Martensite in interstitial-free steel obtained by ultra-high pressure

Jun-Yun Kang, a Seul Cham Kim, b Jeang-Ook Oh, c Heung Nam Han, b,* Kyu Hwan Oh b and Hu-Chul Lee b

a Korea Institute of Materials Science, 797 Changwon-daero, Changwon, Gyeongsangnam-do 641-831, Republic of Korea
b Department of Materials Science and Engineering and Center for Iron & Steel Research, RIAM, Seoul National University, 599 Gwanak-ro, Gwanak-gu, Seoul 151-744, Republic of Korea
c ILJIN Diamond Co. Ltd., 614-2, Oryu-ri, Daeso-myun, Eumsung-gun, Chungcheongbuk-do 369-820, Republic of Korea

Received 19 August 2011; revised 3 October 2011; accepted 3 October 2011
Available online 8 October 2011

An interstitial-free steel was austenitized and cooled in a high-pressure high-temperature apparatus. Despite its inherently negligible hardenability, an interstitial-free steel with a full martensitic structure could be obtained even without quenching. Under atmospheric pressure, the microstructure after austenitization and cooling at an even faster rate consisted of massive ferrite. The thermodynamic stabilization of austenite by high pressure (on the gigapascal scale) led to great retardation of ferrite transformation and resulted in a rare microstructure of martensite in interstitial-free steel.

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Keywords: IF steel; Martensite; Pressure; Transformation; High-pressure high-temperature (HPHT)

Interstitial-free (IF) steel is a steel product in which the interstitial elements C and N are scavenged by the controlled addition of Ti or Nb. The formation of Ti- or Nb-carbonitride precipitates makes the steel matrix almost free of solute interstitials. Besides the scavenging of solute C and N, IF steels inherently contain a very small amount of the interstitial elements in their nominal composition [1]. In general, the content of other substitutional elements is also relatively very low. Although their strength is generally low because of the very lean alloying, cold-rolled and annealed IF steel sheets have excellent formability and especially superior drawability by virtue of their optimal recrystallization textures. Therefore, they have been widely used in the outer shells of cars, household appliances and beverage cans [1–3]. Because of the lean alloying, the hardenability of IF steels should be extremely shallow. By conventional water quenching, therefore, it is generally impossible to observe martensitic transformation in IF steels. Indeed, martensitic structures in IF steels of rather high alloying were observed after rapid quenching with iced brine or water. Martensitic IF steels show greater microstructural refinement [4,5], and this could compensate for their deficit in strength. In this study, an attempt was made to obtain a martensitic structure in IF steels at a rather slow rate of cooling with the aid of ultra-high pressure.

The chemical composition of the IF steel used in this study is presented in Table 1. This steel was designed to have more strength than a typical grade of IF steels. The C and Mn contents are relatively high compared to those of typical ultra-low-carbon (ULC) products, while the C content is still low enough to be fully stabilized by the amount of Ti and Nb added. A relatively large amount of Mn and a small addition of B enhance hardenability. However, the amounts of the two elements are considerably less than those used in Refs. [4] and [5], and therefore the hardenability is also considerably less.

Specimens from a hot-rolled sheet were austenitized at high temperatures and cooled. To perform heat treatment under ultra-high pressure, we used the high-pressure high-temperature (HPHT) technology developed for diamond synthesis [6–8]. In this study, a cube-type anvil press [9] installed in the pilot plant of ILJIN Diamond Co. Ltd. was used to generate the HPHT condition. A coin-shaped specimen with a diameter of 30 mm and a
A thickness of 4 mm was inserted into a reaction cell designed for sintering of polycrystalline diamond composite. The structure of the reaction cell is illustrated in Figure 1. It was pressed on all six surfaces with 85 MPa of pressure by the anvils while it was heated to 1200 °C at 10 °C s⁻¹, held for 10 min, then cooled to room temperature at 8 °C s⁻¹. As the NaCl in the cell melted, the reaction cell experienced an internal hydrostatic pressure of approximately 5 GPa due to the volume expansion of NaCl in melting. After cooling was complete, the external force was removed, the cell was disassembled and the coin-shaped specimen of IF steel was finally retrieved. During the process, the specimen was encapsulated in Ta foil, which prevented any contamination from contact with other materials during the process. However, because Ta is a strong carbide former, it could also scavenge the solute C from the specimen to form a Ta-carbide film on the surface, resulting in a reduction in hardenability.

The microstructure at the mid-thickness region of the specimen core was examined using electron backscattering diffraction (EBSD) and a transmission electron microscopy (TEM). It was also compared with microstructures produced under atmospheric pressure. Figure 2 shows the microstructure of the specimen after the HPHT process. In Figure 2(a), the prior austenite boundary and the subdivision of the prior austenite grains by blocks and packets of laths can be recognized. The average diameter of prior austenite grains was estimated to be 186 µm. In Figure 2(b), a more detailed morphology of individual laths with dislocation tangles can be also observed. These correspond well to the typical characteristics of lath martensite [10]. From Figure 2(c), it is clear that a large proportion of the packet boundaries shows a twin relationship, which has also been reported as a characteristic of low-carbon martensite [11,12]. From these features, it can be concluded that full-lath martensite has been obtained in an IF steel with extremely shallow hardenability, even without quenching, by the use of ultra-high pressure.

