Proceedings of the 5th International Symposium on Steel Science (ISSS 2017)
Nov. 13-16, 2017, Kyoto, Japan: The Iron and Steel Institute of Japan

Effect of grain boundary on the plastic deformation in Fe-C alloys

Katsuya Nakano*[1)], Kunio Hayashi[2)], Kengo Takeda[1)], Seiichiro Ii[3)], Takahito Ohmura[3)]

[1] Steel Research Laboratories, Nippon Steel & Sumitomo Metal Corporation,
20-1 Shintomi, Futtsu, Chiba 293-8511, Japan
[2] Kimitsu R&D Laboratories, Nippon Steel & Sumitomo Metal Corporation,
1-1 Kimitsu, Kimitsu, Chiba 299-1141, Japan
[3] Research Center for Structural Materials, National Institute for Materials Science,
1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

ABSTRACT:
To investigate the effect of grain boundaries and solute carbon on plastic deformation, nanoindentation was performed on the grain interior, and near grain boundaries for interstitial free steel (IF steel) and Fe-120 at.ppmC (C120). The nanohardness of the grain interior (the distance from the grain boundary was over 2 μm) for C120 was higher than that for the IF steel. In contrast, the nanohardness increased when the distance became shorter than 2 μm. Furthermore, the nanohardness was almost the same for each sample when the distance was about 1.0 μm. It is believed that the increase of the Hall-Petch slope with carbon addition in tensile testing is caused by the increase of grain boundary strength with segregation of carbon on the assumption of the dislocation pile-up model. However, the results obtained by nanoindentation suggest that grain boundary strength is almost the same in the two steels. This result suggests that the dislocation movement is important not only near grain boundaries but also within the grain interior.

1. INTRODUCTION

In the 1950s, Hall [1] and Petch [2] reported grain size dependence of yield strength and cleavage fracture stress of iron and steel. The Hall-Petch (H-P) relation is described as:

$$\sigma_y = \sigma_0 + kd^{-1/2} \quad (1)$$

where σ_y is the yield strength, σ_0 is the friction stress, k is the H-P coefficient and d is the grain size. There are several models to explain the H-P relation, the dislocation pile-up model [1,2], the dislocation source model [3], the geometrically necessary dislocation model [4], the composite model [5], and the slip distance model [6]. However, the mechanism is under discussion.

Takeda *et al.* [7] studied effects of interstitial carbon and nitrogen on k of ferritic steels. Carbon increased k, but nitrogen hardly did. It was concluded that the difference stemmed from the number of segregated atoms to grain boundaries. Takahashi *et al.* [8] measured the amount of segregated carbon and nitrogen at grain boundaries using 3D atom probe tomography. They showed that the segregating trend of nitrogen is much weaker than that of carbon and that the increment of k from nitrogen is also lower than that from carbon. Based on the dislocation pile-up model, they concluded that carbon segregation to grain boundaries increased the boundary strength [9], whereas nitrogen segregation did not affect it.

In many previous reports, macroscopic tensile tests were performed to evaluate the H-P relation. However, the results of macroscopic tests contain the effect of several types of grain boundaries. In order to investigate an elementary step of the H-P relation, it is important to focus on a single grain boundary and evaluate the interaction between a dislocation and the grain boundary. Recently several microscopic approaches such as nanoindentation have been performed to investigate the dislocation-grain boundary interaction. In some reports, ex-situ nanoindentation was performed to evaluate the first pop-in, which corresponds to the initiation of plastic deformation [10] and the second pop-in, which corresponds to the strain transmission across the grain boundary [11-13]. In other reports, in-situ compression tests in transmission electron microscopy were conducted and the dislocation-grain boundary interaction was evaluated directly [14].

In this study, we conducted ex-situ nanoindentation and evaluated the mechanical response in

*Corresponding author. E-mail: nakano.8ak.katsuya@jp.nssmc.com, telephone: +81 07035148016.

the vicinity of a single grain boundary in order to investigate the effect of a grain boundary and segregation of carbon on the mechanical properties.

2. EXPERIMENTAL PROCEDURE

2.1. Sample

The chemical compositions of the samples are listed in Table 1. Conventional high purity iron was melted in a vacuum furnace and a 25 kg ingot was obtained. The ingot was homogenized at 1473 K for 3.6 ks and hot rolled to 5 mm. The hot rolled plate surface was ground to 3 mm and cold rolled to 1 mm. The cold rolled sheet was annealed at 973 K for 30～2000 seconds and quenched into water. The H-P coefficients measured by the tensile test were 1038 MPa・$\mu m^{1/2}$ for C120 and 627 MPa・$\mu m^{1/2}$ for the IF steel.

Table1 Chemical composition of the samples used in this study.

(mass ppm)

	C	Al	O	N	Ti
IF steel	20	40	37	8	350
C120	110	30	49	6	-

2.2. Internal friction

Internal friction measurement was performed to evaluate the solute carbon concentration. It was conducted by the free decay method using an inverted torsion pendulum machine under a 100 Pa He atmosphere. The frequency was 1 Hz, and the heating range was from 200 K to 400 K under continuous heating at a rate of 1 K/min.

