Comparison of the Dislocation Density in Martensitic Steels Evaluated by Some X-ray Diffraction Methods

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X-ray diffraction (XRD)-based modified Warren–Averbach (MWA) analysis, in comparison with the Williamson–Hall (WH) analysis, was applied to 0.3 mass% carbon martensitic steels, as-quenched and subsequently tempered at various temperatures, to give their dislocation densities. For the as-quenched martensite, the WH method gives a value of around \(2.0 \times 10^{16} \text{ m}^{-2}\), which could be overestimated. Meanwhile, the MWA method gives a value of around \(6.3 \times 10^{15} \text{ m}^{-2}\), which is below the possible upper limit of dislocation density, \(10^{16} \text{ m}^{-2}\). The MWA-derived value for the as-quenched steel seems to be 1.6–4.8 times higher than those expected from the precedent results derived by transmission electron microscope (TEM) observations. However, considering that the TEM-derived value gives the microscopically local average while the XRD-derived value gives the macroscopic average, such discrepancy between the TEM-derived value and MWA-derived value is tolerable. For the steels tempered at 723 K and 923 K, the MWA and WH methods give comparable values ranging in \(10^{14} \text{ m}^{-2}\), where the rearrangement of dislocation structure is observed by TEM. However, in these steels where the XRD peaks are narrower and the instrumental width of the present XRD system could be significant, care should be taken over the peak width correction.

KEY WORDS: martensitic steels; line broadening; dislocation densities; X-ray diffraction; transmission electron microscopy.

1. Introduction

1.1. Background

In investigating the mechanical properties of martensitic steels, dislocation density is one of the critical key factors that should be evaluated. Morito et al. observed dislocations and evaluated the dislocation density by TEM in Fe–C and Fe–Ni martensitic steels. It was also evaluated by Kehoe et al. in steels with 0.01 to 0.1 mass% carbon. In addition to the TEM method, X-ray diffraction (XRD) peak broadening caused by strain has long been investigated in connection with dislocation density. The XRD peak width, \(\delta\), is related to the strain through the following Williamson–Hall (WH) equation:

\[
\delta \cos \theta = \alpha + 2\varepsilon \sin \theta
\]

Here, \(\theta\), \(\lambda\), \(\varepsilon\), and \(D\) are the diffraction angle, X-ray wavelength, strain, and average particle size, respectively. And \(\delta\) is measured on a scale of \(2\theta\). Then, the dislocation density, \(\rho\), is derived as follows:

\[
\rho = \frac{14.4}{\frac{b^2}{\lambda^4}} \cos \theta \sin \theta
\]
location density in 11Cr–0.1C martensitic steel in order to evaluate the strain energy stored in the steel. The XRD-based method was also used for analysis of the heat treatment effect on the dislocation density in 11.7Cr–0.2C martensitic steels by Pěšíčka et al.,\textsuperscript{16} where the TEM method was also applied and the rather compatible results between the XRD and the TEM method were obtained in deriving the dislocation density. However, it should be kept in mind that in an inhomogeneous system such as martensitic steel, where the dislocation density varies from place to place within the grain on a microscopic scale, the method using XRD gives a macroscopic average value while TEM method gives a microscopic local value.\textsuperscript{16,17}

1.2. Analysis

In the present XRD profile analysis, a variable is used that is referred to as the dislocation contrast factor, which depends on the relative orientation between the Burgers vector and the dislocation line.\textsuperscript{59} Ungár et al.\textsuperscript{10,11} introduced the new scaling factor consisting of dislocation contrast factor and the magnitude of diffraction vector to account for the XRD line broadening in order to suppress the anisotropic line broadening often observed in the conventional WH analysis. The width of a diffraction peak, defined by the full-width-half-maximum (FWHM), which is denoted by $\Delta K$, measured on a scale of the magnitude of the diffraction vector, is related to the dislocation contrast factor, denoted by $C$, through the following modified Williamson–Hall (MWH) equation:\textsuperscript{10}:

