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Nanometer martensite flakes in high-temperature deformation-induced ferrite grains of a low-carbon steel

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High-temperature deformation-induced ferrite grains supersaturated with carbon offer superior properties such as higher elastic modulus and microhardness compared with proeutectoid ferrite because of they contain nanometer martensite flakes. After annealing at 700 °C, fine carbides precipitate due to the decomposition of these nanometer martensite flakes.

Keywords: Nanometer martensite; High-temperature deformation-induced ferrite; Carbon supersaturation; Low-carbon steel; Carbon diffusion

Since the austenite–ferrite transformation is of great importance in refining ferrite grain size, it has been investigated intensively and extensively over recent years. The most frequently mentioned transformation is the deformation-induced ferrite (DIF) transformation because of its simplicity and efficiency in grain refinement [1–3]. Using this transformation, Priestner and co-workers observed local areas of very fine grains in rolled steels [4]. Yada and Matsumura claimed to have produced coils of hot-rolled strip with a ferrite grain size approaching 1 μm [5].

A number of hypotheses regarding the formation of DIF from austenite in steel have been suggested. Generally speaking, the austenite–ferrite transformation during undeformed austenite isothermal decomposition occurs by diffusion-controlled nucleation and growth processes [6,7]. Some researchers [8] have proposed that the DIF transformation is also controlled by carbon diffusion. However, this has been questioned by other researchers [9–11] because of the high percentage of the DIF existing in specimens when the deformation has taken place above Ar_3 and at a high strain rate. In these conditions, carbon cannot diffuse fast enough to maintain the carbon concentration equilibrium between austenite and ferrite. Yang et al. [9,10] found that a high percentage of the DIF was obtained when the strain rate was as high as $350\ s^{-1}$. If the DIF transformation was controlled by long-range carbon diffusion, the carbon concentration in the residual austenite would be high enough to decompose the austenite into pearlite and ferrite. Thus, there would be deformed pearlite in the deformed specimens. However, no pearlite was observed, even though the percentage of the DIF was up to 95%. Yang and co-workers therefore hypothesized that DIF transformation was not controlled by long-range carbon diffusion. Their hypotheses were confirmed by Tong's [12] simulation results of isothermal DIF transformation in a Fe–C binary system, in which there was no obvious directional carbon diffusion between austenite and ferrite grains at a high strain rate. Thus, although there have been various hypotheses about carbon diffusion, none of them have been directly confirmed by experiments. Moreover, because much DIF transformation work by previous researchers was undertaken below Ae_3, the influence of supercooling has been a complicating factor.

In this work, we create conditions to induce high-temperature DIF. (In order to distinguish this material from the DIF induced below Ae_3, we name the DIF induced above Ae_3 as high-temperature DIF.) We will then demonstrate experimentally the presence of high-temperature DIF grains with supersaturated carbon, and thereby reveal the storage location of the excess carbon, hence clarifying the DIF transformation mechanisms.

The chemical composition of the low-carbon steel (Q235) used is 0.13 C–0.19 Si–0.49 Mn–0.012 P–0.013

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S (wt.%). The austenite–ferrite equilibrium transformation temperature Ae_3, calculated by the Thermo-Calc software package, is 848 °C. The hot compression specimens with diameter 8 mm and height 15 mm were machined from a rough slab, normalized at 950 °C for 1 h. The thermal simulation experiments were performed on a Gleeble 3500 machine.

Two routes were adopted here and specimens containing either DIF or proeutectoid ferrite were obtained.

The DIF specimens: After austenitization at 950 °C for 5 min, the specimens were cooled to 850 °C at a cooling rate of 1 $°C\,s^{-1}$ and isothermally held for 30 s at 850 °C. The specimens were then uniaxially compressed to a reduction of 80% at a strain rate of 20 s^{-1} at a target temperature of 850 °C. However, from the recorded temperature–time curve, the temperature increased to 875 °C during this deformation due to the plastic work. Therefore, the temperature of the DIF transformation in this study was much higher than the Ae_3 temperature of 848 °C. Quenching immediately after deformation was considered to be the key to conserve the high-temperature DIF microstructure, because, naturally, the high-temperature DIF can rapidly retransform to austenite above Ae_3. The cooling rate is also important, because the cementite can precipitate from the high-temperature DIF grains supersaturated with carbon, and proeutectoid ferrite can form when the cooling rate is low [13]. In this work, the DIF specimens were quenched immediately after the compression finished not only by the water sprayed from the standard nozzle of the Gleeble 3500, but also by additional iced water poured on by hand. By this means a cooling rate of up to 1500 $°C\,s^{-1}$ was achieved.

