1. Introduction

Pearlitic steels have the highest tensile strength among steel products, exceeding 5 GPa, obtained by loading high level stress through cold-drawing processes, and cold-drawn pearlitic steel wires have been used in many industrial applications, such as suspension cables of bridges and automotive tire cords. Pearlitic steel is reinforced mainly by dislocation hardening and thinning of inter-lamellar spacing, which can be induced by strong plastic deformation. During the drawing process, non-uniform plastic deformation develops along the radial direction of the wire, and the wire surface undergoes more severe plastic deformation than the wire center. Moreover, the pearlitic lamellae become progressively aligned parallel to the drawing direction with increasing drawing strain; thereby, cold-drawn pearlitic steel wires exhibit anisotropic microstructure along the radial and axial directions. Consequently, cold-drawn pearlitic steel wires exhibit non-uniform and anisotropic features of dislocation characteristics, which can cause positional and directional non-uniformity of mechanical properties. Besides, hot-dip galvanizing or bluing treatment, during which the wire is immersed in high temperature atmosphere (typically around 700 K), is performed after the cold-drawing process. It has been reported that some softening occurs because spheroidization of lamellar cementite occurs at high temperature. However, the recovery and annihilation of dislocations have not been discussed in detail. For better understanding of the microstructural changes in cold-drawn pearlitic steels that underwent high temperature treatment, it is essential to characterize the evolution of dislocations as well as cementite lamellae.

Numerous studies on the microstructure of cold-drawn pearlitic steel have been performed for discussing the relationship between structural properties and plastic strain introduced by cold-drawing processes. Zhang et al. observed the dislocation configuration in ferrite lamellae using transmission electron microscopy and reported that the most of the dislocations were spread in the ferrite lamellae with the two ends of the line located at the steps in the ferrite/cementite boundaries. Since the pearlitic lamellae...
align the drawing direction, the dislocation configuration can introduce anisotropic characteristics of dislocations into the cold-drawn wire. On the other hand, quantification of dislocations in deformed lamellae has not been satisfactorily addressed using microscopy because dislocations in cold-drawn pearlitic steels are dense, non-uniform, and tangled. Recently, line-profile analysis using X-ray diffraction (XRD) has been applied for characterizing dislocations in various deformed alloys. In particular, modified Williamson–Hall and Warren–Averbach procedures, which consider dislocation contrasts on the line profiles, can yield reliable microstructural parameters of coherent domain size for diffraction, as well as density and arrangement of dislocations for explaining the work hardening.\textsuperscript{11–13} In our previous studies, cold-drawn pearlitic steels were measured with a laboratory XRD apparatus and were analyzed using modified procedures, and it was suggested that work hardening at small true strains could be mainly attributed to the dislocation hardening, and that increasing carbon composition could explain the increasing dislocation density.\textsuperscript{14} However, the incident X-rays of the laboratory XRD irradiated the entire cross-section of the axial direction of the wire specimens. Consequently, the laboratory XRD method averaged the dislocation characteristics for the cross-section of the axial direction. Besides, to analyze the dislocation distribution in the cross-section of pearlitic steel wires, we developed the line-profile analysis using an energy-dispersive X-ray diffraction method.\textsuperscript{15} A micro-beam of white X-rays made it possible to characterize the dislocation distribution in the wire cross-section, and it was found that the dislocation density was almost constant along the radial direction. However, owing to the low resolution of a reciprocal space associated with energy-dispersive X-ray diffraction method, the errors of the estimated microstructural parameters became somewhat large; especially, those of the dislocation arrangement parameters were crucial, because they were principally determined by the quality of diffraction data. Moreover, the anisotropy of dislocation characteristics was not analyzed in the energy-dispersive X-ray diffraction method. To advance the discussion on how the plastic-strain distribution and the anisotropy of dislocations in the wire cross-section affects the dislocation characteristics, it is essential to perform line-profile analysis using a high-resolution XRD setup with positional scanning function.

In this study, we suggest a line-profile analysis for characterizing non-uniform and anisotropic features of dislocations in cold-drawn pearlitic steels. Instead of the energy-dispersive mode, an angular dispersive mode was adopted to obtain higher resolution in reciprocal space, and high-brilliance X-rays from an undulator source of synchrotron radiation were used. Generally, highly directional X-rays from synchrotron radiation make it difficult to measure diffraction profiles of polycrystalline materials with large grains, and thereby the powder diffraction of standard LaB\textsubscript{6} powder, which is usually used for defining the instrumental profile, cannot be measured in the synchrotron facility. Therefore, an analytical procedure for solving this problem is required. By developing an XRD setup especially for analyzing the anisotropic features of dislocations in pearlitic steel wires, we were able to characterize the non-uniformity and anisotropy of the dislocation density and the dislocation arrangement, as well as the crystallite size in cross-sections of pearlitic steel wires. In addition, XRD measurements at elevated temperatures were also performed for analyzing the evolution of dislocations during annealing.

