Effect of Low Temperature Aging on Hall-Petch Coefficient in Ferritic Steels Containing a Small Amount of Carbon and Nitrogen

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In this study, the effect of aging treatment at 373 K on Hall–Petch coefficient ($k_y$) was investigated by considering the change in friction stress associated with carbide/nitride precipitation in ferritic steels containing 60 mass ppm carbon or nitrogen (C60 and N60). Tensile tests revealed that the $k_y$ monotonously increased with increasing aging time in both steels. Additionally, C60 exhibited a $k_y$ value exceeding that of N60 with respect to the same aging time. The results of the 3DAP analysis and theoretical calculation for the grain boundary segregation of carbon and nitrogen indicated that the $k_y$ corresponded to the amount of carbon and nitrogen existing at the grain boundary. No difference in the effect on increments in $k_y$ between the two elements was observed. The increase in $k_y$ in C60 under the same aging condition was due to the higher amount of segregated carbon when compared with that of nitrogen.

KEY WORDS: grain boundary segregation; age hardening; grain refinement strengthening; ferritic steel; Hall–Petch relationship.

1. Introduction

In polycrystalline metals, the relation between yield strength and grain size is experimentally described by the Hall–Petch relationship1,2 as follows:

\[ \sigma_y = \sigma_0 + k_y d^{-1/2} \] ............................................... (1)

Specifically, $k_y$ in Eq. (1) is the Hall-Petch coefficient and is related to the hardening effect of grain refinement strengthening. Although the value of $k_y$ depends on the modulus of rigidity and chemical composition,3 it is known that $k_y$ is approximately 600 MPa ($\mu$m)$^{-1/2}$ in conventional pure iron and low carbon steel.4,5 However, it is reported that Interstitial Free (IF) steel without solute interstitial elements exhibits a low $k_y$ value of approximately 150 MPa ($\mu$m)$^{-1/2}$.6 The result suggests that the interstitial elements of carbon and nitrogen significantly influence the $k_y$ value even if the amount of the elements is very low. Wilson7 examined carbon and nitrogen contents dependence of $k_y$ in ferritic iron and quantitatively reported that the effect of grain refinement strengthening is significantly increased by tens of ppm of carbon because $k_y$ gradually increases with the amount of solute carbon. The authors also reported that the effect of nitrogen is lower than that of carbon although both elements are interstitial atoms. Thus, they suggested that the effect of solute atoms on $k_y$ depends on atomic species. Takahashi et al.8 examined the grain boundary segregation tendency of carbon and nitrogen in the same specimens as those examined by Takeda et al. through elemental maps with 3DAP. They reported that it is more difficult to segregate nitrogen atoms to grain boundaries and that the concentration of segregated nitrogen is significantly lower than that of carbon. The results in the segregation amounts correspond to the results for $k_y$ obtained by Takeda et al. The result strongly supports the idea that $k_y$ in ferritic steel increases by grain boundary segregation.

However, earlier studies did not quantitatively describe the dependence of the amount of grain boundary segregation. In order to understand the effect of carbon and nitrogen on grain refinement strengthening, it is necessary to reconsider the Hall–Petch relation from the viewpoint of the effect of the amounts of carbon and nitrogen in solution and also the amount of segregated carbon and nitrogen atoms at grain...
boundaries. In this study, aged specimens were prepared to control the extent of grain boundary segregation in ferritic steels containing extremely small amounts of carbon and nitrogen. Subsequently, the correlation between the number of segregated atoms and $k_y$ was quantitatively discussed. Additionally, the Hall–Petch relation was used to accurately evaluate $k_y$ by considering the change in friction stress ($\sigma$) on the right-hand side of Eq. (1).

2. Experimental Procedure

The chemical compositions of the steels used in this study are listed in Table 1. The materials are conventional pure iron containing approximately 60 ppm carbon and nitrogen (C60 and N60) to evaluate the individual effects of the atoms. A conventional Ti-added IF steel in which carbon and nitrogen are completely fixed as Ti (C, N) was used as the base material. Ingots (25 kg) with a 110 mm square cross section were homogenized at 1523 K for 3.6 ks and subsequently hot-rolled to a thickness of 10 mm. The hot-rolled plates were subjected to 90% cold rolling. The cold-rolled materials were annealed at 973 K from 0.015 ks to 3.6 ks to control the ferrite grain size and subsequently quenched into water to suppress the precipitation on cooling. (as-annealed specimen). Subsequently, the specimens were aged at 373 K from 0.6 ks to 60 ks (aged specimen), and various experiments were conducted. Additionally, the specimens were placed in a freezer at 223 K after annealing to suppress the aging at room temperature and were quickly handled by removing the required amount during the experiments.