2.3. Nanoindentation

The nanoindentation test was conducted on a Hysitron Triboindenter (TI900) with a Berkovich type diamond indenter tip. The samples were mechanically and chemically polished to remove the surface damaged layer because the results of nanoindentation are affected by surface roughness and surface damaged layer [15]. Indentation was performed under a load-controlled condition at 300 K. The maximum load was 1～5 mN and the loading rate was 1 mN/s. Nanoindentation was performed on the grains whose surface is normal to the <100> direction.

3. RESULTS AND DISCUSSION

3.1 Internal friction

Fig. 1 shows the relationship between temperature, T and internal friction, Q^{-1}. A peak was observed around 300～320 K. This is the Snoek peak, which is due to stress-induced reorientation of interstitial solute atoms and is described as:

$$Q^{-1} = \Delta \frac{\omega\tau}{(1+\omega\tau)^2} \quad (2)$$

where Δ is the relaxation strength, ω is the angular frequency and τ is the relaxation time. τ shows Arrhenius type temperature dependence and is expressed as:

$$\tau = \tau_0 \exp\left(\frac{E}{kT}\right) \quad (3)$$

where τ_0 is a constant, k is the Boltzman constant and E is the activation energy.

Nowick and Berry showed τ_0, E, and the relaxation strength for nitrogen and carbon per atomic ppm. [16] We calculated the concentration of solute carbon by using these values. The concentrations were 3.8 mass ppm for IF steel and 57.8 mass ppm for C120.

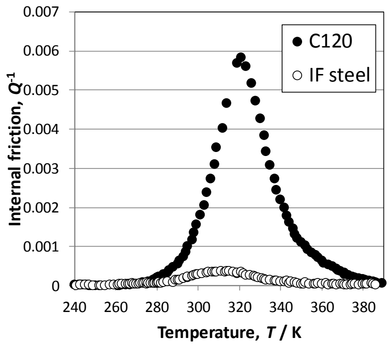

Fig.1 Relationship between temperature, T and internal friction, Q^{-1}

3.2 Nanoindentation

Nanoindentation was performed in the vicinity of a single grain boundary with various distances from the grain boundary as shown on the scanning robe microscope (SPM) image in Fig. 2 (left). The grain boundary was a high angle boundary (>15 °). Fig. 2 (right) shows a cross section profile of a height of the sample surface across the grain boundary. Since the step at the grain boundary is 52 nm, which is comparable to a typical indentation depth, the indented position was chosen at the bottom of the step in the (001) grain side to avoid errors by the free surface of the cliff.

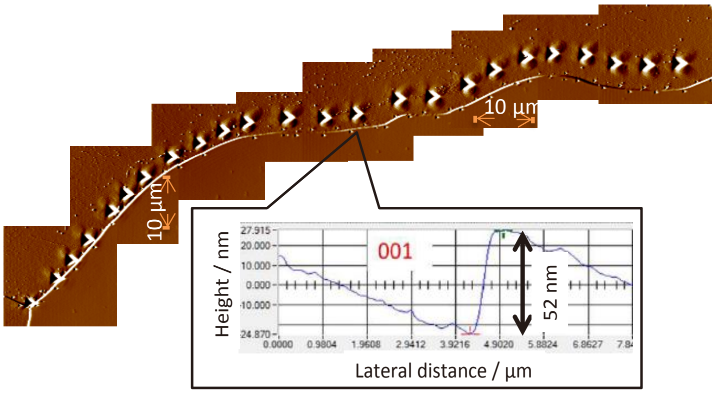

Fig.2 Scanning probe microscopy image of the sample surface after multiple indentations and the cross-section profile among the grain boundary.

Fig. 3 shows typical load-displacement curves for the IF steel. The curves include three cases of distance between the centre of the indent and the grain boundary. Three curves overlap at the initial stage below 125 nm in displacement, while the slope of the curve for 0.7 μm becomes steeper when the displacement is over 125 nm. The penetration depth after unloading is the shallowest and the hardness is the highest for the 0.7 μm case. This change in slope is presumably caused by the grain boundary effect. Using the hemispherical approximation of the plastic zone beneath the indenter [17], the diameter is estimated as 1.25 μm when the penetration depth is 125 nm. As the radius of the plastic zone is comparable to the distance between the indent and the grain boundary, the plastic zone could interact with the grain boundary.

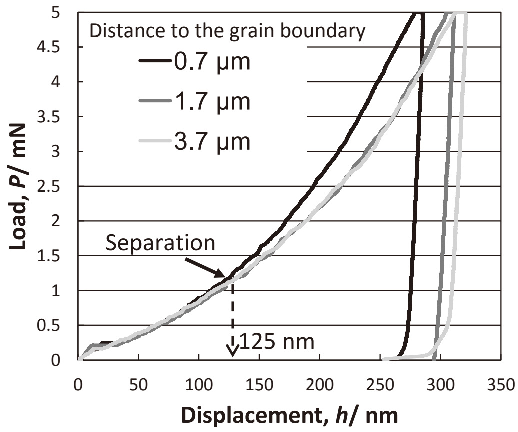

Fig.3 Load-displacement curves for the IF steel with different distances to the grain boundary (0.7, 1.7 and 3.7 μm).