2. Experimental

Straight wires of pearlitic steel with carbon composition of 0.73 mass\% around the eutectoid point were used in this research. The 5.50-mm-diameter patenting rod was cold-drawn down to a diameter of 2.74 mm, corresponding to a true strain of 1.39.

XRD measurements were performed at the BL22XU beam-line in SPring-8 (Japan). Experimental setup is shown in Fig. 1. Because the XRD measurements were performed in a transmission geometry, incident X-rays of 30.036 keV would be mostly absorbed by steel wires. To suppress this absorption, the wire specimens were shaped as plates with a thickness of about 1 mm by cutting along the longitudinal direction of the wire. The incident X-rays were collimated to $200 \times 200 \mu \text{m}^2$ and were irradiated normal to the plate-shaped specimen surfaces. Although about 99.8\% of the collimated X-rays were absorbed by the plate-shaped steel, diffraction profiles with high counting statistics were obtained in a few minutes owing to the highly intense X-rays from the undulator of synchrotron radiation. Diffracted X-rays were collected using two-dimensional detectors (PILATUS 100 K, Dectris). Two individual detectors were used for measuring diffraction profiles along the axial and radial directions of the wire, facilitating the analysis of directional anisotropy of dislocations. For both detectors, the distance between the specimen and the detector plane was 858 mm, whereas the angular resolution for each pixel size ($172 \mu \text{m}$) of the detectors was about 0.011°. Because the detector area was relatively small (about $83.8 \times 33.5 \text{ mm}^2$), up to two Debye rings per image could be obtained. Therefore, the detectors were translated as shown in Fig. 1, so that five images could be collected for constructing the Debye patterns of ferrite from 110 to 310. The individual images were recorded in a cumulative time of 50 s. A one-dimensional scattering profile was extracted from the ring pattern by azimuthally caking $\pm 2^\circ$ fractions. It should be mentioned that the non-uniformity of pixels of the two-
dimensional detector can distort Debye rings. This problem can be significant when the detector is placed at a short distance from a specimen and covers large part of Debye rings. On the other hand, the detector was placed at the long distance of 858 mm, and the caking range for Debye rings was small in this study. Thus, the distortion of the diffraction profiles was negligibly small. An air scattering profile was also measured and was subtracted from the measured profiles of the specimens to reduce the background.

An infrared heating furnace was mounted on the sample stage during high-temperature XRD measurements. Air in the chamber was replaced by argon gas. Figure 2 shows the heating history of the XRD measurements. The XRD measurements were conducted for 600 s after reaching a pre-determined temperature.

The analysis of physical line profiles of ferrite in the pearlitic steels was based on the modified Williamson–Hall and Warren–Averbach procedures, which were presented by Ungár et al.\(^\text{11-13}\) The modified Williamson–Hall equation is:

\[ \Delta K = 1/D \]
\[ + \frac{\pi B^2 b^2}{2} - \rho \left( K(C)^{1/2} \right) + O \left( K(C)^{1/2} \right), \quad (1) \]

where \( K = 2\sin(\theta / 2) / \lambda \) and \( \Delta K = \Delta \theta \cdot \cos(2\theta_{\text{trig}} / 2) / \lambda; \) here, \( \Delta \theta \) is the integral breadth. Additional parameters in Eq. (1) are: \( D \), the crystallite size; \( \rho \) and \( b \), the dislocation density and the size of the Burgers vector, respectively; \( B \), a parameter depending on the outer cut-off radius of the dislocations; and \( O \), standing for a higher-order term in \( K(C)^{1/2} \). The variable \( C \) is the dislocation contrast factor and was explained in the literature.\(^\text{15}\) To estimate the dislocation density and the outer cut-off radius, a further calculation of the line profiles is required using the modified Warren–Averbach method:

\[ \ln A(L) \simeq \ln A^5(L) \]
\[ - (\pi b^2 / 2) \cdot \rho L^2 \cdot \ln(R_e / L) \left( K^2(C) \right) + O \left( K^2(C) \right)^2, \quad (2) \]

where \( R_e \) and \( L \) are the effective outer cut-off radius of dislocations and the Fourier variable, respectively; \( A^5 \) is the size Fourier coefficient; and \( O \) stands for a higher-order term in \( K(C) \). Consequently, \( \rho \) and \( R_e \) can be obtained by analyzing \( A(L) \) with Eq. (2). Furthermore, a dislocation arrangement parameter, \( M = R_e \sqrt{\rho} \), is determined using the values of \( \rho \) and \( R_e \). \( M \) parameters that are smaller or larger than unity indicate a strong or weak dipole character of the dislocation, respectively.\(^\text{16}\) With these analytical procedures, the factors relating to dislocations in the ferrite phase can be obtained.

