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Relationship between dislocations and residual stresses in cold-drawn pearlitic steel analyzed by energy-dispersive X-ray diffraction

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ABSTRACT

We analyzed the dislocation distribution of cold-drawn pearlitic-steel wire by using the line-profile analysis based on the energy dispersive X-ray diffraction (EDXD). Although this line-profile analysis requires a high resolution in reciprocal space, the resolution for EDXD is generally poor due to the energy resolution of the detector. Our analysis demonstrated that the resolution in the reciprocal space can be maximized at small scattering angles. Using the line-profile analysis based on the EDXD, the microstructural parameters such as the crystallite size and the dislocation density of the ferrite phase in the pearlitic steel were successfully analyzed. In addition, the distribution of the residual stress of the ferrite phase of a pearlitic steel wire was also analyzed using the EDXD measurement.

1. Introduction

High-strength pearlitic steel wires, which are widely used as suspension cables for bridges and steel cords for automobile tires, are produced through a cold-drawing process. In this process, structural reinforcements are achieved by work hardening of the microstructures of ferrite and cementite lamellae. The evolution of the microstructural deformation during the cold drawing causes residual stresses, which affects the mechanical properties and durability. A number of studies have analyzed the residual stresses in cold-drawn pearlitic steel using X-ray and neutron diffraction techniques [1–4]. Residual stress analysis using neutron diffraction indicates that the average residual stress in a ferrite phase is compressive with a few hundred MPa along the drawing direction [1]. Recently, it was reported that the residual stress is not uniform through the wire cross section: Although the compressive residual stress exceeds 600 MPa at the core of the wire, the residual stress is greatly reduced at the surface [4]. However, in contrast to the well-researched residual stress, the distribution of the microstructural features, such as dislocations and subgrains, has not been well analyzed in the cross-section of the wire.

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The microstructural features relating to dislocations can be analyzed using line profiles of X-ray or neutron diffraction. Therefore, the diffraction methods can be a promising candidate for analyzing the residual stress and the microstructural features. Tomota et al. successfully characterized the dislocation-induced microstrain and the residual strain for pearlitic steel simultaneously using neutron diffraction [5]. However, although the high transparency of neutron in steel is favorable for non-destructive and in-situ measurements, it is difficult to reduce the size of the incident beam to the micrometer scale. Therefore, the analytical data obtained by the neutron diffraction correspond to the values averaged over the whole cross section of the specimen. High-energy X-rays, which are available from synchrotron radiation, also penetrate steel. These X-rays allow the beam size to be reduced to the micrometer scale. The spatial resolution of the X-ray diffraction (XRD) measurement using a micro-sized incident beam will then permit scanning across the entire cross section of the specimen.

Among the several theories relating to diffraction profiles for microstructural analysis, the modified Williamson–Hall and Warren–Averbach procedures [6–8], based on the crystallographic strain anisotropy of dislocations, provide the most reliable values of dislocation density and subgrain size. However, since a series of diffraction peaks with good counting statistics up to high-index planes need to be collected for analysis, a substantial amount of time is required for the measurements. Moreover, X-ray diffraction intensity is low owing to the micro-sized incident beam, and the whole measurement time will be multiplied with the several measurement points within a specimen. Therefore, to save time, the energy-dispersive X-ray diffraction (EDXD) method, which obtains a series of diffraction peaks without the angular scan, can be a powerful tool for scanning the specimen in the XRD measurement. In previous research, Shibano et al. observed the increase in the full width at half maximum (FWHM) of diffraction profiles measured near the fatigue crack tip in a steel specimen using the EDXD method [9,10]. One of the main drawbacks of the EDXD method, however, is the low resolution of reciprocal space compared to the conventional angular scan method. This is because the energy resolution of a solid-state detector (SSD), which is used for the EDXD measurement, impairs the reciprocal-space resolution. If the line broadening observed in the EDXD measurement is close to that of the instrumental line broadening, it becomes considerably difficult to deconvolute the line profile originating from microstructural defects from the observed line profile. Meanwhile, the reciprocal-space resolution in the EDXD method is a function of scattering angle, and the effect of the energy resolution of the SSD on the reciprocal-space resolution can be reduced at small scattering angles. In fact, at small enough scattering angles, a fine oscillation pattern can be successfully obtained in X-ray reflectivity profiles for thin films on substrates [11,12]. Therefore, we expect that the line-profile analysis can be carried out in the energy-dispersive mode with better reciprocal-space resolution by setting an appropriate scattering angle.

