Effect of Aging on Low-temperature Tensile Properties of Ultra-low Carbon Steel

Norimitsu KOGA,^{1)*} Yuki KANEHIRA,²⁾ Pham Thi Thanh HUYEN,²⁾ Kazuya HORI²⁾ and Osamu UMEZAWA³⁾

1) Faculty of Mechanical Engineering, Institute of Science and Engineering, Kanazawa University, Kakumamachi, Kanazawa, Ishikawa, 920-1192 Japan.

2) Graduate School of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya, Yokohama, 240-8501 Japan.

3) Faculty of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya, Yokohama, 240-8501 Japan.

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The microstructural changes and low-temperature tensile properties of ultra-low carbon steel aged at 443 K were examined, and the relationship between the low-temperature tensile properties and ultra-low carbon state was discussed. Fine cementites of approximately 60 nm were observed at 0.6 ks and coarsened to approximately 800 nm at 600 ks. The yield and tensile stresses at 77 K increased until 6 ks and then decreased. The nominal stress-strain curves of all the specimens at 77 K exhibited low elastic limits, the nominal stress plateaued from an approximate nominal strain of 0.002 and, subsequently, work hardening occurred. In the unaged and 6 ks aged specimens, several twins were generated after the elastic limit, and which increased dramatically in the nominal stress plateau regime, and corresponded to macroscopic yielding. In contrast, the number of twins in the 600 ks aged specimen negligibly increased during macroscopic yielding. Macroscopic yielding occurred in the unaged and 6 ks aged specimens by deformation twinning, while in the 600 ks aged specimen it occurred by slip deformation. In the 6 ks aged specimen, the fine cementites and/or decrease in solid solute carbon enhanced the critical resolved shear stresses of deformation twinning, resulting in the highest strength. In the 600 ks aged specimen, the coarse cementites negligibly enhanced the critical resolved shear stress for slip deformation. Hence, the strength of the specimen aged for 600 ks decreased as compared to the specimen aged for 6 ks, and slip deformation occurred.

KEY WORDS: ultra-low carbon steel; aging; precipitate; low-temperature tensile property; twin.

1. Introduction

Carbon is the most important alloying element for strengthening conventional steels and strongly affects their mechanical properties, even in very small amounts such as tens of ppm. Bake hardening steels use carbon to improve their mechanical properties.^{1–3)} In particular, strain aging occurs during the paint-baking procedure of stamped bake-hardening steels and improves the hardness. Maruyama *et al.*⁴⁾ investigated the segregation of carbon atoms to dislocations and the carbide precipitation behavior during strain aging of ultra-low carbon steels with 43 ppm. They concluded that the amount of carbon atoms segregating to dislocations was approximately 5 ppm, and the strength of the steel is mainly enhanced by carbide precipitates. It has also been reported that ultra-low carbon significantly affects the Hall–Petch coefficient of ferritic steel.⁵⁾ Segregation of carbon atoms to the grain boundary occurs during

low-temperature aging in ultra-low carbon steels, enhancing the grain boundary strength, and hardening the steel through an increase in the Hall-Petch coefficient.^{6,7)} At high temperatures, ultra-low carbon also plays an important role in improving the mechanical properties. Using a small ball rebound hardness test,⁸⁾ Koga *et al.*⁹⁾ demonstrated that a solid solution of ultra-low carbon (approximately 100 ppm) in the matrix significantly enhanced the high-temperature hardness of steels with various carbon concentrations. The effect of ultra-low carbon on the mechanical properties of steels above room temperature has been widely investigated; however, it is still unclear whether ultra-low carbon influences the mechanical properties at low temperatures.