3. Results and Discussion

3.1. Deconvolution of Structural Profile

The measured profiles (\( f_{\text{meas}}^{\text{Fe}} \)) of the ferrite phase are expressed as a convolution of the structural profiles (\( f_{\text{str}}^{\text{Fe}} \)) and the instrumental profiles (\( g_{\text{inst}}^{\text{SPS}} \)) measured at SPring-8.

\[ f_{\text{meas}}^{\text{Fe}} = f_{\text{str}}^{\text{Fe}} \otimes g_{\text{inst}}^{\text{SPS}} \quad \text{(3)} \]

In general, diffraction patterns of LaB\(_6\) powder are used for the instrumental profiles. However, the LaB\(_6\) powder diffraction patterns are not available for the highly directional micro X-rays because of its large grain size. Instead, the CeO\(_2\) powder profiles (\( g_{\text{inst}}^{\text{CeO2}} \)) were used in this study for defining the instrumental profiles. However, the CeO\(_2\) powder profiles include structural profiles (\( g_{\text{str}}^{\text{CeO2}} \)) that originate from structural imperfections, as shown in Eq. (4).

\[ g_{\text{meas}}^{\text{CeO2}} = g_{\text{str}}^{\text{CeO2}} \otimes g_{\text{inst}}^{\text{SPS}} \quad \text{(4)} \]

Thus, \( g_{\text{str}}^{\text{CeO2}} \) need to be evaluated for obtaining \( g_{\text{inst}}^{\text{SPS}} \). \( g_{\text{str}}^{\text{CeO2}} \) were determined from the CeO\(_2\) (\( h_{\text{inst}}^{\text{CeO2}} \)) and LaB\(_6\) (\( h_{\text{lab}} \)) powder profiles, which were measured using the laboratory XRD apparatus.

\[ h_{\text{lab}} = g_{\text{str}}^{\text{CeO2}} \otimes h_{\text{lab}} \quad \text{(5)} \]

A Voigt function, which is a combination of Gaussian and Lorentzian functions, was used in the deconvolution processes of Eqs. (3), (4), and (5). The parameters relating to the Gaussian (\( w_G \)) and Lorentzian (\( w_L \)) widths for \( A = B \otimes C \) were as follows:

\[ w_G^2 (A) = w_G^2 (B) + w_G^2 (C), \quad \text{(6)} \]
\[ w_L (A) = w_L (B) + w_L (C), \quad \text{(7)} \]

Figure 3 shows the \( w_G \) and \( w_L \) of \( g_{\text{str}}^{\text{CeO2}} \) and \( g_{\text{inst}}^{\text{SPS}} \). These fac-
tors for the \( \sigma_{\text{SPS}} \) were smaller than those for the \( \sigma_{\text{COS}} \), indicating that a correction of the instrumental profile is needed when using the CeO\(_2\) powder.

### 3.2. Distribution and Anisotropy of Dislocation Characteristics in the Cold-drawn Wire

Figure 4 shows diffraction patterns obtained at distances of 0, 0.33, and 1 mm from the wire center. The scattering vector in diffraction data was almost parallel to the wire radial direction. Thus, the diffraction patterns shown in Fig. 4 reflect on any microstructure along the wire radial direction. The breadth of the peaks clearly decreased with increasing distance from the wire center, indicating a decrease in microstructural imperfections. The modified Williamson–Hall plots are shown in Fig. 5. Interestingly, the slopes in these plots decreased with increasing distance from the wire center, suggesting that the distortion of the ferrite lattice was relaxed near the surface, whereas the plastic strain of a drawn wire should be generally large near the surface, compared with the center.\(^2\)\(^-\)\(^4\) This effect could be explained by using Eq. (1) and noting that the dislocation density and/or the outer cutoff radius of dislocations decreased with increasing distance from the wire center.

