Line profile analyses of a martensitic steel during continuous and stepwise tensile deformations

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Abstract. Dislocation characteristics in an as-quenched 22SiMn2TiB martensitic steel during tensile deformation were monitored by in-situ time-of-flight neutron diffraction combined with the Convolutional Multiple Whole Profile fitting analysis. Two loading conditions, continuous and stepwise followed by unloading, were adopted in the experiments. The diffraction patterns both in the loading (axial) and the transversal directions were measured simultaneously. The dislocation densities obtained from the experiments behaved differently in two loading conditions and in two measured directions, respectively. The different behaviour was mainly due to the increase of intergranular strains with the increase of deformation, and the profiles measured in the axial direction in the loading condition of stepwise followed by unloading gave most reliable dislocation characteristics among the profiles measured in other conditions.

1. Introduction

The density, the arrangement and the character of dislocations are important factors to understand the mechanical properties of materials. These characteristics are often observed directly using a transmission electron microscope (TEM) [1]. Because the TEM observation needs thin film samples and performs only in a limited area, it is difficult to observe the in-situ evolution of dislocation characteristics in the bulky material during a mechanical test. Meanwhile, the microstructures of materials are also often evaluated from the peak broadening in X-ray or neutron diffraction profiles [2]. In particular, neutron diffraction is a powerful tool for the microscopic evaluation of bulky materials owing to its high permeability.

The dislocation density in a martensitic Fe-18Ni steel have been studied using X-ray [3] and neutron [4] diffraction through the classical Williamson-Hall plot [5]. These studies were done based on the peak width and concluded that the dislocation densities decreased with the increase of plastic deformation. These results have been judged from the decrease of diffraction peak width observed after the plastic deformation. The Convolutional Multiple Whole Profile (CMWP) fitting method [6, 7] is a state of the art technique for line profile analysis based on physically modelled profile functions for dislocations, crystallite size and planar defects. This technique evaluates the whole peak profile including the tail as well as the width. The in-situ neutron diffraction combined with the CMWP fitting method should provide better understanding for the microstructural evolution particularly the dislocation characteristics in steels during deformation.

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Recently, many high-resolution time-of-flight (TOF) neutron diffraction instruments built at high-intensity pulsed neutron sources are available for *in-situ* observation of microscopic characteristics, such as lattice strain, texture and phase transformation, of materials during mechanical tests. However, there are only few studies on the dislocation characteristics of steel have been reported using these instruments. In the present study, neutron diffraction patterns of a martensitic steel during tensile deformation were measured, and the evolution of dislocation characteristics were evaluated using the CMWP fitting. Two types of loading conditions were adopted for the deformation, in order to verify which loading condition is more appropriate for the dislocation characterization.

### 2. Experimental and analyses

#### 2.1. Neutron diffraction measurement

Rod shaped specimens of an as-quenched lath martensitic 22SiMn2TiB steel [8] were used in this study. The diameter and the length of the parallel part were 5.0 mm and 30 mm, respectively. *In-situ* neutron diffraction measurements during tensile tests were performed using the Engineering Materials Neutron Diffractometer TAKUMI [9] at the Materials and Life Science Facility (MLF) of Japan Proton Accelerator Research Complex (J-PARC). The specimen was mounted horizontally in the loading machine sitting on the sample stage of TAKUMI, in which the loading axis was set as 45° to the incident neutron beam, as shown in figure 1. Because TAKUMI has two detector banks that have the scattering angles of +90° and -90°, respectively, the diffracted neutron with the scattering vectors parallel and perpendicular to the loading axis can be measured simultaneously. In figure 1, the geometries of the wave vectors of the incident neutron \( \mathbf{k}_1 \) and the diffracted neutron \( \mathbf{k}_2 \), and the scattering vector \( \mathbf{Q} \) for both the loading (axial) and the transversal directions are described. The gauge volume was restricted to be 5.0 × 5.0 × 5.0 mm³ using the incident beam slit and the radial collimators. The instrumental peak resolution was set to be about 0.3 % by the tuning of incident beam divergence. Two tensile loading conditions were adopted in this study; a continuous loading and a stepwise loading. In the continuous loading, the deformation was applied to the specimen until its fracture in a constant crosshead speed with the initial strain rate of \( 1 \times 10^{-5} \text{ s}^{-1} \). The diffraction patterns measured for the several strain states marked in figure 2 were extracted for the analyses. The time widths for the diffraction pattern extraction were 300 s and 150 s. Because TAKUMI adopted the event-recording technique for the neutron diffraction data acquisition system, the time slicing for extracting intensities in the arbitrary time region from whole data after the measurement was available. In the stepwise loading, the deformation was applied step by step in the same crosshead speed to arbitrary strains followed by unloading for 1800 s. Diffraction patterns related to the unloaded states after deformations were extracted for the CMWP analyses. Figure 2 shows the stress-strain curves of the steel for the different loading conditions.