$$\Delta K = \alpha + \beta K C^{1/2} + O(K^2C) \quad \ldots \ldots (4)$$

$$\beta = \frac{\pi M b^2 \rho^{1/2}}{2} \quad \ldots \ldots (5)$$

Here, $K$, the magnitude of diffraction vector, equals $2 \sin \theta/\lambda$, with $\theta$, $\lambda$, and $\alpha$ corresponding to those in Eq. (1), $b$ and $\rho$ corresponding to those in Eq. (2), $M$ and $O$ being constants depending on the effective cut-off radius of the dislocations.\textsuperscript{10,14,15} It should be noted that with the proper selection of $C$, Eq. (4) gives $\Delta K$ as a monotonous function of $K C^{1/2}$ to suppress the anisotropy in line broadening. Dislocation contrast factor $C$ is replaced by the average version, denoted by $\bar{C}$, for the specific $(hkl)$ diffraction expressed as follows:

$$\bar{C} = \bar{C}_{\text{iso}} (1 - qH^2) \quad \ldots \ldots (6)$$

$$H^2 = \frac{h^2k^2 + k^2l^2 + l^2h^2}{(h^2 + k^2 + l^2)^2} \quad \ldots \ldots (7)$$

Here, $q$ is a constant to be determined experimentally and $\bar{C}_{\text{iso}}$ is the average dislocation contrast factor corresponding to $(h00)$ diffraction determined by crystal elasticity.\textsuperscript{10,11} For $\bar{C}_{\text{iso}} = 0.285$ was employed as the value in pure iron in the literature.\textsuperscript{14,15} Substituting Eq. (6) into Eq. (4), ignoring the third term on the right hand side of Eq. (4), gives

$$\frac{(\Delta K - \alpha)^2}{K^2} = \beta^2 q^{0.285}(1 - qH^2) \quad \ldots \ldots (8)$$

With the proper selection of $\alpha$, the left-hand side of Eq. (8) is given experimentally. The linear fitting against $H^2$ then gives $q$ as an inverse of the intercept on the $H^2$ axis. Thus determined $q$ gives $\bar{C}$ through Eq. (6) for $(hkl)$ diffraction and then leads to the new variable expressed by $K^2C$ ($=K^2\bar{C}$), which is used as an independent variable in the following modified Warren–Averbach (MWA) equation that relates the Fourier coefficients of the diffraction peaks to the dislocation density:

$$\ln A(L) = \gamma - X(L)(K^2\bar{C}) + P(K^2\bar{C})^2 \quad \ldots \ldots (9)$$

Here, $A(L)$ is the real part of the Fourier coefficient of the $(hkl)$ diffraction peak depending on $L$, Fourier length, for which the values ranging from 40 to 90 nm were used. $A(L)$ can be obtained simply by Fourier transformation, where the phase factor is $2\pi L K$, of the Lorentzian that fits the diffraction peak. Constant $\gamma$, and second-order coefficient $P$ in Eq. (9) are shown in the literature.\textsuperscript{10,11} Fitting $\ln A(L)$, with $L$ fixed, against $K^2\bar{C}$, which is referred to as the MWA plot hereafter, gives $X(L)$ as the coefficient of the first-order term in Eq. (9) for a given value of $L$. Then, with various $L$s, Eq. (10) leads to

$$\frac{X(L)}{L^2} = \frac{\rho \pi b^2}{2} \left(\ln(R_e) - \ln(L)\right) \quad \ldots \ldots (11)$$

through which another fitting procedure upon $X(L)/L^2$ against $\ln(L)$ gives $\rho \pi b^2/2$, where $b=0.283$ nm, as the first-order coefficient, and then finally leads to the value of $\rho$.

