Thermal Stability of High Temperature Deformation Induced Ferrite in a Low Carbon Steel

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(Received on June 27, 2006; accepted on October 24, 2006)

A series of unidirectional compression tests for a low carbon steel were performed on a thermal simulating machine Gleeble 3500, and the thermal stability of high temperature deformation induced ferrite (DIF) was studied systematically through the post-treatments such as isothermally hot holding time above the austenite–ferrite equilibrium transformation temperature A_{e3} , cooling rates and annealing temperatures after deformation. The results show that the DIF volume fraction decreases during isothermally hot holding and finally DIF disappears. The DIF grains showed rapid growth at low annealing temperatures ($<200^{\circ}\text{C}$) and relatively stable at middle annealing temperatures ($200\text{--}500^{\circ}\text{C}$), lastly rapid growing at high annealing temperatures ($500\text{--}700^{\circ}\text{C}$). Compared with the microstructure of DIF with martensite in quenched specimens, the proeutectoid ferrite and pearlite appear in controlled cooled specimens at low cooling rates (*i.e.* 10°C/s and 1°C/s). The present experiments revealed that DIF is not stable at the high temperature and hardly conserved in hot mills.

KEY WORDS: DIF; thermal stability; post treatments; low carbon steel.

1. Introduction

Grain refinement of steels has attracted considerable interest because ultra-fine grained (UFG) steels exhibit superior mechanical properties, such as high strength, toughness, long fatigue life and excellent super plasticity. Recently, there are two main groups of laboratory methods for refining ferrite grains down to the ultrafine grain regime.¹⁾ One is severe plastic deformation technique such as equal channel angular pressing (ECAP),²⁾ accumulative roll bonding (ARB)^{3,4)} and bi-directional deformation.⁵⁾ The other is thermo-mechanical process including the DIF transformation (DIF transformation is also called ‘strain induced ferrite transformation’, which is a kind of the solid state phase transformation that is mechanically induced during deformation by the production of new nucleation sites by plastic strain.⁶⁾ In order to distinguish from DIF induced below A_{e3} , we name the DIF induced Above A_{e3} as high temperature DIF.), dynamic recrystallization of the austenite during hot deformation with subsequent austenite to ferrite transformation,⁷⁾ hot rolling in the intercritical region (*i.e.* in the austenite+ferrite two-phase region)⁸⁾ and/or dynamic recrystallization of the ferrite during warm rolling.⁹⁾ Among these methods, the DIF transformation is one of the most important methods for producing low-cost, high strength steels. By the DIF transformation method, the DIF with a limited grain size of around $1\text{ }\mu\text{m}$ was obtained in laboratory scale for low carbon microalloyed and plain carbon steels.^{10–13)} To conserve the DIF, the specimens are usually quenched after deformation at a low temperature

(near A_{r3} temperature) in laboratory research. In the past years, it has been found that the strain, strain rate, deformation temperature and the grain size of primary austenite have great effect on the DIF during deformation or pre-deformation.^{14–16)} In addition, there are several factors such as hot holding time at high temperatures, cooling rates and annealing temperatures, might also influence on the conserving of DIF in practical hot mills. For example, the traditional finish rolling temperatures are normally higher than A_{e3} , there is a delay of about 2–5 s between deformation and subsequently cooling, and a laminar flow cooling at a rate about $20^{\circ}\text{C s}^{-1}$ exists in the strip steels producing. Moreover, the ultimate strip steels are coiled and normal coiling temperature is about 600°C . However, few systematic studies have been conducted on these extrinsic factors. Concerning the effect of hot holding time, especially, almost no knowledge is available in the field of conserving the DIF. Several reports fragmentarily suggest that short hot holding might be a key to produce the UFG structure. For instance, Yang¹⁷⁾ reported that the DIF would retransform partially to the austenite during hot holding at the temperature region $A_{e3}\text{--}A_{r3}$.

In this paper, the effects of the hot holding time on the high temperature DIF as well as cooling rates and annealing temperatures, which are very important but seldom considered before, have been investigated. The thermal stability and the forming mechanism of the high temperature DIF are thus discussed.

2. Experimental Procedures

2.1. Specimens Preparation

A rough slab of the low carbon steel (Q235) was used here. The chemical composition of the steel is shown in **Table 1**. The A_{e3} temperature is 848°C , which is calculated by the ThermoCalc software. The material was homogenized at 950°C for 3 600 s and then air cooled to ensure uniform composition of the starting material. Cylindrical compression specimens with the height of 15 mm and the diameter of 8 mm were machined from the as-received slab. The microstructures of starting specimens are composed of proeutectoid ferrite and pearlite.

