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Quantitative measurement of cementite dissociation in drawn pearlitic steel

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ABSTRACT

Fractional dissociation of cementite was quantified as a function of strain by measuring the volume change of cementite in the pearlitic steel. The amount of carbon dissolved into the ferrite was estimated from the decrease of cementite volume, to correlate with the hardness in different strain level. The hardness showed linear relationship with the carbon dissolved into the ferrite matrix, which is believed to contribute in strengthening the drawn wire. Defects introduced from the deformation were believed to lower the energy barrier of cementite break-ups and to enhance the dissolution of carbon into ferrite.

1. Introduction

High-strength steel wire can be fabricated by imposing high level of strain through drawing processes. For industrial applications such as bridge cables and tire cords, the initial pearlite structure become severely distorted during drawing process, resulting in fibrous colonies and grains along the drawing direction [1,2]. Lamellar spacing (λ) was considered as a representative parameter to express the strength of drawn pearlitic steel, and refinement of lamellar spacing was regarded as one of the foremost ways to strengthen the drawn wires. The correlation between lamellar spacing, λ, and mechanical strengths of drawn pearlitic steel was elucidated by Embury using a Hall–Petch-like equation [3]. The effect of pearlite spacing on strength was demonstrated by changing the λ with Cr addition [1]. The lamellar spacing, without any deformation, can be a good parameter to represent the strength [4,5], but it is hard to define the λ when severe deformations were imposed, which results in the wide variation in the spacing.

The mechanism responsible for the extraordinarily high strength of drawn wire is still uncertain and is a subject of debate. Grain refinement hardening [6,7] and work hardening by cold working [1,8] have been proposed as the main mechanisms that explain the increase in strength with diameter reduction. The most recent study proposed that cementite dissociation under strain could be a possible determinant mechanism for the strengthening [9–14]. Carbon atoms separated from the cementite were believed to form Cottrell atmosphere, thereby hindering the movement of the dislocation. Gavriljuk [9] proposed that dissociation of cementite was limited with strain as dislocations were saturated by carbon atoms.

It was reported that complete dissolution of cementite was achieved with uniformly distributed carbon atoms in a ferrite matrix [12], in which λ, being considered as one of the parameters that represents the mechanical properties of drawn wires, could not be defined. In the range of strain where the λ cannot be defined, the amount of dissociation can be an alternative parameter to represent the strength of the wire.

Another consideration for the strengthening is the fragmentation of the cementite during the drawing process. Even though cementite has a some degree of deformation at high temperature [15], cementite is known as a hard phase and is believed to be the sustaining medium of strength in pearlitic steel. The drawing process at room temperature, however, is known to impose severe deformation on the wire, which inevitably results in bending and breaking of cementite [15]. If cementite becomes fragmented, it may not be a strong medium any more to support the mechanical strength unless it maintains continuity by recrystallization during the drawing process. Even with the fragmentation of the cementite lamellae, pearlitic steel maintains high mechanical strength as high strain applied through the drawing. In this study, the amount of dissociated cementite fraction was quantitatively measured and correlated with hardness to find that the amount of carbon dissolved in the ferrite matrix contributed the strengthening of the drawn wire.

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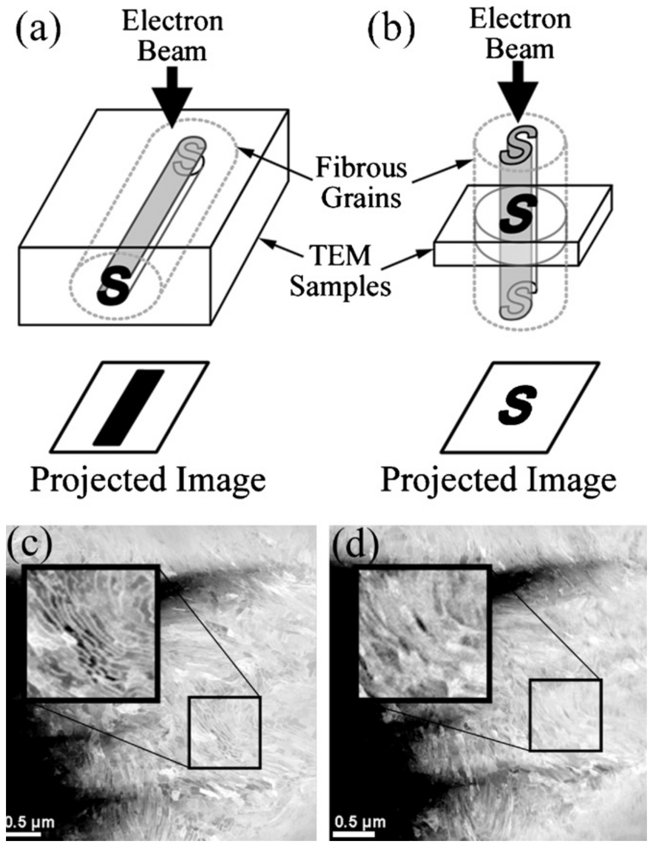

Fig. 1. Schematic view of projected image of lamellae in (a) longitudinal direction and (b) cross-sectional direction. (c) Annular dark field image of cross-sectional view with 0° tilt, and (d) 10° tilt from electron beam direction. When lamellae are aligned parallel to the electron beam, sharp interfaces of cementite/ferrite are visible.