Thus far, the proper selection of $\alpha$ and $q$ in Eq. (8) is necessary to obtain the contrast factor $\bar{C}$ ($=\bar{C}$), which works through Eqs. (9)–(11) and leads to the dislocation density $\rho$. However, it seems to be somewhat laborious to determine $\alpha$ and $q$ through Eqs. (4)–(8). Kunieda et al.\textsuperscript{15} set $\alpha$ so that the linear relationship should hold between the left hand side of Eq. (8) and $H^2$. However, considering that $\alpha$ should not violate the consistency between Eq. (4) and Eq. (8), a recursive iteration method is employed to give $\alpha$ and $q$ in the present analysis. First, the initial value $\alpha_0$ is substituted into Eq. (8). A linear fitting procedure then gives $q_0$ as the inverse of the intercept on the $H^2$ axis. Thus-obtained $q_0$, is then substituted into Eq. (6) to obtain $\bar{C}$ and the quadratic fitting technique is applied to Eq. (4) to fit $\Delta K$ against $K C^{1/2}$ to give renewed value $\alpha_1$ as the intercept on the $\Delta K$ axis. $\alpha_1$ is then substituted into Eq. (8) and the linear fitting procedure works again to obtain the renewed value $q_1$. Looping these procedures until $\alpha_n$ and $q_n$ converge is expected to give the proper selection of $\alpha$ and $q$.

In spite of the development of the line broadening analysis, comparison between the two methods described her, has not been made. Therefore, it is the purpose of the present study to make a quantitative comparison between the two
methods, WH and MWA methods. For the as-quenched steel, the present results were compared with those estimated from the precedent TEM analysis. Furthermore, a series of tempered martensitic steels were investigated to determine their mechanical properties. TEM observation was also performed on some of the specimens to observe the dislocations therein.

2. Experiment

A steel with 0.3 mass% carbon, the chemical composition of which is listed in Table 1, was used. The ingot was prepared by vacuum induction melting. It was then homogenized at 1473 K for 2 h and forged down to a thickness of 15 mm. Bars of 15 mm in width and 180 mm in length were then sampled from the plate. The bars were austenitized at 1153 K for 5 min, and were then water-quenched. Each of the specimens was tempered for 90 min at 453 K, 623 K, 723 K and 923 K, respectively. The test pieces were then machined out for tensile testing with a cross-head speed of 10 \( \times 10^{-3} \) m/min and a gauge length of 0.025 m and for Vickers hardness measurement with a diamond pyramid under the force of 9.8 N. A Rigaku-Denki RINT 1500 system with Cuk_α radiation was used for XRD measurement. A monochrometer employing graphite diffraction was used to eliminate the Cuk_β radiation. The X-ray tube was operated at 40 kV and 150 mA. The diffraction lines were recorded from 2\( \theta \)=35 to 145° with a step of 0.01° to cover the diffractions of (110), (200), (211), (220), (310) and (222). The observed diffraction peaks were corrected by splitting into the peaks diffracted by Cuk_α1 and Cuk_α2 radiations, followed by the Lorentz Polarization correction. The peaks corresponding to Cuk_α1 radiation were fitted with Lorentzian functions and used for the analysis. Then, the corresponding instrumental width was subtracted from each peak width. The instrumental line broadening was evaluated by using the standard reference material of LaB_6 (SRM660a supplied by NIST). The instrumental width depends upon the peak position and it is approximated by a polynomial function. The average instrumental width corresponding to the measured 6 peaks was found to be about 6\( \times 10^{-3} \) nm^{-1}.

The structure of the dislocations in the as-quenched steel, the 623-K tempered steel and the 923-K tempered steel were observed by TEM.

3. Results and Discussion

3.1. Analysis

The tempered Fe–C martensitic steels have been investigated extensively. The hardness, tensile stress and total elongation of the present steels are plotted against the tempering temperature in Figs. 1(a), 1(b) and 1(c), respectively. The Vickers hardness of the as-quenched steel shows about 560 Hv, which is consistent with the result by Grange et al. for as-quenched martensitic steel with 0.3 mass% carbon, and it decreases to around 230 Hv as the tempering temperature rises to 923 K. Optical micrographs of the as-quenched and the 923-K tempered specimens are shown in Figs. 2(a) and 2(b), respectively. The figures show that plain and simple tempered martensitic steels are the objectives of the present analysis.

