed UFF grain being formed is shown in Fig. 1e by TEM image with misorientation map. There are mainly two coarse DT ferrite grains divided by HAB (blue solid lines), and several subgrains. Dislocations are generated in ferrite grains due to straining and gradients of dislocation densities seem to be developed near the grain boundaries of the DT ferrite, possibly because of deformation incompatibilities between the grains. This is accompanied by subgrain formation (i.e., formation of LABs), leading eventually to the development of boundary corrugations or serrations. With straining, higher concentration of deformation would occur near high-angle grain boundaries of ferrite [19], which promotes the accumulation of dislocations and the development of misorientation near the grain boundary area of DT ferrite. The features of UFF formation in this process accord well with those of DRX of ferrite [12].

The mechanism of DRX of DT ferrite is interpreted by Fig. 2. Basically there are two regions in Fig. 2a: a region prior to the onset of DT (No-DT region) and a region after the onset of DT (DT-region). In the DT-region, there is another stage during which DRX (UFF formation) occurs. At the beginning of DT, ferrite grains are transformed from austenite during deformation. The grain size of DT ferrite firstly increases, indicating a grain growth process at a relatively early stage of transformation, but then decreases after a certain strain. The decrease in the ferrite grain size accords well with the development of DRX grains (Fig. 2b) in subsequent deformation.

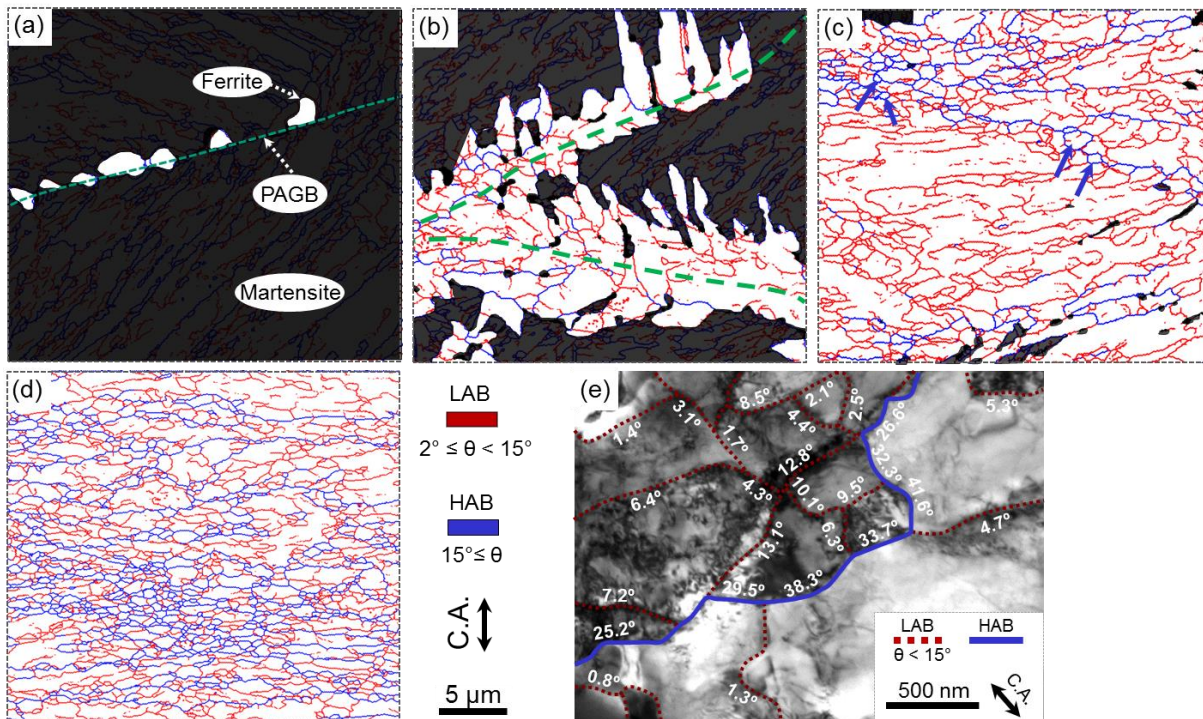


Fig. 1 (a-d) EBSD grain boundary maps of the 10Ni-0.1C steel deformed to different strains of (a) 0.11, (b) 0.36, (c) 0.60, and (d) 1.39 at a strain rate of 10^{-2} s^{-1} at 520 °C. (e) TEM image corresponding to (d). C.A. indicates the compression axis.