The microstructure was observed with an optical microscope and crystallographic orientation was identified by means of electron backscatter diffraction (EBSD) method by using a scanning electron microscope (SEM). The grain size was measured by the quadrature method. Tensile testing was performed at an initial strain rate of $10^{-3}$ s$^{-1}$ for plate test pieces standardized by JIS13B at room temperature to measure the yield strength. In the study, the yield strength of specimens was defined as the lower yield stress. The change in the microstructure with aging time was observed with a transmission electron microscope (TEM), and the change in segregation behavior of carbon and nitrogen was investigated by using a three-dimensional atom probe (3DAP). Atom probe measurements were performed under the following conditions: pulse/DC voltage ratio = 20%, specimen temperature = 65 K in voltage mode and laser pulse energy = 30 pJ, and specimen temperature = 50 K with a laser displaying a wavelength of 355 nm in the laser mode.

The results confirmed that a difference in the quantitative performance between the voltage mode and the laser mode did not exist. The average amount of the grain boundary segregation was obtained by measuring the two high-angle grain boundaries. Additionally, the amount of grain boundary segregation was evaluated by the interfacial excess value that corresponds to the excess segregated atoms per unit grain boundary area. (N was identified as the peak of N$^+$ in the mass-to-charge ratio. Additionally, the electric resistance measurement was performed to estimate the precipitation behavior by using the four terminal method. The wire specimens corresponding to 50 mm$^2$×1 mm$^2$×1 mm$^2$ were used in the electric resistance measurement. The voltage was measured by changing the direction of the current and the average value of electric resistance was obtained after maintaining the specimens in liquid nitrogen (77 K) for 1 min.

3. Results and Discussion

3.1. Effect of Aging Treatment on the Hall–Petch Coefficient

As an example of the observed microstructure, Fig. 1 shows the orientation imaging map and the orientation distribution of crystal grains in the C60 and N60 annealed at

![Orientation imaging maps and inverse pole figures of C60 and N60](image)

**Table 1.** Chemical composition of specimens used in this study (mass ppm).

|   | C | N | Si | Mn | P | S | Al | O | Ti | Fe |
|---|---|---|----|----|---|---|----|---|----|----|
| IF | <10 | <1 | <30 | <30 | <20 | <10 | 50  | 18 | 240 | bal. |
| C60 | 56 | 11 | <30 | <30 | <20 | <3  | 40  | 39 | –   | bal. |
| N60 | 5  | 54 | <30 | 830 | <20 | <3  | 40  | 21 | –   | bal. |

†1 It is reported that N appears as N$^+$ and NN$^+$ with respect to the analysis of atom by 3DAP. However, a possibility exists that the amount of N is underestimated because the peak of NN$^+$ is hidden by that of Fe$^{++}$, and thus it is not possible to quantify the same. If this is true, the nitrogen contribution is considered as slightly lower than that of carbon.
973 K. Both recrystallized ferritic structures were equiaxed. The crystal orientation was randomly distributed. The grain size measured by quadrature method was approximately 18 µm in C60 and 19 µm in N60, and a significant difference between both specimens was not observed. In order to investigate the Hall–Petch relation, the grain size was increased to 55 µm by controlling the annealing time. The grains grew uniformly, and the orientation distribution of crystal grains did not significantly change. Figure 2 shows the nominal stress-strain curves in as-annealed specimens of C60 and N60 with changes in the grain size. Both specimens exhibit the clear yield point and the yield stress increases with respect to grain refinement. A comparison of the same grain size specimens of C60 and N60 indicates that the yield stress of C60 exceeds that of N60. Additionally, Fig. 3 shows the nominal stress-strain curves of C60 and N60 aged at 373 K for a grain size 30 µm. Although the yield stresses of both specimens increase with aging time and the yield point elongation also clearly increases, the yield stress of C60 is still higher.