Figure 3 shows typical microstructures of the same steel after austenitization under atmospheric pressure. Figure 3(a) and (b), which resulted from different cooling rates (8 and 50 °C s⁻¹ respectively), show essentially the same character. They do, however, show a large variation in grain size and highly irregular grain shapes with ragged boundaries. Moreover, many grains contain sub-boundaries and consequently exhibit internal orientation gradients due to the dislocations introduced during transformation. This type of microstructure can be regarded as a massive or quasi-polygonal ferrite, which is a higher-temperature product of austenite decomposition than martensite and is frequently encountered in ULC steels [12–15]. It can also be observed that further subgranular structures evolve as the cooling rate is increased. However, at least up to 50 °C s⁻¹, a clearly aligned subdivision like that in lath martensite cannot be observed. It is generally considered that a martensitic structure in IF steels would be difficult to obtain under atmospheric pressure, even with more rapid cooling [14,15]. Although martensite can be found in high-purity iron with a comparable carbon content, the required cooling rate is several thousand (5000–35,000) °C s⁻¹ [16].

The hardness of the HPHT specimen was 236 HV, while that of the microstructures shown in Figure 3(a)
and (b) was only 81 and 118 HV, respectively. This hardness comparison reflects dramatic strengthening through martensitic transformation based on transformation strain and microstructure refinement. In this case, however, the supersaturation effect of C or N would be diminished.

The criticism might be made that the two specimens treated under atmospheric pressure were austenitized at a lower temperature (1150°C) than the HPHT specimen (1200°C), resulting in decreased hardenability. Strictly speaking, a direct comparison between the two types of microstructures shown in Figures 2 and 3 cannot be justified. However, the cooling rate under atmospheric pressure (50°C s⁻¹) was considerably faster than that under high pressure (8°C s⁻¹), which might counterbalance the effect of the lower austenitization temperature. In Ref. [16], little effect of austenitization temperature was reported. Meanwhile, the HPHT specimen should experience a reduction in hardenability due to the additional scavenging of C by the Ta foil. In spite of these factors, the HPHT process resulted in a martensitic structure which was clearly distinct from the massive ferrite, which provided confirmation that great retardation of ferrite transformation can be achieved using high pressure. It must be emphasized that the cooling rate in the HPHT process was far lower than in a typical quenching situation (greater than approximately 50°C s⁻¹).

Generally, as pressure increases, denser phases are favored in the transformation. From this point, it can be easily inferred that the close-packed face-centered cubic austenite is more stable than the body-centered cubic ferrite under pressure. Moreover, it can be confirmed from phase diagrams that hydrostatic pressure stabilizes austenite [17–20]. Therefore, the retardation of ferrite transformation under pressure is considered to originate from the thermodynamic stabilization of austenite, which has been intensively discussed in a number of studies [19–22]. For example, the equilibrium transformation temperatures (A₃) of high-purity irons decrease from about 910°C to 600–730°C when pressure increases from atmospheric level (about 10⁵Pa) to 5 GPa [17–20]. It is interesting that the latter temperature range falls well within the reported temperature range (540–750°C) of martensitic transformation in high-purity iron at atmospheric pressure [16]. Nevertheless, this cannot support the martensitic transformation in that temperature range under a high pressure of about 5 GPa because pressure also lowers the martensitic transformation temperature as well as the equilibrium one [20–22]. However, it is evident that the reduced driving force for the transformation greatly suppresses the ferrite formation in both aspects of transformation temperature and kinetics, by which martensitic transformation at a low temperature is preferred [20–22].

The great retardation of transformation under pressure has been shown as displacement of the transformation–time–temperature curve [20,21]. Under a very high pressure, an extremely slow cooling rate would be required to obtain a product of diffusional transformation such as polygonal ferrite, even though the cooling actually passes through the temperature regime in which the diffusional transformation can take place. In summary, a rare, fully martensitic structure of IF steel has been achieved without quenching by use of ultra-high hydrostatic pressure. The thermodynamic stability of austenite increases under high pressure, which greatly retards the ferrite transformation in an IF steel with negligible intrinsic hardenability, resulting in the martensitic structure. Currently, the industrial significance of this may be questioned. However, it is worth knowing that the effectiveness of pressure can be considered as a new parameter for microstructure control, in addition to conventional ones such as temperature and plastic strain. The feasibility of a high-pressure metallurgy based upon these observations needs to be investigated.

H.N.H. was supported by a National Research Foundation of Korea grant funded by the Ministry of Education, Science, and Technology (2011-0020493) and POSCO research program (2009Z041).