Ultra-low carbon steels consist of a ferrite (bcc) single phase and exhibit strong temperature-dependent mechanical properties owing to their crystal structure.¹⁰⁾ The strength of ferritic steel significantly increases with decreasing temperature, while the elongation drastically decreases at a critical temperature and brittle fracture occurs. There are 48 slip systems, such as $\{110\} \langle 1-11 \rangle$, $\{112\} \langle 11-1 \rangle$, and

* Corresponding author: E-mail: koga-norimitsu@se.kanazawa-u.ac.jp

{123}<11-1>, in ferritic steels. Above room temperature, the critical resolved shear stress is equal among these slip systems, and various slip systems can operate. However, at low temperatures, the critical resolved shear stress of the {110}<1-11> slip system is lower than that of the other slip systems.¹¹⁾ Thus, the 48 slip systems are restricted to only 12 slip systems at low temperatures. Furthermore, deformation twinning occurs at low temperatures in ferritic steel.¹²⁾ At room temperature, the stacking fault energy in ferritic steels is relatively high and deformation twinning does not occur. However, at low temperatures, deformation twinning occurs and causes brittle fracture through cleavage cracking at the grain boundaries induced by twins.¹³⁾ On the other hand, some alloying elements in ferritic steels, such as Ni and Mn, provide solid solution softening at low temperatures in ferritic steels, even though they enhance strength at room temperature.¹⁴⁾ The mechanical properties, deformation behavior, and effect of solid solute alloying elements on the strength of ferritic steels at low temperatures are significantly different from those at room temperature.

In this study, the microstructural changes and low-temperature tensile properties of ultra-low carbon steel with aging treatment were examined and the relationship between the low-temperature tensile properties and the ultra-low carbon state were discussed.

2. Experimental Procedure

2.1. Material

This study used an ultra-low carbon steel with a carbon concentration of 75 mass ppm. **Table 1** presents the chemical composition of the steel. The steel was cold rolled to a 90 % reduction in thickness, annealed at 973 K for 1.8 ks, and then water-cooled. The annealed specimens exhibited equiaxial recrystallized grains with an average grain size of approximately 50 μm . The annealed specimens were aged at 443 K for 0.06–600 ks and subsequently water-cooled. The aged specimens were maintained at 77 K in liquid nitrogen to prevent further aging at room temperature.

2.2. Tensile Tests

Tensile test specimens with a gage length of 20 mm, width of 4 mm, and thickness of 2 mm were cut from plates where the tensile direction was parallel to the rolling direction (RD). Tensile testing was conducted at an approximate initial strain rate of $5.2 \times 10^{-4} \text{ s}^{-1}$ at 293 K in air and at 77 K in liquid nitrogen. The tensile tests were initiated after the specimens were maintained at the test temperature for 1.8 ks.

2.3. Microstructural Analysis

The microstructure on the surface of the normal direction (ND) specimen was observed using field-emission scanning electron microscopy (FE-SEM). The specimen was polished

with colloidal silica and etched with 3% nital. Electron back-scattered diffraction (EBSD) with FE-SEM was used to analyze the crystal orientation in the ferrite matrix and twins. Data were recorded with the beam scanning step of 0.05 μm . Only data points with a confidence index exceeding 0.1 were used for the crystal orientation analysis using OIMTM software (version 7.0.1). A twin-jet electro-polisher, using methanol-perchloric acid with a volume ratio of methanol, 1-butanol, and perchloric acid of 12:7:1 at 233 K, was employed to prepare the specimens for transmission electron microscopy (TEM) observations.

2.4. Electrical Resistivity Measurements

The electrical resistivity was measured by direct-current four-terminal sensing. The test specimen was cut longitudinally parallel to the RD to obtain samples of 22 mm in length, 10 mm in width, and 2 mm in thickness. The voltage at 77 K was measured while varying the current between 100 and 500 mA, and the electrical resistance was calculated. The electrical resistivity was then calculated from the electrical resistance based on the specimen shape.

2.5. Internal Friction Measurements

Internal friction measurements on 60 mm $\times$ 10 mm $\times$ 1.0 mm samples were conducted to estimate the solute carbon concentration. The internal friction was measured every 5 K from 293 to 473 K with the resonant frequency of 170 Hz using an internal friction measurement device with free vibration (JE-HT, Nihon Techno-Plus Corp.). The measurement time at each temperature was approximately 60 s, and the total measurement time was approximately 1.8 ks. The temperature corresponding to the peak internal friction was approximately 373 K.

2.6. Digital Image Correlation Method

Digital image correlation analysis was performed on the SEM images before and after deformation with a subset of 41 pixels and a step of 3 pixels. VIC-2D software (Correlated Solutions Inc.) was used for the analysis.