2.2. Hot Compression Tests

The thermal mechanic tests were performed on the thermal simulating machine Gleeble 3500 equipped with a radiant furnace and a quenching mechanism for the compressive arrangements. In order to prevent the molybdenum-based tools and the specimens from oxidation, all the tests were carried out in an argon atmosphere. The thermocouples were attached centrally to the sample surface. The temperature, load, time and longitudinal displacements were measured concurrently. The simulator was programmed to perform compression tests with constant velocity rather than constant strain rate by keeping the ram speed constant so as to produce the desired tests.

The thermal cycles used in all phases of this work were shown schematically in **Fig. 1**. After austenitized at 950°C for 300 s, the specimens were cooled to the deformation temperature $T=850^{\circ}\text{C}$ at a cooling rate of 1°C s^{-1} . Isothermally held for 30 s, the specimens were uniaxially compressed to the nominal strain $\varepsilon=80\%$ at the strain rate $\dot{\varepsilon}=20/\text{s}$. The recorded temperature–time curve show that the error of temperature was $\pm 0.5^{\circ}\text{C}$ before deformation and the temperature increased to $875\text{--}880^{\circ}\text{C}$ during the compression due to deformation heating.

Route I (Isothermally hot holding): after the deformation, the specimens were isothermally hot held for 1 s, 2 s, 5 s, 10 s, 60 s, 300 s, 3 600 s and 7 200 s respectively at the deformation temperature $T=850^{\circ}\text{C}$, and then water quenched.

Table 1. Chemical composition of a low carbon steel.

(wt %)				
C	Mn	Si	P	S
0.13	0.19	0.49	0.012	0.013

(Zhaoxia Liu, Dianzhong Li, Shanping Lu, Guiwen Qiao)

Route II (Annealing): the specimens were quenched after deformation, then isothermally tempered at 200°C , 300°C , 400°C , 500°C , 600°C and 700°C for 3 600 s respectively.

Route III (Cooling at different rates): the specimens were controlled cooling after deformation at 1°C s^{-1} and $10^{\circ}\text{C s}^{-1}$, air cooling at a rate of about $25^{\circ}\text{C s}^{-1}$, and quenching at a rate of $1\,500^{\circ}\text{C s}^{-1}$.

The compression specimens were bisected perpendicular to the compressive axis. And then the center section was ground, polished, and etched with a 3% nital solution. The microstructures of the section were observed by optical microscope (OM) and scanning electron microscope (SEM). The grain size was defined as the diameter of a circle with equivalent area and performed on leica Q Grain application systems from leica Microsystems Imaging solutions Ltd. Cambridge, UK.

3. Results and Discussion

3.1. Isothermally Hot Holding

Figure 2 represents the microstructures of the specimens isothermally held for different time after a hot deformation under the nominal strain $\varepsilon=80\%$ and the strain rate $\dot{\varepsilon}=20/\text{s}$ at deformation temperature $T=850^{\circ}\text{C}$. Examination of the microstructures by OM shows a quite distinctive microstructure evolution depending on the hot holding time after deformation. **Figure 2(a)** shows the microstructure of the specimen isothermally held for 1 s after deformation, which is composed of the ferrite grains and lath martensite. The white areas denote the ferrite grains, and dark areas martensite. The ferrite grain size is fine (about $2\text{--}3\,\mu\text{m}$), some even less than $1\,\mu\text{m}$ and homogenous. Polygonal and equiaxed ferrite grains occupy the most part of the specimen. The ferrite grain size in **Fig. 2(a)** is slightly bigger and volume fraction of DIF is slightly less than that without hot holding after deformation. **Figures 2(b)** and **2(c)** show the specimens isothermally held for 2 s and 5 s after deformation respectively. It is easy to find that DIF grain size increases obviously and volume fraction decreases with increasing hot holding time. Because the DIF transformation takes place at a high temperature (above A_{e3} temperature), where the free-Gibbs energy of the austenite is lower than that of ferrite, the austenite is more stable than the ferrite in the undeformed specimens. At the large nominal strain $\varepsilon=80\%$ and strain rate $\dot{\varepsilon}=20/\text{s}$, a significant energy is stored in the material. To lower the system energy, the DIF transformation takes place. The DIF is metastable and readily to retransform to the austenite. Therefore, the DIF volume fraction decreases. On the other hand, high temperature defor-

Fig. 1. Schematic diagrams of experiment schemes: (a) isothermally hot holding at deformation temperature $T=850^{\circ}\text{C}$ for different time, (b) annealing at different temperatures for 3 600 s, (c) cooling at different rates.