2. Experimental procedure

Chemical compositions of the wires used in this study are 0.82C–0.2Si–0.5Mn and 0.92C–0.2Si–0.3Mn–0.2Cr, which were labeled as steel A and steel B, respectively. Sample was heated to austenization temperature followed by lead-patenting at 580 °C, which was used as starting material. Both wires were manually drawn through nine steps up to strains of 1.68 and 1.73 for steel A and steel B, respectively. The wires with higher strains were directly drawn from a continuous, wet-drawing machine to make final diameters of 0.35 mm. The maximum strains obtained were 3.33 and 3.38 for steel A and steel B, respectively. True strains were used in all deformation. At each stage of the strains, samples were taken to measure the hardness and the fraction of dissociated cementite. The Vickers hardness test was conducted using a 1-kg load holding for 5 s. Specimens for transmission electron microscopy (TEM) were prepared by conventional procedures, i.e., grinding and polishing, followed by Ar-ion milling at conditions of 3 kV and 5 mA with liquid nitrogen cooling. The prepared specimens were analyzed using FEI Tecnai F20 and JEOL JEM-3000F electron microscopes, operated at 200 kV and 300 kV, respectively. Longitudinal specimens were prepared to check the fibrous alignment of the cementite and ferrite grains along the drawing direction, and cross-sectional samples were used to observe and measure the fraction of the cementite from the section normal to the drawing direction.

Volume fractions of the cementite were measured from the projected image of cementite in TEM. When the cementite was aligned parallel to the drawing direction, the cementite area in the projected TEM image could be directly linked to the volume fraction regardless of the foil thickness along electron beam path in TEM. The differences in the projected images of longitudinal and cross-sectional views are illustrated in Fig. 1(a) and (b). The projected image from the longitudinal view, observed normal to the drawing direction, gave an erroneous estimation of the fraction because of the overlapping of curved cementite. In the longitudinal view, the projected images inevitably overlapped, irrespective of how the grains were placed, considering that the λ in the fibrous structure of drawn wires can be as small as ~20 nm and the lamellar plates are severely warped. In cross-sectional view, on the other hand, the projected images accurately represented the volume fraction of the cementite from the projected area regardless of the foil thickness by observing the sample parallel to the drawing direction. It is required to align the cementite lamellar planes parallel to the electron beam to convert the projection area to volume fraction with minimum errors. It is an advantage of TEM which can tilt the sample to align the orientation to a specific orientation, i.e., to make cementite lamellar parallel to the electron beam. The effects of alignment of the fiber grains relative to the electron beam in TEM are demonstrated in Fig. 1(c) and (d), where clear boundaries of cementite were visible in annular dark-field (ADF) images when the lamellar planes were accurately aligned along the electron beam. Sharpness of the boundaries was sensitive to the alignment. When the lamellar planes were off 10 degrees from the direction of the electron beam, the boundaries of ferrite/cementite interface plane were smeared and invisible. The sharpness of the lamellae boundaries was the criterion for the accurate alignment of the cementite along the drawing direction. Lamellae widths were traced by hand from the acquired projected images, eliminating ferrite grains of strong contrast from diffraction and cementite with unclear bound-

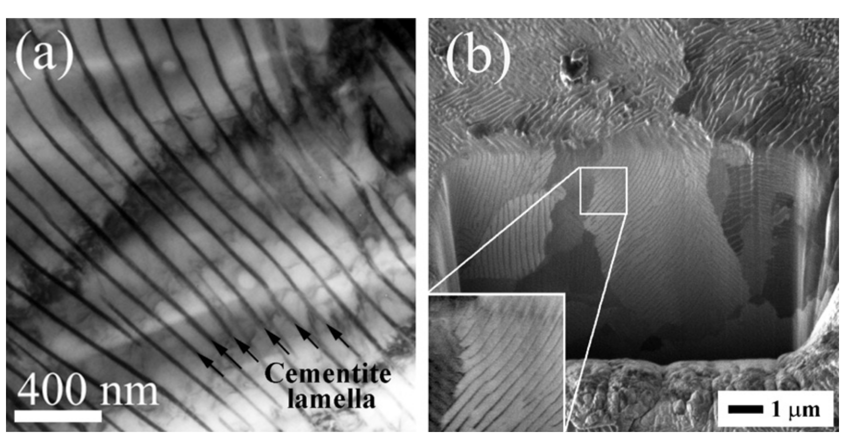

Fig. 2. (a) Comparison of the measurement of cementite fraction from (a) projected view in TEM and (b) ion channeling image from focused ion beam. Measurements were done from colonies and both fractions matched well. Inset in (b) shows the enlarged view of lamellar structure.