A multiscale approach for the deformation mechanism in pearlite microstructure

Experimental measurements of strain distribution using a novel
technique of precision markers.

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Abstract:

Plastic deformation of fully pearlitic steels was investigated using a multiscale approach: experimentally, the finite element method and molecular dynamics. This paper is the first in a series of three papers demonstrating the strain distribution in uniaxial tensile deformation with high-precision markers drawn by electron beam lithography. Strain was measured at loads of 1.98 kN, 2.21 kN and 2.28 kN in tensile deformation. Scanning electron microscopy (SEM) images and strain maps show the plastic deformation of cementite lamellae and homogenous plastic deformation under uniaxial tensile deformation in the area where the cementite lamellae are aligned in the tensile direction. The areas strain enhanced were block/colony boundaries and the areas where the cementite lamellae are inclined approximately 45° to the tensile direction.

Keywords: steels, local deformation, high-precision markers, wire drawing

1. Introduction

Pearlitic steels consist of cementite and ferrite that form lamellar structures. Single-phase cementite is brittle at room temperature, whereas it becomes ductile at high temperatures [1, 2]. The combination structure with such brittle and ductile phases in steels induces interesting mechanical properties. For example, the yield stress of pearlitic steels is an inverse function of interlamellar spacing and their ductility depends on the size of the blocks [3, 4]. It is also reported that pearlitic steels show good work-hardening ability. Using neutron diffraction, Tomota *et al.* [5] elucidated that such significant work hardening is due to stress partitioning between the ferrite and cementite, where internal stresses induced by the misfit of plastic strain between the two phases causes high and low stress states in the cementite and ferrite phases, respectively. However, the fundamental deformation process of pearlitic steels has not been fully elucidated yet, particularly the characteristics of inhomogeneous plastic deformation of ferrite and cementite lamellae. It is essential to elucidate how the plastic deformation of pearlitic steels depends on the direction of the

pearlite lamellae, colonies and blocks. In addition, it has not been clarified yet whether cementite lamellae can be plastically elongated in tensile deformation at room temperature, whereas it has been reported that they deform under cold rolling [6] or wire drawing [7]. One of the difficulties in investigating whether or not cementite lamellae can plastically deform during uniaxial tensile deformation is that cementite lamellae are not innately straight [8] and are fragmented [9]. Therefore, it is not straightforward to determine whether the observed cementite lamellae were deformed during tensile tests [8]. Thus, it is essential to observe and compare the same area in order to investigate plastic deformation of cementite lamellae.

In the present paper, the plastic strain distribution during tensile deformation was investigated in fully pearlitic steels using high-precision markers drawn on the specimen surface by electron beam lithography. In addition, the characteristics of the inhomogeneous deformation of pearlitic steels were also observed using backscattered electrons (BSE) with an angle-selected backscattered electron detector. The purpose of the present

paper is to show experimentally the inhomogeneous behaviour of plastic deformation in pearlitic steels. The strain in the pearlitic steels and cementite lamellae were quantitatively measured for the first time. The results correlate with the modelling and simulation studies on the mechanical behaviours of this kind of characteristic lamellar structure reported in separate papers. [10, 11].

2. Experimental procedures

Tensile specimens were cut from a 5.5 mm diameter patented SWR92A fully pearlitic steel, as shown in Fig. 1(a). The crosshead speed in the tensile test was set to be 0.2 mm/min (Shimazu: AG-IS). The average block size and interlamellar spacing of the samples were 20 μm and 150 nm, respectively. In order to elucidate the distribution of strain in the specimen, precision markers were first drawn on the parallel portion of the specimen surface using electron lithography [12, 13]. The shape of the precise markers was a square with one side 500 nm in length, as shown in Fig. 1(b). The width of the markers was approximately 70 nm. Strain was measured from the

displacement of the vertices of the square. Precise coordinates were put on each vertex of the square lattice in the scanning electron microscope (SEM) images before deformation, and then we measured the relative displacement of each coordinate in the x and y directions (parallel and perpendicular to the tensile direction) after deformation. The BSE images were obtained using an angle-selected backscattered electron detector in an SEM (Carl Zeiss: Ultra-55).