![Figure 1. Schematic drawing of the in-situ neutron diffraction experiment at the TAKUMI.](image-url)
2.2. Line profile analysis

Prior to the CMWP fitting, the behaviour of peak width according to the strain was quickly evaluated by the fitting with a simple Gaussian function. The obtained full width at half maximum (FWHM) values of 110 diffraction peaks for both the axial and the transversal directions are plotted in figure 3 as a function of strain. The FWHM values for all the data decrease largely in the strain range between 0% and 2%. For the continuous tensile loading condition, the FWHM values of the data extracted with the time width of 300 s and 150 s are the same within the error bars. These may show that the peak broadening due to a wider range of applied stress that may appeared in the wider extracting time width is negligibly small. The FWHM values of the continuous loading are larger than those of the stepwise loading.

The line profile analyses using the CMWP fitting method were carried out for the data that were extracted with 300 s time width in order to get better statistics for dislocation characterization. The diffraction peak profiles obtained from LaB₆ powder (NIST, SRM 660a) measured in the same instrumental conditions as the in-situ neutron diffraction measurements were used as the instrumental resolution function for the analyses.

Figure 2. The stress-strain curves of the 22SiMn2TiB steel obtained from tensile deformation tests in the stepwise and the continuous loading conditions for in-situ neutron diffraction measurements. The circles on the curve for the continuous loading data indicate the conditions of the data used for the line profile analysis.

Figure 3. Strain dependences of FWHM values of 110 diffraction peak in (a) the transversal direction and (b) the axial direction.
Figure 4 shows the observed and CMWP fitted diffraction patterns of 22SiMn2TiB steel before deformation. Diffraction peaks that have much smaller intensities in the figure indicate the existence of retained austenite in the specimens. Although these austenite peaks were also fitted, reasonable values could not be obtained due to the small fraction of austenite. The mass fraction of austenite in the specimens estimated by Rietveld refinement was about 3%.