3. Results

3.1. Microstructure of Aged Ultra-low Carbon Steel

Figures 1(a) and **1(b)** show the maximum internal friction and electrical resistivity as a function of aging time at 443 K, respectively. The changes in the maximum internal friction and electrical resistivity against aging time were identical, where both values decreased until 60 ks and then stabilized. The maximum internal friction value strongly correlated with the solute carbon concentration in the ferrite matrix.^{15,16)} Electrical resistivity is influenced by many factors, such as the dislocation density,¹⁷⁾ amount of precipitates,¹⁸⁾ and grain size.¹⁹⁾ However, the main factor influencing the electrical resistivity in this analysis is the solid solute carbon concentration because the aging temperature was significantly lower than the temperature at which grain growth or recovery of dislocations occurs. Therefore, as shown in Fig. 1, the solid solute carbon in the ferrite matrix continuously decreased; specifically, the precipitate continuously increased until 60 ks and then stabilized.

Figures 2(a)–2(f) display TEM images of the specimens

Table 1. Chemical composition of the ultra-low carbon steel (mass%).

C	Si	Mn	P	S	Al	N	Fe
0.0075	<0.003	<0.003	<0.002	<0.0003	0.006	0.0006	bal.

aged at 443 K for 0, 0.06, 0.6, 6, 60, and 600 ks, respectively. Precipitates were not observed at 0 and 0.06 ks (Figs. 2(a) and 2(b)). Considering that the solid solute carbon concentration decreased at 0.06 ks, as shown in Fig. 1, fine precipitates and clusters could be present or segregation of carbon atoms to dislocations and/or grain boundaries could have occurred. Meanwhile, precipitates of approximately 60 nm in length were observed at 0.6 ks (Fig. 2(c)). These precipitates grew with increasing aging time (Figs. 2(d)–2(e)) and were approximately 800 nm in length at 600 ks. The precipitate thickness was approximately 25 nm, even

at 600 ks. As the solid solute carbon concentration continuously decreased until 60 ks, the nucleation and growth of precipitates was assumed to have occurred until 60 ks, after which Ostwald ripening occurred. **Figure 3** shows an enlarged TEM image of the precipitates in the specimen aged at 443 K for 600 ks. The precipitates exhibited a plate-like shape, indicating that the specific precipitation/ferrite interfacial boundaries preferentially grew. The angle between the precipitates was approximately 70° . The plane of the ferrite matrix at the ferrite/precipitate interfacial boundary was $\{110\}_\alpha$. These results are consistent with the characteristics of cementites.²⁰⁾ Furthermore, cementites have been observed in similar alloys under identical aging conditions.⁴⁾ Therefore, these were considered to be cementite.

3.2. Tensile Properties at 293 K and 77 K in Aged Ultra-low Carbon Steel

Figures 4(a) and 4(b) present the nominal stress-nominal strain (SS) curves at 293 K and 77 K, respectively, for the specimens at various aging times. At 293 K, the upper yield stress increased continuously until 60 ks and it at

Fig. 1. (a) Maximum internal friction and (b) electrical resistivity as functions of the aging time at 443 K.

Fig. 3. Enlarged TEM image of the precipitates in the specimen aged at 443 K for 600 ks.

Fig. 2. TEM images of the specimens aged at 443 K for (a) 0 ks, (b) 0.06 ks, (c) 0.6 ks, (d) 6 ks, (e) 60 ks, and (f) 600 ks.

Fig. 4. Nominal stress-nominal strain curves of the specimens aged at various times tested at (a) 293 K and (b) 77 K. (Online version in color.)

