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Small-scale mechanical property characterization of ferrite formed during deformation of super-cooled austenite by nanoindentation

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ABSTRACT

The mechanical properties of dynamically and statically transformed ferrites were analyzed using a nanoindentater-EBSD (Electron BackScattered Diffraction) correlation technique, which can distinguish indenting positions according to the grains in the specimen. The dilatometry and the band slope and contrast maps by EBSD were used to evaluate the volume fractions of two kinds of ferrite and pearlite. Fine ferrites induced by a dynamic transformation had higher nano-hardness than the statically transformed coarse ferrites. Transmission electron microscopy (TEM) showed the dynamic ferrites to have a higher dislocation density than the statically transformed ferrites.

1. Introduction

Recently, there has been increasing demand for the manufacture of ultrafine-grained ferritic steels to satisfy the increasing demand for structural steels with improved strength and toughness [1–9]. The key technology for producing ultrafine-grained ferritic steels is to impose heavy deformation to super-cooled austenite to induce a dynamic formation of ferrite grains [10]. The Strain-Induced Dynamic Transformation (SIDT) of super-cooled austenite has been highlighted as a process that can overcome the limitations of conventional ThermoMechanical Control Processing (TMCP). Both dynamically and statically transformed ferrites coexist in SIDTed steels due to the lower volume fraction of SIDTed ferrite in low carbon steel than the equilibrium ferrite fraction at a given processing temperature [5,7,10].

It is known that the grain size of the dynamic ferrite is finer than that of static one [5]. Since the strength of the fine-grained steel has been described by the Hall-Petch relationship, which is taken the interaction between grain boundary and dislocation into account, the amount and the grain size of dynamic ferrite are very important in achieving the higher strength and toughness of the steel. However, this strength evaluation from the Hall-Petch relationship is not based on the mechanical property itself of the inside of ferrite grain. On the contrary, there might be a possibility that fine dynamic ferrites could show the softer mechanical behavior because this dynamic ferrite transforms at higher temperature than static one. In order to compare the small-scale mechanical characteristics of each ferrite itself by grains, a nanoindentation would be a good candidate.

In this study, quantitative dilatometric analysis was used to determine the volume fractions of dynamic and static ferrite in low carbon steel during deformation and subsequent cooling, respectively. The small-scale mechanical properties of the two types of ferrite were characterized by nanoindentation. A nanoindentater-EBSD (Electron Backscatter Diffraction) correlation technique was used to distinguish the indenting positions according to the grains in the specimen. TEM observations of the dislocation densities showed a difference in nano-hardness between the two types of ferrite.

2. Experimental

The composition of steel used in this study was Fe–0.1C–1.5Mn–0.25Si–0.05V–0.01Ti–0.04Nb–0.0036N (wt.%). The Ae_3 temperature of this steel was estimated to be 810 °C under the para-equilibrium condition using Thermo-Calc [11]. A vacuum induction melted ingot was soaked and hot-rolled to a 15 mm thick plate at temperatures between 1000 and 1200 °C.

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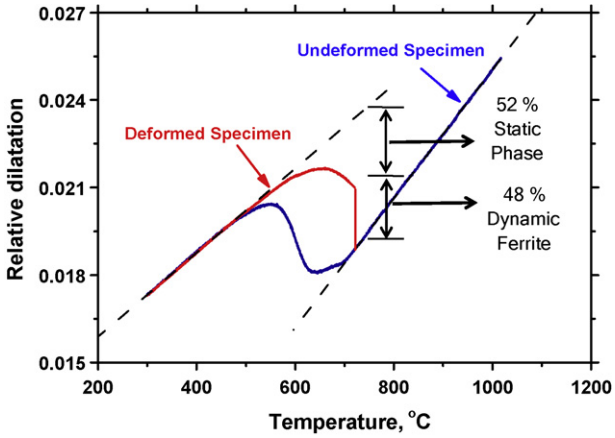

Fig. 1. Comparison of relative dilatation curves between deformed (SIDT) and undeformed specimens during cooling at the rate of 2 °C/s.

Cylindrical specimens, 10 mm in diameter and 15 mm in height, were machined from those plates for thermo-mechanical processing on a Gleeble 3800. The specimens were reheated to 1200 °C for 5 min resulting in an austenite grain size of 77 μm. The specimens were then cooled to 720 °C at a rate of 2 °C/s, and deformed immediately with upto 70% compression at a constant strain rate of 1.0/s. The deformation temperature was chosen to be 20 °C above the Ar_3 temperature of 700 °C. After deformation, the specimens were cooled immediately to room temperature at 2 °C/s. The dilatometric change in the diametrical direction in the middle of the specimens was measured over the entire process, including heating, deformation and cooling.

After thermo-mechanical processing, sample preparation for nanoindentation was performed by mechanical grinding using a diamond suspension and chemical etching with 3% nital solution to expose clearly the grain boundary by SPM (Scanning Probe Microscope) equipped in the nanoindenter. The nano-hardness of the dynamically and statically transformed ferrites was measured using a Hysitron Tribolab nanoindenter. Total 37 indentations were carried out inside grains one by one. The maximum load for each indent was 800 μN. The grains could be classified into dynamic and static ferrite by measuring the grain size in the EBSD image.