Fig. 2. Microstructure changes of the low carbon steel Q235 held for different time during deformation at $T=850^{\circ}\text{C}$, $\dot{\epsilon}=20/\text{s}$ and $\epsilon=80\%$ (a: 1 s, b: 2 s, c: 5 s, d: 10 s, e: 60 s, f: 300 s, g: 3 600 s, h: 7 200 s).

mation and holding are beneficial to carbon diffusion and accelerates grain growth. Then the ferrite morph is also changed accompanying the further grain growth. Figures 2(d)–2(g) show the microstructures of the specimens isothermally held for 10 s, 60 s, 300 s and 3 600 s respectively after the deformation. As illustrated in Fig. 2(d), a microstructure consisting mostly of allotriomorphic ferrite and martensite is produced when the specimen is held for 10 s. An unusual ferrite structure, allotriomorphic ferrite, forms when the specimen is held for 60 s as shown in Fig. 2(e). Numerous Widmanstätten ferrite plates are also found as the hot holding time prolonging (Figs. 2(e)–2(g)) together with the decreasing of allotriomorphic ferrite. The transition from allotriomorphic to plate-like Widmanstätten ferrite is determined by changes in the relative proportions of partially coherent and disordered regions of interface surrounding the ferrite. It is not surprising that after 7 200 s of the isothermally hot holding as shown in Fig. 2(h), there exists almost no ferrite but the austenite as equilibrium theory expected.

With the hot holding prolonging, the phenomenon of bimodal grain size distribution which is normal at temperatures between A_{e3} and A_{r3} ^{18,19)} does not take place above A_{e3} . Formation of various types of microstructures, such as allotriomorphic ferrite, Widmanstätten ferrite and the austenite, rather than single DIF indicates that DIF main-

Fig. 3. Microstructure evolution of the low carbon steel Q235 tempered at different temperatures for 3 600 s: (a) 200°C , (b) 300°C , (c) 400°C , (d) 500°C , (e) 600°C and (f) 700°C .

tains for a very short time when the deformation temperature is above A_{e3} . However, the used hot holding time after rolling in the steels is so short (about 2–5 s) compared with the experimental conditions (10 s, 60 s, 3 600 s, and 7 200 s *etc.*), it is possible to ascertain whether there exists the DIF in the finally products on real practice by heat treatments. But it still deserves to note that in practical bulk hot mills, there is a delay between deformation and subsequently cooling about 2–5 s as previously mentioned. Much ferrite fraction in ultimate microstructure is not formed during the deformation, but decomposed by the austenite during the laminar flow cooling. Therefore, it is controversial whether the properties as reported by previous researchers are totally of DIF steel. For example, Choi and co-workers²⁰⁾ have found that the hardness distribution is not uniform for existing proeutectoid ferrite by nanoindentation techniques.

3.2. Annealing

Figure 3 shows the microstructures of DIF specimens tempered at different temperatures for 3 600 s. The microstructure is composed of a great deal of DIF, a little of martensite and pearlite at low annealing temperature (200°C), the ferrite and pearlite at medium annealing temperatures (200 – 500°C), and the ferrite and globular cementite particles at high annealing temperatures (500 – 700°C). **Figure 4** shows the DIF grain growth during annealing. The curve can be divided into three phases: rapid increasing at first ($<200^{\circ}\text{C}$), enormous stability at medium annealing temperatures (200 – 500°C) and increasing again at high annealing temperatures (500 – 700°C). The initially increasing of the grain size attributes to the UFG, which represents a very high energy density in grain boundary area, and thus a high interfacial stored energy. Therefore, a very rapid grain growth would be presented in the DIF specimens tempered

Fig. 4. Ferrite grain size evolution with the various annealing temperatures for 3 600 s.

Fig. 5. Microstructure changes under SEM of the low carbon steel Q235 annealing at different temperatures for 3 600 s: (a) 600°C and (b) 700°C.

at low temperatures. Enormous stability at medium annealing temperatures is due to the recovery process and martensite decomposition. In the recovery process the subgrains form within the deformed matrix and grow, thus the dislocation density decreases due to the reduction of subgrain boundaries area and, finally, formation of high angle grain boundaries. Because of the martensite decomposition, a fine dispersion of cementite particles presents, these particles can lead to a high dragging force for the migration of high angle grain boundaries due to Zener pinning of the boundaries that increases the recrystallization temperature. However, when the specimens were tempered at relatively high temperatures (500–700°C) as shown in Figs. 3(e) and 3(f), the increase of the ferrite grain growth occurs again. It is because of the coarsening of the cementite particles, leading to a reduction of the Zener drag effect, although cementite particles produce a stabilizing effect on the fine grained ferrite matrix. Microstructure under high magnification in Fig. 5 showed that it is composed of the ferrite and randomly distributed cementites. Some ferrite grains contain so many cementite particles, and others contain none. It is easy to differentiate that the cementite particles intend to concentrate on the grain boundaries or triple points. Figure 5(b) shows the ferrite and relatively coarser cementite compared with those in Fig. 5(a). But the cementite particles are more homogeneously distributed. Although the grain growth is obvious at relatively high annealing temperatures, a uniform dispersion of fine cementite particles within the ferrite grains is more desirable for strengthening for the DIF with fined grain size of 5 μm after tempered at 700°C for 3 600 s.

