Strength Anisotropy and Residual Stress in Drawn Pearlite Steel Wire

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Strength anisotropy is found by tensile and compressive tests for a drawn pearlite steel wire, where the compressive tests are performed for prepared specimens along directions of 0, 45 and 90 degrees with respect to the drawing direction. The influence of annealing on such anisotropic strength is also examined. To make clear the origin of the strength anisotropy, texture and residual strain are measured using neutron diffraction. It is revealed that residual stresses in the ferrite and cementite phases are the cause of strength anisotropy.

KEY WORDS: neutron scattering; pearlite steel; cold working; texture; phase stress.

1. Introduction

Fujita et al. determined that the strength of drawn pearlite steel wire along the drawing direction was higher than that along the transverse direction by performing a skillful tensile test on tiny specimens. However, they were unable to discover the reason for this anisotropy, which is important in the development of advanced pearlite steel wire. Because of the difficulty of the sample preparation, any other report concerning with the strength anisotropy of drawn pearlite steel cannot be found. Then, in this study, anisotropy in the yield strengths of hyper-eutectoid steel specimens prepared under a variety of thermo-mechanical heat-treatment parameters were determined by tension and compression tests.

Two possible reasons for the strength anisotropy are examined, texture and residual stress. Texture was measured using X-ray and neutron diffraction. A specimen annealed at 698 K was found to exhibit fiber texture as intense as that in as-drawn specimens. However, it had almost the same tensile and compressive yield strengths. Residual strain was next measured along five different directions with respect to the drawing direction. Residual stresses are found to develop by inhomogeneous plastic flow of ferrite and cementite plates. The relationship between strength anisotropy and residual stress is determined, giving conclusions that may be of use in the understanding of the ultra-high strength of drawn pearlite steel wire.

2. Experimental Procedures

The chemical composition of steel used in this investigation was 0.853 C, 0.26 Si, 0.82 Mn, 0.011 P, 0.0061 S, 0.0049 N, 0.020 Al, 0.045 Cr and 0.0007 O [mass%]. Round bars of diameter 11 mm were patented to produce a fully pearlite microstructure, and were used as specimen P1. Some of the bars were drawn into 5.5 mm-diameter wire, an area reduction of 75 % (specimen P2). Drawn wire was aged at 423 K for 12.6 ks (specimen P3), at 698 K for 0.6 ks to simulate conditions during Zn-plating (specimen P4), or at 963 K for 18 ks to produce spherical cementite particles (specimen P5). These specimen preparation processes are the same as used in a previous study by Tomota et al. in which specimen preparation and microstructure are described in detail.

Compression tests were carried out at 300 K using a gear type testing system with a crosshead speed of 0.5 mm/min. Specimens of size 3×3×4 mm were prepared such that the angle ψ between the drawing direction and the compression direction was 0, 45 or 90°, as illustrated in Fig. 1. The tensile testing procedure used is reported in Ref. 2).

The texture of P1 (110) was evaluated from (110) pole figures measured by X-ray diffraction using the Schulz reflection method with nickel-filtered Cu Kα radiation. The geometrical arrangement is shown schematically in Fig. 1. The angle between the compression direction and neutron diffraction scanning direction is represented by χ. Both ψ and χ were scanned over a range of 0 to 90° at 5° intervals. Diffraction measurements of strain in crystalline materi-
als are based on precise measurement of the deviations of lattice plane spacing \( d_{hkl} \) of particularly oriented \( hkl \)-crystal planes due to the effect of stress.