Fig. 4 shows the relationship between the nanohardness and the distance between the indent and the grain boundary for the IF steel and C120. The grain boundaries include misorientation angles higher than 15 degrees and the nanohardness of near grain boundaries for both the samples is higher than those of the grain interior. The nanohardness of the grain interior (the distance to the grain boundary is 2 μm or longer) for C120 is 1.25 times higher than that for the IF steel. In contrast, the nanohardness of C120 is almost the same as the IF steel when the distance is less than 1.0 μm. This result suggests that the hardness near the grain boundary for C120 is not a simple addition of the grain boundary effect in the IF steel to the grain interior of C120.

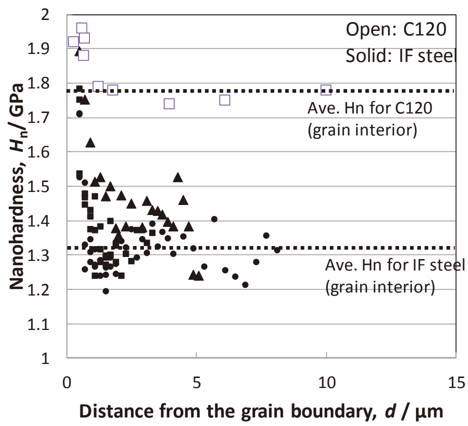

Fig.4 Relationship between nanohardness and the distance to the grain boundary.

To investigate the effect of grain boundaries, the following factors should be considered: one is the deformation inhomogeneity and another is the property of the grain boundary itself. The deformation inhomogeneity depends on the dislocation movement in the grain interior and causes the stress concentration at the grain boundary. The effect of a solute atom on the deformation inhomogeneity was reported [18]. In Fe-Si alloy, the slip plane is limited to {110} and hence cross slip is restricted, leading to a deformation inhomogeneity. In-solution carbon atoms presumably behave likewise. On the other hand, the property of the grain boundary itself corresponds to the effect of a barrier to dislocation glide or the ability of the dislocation source. The effects are presumably determined by grain boundary characters including boundary structure, misorientation angle, segregation of alloying elements and so on.

4. CONCLUSION

The mechanical response in the vicinity of a single grain boundary and the effect of solute atoms on the response were investigated through the nanoindentation technique. Nanohardness in the vicinity of a grain boundary is higher than that in a grain interior. Nanohardness in the grain interior increases with solute carbon, while the nanohardness of the near grain boundary is almost unaffected. These results indicate that it is important to consider the solute effect not only on the grain boundary itself but also on the dislocation motion in the grain interior in studying the effect of solute carbon on the H-P relation.

REFERENCES

[1] E. O. Hall: Proc. Phys. Soc. Lond. B 64 (1951), 747-753.
[2] N. J. Petch: J. Iron Steel Inst. 174 (1953), 25-28.
[3] J.C.M Li: Trans. AIME 227 (1963), 239-247.
[4] M.F. Ashby: Philo. Mag. 21 (1970), 399-424.
[5] H. Conrad: Acta Metall., 11 (1963), 75-77.
[6] U.F. Kocks: Metall. Trans. (1970), 1123-1143.
[7] K. Takeda, N. Nakada, T. Tsuchiyama, S. Takaki: ISIJ Int. Vol. 48 (2008), 1122-1125.
[8] J. Takahashi, K. Kawakami, K. Ushioda, S. Takaki, N. Nakada and T. Tsuchiyama: Scripta Mater., 66 (2012), 207-210.
[9] N. Hansen: Mater. Sci. Eng. A 409 (2005), 39-45.
[10] T. Ohmura, L. Chang, K. Sekido, and K. Tsuzaki: J. Mater. Res., 27(2012), 1742-1749.
[11] M.G. Wang and A.H.W. Ngan: J. Mater. Res. 19 (2004), 2478-2486.
[12] T.B. Britton, D. Randman, and A.J. Wilkinson: J. Mater. Res. 24 (2009), 607-615.
[13] S. Tsurekawa, Y. Chihara, K. Tashima, S.Ii, P. Lejcek: J. Mater. Sci., 49 (2014), 4698-4704.
[14] T. Ohmura, A.M. Minor, E.A. Stach, and J.W. Morris, Jr.: J.Mater. Res., 19 (2004), 3626-3632.
[15] Y.Shibutani and A.Koyama: J. Mater. Res. 19, (2004), 183-188.
[16] A.S. Nowick and B.S. Berry: Anelastic Relaxation in Crystalline Solids, Academic Press, New York, 1972.
[17] M. Itokazu and Y. Murakami: Trans. Jpn. Soc. Mech. Eng. A59 (1993), 2560-2565.
[18] B. Sestak and S. Libovicky: Acta Metall. 11 (1963), 1190-1191.