600 ks decreased to the stress level of the unaged specimen. Although some mechanisms for the increase in the upper yield stress have been proposed,^{21–23)} the segregation of carbon to dislocations, that is, the Cottrell atmosphere, during aging is the main contributor to the increase. In contrast, the tensile strength continuously decreased with increasing aging time. The strengthening by cementites may be lower than the solid solute strengthening by carbon, and the continuous decrease in solid solute carbon concentration with an increasing aging time may result in continuous decrease in tensile strength. On the other hand, Nakanishi *et al.*²⁴⁾ demonstrated that the dislocation density after cold rolling increased with increasing solid solute carbon concentrations. Therefore, the decrease in solid solute carbon concentration due to aging may lead to a decrease in the increase of the dislocation density during tensile deformation, causing the lower tensile strength with an increasing aging time. The SS curves at 77 K and for all aging times exhibited low elastic limits, and the plateau regime appeared from an approximate nominal strain of 0.002, following which work hardening occurred. A similar yielding behavior was observed in duplex-grained ferritic steel, and “micro-yielding” occurred preferentially in the coarse grains,²⁵⁾ which may be the reason for the characteristic yielding behavior of the duplex-grained steel. Herein, we refer to the three deformation stages as the “micro-yield”, “macro-yield”, and “work hardening” stages. The yield and tensile stresses increased until 6 ks and then decreased; however, the specimen aged for 600 ks exhibited higher strength than the unaged specimen. The total elongation did not significantly change as

compared to that at 293 K, indicating that the aging treatment improved the strength-elongation balance of ultra-low carbon steel at cryogenic temperatures. Therefore, the effect of ultra-low carbon on the tensile properties significantly depended on the test temperature.

3.3. Deformation Behavior at 77 K in Aged Ultra-low Carbon Steel

Figure 5 shows (a) a schematic illustration of the SS curve with interruption conditions and SEM images of identical regions (b) before deformation and at the (c) elastic limit, and the (d) micro-yield stage, (e) macro-yield stage, and (f) work hardening stage in the unaged specimen. The elastic limit is defined as the point where the stress and strain linear relationship gradient during elastic deformation changes. Up to the elastic limit, the microstructure did not change (Figs. 5(b) and 5(c)). At the micro-yield stage (Fig. 5(d)), bands appeared continuously across several grains, as indicated by the white arrows. Figure 6(a) shows an inverse pole figure around a band. Another crystallographic orientation grain from that of the matrix was detected inside bands, as indicated by the arrows. Additionally, the pole figure in Fig. 6(b) reveals that the band was a $\{112\} \langle 11-1 \rangle$ deformation twin, which is generally observed in bcc metals.²⁶⁾ Crystallographic orientation analyses were conducted on all bands in Fig. 5, and these were deformation twins. At the macro-yield stage (Fig. 5(e)), the number of deformation twins drastically increased and several twins were not continuous across grain boundaries, but independently occurred inside single grains. At the work hardening stage (Fig. 5(f)), the number of twins did not increase. Figure 7 shows the ϵ_{xx} strain distribution of the 6 ks aged specimen subjected to 0.02 plastic strain. The colors in Fig. 7 denote the strain, and twins were observed, as indicated by black arrows. The high strain regions corresponded with these twinned regions, while the other regions were hardly deformed, indicating that deformation twinning generated high strain. It has been theoretically calculated that twinning generates high strain,²⁷⁾ therefore, the generation of twins is closely related to micro- and macro-yielding.

Figure 8 shows (a) a low magnification SEM image of Fig. 5(d); (b) a schematic illustration of the relationship between the tensile direction, twin plane, and shear direction; and (c) the ϵ_{xx} strain distribution in the specimen subjected to testing to the elastic limit (Fig. 5(c)). Two twin nucleation sites were observed at the triple junction points of the grain boundaries, as indicated by the white and black dotted circles. Twins were continuously generated across the grain boundary, as indicated by the white arrows in Fig. 8(a). The resolved shear stress (τ_{RSS}) to the shear direction on the twin plane was calculated using the following equation.

$$\tau_{RSS} = \sigma \cos \phi \cos \theta, \dots \dots \dots (1)$$

where ϕ and θ are the angles between the tensile direction and the ND of the twin plane and the shear direction, respectively, and σ is the applied stress, as shown in Fig. 8(b). τ_{RSS} is dominated by $\cos \phi \cos \theta$; therefore, the difficulty of deformation twinning depends on $\cos \phi \cos \theta$, similar to the Schmid factor of slip systems. The expression of $\cos \phi \cos \theta$ is called the “Schmid factor for deformation twinning (SF_{twin}).” The

