Elastic Strains of Cementite in a Pearlite Steel during Tensile Deformation Measured by Neutron Diffraction

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Lattice plane strain, i.e., elastic strain, in cementite plates embedded in the ferrite matrix was measured by in situ neutron diffraction during tensile deformation for a hypereutectoid pearlite steel. The employment of time-of-flight method and microstructure control enable us to measure the shift of cementite peaks along tensile and transverse directions at the applied stress up to 1.6 GPa. The highest elastic strains of cementite determined was approximately 0.015. Heterogeneous plastic deformation between ferrite and cementite as well as among ferrite blocks are discussed.

KEY WORDS: neutron diffraction; pearlitic steel; cementite; strength; tensile deformation.

1. Introduction

Much attention has been paid on the maximum strength of cementite in steel. The cementite strength was measured to be 4–8 GPa by bending test of tiny cementite plates extracted from a cast iron while 3.2 GPa is the highest value observed so far by compression test of bulky fine-grained cementite. Cementite plates in pearlite structure bend and/or elongate easily by plastic deformation but the strength of cementite has not been made clear. By employing the in situ neutron diffraction, the strength of cementite plates embedded in the ferrite matrix was predicted from the elastic strains of the ferrite matrix determined from the (110) lattice spacing measurement and the highest stress loaded in cementite was estimated to be approximately 5 GPa. It is, however, not verified directly from the cementite peak shift. When we used patented samples to measure such cementite peak shift by using an angler dispersion (AD) method, the tensile strain corresponding to approximately 3 GPa was observed in cementite. Then, the direct measurements of cementite lattice plane strains along the tensile and transverse directions were performed by using a time-of-flight (TOF) neutron diffraction during tensile loading in this study. To increase the strength of pearlite, a patented specimen was swaged to decrease the lamellar spacing and then annealed to remove the residual stress.

2. Experimental Procedures

Chemical compositions of a steel used are 0.82C, 0.23Si, 0.73Mn, 0.008P and 0.009S in mass%. To obtain fully pearlitic structure, specimens were austenitized at 1273 K for 0.6 ks followed by isothermal holding at 823 K for 1.2 ks and air cooling. To make lamellar spacing finer with little residual phase stress, cylindrical specimens with 11 mm in diameter were swaged to 6 mm, i.e., true strain of 1.2, followed by reheating at 673 K for 1.8 ks; lamellar spacing became finer, the 110 fiber texture was evolved, and residual phase stress was relaxed by the annealing. Cylindrical tensile specimens with 3 mm in diameter and 25 mm in gauge length were machined. In situ neutron diffraction experiment during tensile test was performed with a TOF method by using a powder diffractometer, Sirius, at the High Energy Accelerator Research Organization (KEK). A tensile jig shown in Fig. 1(a) was prepared in such a way that the central position of a specimen did not move even when the specimen was elongated. The geometrical arrangement for the strain measurements in the axial and transverse directions of the specimen is illustrated in Fig. 1(b). The applied load was kept constant during the measurement. The sampling volume for the neutron diffraction within the parallel portion of the tensile specimen was about 70 mm3. Peak shifts of ferrite and cementite diffractions with tensile straining were converted to elastic strains (εhkl) by

$$
\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0} \quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (1)
$$

where $d_{hkl}$ and $d_{hkl}^0$ refer to (hkl) spacing, and reference spacing (stress-free spacing), respectively.
3. Results and Discussion

3.1. Characteristics of the Specimen

Microstructure observed by SEM is shown in Fig. 2, which consists of pearlite. The declinations of lamellar colonies are nearly uni-directional. In the previous work\(^4\), the annealing at 673 K was found to relax the residual phase stress evolved during plastic deformation but hardly to make cementite plates spherical. Overall diffraction profiles are shown in Fig. 3. As is seen, the intensity of cementite peaks is weak compared with that of ferrite. The diffractions from a few (hkl) cementite planes overlap each other (see an enlarged figure inserted in Fig. 3). According to the X-ray pole figure measured for the present specimen, the (110) fiber texture was evolved. The texture is confirmed from the difference in diffraction intensities in the axial and transverse directions; as is observed in Fig. 3, the intensity of (110) ferrite in the axial direction is extremely high because of the (110) fiber texture.

Figure 4 shows the stress–strain curves of the as-patented and the swaged-annealed specimens, in which applied stresses kept constant during the neutron diffraction measurements are shown.