Lattice strain \( \varepsilon_{hkl} \) can be calculated from the difference between measured lattice spacing \( d_{hkl} \) and the stress-free lattice spacing \( d_{0hkl} \). These can be calculated from the shift in the angular positions of diffraction peaks \( q_{hkl} \) according to:

\[
\varepsilon_{hkl} = \frac{d_{hkl} - d_{0hkl}}{d_{0hkl}} = -\cot(\theta_{hkl}) \Delta \theta_{hkl},
\]

Residual phase strains of specimens P1 to P5 were measured by conventional \( q-2q \) neutron diffraction using RESA at JAERI. Lattice plane spacing of the (110) ferrite in the samples was measured using specimen rotation angles of 0, 22.5, 45, 67.5 and 90° with respect to the drawing direction. Example diffraction profiles are shown in Fig. 2. As has been demonstrated by Acker et al., Voight functions give a better fit to the diffraction curves of P2 specimens than the commonly used Gaussian. However, as only residual stress is to be measured, diffraction peak positions are determined using Gaussian fits for simplicity, with the results shown in Fig. 2. Stress-free lattice spacing \( d_{0hkl} \) was taken from sample P5, which was well annealed and assumed to be stress-free, as was successfully done in Ref. 2). The stress-free lattice spacing was almost identical to the ASTM value for pure iron. For specimen P2, (200) and (211) spectra were also measured. Because of the intense texture in P2, the statistically reliable (110) profile was easily obtained in 4.5 ks (see Fig. 2(a)) but the (200) profile was difficult to analyze under the same amount of exposure time (see Fig. 2(b)). However, if discussion is limited to peak shift, \( i.e. \), lattice plane strain, the results are useful.

3. Results

Figure 3 shows microstructures of specimen P4. The cementite plate was apparently recognized even after drawing by 75% of reduction in area and heat treated at Zn-plating simulated condition. As is observed in Fig. 3, pearlite colonies are elongated along the drawing direction. When sever plastic deformation was given to a specimen with pearlite structure, cementite plates have been reported to become nano-sized particles and to dissolved partially into the ferrite matrix. By the TEM observations of these specimens, the cementite plates become small spherical particles partially but recognized even after the drawing and annealing.

Figure 4(a) shows the (110) pole figure of the ferrite phase of specimen P1 obtained by X-ray diffraction. No areas of high pole density can be seen, indicating a random distribution of orientations. The evolution of texture due to drawing from 11 to 5.5 mm diameter can be seen in the (110) ferrite phase. Pole densities were projected onto a plane perpendicular to the extrusion direction and normalized against mean pole density. A distribution circularly symmetric about the center is observed, with a maximum intensity of 6.8 times average at the center. Another concentric circle distribution is apparent at an inclination of approximately 60° from the center. This represents the formation of (110) fiber texture in the ferrite phase. The pole figure of specimen P4 is quite...
similar to that of specimen P2, as shown in Fig. 4(c).

Figure 5 shows the flow curves observed by the compressive test for specimen P2. They are apparently dependent on the angle $\psi$. The yield strength along 90° is higher than that along 0°, which is the reverse result for the tensile strength reported by Fujita et al.\textsuperscript{1}) The flow stress along 45° is between them. Judging from Fig. 5, the 0.2% (0.002) flow stress is appropriate to be employed for such a comparison. Thus, the 0.2% proof stresses observed by the compression test are tabulated in Table 1 in which the tensile 0.2% proof stresses reported in Ref. 2) are also listed.

A difference in tensile and compressive strengths is found in P2 and P3, whereas, tensile and compressive strengths are almost same for the other specimens.

Figure 6 shows ferrite (110) lattice plane strain as a function of measuring direction (angle $\psi$ in Fig. 1). As can be seen, residual elastic strains in specimens P2 and P3 are relatively large, and exhibit a strong direction dependence compared to those in P1, P4 and P5.

4. Discussion

4.1. Effects of Annealing on Texture, and Its Relationship to Strength Anisotropy

The (110) fiber texture is evolved by the drawing and it hardly changes by annealing at 698 K. Hence, the specimens P2 and P4 exhibit the similar pole figures in Fig. 4. Although the texture is suspected to induce anisotropic strength, Table 1 reveals that such a strength anisotropy is found in specimen P2 but not in P4. It is evidently concluded that the texture is not the reason of the strength anisotropy observed in specimens P2 and P3.