Fig. 5. (a) Schematic illustration of a stress-strain curve with interruption conditions, and SEM images of an identical region (b) before deformation, at the (c) elastic limit, (d) micro-yield stage, (e) macro-yield stage, and (f) work hardening stage in the unaged specimen.

activated twin system was determined from the SEM image and crystallographic data measured by EBSD, and then the SF_{twin} of the twin system was calculated from the crystal orientation along the tensile direction. These resultant SF_{twin} are shown in Fig. 8(a), and were relatively small (below 0.4), indicating that the macroscopic applied stress hardly induced these twins. The ϵ_{xx} strain distribution in the specimen subjected to the elastic limit (Fig. 8(c)) demonstrated that the twin nucleation site corresponded to the high-strain region at the triple junction point. Furthermore, it has been reported that the triple junction points of grain boundaries tend to be high-strain regions.²⁸⁾ Therefore, it can be assumed that the stress concentration at the grain boundary led to the nucleation of the twin, and then the generated twin induced a twin in the neighboring grain, resulting in continuous twins across the grain boundary.

Figure 9 shows the length of the twin boundaries per unit area at the micro-yield, macro-yield, and work hardening stages in the unaged, 6 and 600 ks aged specimens. The observation area was 1 mm × 1 mm. The deformation

twins occurred until the micro-yield stage independent of the aging conditions. At the macro-yield stage, deformation twins increased in the unaged and 6 ks aged specimens, but it negligibly increased in the 600 ks aged specimen. The length of the twin boundaries per unit area at the work hardening stage was equal to those at the macro-yield stage in all the specimens. These results suggest a deformation process in which micro-yielding occurred in all the specimens by deformation twinning induced by local stress concentration at the grain boundary. In the unaged and 6 ks aged specimens, macro-yielding occurred as a result of deformation twinning, and then changed to slip deformation at the work hardening stage. However, in the 600 ks aged specimen, macro-yielding occurred due to slip deformation because the length of the twin boundaries per unit area were constant independent of the deformation stage. The plateau regime within the macro-yield stage was due to the drastic increase in twins in the unaged and 6 ks aged specimens. The 600 ks aged specimen also exhibited a plateau regime, although the number of twins negligibly increased during the macro-yield

Fig. 6. (a) Inverse pole figure of ferrite phase and (b) pole figure of matrix and twin. (Online version in color.)

Fig. 7. ϵ_{xx} strain distribution of the 6 ks aged specimen subjected to 0.02 plastic strain. (Online version in color.)

Fig. 9. Length of twin boundaries per unit area at the micro-yield, macro-yield, and work hardening stages in the unaged, 6 ks and 600 ks aged specimens.

Fig. 8. (a) Low magnification SEM image of Fig. 5(d); and (b) schematic illustration of the relationship between the tensile direction, twin plane, and shear direction; and (c) the ϵ_{xx} strain distribution in the specimen subjected to testing to the elastic limit (Fig. 5(c)). (Online version in color.)

stage. It is possible that the drastic increase in dislocations at the macro-yield stage resulted in the plateau regime, according to the Johnston-Gilman model;^{29,30)} however, details of this were unclear in this study. Thus, the change in the dislocation density during deformation must be measured to clarify the mechanism.

4. Discussion

Table 2 summarizes the macro-yielding stress at 77 K, the solid solute carbon concentration, volume fraction of cementite, length of cementite, thickness of cementite, increase in strength by precipitation strengthening, and deformation mechanism during macro-yielding in the unaged, 6 and 600 ks aged specimens. The solid solute carbon concentration was estimated from the maximum internal friction according to the following equation proposed by Sekino *et al.*:³¹⁾