DIF specimens were sectioned through the center parallel to the compression axis. The sections were ground, polished and etched in a 3% nital solution. Under the microscope, the outer region of the DIF specimens were seen to be composed of the high-temperature DIF and a little lath martensite, and the center region was composed totally of the high-temperature DIF grains.

In the outer region of the DIF specimens (Fig. 1a) the carbon concentration in the quenched martensite and the high-temperature DIF grains was compared by electron probe microanalysis (EPMA). To achieve optimal conditions for the measurement of light elements such as carbon, sensitivity was maximized by the use of a 10 kV accelerating voltage and 0.5 nA as reported by Refs. [14,15]. The center part of the specimens (Fig. 1a), where only the high-temperature DIF grains existed, was studied by X-ray diffraction (XRD) measurements using a Rigaku D/Max-RC with a Cu target. Microstructural observations were carried out using a JEOL JEM 2010 transmission electron microscope (TEM). Nanoindentation tests were carried out with a Nano Indenter XbTM (MTS, USA) with a Berkovich tip loading weight of 980 μN. The continuous stiffness measurement (CSM) method was used. After the tests, the specimens were etched with 3% nital and then observed under a scanning electron microscope (SEM) to identify the locations of the residual indentations.

The proeutectoid ferrite specimens: After austenitization at 950 °C for 5 min, the specimen was directly cooled to room temperature at a cooling rate of 1 $°C\,s^{-1}$. The specimens were sectioned perpendicular to their axis. The sections were ground, polished and etched in a 3% nital solution. Under the microscope, the specimens were seen to be composed of proeutectoid ferrite and pearlite. The proeutectoid ferrite specimens were studied by XRD and nanoindentation (Fig. 1b).

To compare the carbon concentration in the high-temperature DIF grains with the parent austenite (residual austenite transformed into martensite during quenching), an EPMA line analysis of the carbon distribution across the section of the DIF specimen is shown in Figure 2 [16]. The concave areas denote the high-temperature DIF grains and are labeled a, c, d, g, h, i, j and k while the convex areas denote martensite and are labeled b, e and f. Figure 2 illustrates that there is almost no difference between the carbon concentration of the high-temperature DIF grains and martensite grains, in contrast to expectations based on the carbon concentration of ferrite which is two orders of magnitude lower than that of quenched martensite. The high-temperature DIF grains are therefore supersaturated with carbon.

The XRD method involving step scan techniques was also used here to compare the parameters of the DIF grains with that of proeutectoid ferrite. A software program (MDI Jade 5.0 Materials Data, Inc.) was used to obtain the lattice parameters for the high-temperature DIF grains and proeutectoid ferrite. From Table 1 it was found that the lattice parameter 0.286967 nm of the DIF was significantly larger than that of proeutectoid ferrite at 0.286865 nm. Using the relationship proposed by Bhadeshia et al. [17] which relates the ferrite lattice parameter to alloying element concentration,

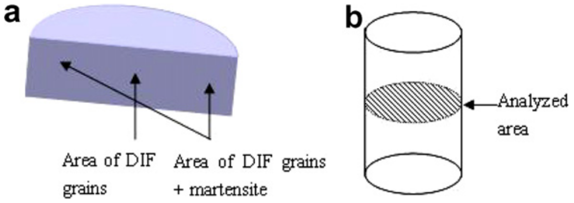

Figure 1. The shape of (a) the DIF specimen, (b) the proeutectoid ferrite specimen.

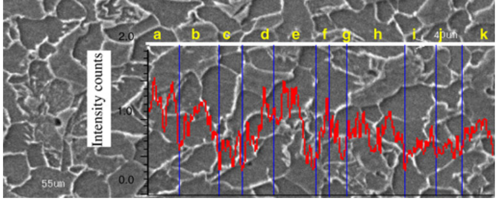

Figure 2. EPMA line analysis of the carbon concentration in the high-temperature DIF (a, c, d, g, h, i, j, k) and the water-quenched martensite (b, e, f) in the border of the DIF specimen, showing very similar carbon concentrations in the high-temperature DIF and the quenched martensite.