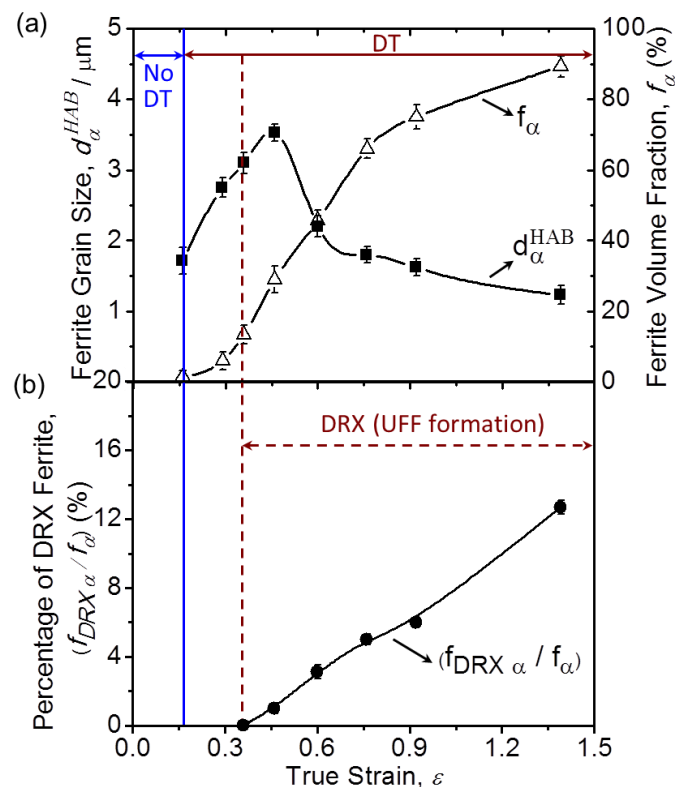


Fig. 2 Variations of (a) ferrite grain size (counting only HABs), volume fraction of the dynamically transformed ferrite and (b) volume fraction of the DRX ferrite as a function of hot-compression strain.

3.2. Unconventional Temperature Dependence of DRX of DT Ferrite.

The temperature dependence of DRX of DT ferrite is further investigated. Figure 3a-3c shows EBSD grain boundary maps of specimens deformed to a strain of 0.92 at different temperatures. At 560 °C (Fig. 3a), the ferrite mostly shows coarse and elongated morphologies. The volume fraction of DT ferrite is 52%, and only a few DRX grains having fine and equiaxed morphologies can be observed, as marked by blue arrows. At 520 °C (Fig. 3b), the fraction of DRX grains increases compared to that at 560 °C. At 440 °C (Fig. 3c), on the other hand, the fraction of DRX grains decreases. Most of the grains are elongated and contain a large amount of LABs, exhibiting more deformed structures. The HABs surrounding ferrite grains at 440 °C are more like geometrically necessary boundaries (GNB) [13] rather than being formed by DRX mechanism. Figure 3d summarizes the changes in the grain size, volume fraction of DT ferrite and volume fraction of DRX ferrite as a function of deformation temperature. At temperatures from 440 °C to 520 °C, the ferrite transformation (solid rectangles) is completed after a strain of 0.92. The grain size (solid circles) of DRX ferrite increases with increasing temperature from 440 °C to 560 °C. It is interesting that the fraction of DRX ferrite (solid triangles) exhibits a peak value at 520 °C, indicating an optimal temperature for the formation of DRX grains.