3.2. Mechanical Properties

The 0.2% proof stress, tensile strength, and uniform elongation were, 808 MPa, 1206 MPa and 6.5% for the as-patented specimen, while 1423 MPa, 1698 MPa and 4.4% for the swaged-annealed one, respectively. The higher strength of the latter specimen is achieved by finer lamellar spacing and rearrangement of lamellar direction. To examine the strength of cementite, a specimen with a higher strength is preferable. Then, neutron diffraction measurements were preformed for the swaged-annealed specimen at the applied stresses indicated by #1 to #8 in Fig. 4.
3.2. Diffraction Profiles Obtained by *in Situ* Neutron Diffraction during Tensile Deformation

Figure 5 shows the change in ferrite (110) diffraction profile with the applied stress. It is found from #1 to #4 that the peak moves towards the wider spacing side in the axial direction while the narrowest in the transverse. After #5, the change becomes smaller. The change from #7 to #8 is corresponding to unloading, so that the spacing moves back towards the starting value. It is also observed that the residual strain remains after tensile plastic deformation. Such peak shifts with loading and unloading were examined for other diffraction peaks of (200), (211), (220), (310) and (321).

The peak shifts with the applied stress for cementite are presented in Fig. 6. The general trend in peak shifts is similar to Fig. 5; shifting to the wider spacing side in the axial direction with loading while the narrower in the transverse, and returning back upon unloading.

3.3. Elastic Strains in Cementite and Ferrite under the Applied Stress

Daymond and Priemeyer have claimed that the use of Rietveld refinement for overall diffraction profiles is of use for pearlite. However, we encountered difficulty to perform the Rietveld fitting, because the elastic strains of individual $(hkl)$ planes are influenced by elastic anisotropy in ferrite and cementite. The strong $(110)$ fiber texture of the present specimen also makes it difficult to employ the Rietveld refinement. Hence, single peak fitting was employed here to determine the elastic strains for individual diffraction planes. The details in the curve fitting procedure were already reported; a convolution curve consisting of three functions were used.

Figure 7 shows the lattice plane strains in the ferrite matrix as a function of the applied stress: (a) the axial and (b), (c) the transverse directions.

![Fig. 5. Change in diffraction profiles with applied stress obtained from the ferrite matrix: (a) the axial and (b) the transverse directions.](image1)

![Fig. 6. Change in diffraction profiles with applied stress obtained from cementite: (a) the axial and (b) the transverse directions.](image2)

![Fig. 7. Lattice plane strains in the ferrite matrix as a function of the applied stress: (a) the axial and (b), (c) the transverse directions.](image3)
ficient is dependent on crystal orientation. Strange is the result observed after the yielding; two types shown in Figs. 7(b) and 7(c) are found. If one imagined tensile deformation of a simple two-phase material, a grain of the softer constituent would elongate more plastically, leading to produce the compressive internal stress in the tensile direction while the tensile in the transverse after unloading. In such a case, the combination of Figs. 7(a) and 7(b) is acceptable. Thus, the results obtained from (110) and (220) shown in Fig. 7(c) is a little puzzling. This point will be discussed in the following Sec. 3.4.

The strains of cementite were determined from (212), (122) and (201) peaks, because these peaks do not overlap with each other or ferrite reflections. The strains determined in cementite were plotted in Fig. 8 as a function of the applied stress. The results after the yielding are just the reverse of the combination of Figs. 7(a) and 7(b), indicating good agreements with the predictions by a simple two-phase material model; the strong cementite plates bear higher stress after the yielding, resulting in high work-hardening of the pearlite steel. It must be the first experimental evidence that cementite deforms elastically by 1–2%. It should also be noted that the high residual strain remains in cementite after tensile deformation, which have been speculated from the results for the ferrite matrix.5,6)

3.4. Residual stress after tensile plastic deformation

Figure 9 indicates the residual elastic strain determined by comparison between the diffraction profiles obtained before and after tensile deformation. As is seen in Fig. 9(a), the compressive strains remain in the ferrite while tensile in cementite in the axial direction. In the transverse direction shown in Fig. 9(b), strains in cementite are commonly compressive showing the reverse features of the results in the axial direction. However, the ferrite strains in the transverse direction are complicated. The (110) and (220) strains show compressive while the other (200), (211) and (310) tensile.

Figures inserted in Fig. 9 show the results obtained by another work using as-patented pearlitic steel with little texture.6) The residual strains for all (hkl) ferrite examined are compressive in the axial direction while tensile in the transverse direction. Comparing with the present (110) fiber textured specimen, strains in the axial direction are commonly compressive although the trend in their magnitudes is different from each other. A difference is obvious in the transverse direction. For instance, the (110) strain in the little textured specimen is tensile but compressive in the present specimen. The results obtained in the drawn pearlite wire with a strong (110) texture are exactly similar to the present specimen.8) Therefore, the heterogeneous deformation among individual blocks resulting in residual stresses is strongly influenced by texture.

4. Conclusion

Stress partitioning between ferrite and cementite in a pearlite steel is confirmed by strain measurements for both constituents with *in situ* neutron diffraction during tensile deformation. The stress actually working in the ferrite matrix is almost limited to the yield strength that is dependent mainly on lamellar spacing. The elastic strain that cementite plates in a pearlite steel can bear, is nearly 0.015 under the applied stress of 1.6 GPa.

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