3.3. The Cooling after Deformation

Figure 6 shows the microstructures changes under SEM of specimens quenched and controlled cooling at different rates after the deformation. Figure 6(a) shows the speci-

Fig. 6. Microstructure changes of the low carbon steel Q235 deformed at $T=850^{\circ}\text{C}$, $\dot{\epsilon}=20/\text{s}$, $\dot{\epsilon}=80\%$ and at different cooling rates (a) quenching at a rate of about $1500^{\circ}\text{C s}^{-1}$, (b) air-cooling at a rate of about $25^{\circ}\text{C s}^{-1}$, (c) controlled cooling at a rate of $10^{\circ}\text{C s}^{-1}$, (d) controlled cooling at a rate of 1°C s^{-1} .

mens that are quenched immediately after the deformation. The microstructure is composed of very fine ferrite grains. Polygonal and equiaxed ferrite grains occupy great part of the DIF specimen. There is no cementite precipitated in the specimen resolved by SEM. In contrast, the coarser ferrite and a light-etching phase identified as iron carbide (cementite) as shown by arrow are found in air-cooled specimens in Fig. 6(b). It should be noted that the mean ferrite grain size is not changed obviously. As reported in Refs. 20)–22), the DIF is not stable for high carbon concentration compared with proeutectoid ferrite. The carbon will diffuse during the cooling. Then, the cementite will form when carbon diffuses sufficiently to grain boundaries and triple points, where dense dislocations congregate. The cementite will act as Zener pinning to inhibit the grain growth. With the cooling rate decreasing, the ferrite grain size increases and the ferrite volume fraction decreases. Figures 6(c) and 6(d) show the microstructure is composed of ferrite and light-etching phase identified as lamellar pearlite. Yang *et al.*^{23,24)} found that no pearlite has been observed in DIF specimens, even though the percentage of the DIF was up to 95%. In our quenching specimens, there is no pearlite, either. Then, the pearlite and ferrite in Figs. 6(c) and 6(d) are decomposed by newly formed austenite during cooling. This is because that the time from A_{e3} to A_{r3} is long enough for retransformation of the ferrite to austenite when the cooling rate is lowered to 10°C/s and 1°C/s . Therefore the ferrite is not DIF any more, but is proeutectoid ferrite. For the stored energy has been depleted by DIF transformation and the retransformation from DIF to austenite, it is reasonable that the grain size of proeutectoid ferrite will be same to the normal ferrite decomposed by undeformed austenite.

Since decreasing cooling rates will prolong the staying time between A_{e3} and A_{r3} , the retransformation from the DIF to austenite will happen. And the newly formed austenite will likely decompose into proeutectoid ferrite during cooling. Thus the normal laminar flow cooling in actual strip steel schedule is not easy to realize by the DIF transformation methods. Increasing the cooling rates in

post-treatments is especially important to conserve the DIF.

4. Conclusions

The thermal stability of high temperature DIF is rather low. By the DIF transformation method, it is hard to obtain UFG DIF in bulk steels by the usual post-treatments for the following points:

(1) Increasing hot holding time, the grain size of the DIF increases and the volume fraction decreases obviously. When hot holding time is longer than 10 s, various types of ferrite as allotriomorphic ferrite and Widmanstätten ferrite, appear. If hot holding time is longer than 7 200 s, the ferrite will be totally retransformed to the austenite.

(2) The microstructure is composed of the DIF, a little of martensite and pearlite at low annealing temperature (200°C), the ferrite and pearlite at medium annealing temperatures (200–500°C), and the ferrite and globular cementite particles at high annealing temperatures respectively (500–700°C). The ferrite grain growth is rapid both at low and high annealing temperatures, but relatively stable at medium annealing temperatures.

(3) Compared with the microstructure of DIF and lath martensite in the quenched specimens, the specimens at low cooling rates of about 1°C/s and 10°C/s composes of proeutectoid ferrite and pearlite. There is no obvious ferrite grain growth occurring in air cooled specimens at a rate of about 25°C/s for cementite precipitating.

Acknowledgement

This work is supported by National Natural Science Foundation of China (No. 50471073).

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