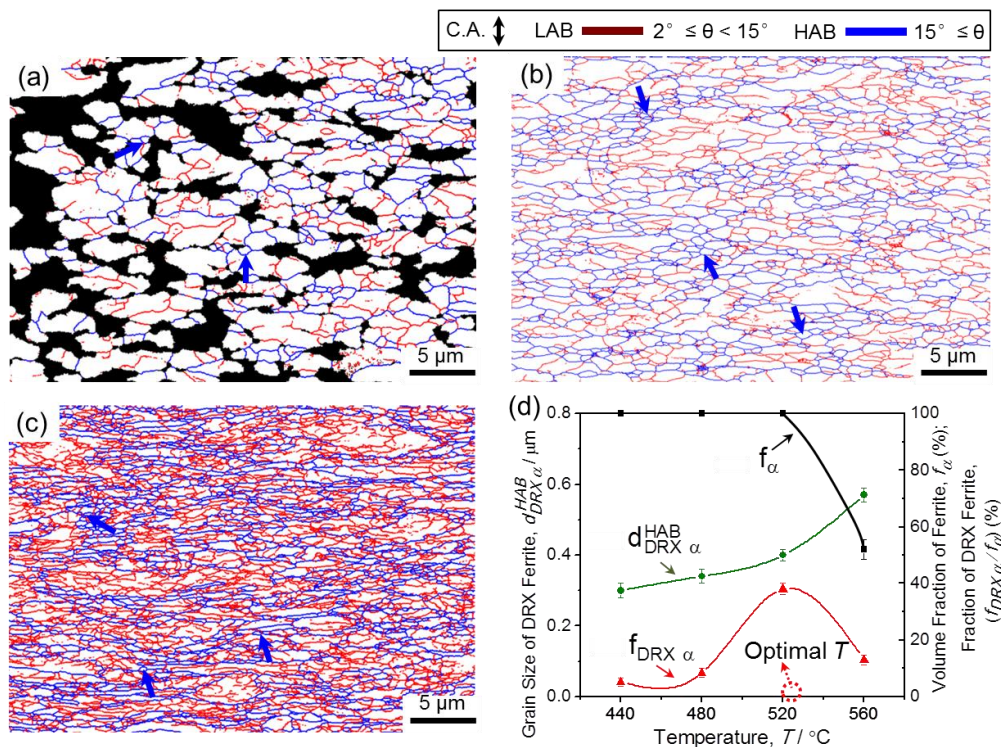


Fig. 3 (a-c) EBSD grain boundary maps of the specimens with an austenite grain size of 10 μm deformed to a strain of 0.92 at a strain rate of 10^{-2} s^{-1} at different temperatures. Non-ferrite phases were painted in black. C.A. indicates the compression axis. (d) Variations of the mean grain size ($d_{DRX\alpha}$), volume fraction of DRX grains ($f_{DRX\alpha}$) and volume fraction of dynamically transformed (DT) ferrite (f_{α}), as a function of the deformation temperature.

The reasons for the unconventional temperature dependence of the DRX of DT ferrite are explained as follows. It has been known that lowering deformation temperature (above the nose temperature in transformation kinetic C-curve) shortens the incubation period for the onset of DT and accelerates its kinetics [14]. At 560 °C, the driving force and nucleation density for ferrite transformation are low, but the diffusivity of atoms is high. Therefore, the grain growth of DT ferrite is rather dominant than nucleation, resulting in a large grain size. The combining effect of large grain size and temperature retards the onset of DRX. At 520 °C, both the driving force and the nucleation density for ferrite transformation increase, which leads to faster nucleation kinetics in DT. The fast

nucleation results in finer ferrite grain size due to the sufficient impingement effect between ferrite grains. The fine ferrite grain size enhances the initiation of DRX and accelerates its kinetics [7,15]. At 440 °C, the diffusivity of atoms is so low that DRX is suppressed. The DRX behavior of DT ferrite exhibits a C-curve according to the deformation temperature, which implies an optimized temperature for the progress of DRX, to be more specific, for the formation of UFF grains.

3.3. New Strategy for Fabricating UFG Steel without High-Strain Deformation.

The novelty of the DRX of DT ferrite is that the initial grain size of ferrite formed by DT (prior to DRX) could be very small. This means that the required strain for the initiation of DRX and fabrication of fully ultrafine microstructure can be greatly reduced [7,15]. The present study has proved two important points: (1) DT can greatly affect the subsequent DRX behavior; (2) There exists an optimal temperature at which the kinetics of DRX of DT ferrite is the fastest. These two points give a new grain refinement strategy by controlling DT and DRX. That is, at the optimal temperature, DRX should be enhanced by accelerating DT kinetics and consequently leads to finer grains.

Figure 4a schematically illustrates a new TMCP route for grain refinement. The novelty of the new TMCP route is that at a relatively low temperature (T_{d2}), a small pre-deformation is applied for accelerating DT as well as subsequent DRX at a high temperature (T_{d1}). As a result, the final ferrite structure can be significantly refined to 0.46 μm with 75% HABs by applying only a true strain of 0.92. Figure 4b shows a possibility of further grain refinement through the new TMCP route with a true strain of 1.39. The fraction of HABs is 70% and the mean grain size is 0.35 μm .