$$C(\text{ppm}) = kQ^{-1} \times 10^{-4}, \dots\dots\dots (2)$$

where C is the solid solute carbon concentration; k is a constant ranging from 1.5 to 1.8, and $k = 1.65$ was adopted in this calculation, and Q^{-1} represents the maximum internal friction. Here, the solid solute carbon concentration of the unaged specimen must be underestimated because the specimen was subjected to high temperatures during the internal friction measurement, that is, aging occurred. Therefore, the actual solid solute carbon concentration should be greater than 27 ppm in the unaged specimen. However, the aging time was longer than the measurement time (1.8 ks) for the 6 and 600 ks aged specimens; hence, the estimated solid solute carbon concentration should have a certain accuracy. The volume fraction of cementite was calculated from the carbon concentration precipitated as cementite, *i.e.*, subtracting the solid solute carbon concentration from 75 ppm, and from the densities of ferrite (7.87 g cm^{-3} ³²⁾) and cementite (7.68 g cm^{-3} ³³⁾), under the assumption that the saturated solid solute carbon concentration was 0 ppm at 443 K. The increase in strength by precipitation strengthening was calculated using the following Ashby-Orowan equation³⁴⁾

$$\Delta\sigma = 0.84 \left(\frac{1.2Gb}{2\pi L} \right) \ln \left(\frac{d}{2b} \right), \dots\dots\dots (3)$$

Where G and b are the shear modulus and Burgers vector, equal to 80 GPa and 0.25 nm in ferritic steels, respectively; d is the average cross-sectional diameter of the precipitate on the slip plane; and L is the distance between the precipitates. In the case of plate-like precipitates, L and d can be calculated using the following equations:³⁵⁾

$$L = \left\{ \frac{2fsin\theta}{\pi rt} \right\}^{-1/2}, \dots\dots\dots (4)$$

$$d = \frac{\pi}{4} 2r, \dots\dots\dots (5)$$

where f is the volume fraction of the precipitate; r and t are the radius and thickness of the precipitate, respectively, and half the length of the cementite was adopted as r in this study; and θ is the angle between the precipitate and slip plane, and was assumed to be 60° assuming that the habit plane of ferrite is $\{110\}$, and a $\{110\} < 1-11 >$ slip system, which differs from the habit plane, are activated.

The unaged specimen exhibited the lowest strength at 77 K, although it exhibited the highest tensile strength at 293 K. Macro-yielding in the unaged specimen occurred because of deformation twinning; therefore, at 77 K, the critical resolved shear stress (τ_c) of slip deformation is larger than that of deformation twinning at the macro-yield stage, as shown in **Fig. 10(a)**. As summarized in Table 2, aging up to 6 ks increased the macro-yielding stress and decreased the solid solute carbon concentration. Additionally, fine cementites formed, while the deformation mechanism was unchanged from that of the unaged specimen; specifically, deformation twinning occurred. This suggests that the τ_c of both slip deformation and deformation twinning increased with aging until 6 ks, as shown in **Fig. 10(b)**. Because precipitates inhibit the formation of twins,³⁶⁾ it is possible that fine cementite enhanced the τ_c of deformation twinning. Another possible reason for the increase in τ_c of deformation twinning is that the decrease in solid solute carbon concentration with aging may increase the stacking fault energy. According to the Cottrell and Bilby model,³⁷⁾ stacking fault energy plays an important role in the nucleation of twins of bcc metals. Therefore, if solid solute carbon decrease the stacking fault energy, the τ_c value of deformation twinning will increase with increasing aging time. The relationship between the τ_c of deformation twinning and the precipitates, and the effect of solid solute carbon on the stacking fault energy has not yet been discussed, and further experimental and computational investigations are necessary.

The 600 ks aged specimen exhibited a lower macro-yielding stress than the 6 ks aged specimen, and the deformation mechanism during macro-yielding changed from deformation twinning to slip deformation (Table 2). Thus, the decrease in τ_c of slip deformation is the reason for the decrease in the macro-yielding stress in the 600 ks aged specimen, as shown in **Fig. 10(c)**. The solid solute carbon

Table 2. Macro-yielding stress at 77 K, the solid solute carbon concentration, volume fraction of cementite, length of cementite, thickness of cementite, increase in strength by precipitation strengthening, and deformation mechanism during macro-yielding in the unaged, 6 and 600 ks aged specimens.