As described above, it was revealed that the effect of carbon on the yield stress exceeds that of nitrogen, and the yield stress increases with aging treatment across all the specimens. However, friction stress (σ₀) and grain refinement strengthening (k_yd⁻¹/₂) exist in the strengthening mechanism as shown in the Eq. (1). Thus, they are separately required to evaluate the mechanism. The friction stress is the resistance force at the time of dislocation movement and is based on the solid solution strengthening of ferrite matrix only when the temperature and strain rate are constant. Therefore, the carbon and nitrogen concentration in ferritic matrix decrease when the carbide and nitride are precipitated by aging treatment. Additionally, the σ₀ also decreases. Furthermore, the possibility exists that k_y in the Hall–Petch relation changes due to the effect of σ₀. The σ₀ of each aged specimens is required to precisely estimate the change in k_y with aging though it is difficult to experimentally measure the σ₀ that corresponds to the yield stress of infinitely large grain sizes of polycrystalline metals. Thus, in this study, the carbon and nitrogen concentrations were experimentally measured, and the σ₀ was calculated by Eq. (2) as follows:

\[ \sigma_0 / \text{MPa} = 40 + 4.500 \times (X_C + X_N) \] .......... (2)

where \( X_C \) and \( X_N \) are the contents of C and N in mass%.

Cracknell calculated Eq. (2) from the value of the intercept for the Hall–Petch relation in the ferritic steel given changes in the amount of \((C + N)\) from approximately 0.004 to 0.026 mass% and increases in the nitrogen concentration due to the nitriding treatment. However, Eq. (2) does not accurately indicate the effect of solute carbon and nitrogen because precipitation is generated in the specimens. Addi-
tionally, it should be noted that the equation was derived by changing the amount of nitrogen in low carbon steel. In this study, it is assumed that Eq. (2) holds. The change in the amount of solute atoms with aging was evaluated by the electric resistance measurement\(^{13-15}\) that is detected with high sensitivity. **Figure 4** shows the change in the electric resistivity in the aged specimens (grain size: approximately 20 μm). The electric resistivity was measured at the liquid nitrogen temperature after each aging treatment. The electric resistivity of IF steel did not change with aging. Conversely, those of C60 and N60 did not exhibit significant changes until the occurrence of 6 ks aging, although they exhibited a significant decrease after the same. Thus, it is considered that the carbide and nitride were precipitated and the amounts of solute carbon and nitrogen decreased in more than 6 ks aging. In over 10\(^3\) ks, the electric resistivity was leveled, and the amount of the atoms decreased to the solid solubility limit at 373 K. The grain boundary segregation of carbon and nitrogen was also confirmed before 6 ks aging without the change in the electric resistivity as described above. However, it is assumed that the effect on the electric resistivity did not appear because the volume ratio of the grain boundary is low with respect to the grain size of 20 μm\(^{16}\) and the decrease in the solute solution in the grains by grain boundary segregation is also extremely low. **Figure 5** shows the TEM microstructure of the aged C60 and N60. The precipitates were barely observed before 6 ks aging, although the precipitates in both specimens aged for 60 ks were observed. This result matches that of the electric resistivity. The structural analysis indicated that the dendritic precipitates in C60 corresponded to cementite (Fe₃C) and that the massive precipitates in N60 were Fe₄N \(^{17}\). However, the nucleation frequency of the precipitates was not high, and they were sparsely dispersed in the matrix. Additionally, several precipitates exhibited large sizes ranging from several dozen to several hundred nm. Thus, this did not appear to significantly contribute to the strength. Specifically, when the change in hardness in grains with aging was investigated, the results confirmed that the hardening did not occur after the precipitation commenced. The hardness decreased only due to decreases in the amount of solid solution. From the above measurement results, the change in the amount of the solute carbon and nitrogen with aging were calculated. It was assumed that all carbon and nitrogen were solid-soluted in the matrix of the specimens before aging treatment because the specimens were quenched from the ferrite single phase region. Although parts of carbon and nitrogen were segregated to the grain boundary, the effect on the ferrite matrix concentration is considered as low in the range of the grain sizes used in the study (approximately 16 μm–55 μm).\(^{15}\) Additionally, it was assumed that all solute carbon and nitrogen were precipitated at the lower limit value of the electric resistivity, because the solid solubility limit of carbon and nitrogen at 373 K is extremely low and less than 1 ppm. **Figure 6** shows the amount of the solute carbon and nitrogen with respect to the aging time. It is converted to \(\sigma_0\) by substituting the calculated solute amount of each aging specimen in Eq. (2). The obtained \(\sigma_0\) of each specimen was fixed as each intercept. **Figure 7** shows the Hall–Petch relation obtained by considering a linear relationship with the least-square method. It was assumed that solid solute carbon and nitrogen are not present in IF steel, and thus the \(\sigma_0\) of IF steel was 40 MPa as indicated by Eq. (2). A linear relationship is confirmed between the reciprocal of square root of grain size and yield stress on C60 and