The variation of the diffraction peaks, (110), (200) and (310) are shown in Figs. 3(a), 3(b) and 3(c), respectively, where the peaks are corrected by Cuk_α1 splitting and the Lorentz-polarization scheme, but are yet to be corrected by the instrumental width. Each peak is clearly shown to become narrower and more intense in the tempered specimen than in the as-quenched type. The precise analysis of the
diffraction peak profile requires detailed discussion.\textsuperscript{30,31) Though some of the peaks are irregularly shaped, which might be better fitted with the pseudo Voigt function, a weighted linear combination of Lorentzian and Gaussian function,\textsuperscript{30) simple Lorentzian fitting is employed in the present analysis.\textsuperscript{14,15) The instrumental width, which is not a constant value, is then subtracted from the peak width. The variation of the corresponding corrected peak width, corrected additionally with instrumental peak width, is shown in Fig. 4. As the tempering temperature rises, especially at 723 K, the peak width steeply decreases to be ranging below $4 \times 10^{-2}$ nm\(^{-1}\), where the instrumental width, $6 \times 10^{-3}$ nm\(^{-1}\) on average, could be rather significant. Thus, the peaks of the specimens tempered at 723 K and 923 K are so narrow that they may be keen to the errors included during measurement.

In showing the WH plots, Eq. (1) was reformulated in terms of magnitude of diffraction vector $K$. Using the relationship $K = 2 \sin \theta / \lambda$, Eq. (1) is equivalently written as:

$$\Delta K = \Delta \theta / \lambda = \alpha + e K \quad \text{..................(12)}$$

Here, the left hand side is FWHM measured on a scale of $K$. WH plots are shown in Fig. 5 for the as-quenched and the 723-K tempered specimens.

In order to obtain the MWH plot expressed by Eq. (4), the previously-mentioned recursive iteration method mentioned before was applied. The final plots to give the converged $q$ in Eq. (8) are shown in Figs. 6(a) and 6(b) for the as-quenched and the 723-K tempered specimens, respec-

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig3.png}
\caption{XRD peaks of as-quenched steel (dotted line) and 723-K tempered steel (solid line). (a), (b) and (c) correspond to (110), (200) and (310) diffraction peaks, respectively.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig4.png}
\caption{Variations of diffraction peak width plotted against tempering temperature. Circles, rectangles and triangles show (110), (200) and (310) peak width, respectively.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig5.png}
\caption{WH plots of (a) as-quenched and (b) 723-K tempered steel. Plots are not monotonous because of the anisotropy in peak widths. Equation (12), which is equivalent to Eq. (1), is used for plotting.}
\end{figure}
The reciprocal of the intercept on the $H^2$ axis in each figure gives $q$. Then the dislocation contrast factors are obtained through Eqs. (6) and (7), leading to MWH plots, which are shown in Fig. 7. In these figures, the value of abscissa, $KC^{1/2}$, of each diffraction in one specimen differs from that in the other mainly due to the difference in the $C$ value. Although neglecting the highly deviating points in the WH plot is reported to work in evaluating the dislocation density, in the present analysis, the literal WH analysis with all points is compared with the MWA analysis. The intercept on the $D_K$ axis in Fig. 7, representing the particle size broadening through Eq. (2), is in the order of $10^{-1}$ for the as-quenched specimen shown in Fig. 7(a). The observed peak widths for the as-quenched specimen are in the order of $10^{-2} - 10^{-1}$ nm. Thus, it is possible to consider the particle size broadening to be negligibly small. Pěsíčka et al. also found it negligible in investigating the tempered martensite ferritic steels. However, as shown in Fig. 7(b), for the 723-K tempered specimen, the observed widths decrease to the order of $10^{-3} - 10^{-2}$ nm, which is comparable with the instrumental broadening. Then the intercept on the $\Delta K$ axis increases to the order of $10^{-1}$, which is not small enough to be ignored. Discussion on the particle size requires a detailed analysis of the peak profile. However, in the present analysis, as the particle size broadening term does not directly affect the dislocation density, it is possible to ignore the term also in the 723-K tempered specimen. Now that the dislocation contrast factor $C$ has been obtained, it is possible to build
up a new scaling variable $K^2C$ which scales the MWH plot expressed by Eq. (9). The corrected diffraction peaks approximated by Lorentzians underwent Fourier-transform to give the component $A(L)$, on the left hand side of Eq. (9). The corrected MWA plots for the as-quenched specimen, shown in Fig. 8(a), are highly nonlinear, on the other hand, those for the 723-K tempered specimen, shown in Fig. 8(b), are rather straight. The MWA plot gives $X(L)$ in Eq. (9) as the coefficient of the first order term of $K^2C$. Then, according to Eq. (11), plotting $X(L)/L^2$ against $\ln(L)$ gives dislocation density through its coefficient of $\ln(L)$. Figure 9 shows these $X(L)/L^2-\ln(L)$ plots.