The strain in each square was evaluated as follows. First, each square was divided into four triangles by connecting two vertices on a diagonal line. We selected one triangle and assumed that any point inside the triangle, given as (x^i, y^i) , before deformation moved to another point, given by $(\underline{u}^i, \underline{v}^i)$, after deformation. We regard the relationship between these coordinate as linear, such that

$$u^i = a + bx^i + cy^i, \quad (1)$$

$$v^i = d + ex^i + fy^i, \quad (2)$$

where a , b , c , d , e and f are real numbers. Here, the coordinates of the vertices of one of the triangles before and after deformation were defined as

$(x_0^1, y_0^1), (x_0^2, y_0^2), (x_0^3, y_0^3)$ and $(x^1, y^1), (x^2, y^2), (x^3, y^3)$, respectively. The displacement components of each vertex were

$$u^1 = x^1 - x_0^1, \quad v^1 = y^1 - y_0^1, \quad (3)$$

$$u^2 = x^2 - x_0^2, \quad v^2 = y^2 - y_0^2, \quad (4)$$

$$u^3 = x^3 - x_0^3, \quad v^3 = y^3 - y_0^3. \quad (5)$$

Substituting Eqs. (3), (4) and (5) into Eqs. (1) and (2) gives the values for a , b , c , d , e and f . The Green–Lagrange strain is given by the differential forms of Eqs. (1) and (2):

$$\varepsilon_{xx} = \frac{\partial u^i}{\partial x} + \frac{1}{2} \left(\frac{\partial u^i}{\partial x} \frac{\partial u^i}{\partial x} + \frac{\partial v^i}{\partial x} \frac{\partial v^i}{\partial x} \right), \quad (6)$$

$$\varepsilon_{yy} = \frac{\partial v^i}{\partial y} + \frac{1}{2} \left(\frac{\partial u^i}{\partial y} \frac{\partial u^i}{\partial y} + \frac{\partial v^i}{\partial y} \frac{\partial v^i}{\partial y} \right), \quad (7)$$

$$\gamma_{xy} = \frac{1}{2} \left(\frac{\partial u^i}{\partial y} + \frac{\partial v^i}{\partial x} \right) + \frac{1}{2} \left(\frac{\partial u^i}{\partial x} \frac{\partial u^i}{\partial y} + \frac{\partial v^i}{\partial x} \frac{\partial v^i}{\partial y} \right), \quad (8)$$

$$\varepsilon_{eq} = \sqrt{\frac{4}{3} (\varepsilon_x^2 + \varepsilon_x \varepsilon_y + \varepsilon_y^2) + \frac{1}{3} \gamma_{xy}^2}, \quad (9)$$

where ε_i ($i = xx, yy$), γ_{xy} and ε_{eq} are the average of the total normal strain, the shear strain and the equivalent strain of each triangle after unloading,

respectively. The strain of each square is the average value of the strain from all four triangles in the square.

3. Results and Discussion

3.1 Strain distribution during uniaxial tensile deformation

Figure 2 shows a load–elongation curve in a tensile test, which includes the stiffness of the tensile test machine. The fully pearlitic steel shows significant work hardening just after yielding, which is due to the stress partitioning between the cementite and ferrite phases [14]. Another tensile test was performed using a different specimen from that used in Fig. 2. The applied load was terminated and unloaded at loads of 1.98 kN, 2.21 kN and 2.28 kN. The strain distribution was measured from the deviation of the vertices of the markers on the specimen surface.