3. Results and discussions

From the CMWP fitting, dislocation density (\( \rho \)), dislocation character (edge or screw), crystallite size, etc. were obtained. In this study, results concerning \( \rho \) and \( M^* \) will mainly be discussed. The value of \( M^* \) being larger than 1.0 indicates a random arrangement, and that lower than 1.0 indicates a dipole arrangement or a strongly correlated arrangement of dislocations [10, 11]. The values of \( \rho \) and \( M^* \) obtained from the CMWP fitting are shown in figure 5. In the transversal direction, almost the same behaviors of \( \rho \) and \( M^* \) between the stepwise and continuous loadings are found in figures 5 (a) and (c). The \( \rho \) values increase gradually from \( 5.0 \times 10^{15} \) m\(^{-2}\) to \( 10^{16} \) m\(^{-2}\) order with the increase of plastic strain. The \( \rho \) value before deformation is slightly larger than the value from TEM observation of the lath martensitic steel with similar carbon content (0.18 mass %) [12], but considering the physical form (bulk or thin-foil) of the specimens, these values are in good agreement. Meanwhile, the ordering of the dislocation arrangement by the deformation was found from the largely decreasing of \( M^* \) according to the increasing strain. The strong correlation of dislocations gives the narrower widths and the sweeping tails of diffraction peaks, i.e., the peak shape changes from the Gaussian to the Lorentzian. [10]. The FWHM values shown in figure 3 are reflecting this phenomenon. On the other hand, different behaviors of \( \rho \) for the continuous and stepwise loadings were found in the axial direction as shown in figure 5 (b). Although \( \rho \) for the stepwise loading hardly changes during deformation, \( \rho \) for the continuous data increases apparently. More detailed discussions about the relationship between the dislocation characteristics and the work-hardening mechanism of the steel can be found in another paper [13]. In the stepwise loading condition, the diffraction peak profiles that were used for the analysis were not affected by the elastic strain because the diffraction intensities during the unloaded states were used for the analysis. In contrast, in the continuous loading condition, the elastic strain caused by the applied load may affect to the observed diffraction peak profiles. If the homogeneity of elastic strain in a specimen is sufficiently high, only the peak shift will be observed in a diffraction pattern. Otherwise, peak broadening due to elastic strain heterogeneity caused by an applied load will appear. The effect of elastic strain heterogeneity to the peak width can be found in the larger FWHM values for the continuous loading data in figure 3. The effects of the elastic strain for the diffraction peak profile should be eliminated in order to evaluate the dislocations more accurately through the line profile analysis. The stepwise loading condition is more appropriate than the continuous one for the \textit{in-situ} diffraction measurement aiming at the microstructural study of the steels during tensile deformation.
The dislocation density \( \rho \) in the axial direction is smaller than that in the transversal direction for the stepwise condition. Here, the scattering vector of neutrons measured in the axial direction lie in the loading axis as mentioned above. In other words, the lattice planes that contributed to the diffraction signal were always normal to the loading axis. The crystal grains that contributed to a certain \( hkl \) diffraction peak received the load in a same crystal orientation. In contrast, scattering vector in the transversal direction is perpendicular to the loading axis. The lattice planes that contributed to the diffraction signal were parallel to the loading axis. Because the orthogonal direction to the scattering vector is not always parallel to the loading axis, the crystal orientations of the loading direction are different for different sets of grain families. The peak profiles that were observed in this direction were highly affected by intergranular strains in this situation. The dislocation densities that were evaluated from the transversal data are larger than that were evaluated from the axial data due to the large elastic strain heterogeneity of the intergranular strain that might cause peak broadening. For this reason, the diffraction pattern measured in the axial direction should be used for the analysis to investigate the behaviour of the dislocation density during tensile deformation.

**Figure 5.** The dislocation density of the 22SiMn2TiB steel in (a) the transversal direction and (b) the axial direction, and the dislocation arrangement parameters in (c) the transversal direction and (d) the axial direction obtained from the line profile analyses using the CMWP fitting method.
4. Summary
Line profile analyses of 22SiMn2TiB martensitic steels during tensile deformations were performed. Both the continuous loading and the stepwise loading conditions were adopted for the in-situ neutron diffraction measurements using the high resolution and the high intensity time-of-flight (TOF) neutron diffractometer TAKUMI at the MLF of J-PARC. The analyses of the diffraction profiles measured with these two conditions in the axial direction gave different behaviours of the dislocation densities. The dislocation density $\rho$ and the dislocation arrangement parameter $M^*$ in the axial direction obtained from the continuous loading are larger than those from the stepwise loading, because the elastic strain (lattice strain) heterogeneity was introduced in the diffraction profiles during the data collection under applied stress. The elastic strain heterogeneity is also larger in the transversal direction than in the axial direction leading to the larger observed dislocation density. The diffraction peak profiles measured in the unloaded state and in the axial direction thus should provide more reliable information of dislocation characteristics through line profile analysis.

The line profile analysis for in-situ TOF neutron diffraction data using the CMWP fitting method is effective for microstructural study of steels in mechanical tests. Further verification of experimental conditions, such as a search of the standard sample for the instrumental resolution function and an optimization of the incident beam divergence, is needed in order to evaluate dislocation characteristics more accurately.

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