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\(^{12}\) In a short period of time with respect to low temperature aging, the precipitation of \(\varepsilon\)-carbide and Fe₄N are reported in previous studies.\(^{25-27}\)
N60 in all aging conditions. Thus, the yield stress of the aging specimens is determined by the Hall–Petch relation. If errors exist in the estimation of $\sigma_0$, the magnitude of the error corresponds to a maximum of approximately 25 MPa, and the change in the effect on $k_y$ is considered as low. As shown in the results in Fig. 7, the slope or the Hall–Petch coefficient in C60 and N60 exhibits a tendency to increase with aging time. Figure 8 shows the relation between $k_y$ and aging time. In the as-annealed specimens, $k_y$ of N60 is almost equivalent to that of IF steel although the $k_y$ of C60 significantly exceeds that of IF steel. The results are consistent with those obtained by Takeda et al. In both specimens, $k_y$ increases due to the aging treatment. Although the effect of nitrogen on $k_y$ was considered as low in the past, the results indicated that the effect of nitrogen is promoted by the aging treatment and that nitrogen also increases $k_y$ similar to carbon. However, the $k_y$ of C60 is higher for all aging times when both specimens are compared. It should be noted that only a slight change was observed in the electric resistivity of both specimens by 6 ks aging with respect to the result of electric resistivity (Fig. 4) although the $k_y$ values significantly increase. The facts suggest that the carbide and nitride are independent factors with respect to increases in $k_y$, and the grain boundary segregation by a low amount of the solute carbon and nitrogen increases the grain refinement strengthening ability.

### 3.2. Relation between Hall–Petch Coefficient and the Amount of Grain Boundary Segregation

The pile-up model is considered as a theory to explain grain refinement strengthening in polycrystalline metals. The movement of the dislocation generated from dislocation source ceases at the grain boundary, and the dislocation array is formed by the pile-up of the subsequent dislocation in the rearward. The model is based on the idea that the stress by the dislocation array is concentrated to the grain boundary and the yielding phenomenon occurs by generating the dislocation from the grain boundary when the increase in stress exceeds the critical value required for secondary dislocation release from the grain boundary. Wilson et al. investigated the dislocation behavior in the yielding and reported that the grain boundary play a dual role as a barrier of the dislocation movement and the dislocation source. They also reported that the Hall–Petch coefficient increases due to the grain boundary segregation of carbon with the aging treatment. The reason for the increase in $k_y$ in this study is considered as the effect of the grain boundary segregation. Therefore, the fact that the segregation tendency of carbon exceeds that of nitrogen successfully
In a previous study, the authors investigated the amount of grain boundary segregation in as-annealed specimens of C60 and N60 with atom probe tomography (APT) and subsequently reported that the amount of grain boundary segregated carbon in C60 was approximately $7.6 \times 10^{-18}$ m$^{-2}$ and that of nitrogen in N60 was approximately $2.1 \times 10^{-18}$ m$^{-2}$ when the amount was expressed in terms of the number of atoms per unit grain boundary area of 1 nm$^2$. Additionally, the authors reported that 5 ppm carbon that is inevitably mixed caused the grain boundary segregation of $1.6 \times 10^{-18}$ m$^{-2}$ and the segregation tendency of carbon was significantly higher than that of nitrogen. Given that differences in the segregation amount persist between carbon and nitrogen at the time of annealing, there are differences in the grain boundary segregation behavior between both atoms with aging. Thus, the amount of the grain boundary segregation in the aged specimens was also quantitatively evaluated with APT. As an example of the analysis results, Fig. 9(a) shows the 3D elemental maps of carbon and nitrogen in C60 aged for 6 ks, and Fig. 9(b) shows the measurement results of the amount of grain boundary segregation in C60 and N60 of the as-annealed and those aged at 373 K for 6 ks. The vertical axis indicates that the segregation amount is the Interfacial excess. In the 373 K-6 ks specimens, the precipitation was not conformed although the $k_y$ values increased to $5.8 \times 10^{-18}$ m$^{-2}$, which is approximately thrice that of the as-annealed specimen. Thus, the increase in grain boundary segregation amount corresponds to that of $k_y$, and the results strongly support the idea that $k_y$ increases with the grain boundary segregation of carbon and nitrogen. Figure 10 shows the relationship of the total amount of grain boundary segregation of carbon and nitrogen with $k_y$. The $k_y$ and the segregation amount with respect to four points of C60 and N60 in figure are the measured values. The two points of C30 (Fe-28 mass ppm C-11 mass ppm N) and N30 (Fe-11 mass ppm C-24 mass ppm N) are shown as reference data, and the $k_y$ reported by Takeda et al. were adopted, and the values estimated by McLean’s formula as shown below correspond to the grain boundary segregation amount.

