Note

Strengthening of Pearlitic Steel by Ferrite/Cementite Elastic Misfit Strain

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The strength of pearlitic steel was clearly reduced by annealing, even though cementite stably maintained a lamellar structure. In response, lattice strain of the ferrite phase in pearlite monotonically decreased with increasing annealing time. As a result, a good linear relationship was established between the strength and ferrite lattice strain independent of the interlamellar spacing and morphology of cementite. This suggests that the ferrite/cementite elastic misfit strain contributes to the high strength of pearlitic steel.

KEY WORDS: pearlite; strength; elastic misfit strain; lamellar eutectoid structure.

1. Introduction

Pearlitic steel exhibits high strength and good wear resistance at a low production cost, and is therefore used in many engineering applications, *e.g.*, cutting tools, high-strength wires, and railroad rail. In addition to having a eutectoid fine lamellar structure composed of ferrite and cementite, pearlite has hierarchical substructures referred to as colony and block. Colony is an area where cementite lamellae arrange unidirectionally, while block (or nodule) is a larger area where ferrite and cementite have identical crystallographic orientations, maintaining a specific orientation relationship.^{1–4)} In 1976, Marder *et al.* investigated which microstructural unit dominates the mechanical properties of pearlitic steel and consequently proved that its strength is solely dependent on the interlamellar spacing of cementite, and is independent of prior austenite grain size or block diameter. In particular, they emphasized that yield strength (0.2% proof stress) and fracture strength increase inversely proportional to the interlamellar spacing.⁵⁾ Since then, it has been apparently believed that the strength of pearlitic steel

should be explained on the basis of a dislocation bowing model between cementite lamellae: the so-called Orowan mechanism.^{6,7)} About 10 years ago, Tomota *et al.* directly investigated the individual elastic–plastic transition behaviors of ferrite and cementite in full pearlite by using in-situ neutron diffractometry. As a result, they demonstrated that the macroscopic yield strength of pearlitic steel corresponds to the onset of plastic flow in the ferrite matrix; furthermore, the stress necessary for this clearly increases with decreasing interlamellar spacing.⁸⁾ This enables us to understand that the yielding of pearlitic steel stems from that of the ferrite matrix, although the Orowan mechanism has not been proven to be appropriate for explaining ferrite strengthening in pearlitic steel.

On the other hand, we recently discovered using the electron backscatter diffraction (EBSD) method that crystal orientations of ferrite and cementite in pearlite are not completely identical but slightly rotated even in a block.³⁾ We then clarified that the crystal rotation cannot be attributed to high-density dislocations but the elastic ferrite/cementite misfit strain.⁴⁾ Zhou *et al.* had already reported that the ferrite/cementite misfit is so significant that the interfacial structures, *i.e.*, microscopic steps and misfit dislocations, are introduced at lamellar interfaces during pearlitic transformation.⁹⁾ Therefore, our finding suggests that the misfit strain is not completely accommodated by the interfacial structures and thus mostly remains as elastic strain, leading to a clear crystal rotation in pearlite. Considering that the residual elastic misfit strain is high enough to cause crystal rotation detectable by EBSD, it would have some influences on the strength of pearlitic steel. In this study, changes in the amount of elastic misfit strain with respect to pearlitic transformation temperature and annealing time after pearlitic transformation were evaluated by X-ray diffractometry (XRD). Moreover, the effect of elastic misfit strain on 0.2% proof stress of pearlitic steel is discussed in this paper.

2. Experimental Procedures

The material used in this study was commercial hyper-eutectoid steel with the chemical composition of Fe-0.9C-0.9Mn-0.4Si (mass%). This steel was solution-treated at 1 123 K for 1.8 ks and then directly subjected to isothermal holding at 823–973 K, followed by water-cooling to obtain as-transformed full pearlite with varied lamellar spacing. The isothermal holding time was set as short as possible to complete pearlitic transformation at each temperature (*e.g.*, 60 s at 823 K and 900 s at 973 K). Additionally, the specimens isothermally transformed at 823 and 973 K were directly annealed at the same temperatures for up to 360 ks after the completion of isothermal pearlitic transformation. Microstructures of the specimens were observed using scanning electron microscopy (SEM; S-4300SE, Hitachi High-Technology Corp.) and transmission electron microscopy (TEM; JEM-2010F, JEOL Ltd.). In particular, the deeply etched area where cementite lamellae existed vertically to the observation surface was selectively observed to measure the average interlamellar spacing (*S*). Additionally, cementite spheroidization that develops during additional annealing was evaluated by microstructural observation and the degree was determined according to its aspect ratio.¹⁰⁾ The elastic misfit strain in pearlite shows anisotropy within a pearlite colony but it could be regarded as being isotropic within a block because of the randomly orientated colonies.⁴⁾ Accordingly, XRD peaks are broadened while maintaining identical diffraction peak angles when the X-ray beam diameter is much larger than a pearlite block diameter (30–50 μm in this material)^{4,11)}. Therefore, the absolute amount of misfit strain was evaluated as lattice strain using XRD that had an X-ray beam diameter of 3.0 mm (RINT-2100, Rigaku