Figures 3(a)–(c) show equivalent strain maps in which the tensile tests terminated and unloaded at loads of 1.98 kN, 2.21 kN and 2.28 kN, respectively. The tensile direction was horizontal in the figure. The measured area was approximately $100 \times 50 \mu\text{m}^2$. Histograms of the

equivalent strain from Figs. 3(a), (b) and (c) are shown in Figs. 3(d), (e) and (f), respectively. The statistical data are shown in Table 1. The lognormal fit curves are superposed upon each histogram of strain in Figs. 3(d), (e) and (f), exhibiting shapes of strain distribution that are close to those of the lognormal distribution. These results indicate that the values of the strain median are smaller than the strain average, showing that a small number of areas with large strains contributed to the increase in the average of the strain. It also indicates that the increase in the strain in fully pearlitic steels is a multiplicative process. The strain maps indicate inhomogeneous plastic deformation of the specimen. Figures 3(a)–(c) indicate that the strain does not uniformly increase as tensile deformation proceeds. Some areas in the figures show strains of more than 0.3, whereas 66% of the measured area shows strains less than the average strain of 0.066 in Fig. 3(b). The increases in the standard deviation from Figs. 3(d) to (f) indicate that the inhomogeneity of the local plastic deformation is exaggerated as the strain increases. That is, the area indicated by arrows in Fig. 3(a) where large strain had already accumulated continued being deformed to show a large

strain in Fig. 3(c).

Figures 3(g)–(i) show log–log plots of the strain versus count given in Figs. 3(d)–(f), respectively, indicating convex curves as expected from the shape of the histograms. The counts were subject to a power-law over magnitudes of strain of 0.04, 0.05 and 0.06 in Figs. 3(g)–(i), respectively, the values of which are close to the average strain of the observed areas, as shown in Table 1. The slopes of the regression lines decrease as the deformation proceeds, although the data scatter at large strains in Figs. 3(h) and (i) due to the lack of enough data points for the large strains. The power-law relations are also seen in the relationship between the magnitude of stress drop versus frequency in the Portevin–LeChatelier effect [15] and the magnitude of acoustic energy burst versus frequency in single crystalline ice [16]. It is worth pointing out that the strain versus count has the same log–log relation at higher strains as those seen in scale-free phenomena, the mechanism of which needs to be discussed further. The results may suggest that the strain distribution during work hardening should have some relationship to fractals.

In order to examine the origin of inhomogeneous strain distribution in pearlitic steels, four characteristic areas were investigated. BSE images and strain maps obtained from those three areas are shown in the next subsection.

3.2 Characteristic strain maps according to the direction of cementite lamellae

Figures 4(a)–(c) show BSE images and strain maps of the areas where cementite lamellae are seen to be aligned along an incline of approximately 45° in the tensile direction, along which the shear stress takes the maximum value if the lamellae are ideally arranged to be perpendicular to the observed plane. The contrast of the ferrite–cementite lamellae become sharper as deformation proceeds, indicating an increase of surface roughness with strain. Figures 4(d)–(f) show strain maps at loads of 1.98 kN, 2.21 kN and 2.28 kN, respectively. As seen in Figs. 4(c), (f) and (i), meshes with high strains greater than 0.2 appear along the ferrite–cementite lamellae. Frequency histograms of the strain from the areas are shown in Figs. 4(g)–(i),

with the curves of the strain distributions from the all areas shown in Figs. 3(d)–(f), demonstrating that the strain distributions in those areas moved to greater strain as deformation proceeded. The average strain shown in Fig. 4(f) reaches approximately 0.18, which is much larger than the average strain of 0.079 shown in Fig. 3(c) of all the areas measured. These results suggest that plastic deformation in the area where cementite lamellae lie along a 45° incline to the tensile direction should easily occur along the ferrite lamellae. The ferrite lamellae are the soft phase parallel to the plane subjected to nearly the maximum shear stress due to the tensile load. The flow stress needed for slip deformation in the ferrite lamellae should depend not only on their geometry (lamella direction, thickness and continuity) but also on their crystal orientation.