In this work, the distributions of microstructural parameters across the section of a cold-drawn pearlitic-steel wire were determined using the line-profile analysis combined with the residual stress analysis. We employed the EDXD using high-energy white X-rays for these analyses. This article contains our description of the experimental conditions for the line-profile analysis in the EDXD mode.

2. Experimental

A straight wire of pearlitic steel with carbon compositions of 0.84 mass% around the eutectoid point were produced for this research by Tokyo Rope Mfg. Co., Ltd. The chemical composition (in mass percentage) of the steel was 0.84% C, 0.21% Si, 0.75% Mn, 0.021% P, and 0.005% S. The wire was cold drawn from the initial radius of 2.30 mm to 2.03 mm (true strain: 0.25). Fig. 1 shows the example of the ferrite and cementite lamellae structure of the drawn specimen observed by transmission electron microscopy (TEM). The TEM image was taken at the center of the wire. Since the plastic deformation was small at the true strain of 0.25, the texture evolution was weak. Consequently, the lamellae structure was not necessarily parallel to the axial direction of the wire. The ferrite phase, about 100 nm thick, was sandwiched between thin cementite layers. In addition, the distance between dislocations is about a few tens of nanometers, suggesting that the dislocation density can be roughly estimated to be 1×10^{15}–2.5×10^{15} m^{-2}. For the XRD measurements, the wires were cut perpendicular to the axial direction at the length of 3 mm.

EDXD measurements were carried out at the BL28B2 beamline of SPring-8 (Japan). The diffraction patterns for both the line-profile analysis and the residual stress analysis were collected with a Ge-SSD (GLP-16195/10P4, ORTEC) mounted on the diffractometer. Energy calibration of the Ge-SSD was conducted using diffraction patterns of LaB_6 powder (SRM 660b, NIST).

Stress analysis was carried out using the 110 reflection from a ferrite phase. The scattering angle 2θ was fixed at 3.2°, where the 110 reflection appears at high energy over 100 keV.

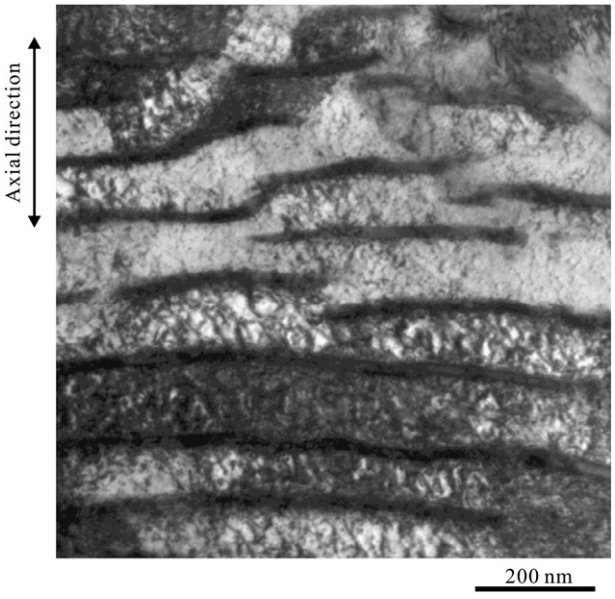

Fig. 1 – Transmission electron microscope micrograph of the pearlitic lamellae deformed by the cold-drawn wire used in this study.