The dislocation density, dislocation arrangement parameter, and crystallite size were estimated from the diffraction profiles for the wire radial direction, and the results are shown in Fig. 6. It is noteworthy that the dislocation density was almost uniform, irrespective of the distance from the wire center, whereas the shear deformation near the surface was more severe compared with that at the wire center.

Fig. 4. Diffraction profiles for the radial direction at distances of 0, 0.33, and 1 mm from the wire center.

Fig. 5. Modified Williamson–Hall plots for different values of radial distance from the wire center.

Fig. 6. Dislocation density, dislocation arrangement parameter (\(M\)), and crystallite size, vs. the distance from the wire center.
center. By contrast, the dislocation arrangement parameter, $M$, decreased with increasing distance from the wire center, suggesting further development of dislocation dipoles near the surface. The arrangement of dislocation dipoles reduces the strain field of dislocations; therefore, the decrease in the slopes of the modified Williamson–Hall plots in Fig. 5 reflects the development of the dislocation dipoles. Because cell walls comprise dislocation dipoles in higher density, cell structures are likely to develop near the wire surface. In accordance with the development of cell structures, the crystallite size became larger near the surface than at the wire center. This may be because the cell interior was enlarged by the dislocation rearrangement resulting from the development of cell structures. It can be concluded that the difference in the shear deformation along the wire radial direction primarily affects the development of dislocation cell structures rather than the dislocation density.

To discuss the directional anisotropy of the dislocation characteristics, diffraction patterns obtained along the wire axial direction were also analyzed. Figure 6 shows the dislocation density, the dislocation arrangement parameter, and the crystallite size for the microstructures along the wire axial direction. In our previous work using the laboratory XRD and the energy dispersive XRD, dislocation characteristics of a specific specimen orientation were analyzed. On the other hand, the XRD system employed in this study revealed the anisotropy of the dislocation characteristics. The parameters for the axial direction changed in a similar manner to those for the radial direction; however, the dislocation density along the axial direction was found to be higher than that along the radial direction. Generally, high dislocation density induces crystallite refinement; nevertheless, our observation indicated that the crystallite along the axial direction was larger than that along the radial direction. This effect may stem from lamellar layer structures aligned parallel to the axial direction. A thin spacing of the lamellae probably prompted the crystallite refinement along the radial direction.

The microstructural anisotropy indicated in Fig. 6 can induce anisotropy of mechanical properties of cold-drawn wires. Therefore, the Vickers hardness was examined for the axial and radial directions of the wire specimen. In Fig. 7, we compare the Vickers hardness distributions in longitudinal and diametrical planes of the wire specimen. The Vickers hardness in the diametrical and longitudinal planes is associated with microstructures in the axial and hoop directions, respectively. It should be mentioned that the characteristics of applied stresses in the hoop and radial directions can be similar in drawing processes because residual stresses along these directions remain similar to a certain extent. Thus, the Vickers hardness in the longitudinal plane can be typically applied for discussing the relationship between the hardness and the dislocation density in the radial direction. There was almost no variation in the Vickers hardness between the wire center and the wire surface, in both planes. This is consistent with the distribution of the dislocation density. On the other hand, the Vickers hardness in the diametrical plane was approximately 20 HV higher than that in the longitudinal plane. This may correlate with higher dislocation density in the axial direction compared with that in the radial direction. The contribution of the dislocation hardening can be estimated as:

$$\sigma(\varepsilon) = M_f \alpha G b^{1/2}, \quad \text{..................(8)}$$

where $M_f$ is the Taylor factor (3), $\alpha$ is a constant (0.24), $G$ is the shear modulus of ferrite (77.5 GPa), and $b$ is the Burgers vector (0.249 nm). The relationship between the hardness, $H_v$, and the yield strength, $\sigma_y$, is, approximately:

$$H_v \approx 3\sigma_y, \quad \text{..................(9)}$$

Using Eqs. (8) and (9), the difference between the hardness values in the axial and radial directions can be estimated to be 26 HV from the average dislocation density in the radial $(2.20 \times 10^{15} \, \text{m}^{-2})$ and axial $(2.82 \times 10^{15} \, \text{m}^{-2})$ directions, given in Fig. 6. The estimated value is almost comparable to the Vickers hardness difference measured in the diametrical and longitudinal planes. It is likely that the hardness anisotropy in Fig. 7 mostly originates from the anisotropic feature of the dislocation density. Thus, it was demonstrated that the microstructural anisotropy in Fig. 6 can explain the anisotropy of mechanical properties of the wire specimen. It should be mentioned that various metal processing such as rolling and pressing can induce the anisotropy of mechanical properties depending on the loading directions. The line-profile analysis based on the micro-beam XRD system used in this study can apply to these materials for discussing the relationship between the anisotropy of dislocation characteristics and the anisotropic mechanical properties.