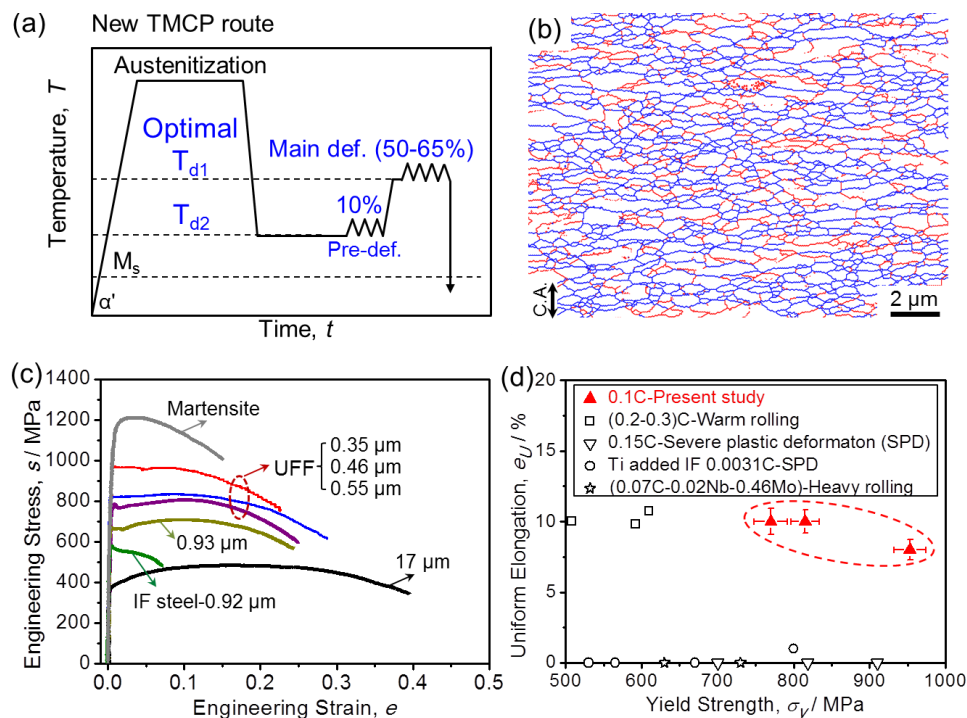


Fig. 4 (a) New TMCP route using DT and DRX for grain refinement; (b) EBSD grain boundary map of ferrite structures with a mean grain size of 0.35 μm obtained by the new TMCP route; (c) Mechanical properties of the UFF specimens processed in the new TMCP route, the specimen with coarse ferrite grains (17 μm), the specimen with full martensite, and an UFG IF steel (0.92 μm) obtained by accumulative roll-bonding and subsequent annealing [16]; (d) Comparison of the mechanical properties between the present UFF structures and those in previous works [17-20].

Figure 4c shows engineering stress-strain curves of the specimens processed in the new TMCP route (marked as UFF, mean grain sizes of 0.55–0.35 μm), the specimen with a statically transformed coarse ferrite grains (17 μm), the specimen with full martensite of the present 10Ni-0.1C steel, and an

UFG interstitial free (IF) steel (0.92 μm) obtained by accumulative roll-bonding and subsequent annealing. Compared to the specimen with statically transformed coarse ferrite, the UFF specimens showed significantly higher yield strength of 770-953 MPa and tensile strength of 810-973 MPa, which are much superior to the UFG IF steel and even close to the yield strength of martensite (1100 MPa). It has been known that UFF ferrite usually shows limited uniform elongation due to early plastic instability [21]. In such a context, it is rather surprising that the present UFF maintains large tensile ductility, i.e., uniform elongation of 8-10% and total elongation of 23-29%. Compared to the results in literatures [17-20], the plots of uniform elongation and yield strength of the present UFF structures are out of the trend in UFF steels obtained through processes including heavy deformations (Fig. 4d). And importantly, the present TMCP route combining DT and DRX mechanisms requires so far the smallest strain to achieve UFF structures.

Conclusions

We could obtain homogeneous UFF structures having a mean grain size down to 0.35 μm through a new TMCP route combining DT and DRX mechanisms, which required a true strain of only 0.92-1.39 in hot deformation. The DRX of DT ferrite showed an unconventional temperature dependence, which suggested the existence of an optimal condition for grain refinement. The obtained UFG steel exhibited superior mechanical properties with a tensile strength of 970 MPa and the total elongation over 20%. The present study could bring new excitement to the production of UFG steels without high-strain deformation in industries.

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