3.2. Dislocation Density

The dislocation density obtained from WH and MWA analysis is shown in Fig. 10. For the specimens tempered below 623 K, the WH results show values around $1.2 \times 10^{16}-2.0 \times 10^{16}$ m$^{-2}$, while MWA results show $2.5 \times 10^{15}-6.3 \times 10^{15}$ m$^{-2}$. The WH values are somewhat higher than the upper limit value $1 \times 10^{16}$ m$^{-2}$ estimated by Nakashima et al.\textsuperscript{32} The MWA plot in Fig. 8(a), corresponding to the as-quenched specimen with the dislocation density of $6.4 \times 10^{15}$ m$^{-2}$ by the MWA analysis, shows a highly nonlinear curve. The high nonlinearity as such is observed also in the 453-K tempered and the 623-K tempered specimens, whose MWA-derived dislocation densities are $5.0 \times 10^{15}$ m$^{-2}$ and $2.5 \times 10^{15}$ m$^{-2}$, respectively. On the other hand, the 723-K tempered specimen, as in Fig. 8(b), shows a moderate nonlinearity, whose MWA-derived dislocation density is $6.8 \times 10^{14}$ m$^{-2}$. These observations seem to imply that, in the present MWA analysis, the coefficient of the third term on the right hand side of Eq. (9) seems to be significant in the case where the dislocation density exceeds $10^{15}$ m$^{-2}$. The ultrafine-grain ferritic steels investigated by Yin et al.\textsuperscript{14} show $2 \times 10^{13}-1.4 \times 10^{14}$ m$^{-2}$, where the second-order term is negligible in Eq. (9). However, the results of Kunieda et al.\textsuperscript{15} show that the as-quenched state of 11Cr–0.1C martensitic steel requires a quadratic fitting of the MWA plot to give a value of $5 \times 10^{14}$ m$^{-2}$. Hence, the linearity of the MWA plot is not necessarily constrained only by dislocation density but by carbon content or other factors as well.

It is important to compare the present results with those of the TEM analyses. As noted previously, Morito et al.\textsuperscript{31} Kehoe et al.\textsuperscript{41} and Noström\textsuperscript{5} investigated the variation of the dislocation density against the carbon content in the as-quenched martensitic steels. They showed that it is linearly dependent on the carbon content. In Fig. 11, the TEM-derived results in the literature for the as-quenched martensitic steels are compared with the present results. The MWA method gives dislocation density of about $6.3 \times 10^{15}$ m$^{-2}$ for the as-quenched specimen, which is relatively higher than the value of about $1.3 \times 10^{15}$ m$^{-2}$ derived by in-
interpolating the results along the carbon content presented by Morito et al., the value of about $3.8 \times 10^{15}$ m$^{-2}$ and $2.7 \times 10^{15}$ m$^{-2}$ given by linearly extrapolating the results presented by Kehoe et al. and Noström, respectively. These discrepancies might be inevitable because of the inhomogeneous distribution of dislocations in martensitic steels. Breuer et al. evaluated the dislocation density in Co-based alloy using XRD profile analysis and the TEM method, where they found the tendency for the XRD method to give a higher value than the TEM method when the dislocation distribution is inhomogeneous. Meanwhile, the WH method gives a difference higher by nearly an order of magnitude. These observations lead to the conclusion that, as far as the as-quenched specimen is concerned, MWA analysis is reliable as compared with the WH method when the dislocation distribution is inhomogeneous.