Table 1. Measured tensile and compressive yield stresses of the different samples (GPa).

| Specimen | Tension | Compression |
|----------|---------|-------------|
|         | $\psi=0^\circ$ | $\psi=45^\circ$ | $\psi=90^\circ$ |
| P1       | 1.00    | 1.05        | 0.98          | 1.05        |
| P2       | 2.05    | 1.45        | 1.53          | 1.58        |
| P3       | 2.15    | 1.55        | 1.72          | 1.77        |
| P4       | 1.60    | 1.60        | 1.67          | 1.52        |
| P5       | 0.70    | 0.57        | 0.69          | 0.58        |

Fig. 6. Residual (110) lattice plane strains of specimens P1 to P5 as a function of measuring angle.
4.2. Effect of Residual Phase Stress on Strength Anisotropy

Since specimens were subjected to drawing, two commonly known origins of residual stresses are thought to exist, “macroscopic residual stress” due to differences in the plastic flow between the outer and inner parts of the wire, and “phase stress” caused by misfit strain between ferrite and cementite. Macroscopic residual stress and phase stress have been investigated in pearlite steels using X-ray diffraction and more recently using neutron diffraction. The gauge volume of neutron diffraction used in the present study covers the whole cross-section of wire, so that the macroscopic stresses are cancelled out and that the phase stress averaged in the ferrite matrix are measured. The results shown in Fig. 6 are such phase stresses in the ferrite matrix.

In Fig. 6, the residual elastic strain determined from (110) spacing in specimens P2 and P3 increases with increasing of the angle $\psi$ from 0 to 67.5°. The value at 90° is lower than that at 67.5°, which is somewhat puzzling. Then, we examined the case of uniaxial tensile plastic deformation and obtained the results inserted in Fig. 6 where the residual elastic strain determined from (110) spacing after tensile deformation for the specimen P1 and P4 are plotted. As is clearly seen, the tendency found for P4 is quite similar to the present drawing case. Because the texture of P1 is not obvious, the lower (110) lattice plane strain at 90° in P4 is postulated to stem from the textured wire and “macroscopic residual stress” due to differences in macroscopic residual stress and the drawing direction $\psi$ degree.

The yield stress anisotropies of specimens P2 and P3 are therefore thought to be related to residual strain. The large residual compressive strains in P2 and P3 appear to contribute to higher tensile yield strengths and lower compressive yield strengths.

Because of sharp (110) texture of specimen P2, the residual strain in the ferrite matrix of P2 may be estimated to be $-0.002$ at 0° and approximately $+0.0008$ at 90° from Fig. 7 which will be discussed later in 4.3 (0.0008 is the average of residual strains from (110), (200) and (211) which is used for the first approximation.). The average residual stress tensor in the ferrite matrix is thus estimated using Hooke’s equation taking Young’s modulus $E=210$ GPa, the Poisson ratio $\nu=0.28$, $\varepsilon_{11}=-0.002$ and $\varepsilon_{22}=\varepsilon_{33}=+0.0008$, where $\chi_i$ is taken as the drawing direction giving:

$$\sigma_i=\begin{bmatrix} -370 & 0 & 0 \\ 0 & 89 & 0 \\ 0 & 0 & 89 \end{bmatrix} \text{ (MPa) } \quad (2)$$

Using the Tresca yielding criterion, which defines a relationship between the principal stresses $\sigma_1>\sigma_2>\sigma_3$ and the yield strength of ferrite without residual stress $2k$, as $|\sigma_1-\sigma_3|=2k$, leads to the following expressions for tensile yield stress $\sigma_{11}^{\text{TEN}}$ and compressive yield stress $\sigma_{11}^{\text{COMP}}$:

$$\sigma_{11}^{\text{TEN}}=2k+370+89 \text{ (MPa)} \quad (3a)$$

$$\sigma_{11}^{\text{COMP}}=-2k+370+89 \text{ (MPa)} \quad (3b)$$

Inputting the observed values of $\sigma_{11}^{\text{TEN}}$ and $\sigma_{11}^{\text{COMP}}$ from Table 1 into Eq. (4) gives $\sigma_{22}^{\text{COMP}}=1.75$ GPa, which is not so different from the observed 1.58 GPa (see Table 1). In the case of P3, Eq. (4) gives 1.85 GPa, which is even closer to the observed value of 1.77 GPa.

