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ScienceDirect

Acta Materialia 85 (2015) 243–249

Acta MATERIALIA

www.elsevier.com/locate/actamat

Effect of prior martensite on bainite transformation in nanobainite steel

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Received 15 October 2014; revised 20 November 2014; accepted 20 November 2014

Abstract—Nanobainite transformation behavior was comparably studied using in situ neutron diffraction measurements, scanning electron microscopy and electron backscatter diffraction observations for two heat treatments: with and without partial quenching before isothermal holding at 523 or 573 K. Prior martensite transformation was found to accelerate the subsequent nanobainite transformation. Bainitic lathes formed adjacent to a pre-existing martensite plate exhibited an almost identical orientation. Dislocations introduced in austenite due to stress relaxation of transformation strains are believed to assist bainite transformation accompanying variant selection. Diffraction profiles of austenite were found to show symmetric broadening with martensite transformation whereas nonsymmetric broadening occurred with nanobainite transformation, indicating the generation of two populations of austenite. Diffraction line broadening analysis using the convolutional multiple whole profile method provided a dislocation density of 1.51×10^{15} m^{-2} in austenite after partial martensite transformation.

Keywords: Martensite; Bainite transformation; Neutron diffraction; Crystallography; Steel

1. Introduction

Multiphase steels with different mechanical properties have been the subject of considerable attention because of their excellent combination of strength and ductility/toughness [1,2]. To obtain multiphase structures, various heat treatments have been developed such as intercritical annealing followed by quenching to produce dual-phase steels [3], intercritical annealing followed by isothermal holding for transformation induced plasticity (TRIP) steel production [4] and quenching and partitioning (Q&P) for high strength steels [5–7]. The realization of an ultra-fine-grained multiphase structure is a necessary target of steel design, where heat treatment must be optimized.

Recently, a nanobainite steel with a microstructure consisting of nanoscaled laths/plates of bainitic ferrite and carbon-enriched film austenite has been found to exhibit a tensile strength greater than 2 GPa and a fracture of ~30 MPa m$^{1/2}$ [8–10]. The formation of nanobainite by isothermal holding at 473–673 K shows a very slow transformation rate [9–11]. This treatment method is favorable for producing large mechanical components with small residual stresses. However, the heat-treatment time must be reduced in many cases. We have found that a small amount of ausforming at a low temperature (e.g., 573 K) significantly accelerates nanobainite transformation [12,13]. The dislocation structure introduced in austenite by low temperature ausforming is found to assist bainite transformation with strong variant selection where partial dislocations introduced by ausforming play an important role for bainite transformation [13].

Achieving such bainite transformation acceleration without plastic deformation is desired. Shibata et al. [14] and Miyamoto et al. [15] reported that martensite transformation introduces dislocations not only in the martensite grain itself but also in the austenite near the interface. Hence if small amounts of martensite were to be introduced prior to isothermal holding, bainite transformation would be accelerated by dislocations in the vicinity of the interface between martensite and austenite. By employing up-quenching heat treatment similar to that employed in Q&P treatment, fresh martensite would be tempered to be ductile.

The effects of pre-existing transformation products on subsequent transformation have recently been investigated. Santofimia et al. [16] reported that partial ferrite formation upon cooling suppresses martensite transformation because the supercooled austenite is stabilized by carbon enrichment. In the case of ferrite transformation, dislocations do not exist near the interface between ferrite and austenite. Zhu et al. [17] suggested that bainite transformation kinetics are influenced by the competition of the acceleration effect by the interface between ferrite and austenite as a nucleation site of bainite lath and the retardation effect by higher concentrations of alloy elements near the interface. In this case, dislocations do not exist in the vicinity

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http://dx.doi.org/10.1016/j.actamat.2014.11.029
1359-6462/

of the interface. Contrary to these cases, Kawata et al. [18] observed that bainite transformation was accelerated by pre-formed martensite. They have claimed that the acceleration of bainite transformation is caused by an increase in nucleation sites at the martensite/austenite interface. It is suspected that the dislocations emitted into austenite by martensitic transformation assist in the nucleation of bainite lathes.

Applying the quenching–heating technique, i.e., partial quenching, is preferred, and it is followed by isothermal holding for bainite transformation treatment, to nanobainite formation for the production of small-sized mechanical parts. The measurement of the dislocation density in austenite prior to the onset of bainite transformation is a critical issue. Recent developments of high-intensity and high-resolution neutron diffractometers enable us to determine the globally averaged microstructural parameters with a short time slicing interval. Various in situ experiments have been conducted for phase transformation behavior [11], mechanical behavior [19,20] and thermomechanically controlled processing [13,21,22]. By analyzing the diffraction patterns, the quantitative values of phase volume fraction and lattice parameter can be obtained, which are used for the determination of kinetics, carbon concentration partitioning, etc. Recently developed line broadening analyses [23–26] have interestingly made it possible to obtain a measure of the dislocation density with substructure from neutron diffraction profiles.