	Macro-yielding stress at 77 K (MPa)	Solid solute carbon concentration (ppm)	Volume fraction of cementite ($\times 10^{-4}$)	Length of cementite (nm)	Thickness of cementite (nm)	Increase of strength by precipitation strengthening (MPa)	Deformation mechanism during macro-yielding
Unaged	570	>27	—	—	—	—	Twin
6 ks	670	11	9.8	110±20	10±3	17±3 MPa	Twin
600 ks	610	8	10.2	800±200	26±5	5±1 MPa	Slip

Fig. 10. Schematic illustration of the critical resolved shear stress for slip deformation and deformation twinning in the unaged, 6 ks and 600 ks aged specimens. (Online version in color.)

concentrations of the 6 and 600 ks aged specimens were similar (Table 2), indicating that the increase in the size of cementite led to a decrease in τ_c of slip deformation. The difference in the estimated strength increase by precipitation strengthening of cementites between the 6 and 600 ks aged specimens was approximately 10 MPa, as shown in Table 2, which is close to the difference in their macro-yielding stresses (60 MPa). The difference in the shear modules between the matrix and precipitates, and the strain field derived from a misfit between the matrix and precipitates also influences the precipitation strengthening,^{38,39)} however, these factors were ignored in the present estimation, which

may have caused the precipitate strengthening effect to be underestimated. The 600 ks aged specimen exhibited higher macro-yielding stress than that of the unaged specimen, although the solid solute carbon concentration in the 600 ks aged specimen was considerably lower than that in the unaged specimen. This implies that the solid solute carbon negligibly strengthened or softened the steel at 77 K. A similar temperature dependence of solid solute strengthening by carbon or nitrogen has been reported.^{40–42)}

In the unaged and 6 ks aged specimens, deformation twinning changed to slip deformation at the work hardening stage. The τ_c value of deformation twinning increased significantly with decreasing grain size.⁴²⁾ The deformation twin divided the grains, and an increased number of deformation twins resulted in grain refinement. Deformation twins also enhance the critical stress of slip deformation because the twin boundary interrupts dislocation gliding. Sakui *et al.*⁴³⁾ demonstrated that the dependence of the grain size on the yield stress by deformation twinning was larger than that by slip deformation. Therefore, grain refinement by deformation twins increased the τ_c of deformation twinning more than that of slip deformation, causing a change in the deformation mechanism at the work hardening stage.

The addition of ultra-low carbon in ferritic steels strongly influences the mechanical properties not only above room temperature, but also at low temperatures. At low temperatures, the τ_c values of both deformation twinning and slip deformation changed with aging treatment; therefore, the change in strength with aging treatment at 77 K differed from that at 293 K.

5. Conclusion

The microstructural changes, and low-temperature tensile properties of ultra-low carbon steel with various aging treatments were examined and the relationship between the low-temperature tensile properties and ultra-low carbon state was discussed. The main results are summarized as follows:

(1) The maximum internal friction and electrical resistivity decreased with increasing aging time at 443 K, indicating that the solid solute carbon concentration decreased. Fine precipitates were observed at an aging time of 0.6 ks, and these grew to approximately 800 nm at 600 ks. It was concluded from the shape of the precipitates that these were cementite.

(2) At 293 K, the tensile stress decreased continuously with aging. At 77 K, both the yield and tensile stresses increased until the aging time of 6 ks, and then decreased until 600 ks. Under all aging conditions, the nominal stress–nominal strain curves at 77 K exhibited low elastic limits, and the nominal stress plateau regime appeared at a nominal strain of approximately 0.002, after which, work hardening occurred.

(3) In the unaged and 6 ks aged specimens, several deformation twins occurred after the elastic limit and remarkably increased in the nominal stress plateau regime, indicating macroscopic yielding. At the work hardening stage, the number of deformation twins increased slightly. Therefore, macroscopic yielding occurred because of deformation twinning. In contrast, in the 600 ks aged specimen, deformation twins negligibly increased at the macroscopic

yield, indicating that macroscopic yielding occurred due to slip deformation.

(4) The 6 ks aged specimen exhibited the highest strength, indicating that the critical resolved shear stress of deformation twinning increased as a result of fine cementites and/or a decrease in the solid solute carbon concentration. In the 600 ks aged specimen, the cementite coarsened, which led to a decrease in the critical resolved shear stress of slip deformation. Hence, the deformation mechanism changed from twinning to slipping in the 600 ks aged specimen and the strength decreased from that of the 6 ks aged specimen.

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