An EBSD (HKL Nordlys Channel 5) system was used to measure the crystallographic orientation and size of each ferrite grain. A total area of 1400 × 1000 pixels was indexed with a step size of 0.1 μm. The indentation positions were determined by the grains using both EBSD and SPM image. In addition, the dislocation densities in the dynamically- and statically-transformed ferrites were observed by TEM (Tecnai F20). Thin foils were prepared for the TEM observation by mechanical polishing and electropolishing with a solution containing 10% perchloric acid and 90% methanol followed by Ar^+ ion milling to remove the oxidized or contaminated surface layer.

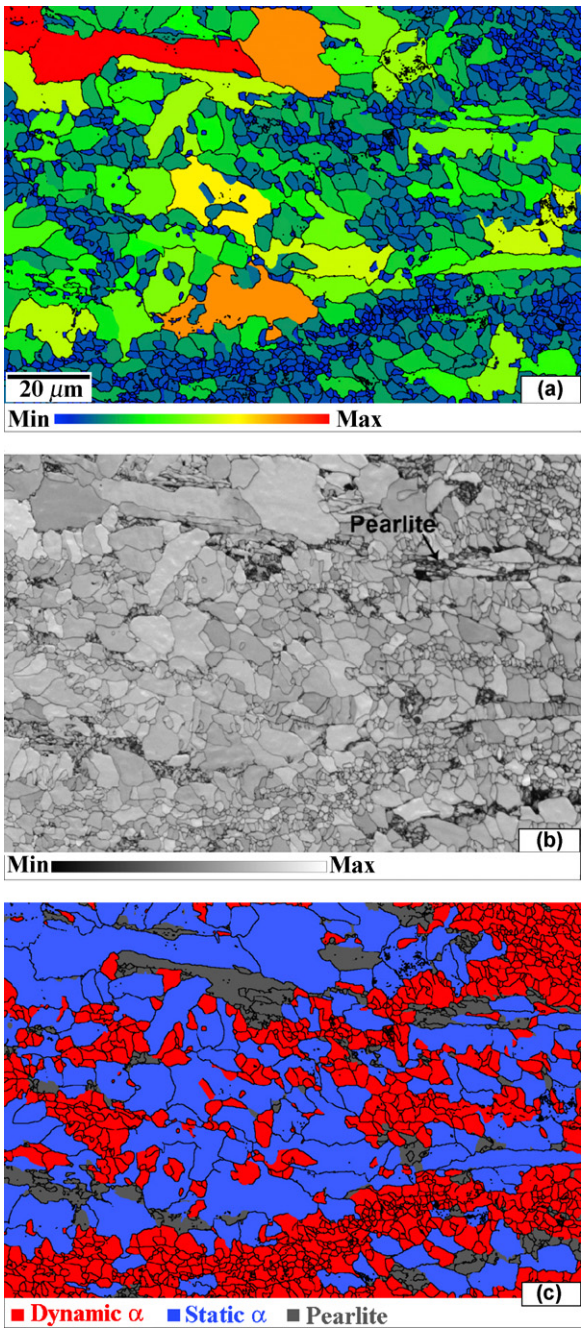

Fig. 2. (a) Grain size map measured by EBSD for the specimen undergone SIDT, (b) band contrast and slope combined map for classifying pearlite and (c) image of microstructure classified into the dynamically and statically transformed ferrites and the pearlite.

3. Results and discussion

The austenite remaining in the specimen that is subject to immediate cooling to room temperature after deformation at above the Ar_3 temperature and below the Ae_3 temperature undergoes a static austenite-to-ferrite and austenite-to-pearlite transformation. The volume fraction of the statically transformed phase after SIDT could be evaluated by a comparison with the relative dilatation curve during continuous cooling of the specimen without deformation. Fig. 1 shows the relative dilatation curves for both the deformed and undeformed samples during cooling at 2 °C/s. The volume fraction of the statically transformed phase, f_S, in the SIDTed specimen was determined by applying the lever rule [12–17] to the dilatation curves. For the lever rule, the dilatations curve of the undeformed specimen was used as the linear segment in the single austenite region. The relative dilatometric curve was used to compensate for large changes in the length of the dilatometric specimen due to the heavy compressive deformation. The volume fraction of dynamic ferrite, f_D, was obtained from the equation, $f_T = f_S + f_D$, where f_T is the volume fraction of the total transformed phase. In this case, the volume fractions of dynamic ferrite and the static phase were determined to be 0.48 and 0.52, respectively. The volume fraction of the statically-transformed pearlite after SIDT was determined to be approximately 0.1 from optical microscopy and the combined band contrast and slope map [18] obtained from EBSD, as shown in Fig. 2(b). Therefore, the volume fraction of static ferrite is 0.42.