4. Conclusions

In summary, in order to evaluate the dislocation densities in 0.3 mass%-C as-quenched and tempered martensitic steels, the MWA method and the conventional WH method were compared. TEM observation was also performed on some of the steels.

(1) The MWA method gave values ranging from $6.3 \times 10^{15}$ m$^{-2}$ to $8 \times 10^{14}$ m$^{-2}$, are given by each method. The broadening anisotropy of the WH plot shown in Fig. 5(b) might become less effective because of the narrow range of the peak width distribution. However, the narrower the peaks are, the more sensitive they are to the extrinsic errors involved during measurement or analysis, especially in case where the instrumental width is comparable with the observed width.

In order to observe the structure of dislocations, the as-quenched specimen, the 623-K tempered specimen, and the 923-K tempered specimen were observed by TEM. Although the present analysis is not precise enough for a quantitative investigation of the dislocation structure, the results are shown in Fig. 12. In the as-quenched specimen, where the dislocations originate in the plastic accommodation of transformation strain, Pešićka et al. observed aggregated dislocations similar to Fig. 12(a) in their 12Cr–0.2C martensitic steel, where they found the dislocation density too high to be evaluated. However, they tried to evaluate at different locations to give the density of $8 \times 10^{14}$–$1.2 \times 10^{15}$ m$^{-2}$. Therefore, considering that such aggregation is observed in most of the views in the present specimen, it is probable that the dislocation density of the present as-quenched specimen is higher than $1.2 \times 10^{15}$ m$^{-2}$ and the MWA-derived value of $6.3 \times 10^{15}$ m$^{-2}$ could be tolerable. In the 623-K tempered specimen, where carbide precipitation is expected, most of the views show such structures as shown in Fig. 12(a), but some show, as in Fig. 12(b), recovery and carbide precipitation possibly giving rise to a decrease of the dislocation density and a rearranged dislocation structure, which implies a moderate decrease in the dislocation density as compared with the as-quenched specimen, Fig. 12(a). Therefore, it is possible to estimate the dislocation density to be still in the order of $10^{15}$ m$^{-2}$, where the MWA-derived value of $2.5 \times 10^{15}$ m$^{-2}$ could be tolerable. In the 923-K tempered specimen, most of the views show a totally different rearranged structure of dislocations as shown in Fig. 12(c). The rearrangement of dislocations suggests that the dislocation density decreased by more than an order of magnitude. As compared again with the observation by Pešićka et al. of the specimen with low dislocation density, it is possible to estimate the density from Fig. 12(c) to be in the order of $10^{14}$ m$^{-2}$, which is consistent with the MWA-derived value. Although the 723-K tempered specimen did not undergo TEM observation, it is possible that the collective rearrangement of the dislocation structure synchronously occurs with the steep decrease in the dislocation density at the tempering temperature of 723 K.

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10^{15} for as quenched steel down to 3 \times 10^{14} \text{ m}^{-2} for the 923-K tempered type. Meanwhile, for these steels, the WH method gave values ranging from 2.0 \times 10^{16} for the as-quenched steel down to 3.5 \times 10^{14} \text{ m}^{-2} for the 923-K tempered type.

(2) For the steels tempered below 623 K, including the as-quenched type, the WH method gives values exceeding 10^{16} \text{ m}^{-2}, which can be overestimated. Meanwhile, the MWA method gives values below 10^{16} \text{ m}^{-2}, which are acceptable.

(3) For the specimens tempered at 723 K and 923 K, both methods give comparable values in the range of 10^{14} \text{ m}^{-2}.

(4) Comparison with the expected values from the precedent TEM results for the as-quenched specimen shows that the MWA method gives a value 1.6–4.8 times higher than the TEM method. However, they are still within the same order of magnitude. Considering the inhomogeneity of the dislocation distribution, the observed discrepancies are tolerable.

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