Figures 5(a)–(c) show BSE images at loads of 1.98 kN, 2.21 kN, and 2.28 kN, respectively, of the area where the cementite lamellae are closely aligned in the tensile direction. The average strain in the area is 0.03, 0.03 and 0.06 in Figs. 4(d), (e) and (f), respectively. The values of strain in this area are smaller than those measured across the entire area in Figs. 3(a)–(c). Figure

5(j) shows an enlarged image of Fig. 5(c) at an average strain of 0.06, indicating neither inhomogeneous deformation nor breaking of the cementite lamellae. The histogram in Fig. 5(i) shows that the strain measured is much larger than the elastic strain. Simple elastic deformation cannot bear such a large strain, so plastic deformation of the cementite must be contributing to the elongation.

Although the mechanism behind the plastic deformation of cementite has not been fully elucidated, it has been reported that one of the slip planes of cementite is (001)[†] [17, 18], in which atomic bonds in those planes are all between Fe–Fe, which are weaker than those between Fe–C. The lack of active slip systems reduces deformability in cementite as intermetallic compounds. Inoue and Ogura [2] and Inoue *et al.* [19] demonstrated the onset of slip deformation at room temperature around indents in a cementite single phase. They investigated slip traces around the indent and suggested that the (100), (010) and (110) planes should be slip planes even at room

[†] The crystal structure of cementite is orthorhombic. Lattice parameters are $a = 4.525 \text{ \AA}$, $b = 5.090 \text{ \AA}$, $c = 6.743 \text{ \AA}$. The c axis is taken to be the [001] direction here.

temperature. Tagashira *et al.* [6] demonstrated using TEM that dislocations in cementite are on the $(12\bar{1})$ planes in cold-rolled pearlitic steels. These investigations suggest that the activated slip systems of cementite in pearlitic steels are not so simple and are strongly affected by the triaxiality of stress fields expanding around the cementite.

Figures 6(a)–(c) show images of another area where cementite lamellae are lying predominantly parallel to the tensile direction. Strain maps and frequency histograms of the strain in the area are also shown. Sharp bands are seen as deformation proceeds along the direction inclined approximately 40° to the tensile direction. Precision markers were sheared by such bands, which penetrated the cementite lamellae. Ohashi *et al.* [11] calculated the strain distribution between ferrite and cementite lamellae using finite element method (FEM) calculations. Parallel layers representing ferrite and cementite phases lay along the tensile direction, forming multilayers. They showed that the characteristics of the deformation depend on the work-hardening ability of the ferrite. The plastic deformation of the ferrite layers restrains inhomogeneous deformation of the cementite layers when

the ferrite layers have high work-hardening ability, thus making the cementite layers primarily capable of homogenous plastic deformation. Shear bands are formed in the cementite layers as the deformation proceeds. When the ferrite layers do not have work-hardening ability, shear bands are formed in the cementite layers even at the very beginning of the deformation. The FEM calculations correspond to characteristics such as homogeneous plastic deformation and shear band formation, as shown in Figs. (5) and (6). The calculations suggest that the varying characteristics between homogeneous deformation and shear bands should be due to local differences of the work hardening in pearlitic steels.

Morikawa *et al.* [20] examined the characteristics of shear bands developed in cold-rolled austenitic stainless steel with a low stacking fault energy, and they showed that shear bands are formed in the twin-matrix fine lamellae structure. It was indicated that shear bands were formed when full work hardening occurred and that a kind of plastic instability takes place [21]. Tagashira *et al.* [6] also demonstrated that the microstructure of shear bands developed in pearlitic steels after cold-rolling with a reduction of more

than 70%, showing that the cementite formed nanocrystals after the cold rolling. In fully pearlitic steels, slip deformations are strongly restricted in the colonies with cementite lamellae lying along the tensile direction, so that shear bands tend to develop in fully pearlitic steels.