3.3. Annihilation of Dislocations at Elevated Temperatures

The XRD measurements at elevated temperatures were performed halfway between the wire center and the wire surface, and diffraction patterns were obtained in the wire axial direction. Figure 8 shows variations in the diffraction patterns around the 110 reflection of the ferrite phase. The 110 reflections at a peak position of 4.4 nm$^{-1}$ shifted to lower $K$ with increasing temperature, indicating increased lattice spacing. This can be explained by thermal expansion. On the other hand, diffraction peaks of the cementite phase shifted to higher $K$ with increasing temperature, suggesting
decreased lattice spacing in the cementite phase. It has been reported that a large tensile stress emerges along the axial direction in the cementite phase of cold-drawn pearlitic steels.23) This implies that the large tensile stress relaxes with increasing temperature. The inset in Fig. 8 shows the temperature dependence of the full width at half maximum (FWHM) at a peak of around 4.2 nm$^{-1}$, for the cementite phase. The FWHM clearly decreases with increasing temperature, indicating a recovery due to the micro-strain relaxation or the crystallite growth or both. Nearly constant recovery rate was observed for temperatures up to 633 K.

Thermal vibration at crystal lattice points at high temperature induces a change in line profiles of the XRD pattern. This effect should be removed from the measured profile in the line-profile analysis. Thus, the contribution of the thermal effect was evaluated using a standard specimen. The patenting material was annealed at 773 K for 1 h for obtaining the standard specimen. The line-profiles of the ferrite phase in the standard specimen were measured according to the temperature program shown in Fig. 2. The diffraction peaks of the standard specimen at elevated temperature were fitted using the Voigt function, and the dependences of the Gaussian ($w_G$) and Lorentzian ($w_L$) widths are summarized in Fig. 9. With increasing temperature, the width of the Lorentzian part increased, whereas that of the Gaussian part decreased. In addition, the variations in these widths depended on the reflection indices. Using Eqs. (6) and (7), the thermal effect was eliminated from the measured profiles.

Figure 10 show the temperature dependence of the dislocation density, the dislocation arrangement parameter, and the crystallite size. A decrease in the dislocation density started when the temperature was increased to 473 K and continued gradually up to 573 K; subsequently, annihilation of dislocations proceeded significantly. The dislocation arrangement parameter, $M$, also increased slowly up to 573 K. The increase in the $M$ value is likely to be caused by the increasing fraction of randomly arranged dislocations. This may be explained by preferential annihilation of dislocations in cell walls, where dislocation dipoles with
low \( M \) values accumulate. The \( M \) value increased distinctly above 573 K in accordance with increasing annihilation of dislocations. Elimination of cell walls can enlarge the coherent domains for X-ray scattering; in this case, the crystallite size should increase with such changes in the dislocation density and the dislocation arrangement parameters. The growth behavior of the crystallites also suggests that the dislocation recovery preferentially occurred at the cell walls. It is interesting to note that the recovery of the ferrite phase progressed differently from that of the cementite phase. Whereas the cementite recovery progressed at a nearly constant rate with increasing temperature, the ferrite recovery rate changed at 573 K. Therefore, the microstructural modification of the ferrite phase likely occurs independently of that of the cementite phase.

4. Conclusions

The distribution and the anisotropy of dislocation characteristics in cold-drawn wires of pearlitic steels were analyzed using XRD line-profile analysis. The main concluding remarks are as follows:

1. While the dislocation density changes little, irrespective of the radial distance from the wire center, the cell structure evolves toward the surface from the wire center. Higher plastic strain near the surface enhances the rearrangement of dislocations, resulting in the development of microstructural anisotropy, in which cementite/ferrite lamellae align parallel to the wire axial direction.

2. The dislocation density in the wire axial direction is higher than that in the radial direction. This may originate from significant micro-structural anisotropy, in which cementite/ferrite lamellae align parallel to the wire axial direction.

3. The recovery of the ferrite phase progressed slowly up to 573 K and became significant above this temperature. This behavior is less related to the recovery of the cementite phase.

4. The changes in the dislocation arrangement parameters and the crystallite size for the ferrite phase with increasing temperature suggest that the dislocations in the ferrite phase annihilate preferentially at the cell walls at elevated temperature.

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