Thus, phase stress appears to be the main reason for yield strength anisotropy. The residual stress tensor of Eq. (2) needs to be balanced with that of cementite. The phase stress of cementite is estimated to be

$$\sigma_{11}^{\text{cementite}}=\sigma_{11}^{\text{ferrite}}(1-\gamma)/\gamma \quad (5)$$

where $\gamma$ refers to the volume fraction of cementite, which is 0.14 in this case. Taking $\sigma_{11}^{\text{cementite}}=370$ MPa into Eq. (5) then gives $\sigma_{11}^{\text{COMP}}=2273$ MPa, which compares well with values reported in Refs. 2 and 3.

The above interpretation means that the yielding surface in the stress-space moves corresponding to the phase stresses given by Eq. (2). Hence, it is acceptable that the yield strength at 45° is between those of 0° and 90° and close to that at 90° for the specimen P2 or P3 in Table 1.

4.3. Effect of Crystal Orientation on Residual Stress

Residual strains in the (200) and (211) lattice planes of specimen P2 were measured with the expectation that features of orientation dependence would be observed. Results are shown in Fig. 7. As can be seen, the dependence of measuring direction exhibited by the (200) and (211) strains is different to that of (110), although an overall trend for strain to increase (compression to tension) as the measuring angle is increased from 0 to 90° is exhibited in all of the measurements. The datum for (200) at 0° is connected by a dashed line because the diffraction intensity of the
measurement was extremely low. Taking the 110 fiber texture into consideration, the averaged residual strain in the ferrite matrix is roughly estimated to be \( \frac{0.002}{H_{1100}} \) at 0° while 0.0008 at 90° which were used for calculation in Sec. 4.2.

Figure 8 illustrates how residual strain is distributed through two kinds of grains, one softer and one harder, after plastic deformation. In this study, softer grain coincides with ferrite phase and harder coincides with cementite, respectively. As can be seen in Figs. 8(a) and 8(b), the softer grains show preferential plastic flow resulting in compressive strain at 0° and tensile strain at 90°. The residual stresses (or strains) are illustrated in Figs. 8(c) and 8(d) and summarized as Mohr’s circles in Figs. 8(e) and 8(f). In the case of phase stress, residual stress in the ferrite phase is compressive at 0° and becomes tensile at 90°, following the Mohr’s circle shown in Fig. 8(e).

On the other hand, the microstructure of pearlite is divided into blocks of what used to be austenite. Each block consists of several lamellae of ferrite and cementite plates with identical ferrite orientations. From the result of this study, two new origins of residual stress are proposed, “block stress” caused by the dependence of flow stress on orientation in the ferrite matrix, and “lamellar stress” caused by differences in flow stress between ferrite–cementite lamellae with different inclinations. The block stress and phase stress were thought to be intermixed and resulted in the feature of Fig. 7. A discussion of lamellar stress and rather minor variations in residual strains between (110), (200) and (211) oriented grains, i.e., block stress, will be presented in a later publication.8)

5. Conclusions

As-drawn specimens and those aged at 423 K after drawing were found to exhibit large differences between tensile and compressive strengths. Strength anisotropy was confirmed by compression tests of specimens prepared at directions of 0, 45° and 90° with respect to the drawing direction, with the anisotropy found to disappear in specimens annealed at temperatures over 698 K. Although as-patented specimens exhibited weak texture, strong (110) fiber texture was produced by the drawing process, and was not removed by annealing at 698 K. Residual lattice plane strains were observed in as-patented specimens, and although not relaxed by annealing at 423 K, relaxed to a negligible level at 698 K. Because the specimen that was annealed at 698 K exhibited (110) texture, little residual stress and little yield strength anisotropy, residual stresses in the ferrite and cementite matrices are believed to be the cause of strength anisotropy in as-drawn wire. Consequently development of residual stress may be responsible to produce the ultra-high strength pearlite steel wire. The influence of selected \((hkl)\) on lattice plane strain is thought to be caused by heterogeneous deformation among blocks with different orientations accompanying block stress.

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