$$X_i = \frac{X_i^0 - \sum_{j=1}^{M-1} X_j^0}{\sum_{j=1}^{M-1} X_j} = \frac{X_i}{1 - \sum_{j=1}^{M-1} \exp \left(-\frac{\Delta G_j}{RT} \right)} \cdots (3)$$

$X_i$ denotes the solute concentration [at%] of the atom $i$, $X_i^0$ denotes the grain boundary concentration [at%], $X_i^*$ denotes the maximum segregation concentration [at%], $\Delta G_i$ denotes the Gibbs free energy of segregation, $R$ denotes the gas constant, and $T$ denotes temperature. As shown in Fig. 10, $k_y$ increases with the grain boundary segregation amount (C + N). The results indicate that $k_y$ is organized uniformly as a function of the total amount of carbon and nitrogen, thereby suggesting that the difference of grain boundary strengthening ability between carbon and nitrogen is low. Thus, the reason as to why the $k_y$ of C60 exceeds
that of N60 in the same aging conditions is potentially because the amount (C + N) of grain boundary segregation differs across the specimens. However, the grain boundary tendency of C significantly exceeds that of N, and the segregation behaviors in both specimens are different. The reason for this is that there exists a possibility for the difference in the driving force for segregation due to the solubility limit\(^{29}\) or the interaction between the grain boundary and the atoms of carbon and nitrogen. Conversely, approximately 0.08 mass% of Mn is mixed in N60 as an impurity at the time of melting. Therefore, studies examining the interaction of Mn–C\(^{21-23}\) suggest that it is difficult to conclude that the Mn–N attractive interaction\(^{24}\) does not affect the behavior of segregation and precipitation. It is not possible to ignore the effects of substitutional atoms on the diffusion of low amounts of interstitial atoms in iron lattice even in low amounts. However, this should be preceded by clarifying the effect of the substitutional atoms with respect to the Hall–Petch relation.

4. Conclusion

In this study, the effects of low temperature aging at 373 K on the Hall–Petch coefficient (\(k_y\)) of ferritic steel with approximately 60 ppm carbon or nitrogen are investigated. Additionally, the relation with the grain boundary segregation behavior was discussed. Thus, the following conclusions were obtained.

(1) The Hall–Petch coefficient (\(k_y\)) of as-annealed nitrogen steel was very low and at the same level as that of IF steel. However, the Hall–Petch coefficient (\(k_y\)) of carbon steel clearly exceeded that of IF steel. Additionally, \(k_y\) in both specimens increased due to the aging treatment and the \(k_y\) of carbon steel always exceeded that of nitrogen steel at the same aging treatment conditions.

(2) The increase in \(k_y\) with aging is caused by the grain boundary segregation of carbon and nitrogen, and the tendency corresponds to the change in the amount of grain boundary segregation. Although the effects of carbon and nitrogen on \(k_y\) are equivalent, the \(k_y\) value is determined by the total segregation amount of carbon and nitrogen (C + N [nm\(^{-2}\)]).

(3) The reason that the \(k_y\) value of carbon steel exceeds that of nitrogen steel at the same aging conditions is potentially because the grain boundary segregation tendency of carbon is higher than that of nitrogen. Additionally, the segregation amount of carbon exceeded that of nitrogen across all aging times.

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