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Corp.). In order to quantify the lattice strain in ferrite matrix (ϵ_L), full width at half maximum (β) measured by XRD at each diffraction angle (2θ) was plotted according to the following Williamson–Hall equation^{†, 12)}

$$\beta \frac{\cos \theta}{\lambda} = \frac{0.9}{D} + 2\epsilon_L \frac{\sin \theta}{\lambda} \dots\dots\dots (1)$$

where D and λ denote the crystallite size and X-ray wavelength (Cu-K α , 0.15418 nm), respectively. In this analysis, (200) diffraction was deselected owing to high anisotropy and the artifact that came from the equipment itself was removed using β for fully annealed interstitial atom free (IF) steel. Tensile testing was carried out at an initial strain rate of $5.6 \times 10^{-4} \text{ s}^{-1}$ for plate test pieces with a gauge size of $6^l \times 3^w \times 1^t \text{ mm}^3$.

3. Results and Discussion

Figure 1 shows SEM images of specimens isothermally transformed at different temperatures (a–d) and subsequently annealed at 823 K (e–h) at different magnifications. Pearlritic transformation had completely finished in all as-transformed specimens, and thus, they show a full lamellar structure (a–d). Their interlamellar spacing decreased with decreasing temperature and the average value was measured at (a) 360, (b) 118, (c) 80, and (d) 54 nm, respectively. The finest lamellar structure (d) was stably maintained until additional annealing for 600 s (e), but then it gradually disintegrated and partially spheroidized with prolonged annealing (f, g). Eventually, cementite was completely spheroidized by annealing for 360 ks (h). The changes in the nominal stress–strain curve depending on the pearlitic transformation temperature (a) and annealing time at 823 K (b) are represented in **Fig. 2**. As is well known, both yield strength and tensile strength significantly increased with decreasing transformation temperature (a). According to previous knowledge, the increase in strength was interpreted in terms of the refinement of interlamellar spacing. Indeed, it was confirmed that the change in 0.2% proof stress depending on transformation temperature can be expressed as a function of the inverse of interlamellar spacing, as reported in previous studies.^{5–7)} In addition to transformation temperature, the strength of pearlitic steel continuously reduced with annealing time (b). Notably, the strength clearly reduced even by short-term annealing for 60, 300, and 600 s, which did not cause any change in interlamellar spacing (see Figs. 1(d) and 1(e)). This result suggests that the well-known relationship between strength and interlamellar spacing in pearlitic steel is a spurious correlation, and the strength of pearlitic steel should be dominated by another factor that would vary with interlamellar spacing in the as-transformed state but without it during additional annealing. Further, it is also interesting that the yielding behavior seemed to gradually transit from continuous- to discontinuous-type with decreasing strength owing to the lowering of transformation temperature (a) and the prolongation of annealing time (b). Therefore, the yield strength of pearlitic steel was evaluated as 0.2% proof stress, as described below.

Similar to **Fig. 2**, the change in the lattice strain of pearlitic ferrite depending on pearlitic transformation temperature and annealing time at 823 K is shown in **Fig. 3** as a function of (a) the inverse of interlamellar spacing and (b) annealing time. In this figure, data for industrial pure iron containing 60 ppm C are added to the left side in (a). From (a), it was found that the lower transformation temperature

Fig. 1. SEM images showing (a–d) as-transformed and (e–h) annealed pearlite. (a) 973 K, (b) 923 K, (c) 873 K, (d) 823 K, (e) 823 K–0.6 ks, (f) 823 K–3.6 ks, (g) 823 K–10.8 ks, (e) 823 K–360 ks.

Fig. 2. Change in nominal stress–strain curve depending on (a) the pearlitic transformation temperature and (b) the annealing time at 823 K.

is the higher lattice strain ferrite matrix has, and that the lattice strain is inversely proportional to interlamellar spacing. Given that the area of the ferrite/cementite lamellar interface in a unit volume is roughly estimated to be $2/S$,¹⁴⁾ this good linear relation suggests that the lattice strain is generated by the ferrite/cementite misfit. In addition to the interlamellar spacing, the lattice strain monotonically decreased with annealing after pearlitic transformation (b). The lattice strain decreased even by short-term annealing before the onset of cementite spheroidization. This implies that the misfit strain was accommodated because of short-range atomic diffusion near the ferrite/cementite interface because no increase in the density of misfit dislocations was observed by TEM.⁹⁾ Since the decreasing lattice strain seemed to correspond to strength reduction, as shown in **Fig. 2(b)**, the 0.2% proof stresses of all specimens are plotted as a function of the lattice strain of the ferrite matrix in **Fig. 4**. It is surprising that a good linear relationship was established between these parameters although the interlamellar spacing and morphology of cementite were completely different among specimens. In other words, strength can be easily predicted by the ferrite lattice strain in not only lamellar but