3.3 Misfit of materials at block/colony boundaries

As shown so far, the amount and distribution of strain depends on colonies. If the strain is not uniform, misfit of strain at the colony or block boundaries results. Figures 7(a)–(c) show strain maps including a block or colony boundary at loads of 1.98 kN, 2.21 kN and 2.28 kN, respectively. The direction of the cementite lamellae in the right half of each figure is quite different from that in the left half, indicating that either a block or a colony boundary lies at the centre of the figure. It could not be clarified whether the boundary in the figure was a colony or a block boundary using electron backscatter diffraction (EBSD) in this study due to the large strain, so the boundary here is denoted as a “block/colony boundary”. Strains greater than 0.35 appear along the boundary in the strain map in Figs.7(e) and (f). Strain

maps in Figs. 7(d)–(f) show that the strain distributions in the left half and the right half are also different while strain accumulates at the boundary in the centre of the maps. A crack is formed and the markers are locally fractured along the boundary. The appearance of such large strain in the strain maps and histograms is due to the crack formation around the block/colony boundary. It is expected that large misfits of plastic strain are induced around such boundaries by the differences of lamellae direction or crystal orientation. These misfits could also be a result of the accumulation of dislocations at smaller strains, but they develop sufficient stress concentration to form a crack along the boundary, as shown in Figs. 7(c) and (f).

4. Conclusion

The deformation process of fully pearlitic steel was investigated by measuring strain from the change in the position of precision markers on the specimen surface. The following results were obtained:

- (1) The distribution of strain is inhomogeneous, at least in the area of

approximately $100 \times 50 \mu\text{m}^2$. The strain distributions were close to a lognormal distribution. The log–log plots of strain and strain count show a power-law over average strain.

- (2) Plastic deformation of cementite lamellae was observed where cementite lamellae were aligned parallel to the tensile direction at room temperature. The local areas deformed inhomogeneously with shear bands or homogeneously without shear bands.
- (3) The strain accumulated areas were block/colony boundaries and the areas in which cementite lamellae were aligned along an incline approximately 45° to the tensile direction.

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Table 1 Statistical data obtained from strain maps.

Load terminated	No. data	Average	Median	St. Div.
(a) 1.98kN	19701	0.038	0.031	0.023
(b) 2.21kN	19371	0.066	0.054	0.051
(c) 2.28kN	19379	0.079	0.066	0.079

Figure 1 (a) Schematic drawing of the tensile specimen. The specimen thickness was 1mm. The specimen was cut from the centre of a 5.5mm diameter bar. (b) SEM image from the undeformed surface showing the 50μm square high-precision markers. The width of each marker was approximately 70μm.

Figure 2 Load–elongation curve of fully pearlitic steel using the specimen in Fig.1. Crosshead speed was set to be 0.2 mm/min. The curve includes the stiffness of the tensile machine used. The values of strain were measured at A, B and C at loads of 1.98kN, 2.21kN and 2.28kN, respectively.

Figure 3 Strain maps and histograms of equivalent strain after unloading where the tensile direction is horizontal to the figure. (d), (e) and (f) are histograms of the strain maps shown in (a), (b) and (c), respectively. Log-log plots of (d), (e) and (f) are shown in (g), (h) and (i), respectively. Inhomogeneous deformation is already seen in (a). Statistical data from each strain map are show in Table 1.

Figure 4(a), (b), and (c): Enlarged BSE images of the area where cementite lamellae lie along 45° incline to the tensile direction at loads of 1.98kN, 2.21kN and 2.28kN, respectively. (d), (e), and (f): Strain maps of the equivalent strain after unloading. Their histograms are shown in (g), (h) and (i), respectively.

Figure 5(a), (b), and (c): BSE images of the area where cementite lamellae are along the tensile direction at loads of 1.98kN, 2.21kN and 2.28kN, respectively. (d), (e), and (f): Strain maps of the equivalent strain after unloading. Their histograms are shown in (g), (h) and (i), respectively. (j): Enlarged image of (c).

Figure 6(a), (b) and (c):BSE images of the area where cementite lamellae are along the tensile direction at the load of 1.98kN, 2.21kN and 2.28kN, respectively. Their strain maps are shown in (d), (e) and (f), respectively. Shear bands developed even at the very early stage of plastic deformation.

Figure 7(a), (b) and (c) show BSE images around a block/colony boundary where large strain accumulated at the load of 1.98kN, 2.21kN and 2.28kN. Their strain maps are shown in (d), (e) and (f), respectively.