In the present study, phase transformation kinetics, carbon distribution and dislocation density/structure were examined using in situ neutron diffraction during heat treatment. Morphologic and crystallographic features were investigated, and the effect of partial quenching on bainite transformation was discussed.

2. Experimental

The chemical composition of the steel used in this study was Fe–0.79C–1.98Mn–1.51Si–0.98Cr–0.24Mo–1.06Al–1.58Co (wt.%). The samples were prepared by vacuum induction melting [8,27]. The ingot was homogenized at 1473 K for 14.4 ks, followed by hot-rolling in the temperature range 1272–1473 K to reduce the thickness from 40 mm to 10 mm through ten successive passes. Cylindrical specimens of 32 mm length and 8 mm diameter were prepared by spark wire cutting for in situ neutron diffraction experiments. The in situ neutron diffraction experiments were performed using a dilatometer installed in the engineering neutron diffractometer, TAKUMI, at the Japan Proton Accelerator Research Complex (J-PARC). The neutron diffraction profiles were recorded by two detector banks simultaneously with dilatometry and temperature measurements. The details of the diffraction instrument [28] and the dilatometer [11] are reported elsewhere.

Two series of heat treatment processes were carried out: (a) the direct isothermal transformation (DIT) process and (b) the quenching and bainite transformation (QBT) process. The heat schedules for in situ neutron diffraction experiments are presented in Fig. 1. In QBT, a specimen was austenitized at 1173 K for 1.2 ks before rapid cooling to 383 or 350 K (below M_s: martensite transformation start temperature), then heated up to 523 or 573 K for isothermal holding and finally cooled to room temperature. There

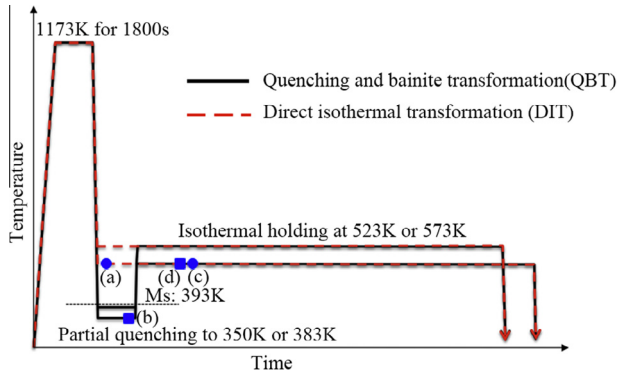

Fig. 1. Schematic illustration of heat treatments: partial quenching followed by isothermal bainite transformation (QBT) and direct isothermal bainite transformation (DIT). Blue solid squares and circles indicate the stages where the diffraction profiles shown in Fig. 2 were obtained. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

is no partial quenching process for DIT treatment, and a specimen was cooled from 1173 K directly to 523 K or 573 K for isothermal bainite transformation.

The Z-Rietveld code [29] was employed for Rietveld refinement to determine the phase fraction and lattice parameters of each constituent. The time slicing interval for the profile analysis was set as 60 s. Here the convolutional multiple whole profile (CMWP) method was employed for profile analysis to determine the dislocation density and substructure. The CMWP program developed by Ungár and coworkers [25,26] provides the density (ρ), arrangement (M^*) and character (q) of dislocations. To subtract the instrumental effects, the diffraction profile of the single austenite phase obtained at 1173 K was used as the instrumental profile, which hardly changed even when measured at 523 or 573 K before the onset of transformation (the supercooled austenite state).

After the in situ neutron diffraction experiments, the specimens were sectioned transversely and polished following a conventional metallographic technique. A 2% Nital etchant was used to reveal the microstructure. Microstructural characterizations were carried out by means of scanning electron microscopy (SEM) using a Hitachi 4300 microscope. The specimens for electron backscatter diffraction (EBSD) examination were prepared by mechanical grinding and then polished with a colloidal silica slurry. EBSD measurements were performed under the following conditions: acceleration voltage of 20 kV, tilt angle of 70°, working distance of 29 mm and step size of 0.1 μm. EBSD data were post-processed by means of the commercial TSL® software.

3. Results and discussion

3.1. Phase transformation behavior during DIT and QBT

The diffraction patterns at different stages of DIT and QBT marked with blue circles and squares, respectively, in Fig. 1 are presented in Fig. 2. Fig. 2a and b shows the patterns observed before and after the onset of bainite transformation at 523 K, respectively. As seen in these figures, austenite peaks before the onset of transformation are