[†]: Modified Williamson–Hall and modified Warren–Averbach methods have been applied as new X-ray analytical methods to accurately evaluate not only dislocation density but also the type of dislocations present in a crystal, taking into account the dislocation contrast factors.¹³⁾ However, there is a small difference between the results calculated using the classical and modified methods for a body-centered cubic crystal; therefore, the classic method was simply adopted in this study.

Fig. 3. Effects of (a) interlamellar spacing and (b) annealing time on lattice strain of ferrite in pearlitic steel.

Fig. 4. Relation between 0.2% proof stress and lattice strain of ferrite in pearlitic steel with various interlamellar spacing and morphology of cementite.

also spheroidized pearlitic steels. This liner relationship is described by the following equation.

$$\sigma_{0.2} = 240 + 230 \times 10^3 \varepsilon_L \quad (\text{MPa}) \quad (2)$$

It is of practical interest that the proportionality constant is very close to the Young's modulus of ferritic iron.¹⁵⁾ The lattice strain, ε_L , in the Williamson–Hall Eq. (1) originally comes from the Bragg's Eq. (3). Further, full width at half maximum, β , is obtained by the differentiation of Eq. (3) with respect of d , as shown below.¹⁶⁾

$$2d \sin \theta = n\lambda \quad (n = 1, 2, 3 \dots) \quad (3)$$

$$2\Delta d \sin \theta + 2d \Delta \theta \cos \theta = 0 \quad (4)$$

$$\Delta \theta = \left| \frac{\Delta d}{d} \right| \tan \theta \quad (5)$$

$$\beta = \Delta(2\theta) = 2\Delta\theta = 2 \left| \frac{\Delta d}{d} \right| \tan \theta = 2\varepsilon_L \tan \theta \quad (6)$$

where d is the lattice spacing. A comparison of Eqs. (1) and (6) indicates that ε_L defines the normal strain in the crystal lattice, $\Delta d/d$, in principle. Therefore, the experimental fact that the proportionality constant in Eq. (2) is close to the Young's modulus of ferritic iron implies that the elastic misfit strain generated upon pearlitic transformation acts as a strengthening factor for the ferrite matrix. Considering that the X-ray diffraction peaks for pearlitic ferrite are broadened while maintaining identical diffraction peak angles, it can be concluded that the elastic misfit strain is composed of both tensile and compressive components. Moreover, they are distributed in a balanced manner within a certain region, probably corresponding to a colony or block diameter. According to dislocation theory, the tensile stress field assists a short-range motion of dislocation, but a long-range motion of dislocation is controlled by the compressive stress

filed, i.e., athermal stress.¹⁷⁾ Therefore, the macroscopic yield strength (0.2% proof stress) of pearlitic steel increases with increasing the absolute amount of ferrite lattice strain. It can be concluded that the strength of pearlitic steel is determined by the macroscopic yield strength of the ferrite matrix strengthened by ferrite/cementite elastic misfit strain, although, of course, this should not be simply applicable to heavily deformed pearlitic steel. It should also be noted that the first term in Eq. (2) (240 MPa) denotes the 0.2% proof stress of pearlite with infinitely coarse interlamellar spacing but much higher than that of pure iron (see Fig. 4). This is because the stress partitioning between ferrite and cementite occurs even at 0.2% strain. That is, the rigid cementite phase increases the flow stress after the onset of ferrite yielding, as proposed by Tomota *et al.*⁸⁾

4. Conclusions

In order to understand the strengthening mechanism of pearlitic steel more deeply, the effect of elastic misfit strain on the strength of pearlitic steel was investigated while changing the ferrite lattice strain by heat treatment. The following results were obtained:

(1) The strength of pearlitic steel was clearly reduced even by short-term annealing where cementite stably maintained a lamellar structure. This implies that the strength of pearlitic steel cannot be fully explained by the interlamellar spacing.

(2) The ferrite lattice strain in as-transformed pearlite increased with decreasing transformation temperature, similar to the interlamellar spacing. However, it also monotonically decreased by annealing after pearlitic transformation owing to short-range atomic diffusion.

(3) A good linear relationship was established between the ferrite lattice strain and 0.2% proof stress in pearlitic steel regardless of the interlamellar spacing and cementite morphology. This suggests that the ferrite/cementite elastic misfit strain strengthens the ferrite matrix, and this contributes to the high strength of pearlitic steel.

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