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Atomically resolved cementite dissolution governed by the strain state in pearlite steel wires

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ABSTRACT

Atom probe tomography was performed to study cementite dissolution as a function of the strain state of pearlite colonies with respect to the applied external strain in severely drawn pearlite steels. We prove that the migration of carbon from cementite into ferrite in the colonies increases under iso-stress conditions relative to that under iso-strain conditions at strains above 1.26. Our findings indicate that the strain state is an important microstructural component that controls the carbon redistribution in steel wires. We propose that the carbon–dislocation interaction is likely to be more prevalent under iso-stress conditions than in the iso-strain state.

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In pearlitic steel that is subjected to severe plastic deformation (SPD), a key issue is to understand the correlations between the microstructural properties and their extremely high strength. Among existing nanostructured high-strength materials, heavily cold-drawn pearlitic steels exhibit a tensile strength of 7 GPa [1–3]. The origin of the extraordinary strength of such steels has been commonly ascribed to the initial lamellar spacing, the prior austenite grain size, and the pearlitic colony size [4]. In addition to these morphological parameters, microstructural factors such as the dislocation density in ferrite and the number of elements in the solid solution have contributed to the strength of pearlite. More recently, Herbig [5] proposed that the dissolved C atoms tend to immediately decorate the evolving sub-grain and nano-grain structure in the ferrite. This effect can lead to a lowering of the associated interfacial energies, and hence a stabilization of these nanostructured and columnar crystals inside the ferrite at high drawing strains [5]. To prove the mechanisms responsible for cementite decomposition, numerous experimental studies using the experimental methods of transmission electron microscopy (TEM) and atom probe tomography (APT) have been reported [1–3,6–11]. For instance, Li [3,10] suggested that based on APT results, the dislocation density in ferrite layers for severely plastic-deformed pearlitic steels might be the cause underlying

cementite decomposition at non-equilibrium. Takahashi [8] also presented the effects of the crystallographic texture of pearlite on the properties of hypoeutectoid steels, based on the results from electron back-scatter diffraction (EBSD). In a recent study, Nematollahi [12] proposed that from the thermodynamic point of view the strain state of the cementite and ferrite phases in pearlite microstructures, i.e., the iso-stress and iso-strain conditions, was strongly dependent on their orientation with respect to the applied strain. In their calculations, two orientations of pearlite colonies were considered; one was an applied strain normal to the ferrite–cementite interface (in the iso-stress state), and the other was the case of an applied strain along the interface (in the iso-strain state). However, the prediction of a greater enhancement in the C concentration within the ferrite phase in the iso-stress state than in the iso-strain state still requires atomic-scale experimental proof.

Therefore, a specific challenge for APT experiment lies in identifying the cementite dissolution, especially the severe deformation-induced redistribution of C with respect to the applied drawing axis under iso-strain and iso-stress conditions. This study enables us to provide direct insights into the migration of C atoms into ferrite for pearlite colonies under the two strain state. Another important goal is to better understand the mechanism underlying the cementite decomposition in nanostructured materials.

The materials studied in this work are commercial hypereutectoid steels with mass-fraction compositions of Fe–0.8C–0.30Si–0.40Mn (wt.%). The wires had an initial diameter of 1.7 mm and were

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patented with an austenitization treatment at 1250 K for 60 s followed by a standard heat treatment for a pearlitic transformation. The wire samples were cold-drawn to true (logarithmic) strains ε of 0 (as-patented), 0.63, 1.26, 1.82, 2.38, 2.96, and 3.48. Measurements of the lamellar spacing and cementite thickness were performed by TEM (JEM 2010F) at an accelerating voltage of 200 kV. The TEM samples were prepared using a dual beam-FIB (FEI Helios NanoLab 650). The samples for APT experiments were prepared using FIB (FEI Helios NanoLab 650) so that the tips with the cementite plates/particles were perpendicular to the cold-drawn axis [13,14]. As reported previously [10,15], the plastic deformation at the surface of the samples can be more severe than in the centre of the wire because of the difference in friction between the wire and the drawing tools. Therefore, all APT tips were taken from the quarter through-thickness position of the wires. APT analyses were conducted using a laser-assisted wide-angle APT (LAWATAP™, Cameca Instruments), applying laser pulses with a 525-nm wavelength, 0.2-nJ pulse energy and 100-kHz pulse repetition rate. The measurements were conducted at a tip temperature of 60 K. In this study, the C peaks were detected at mass-to-charge ratios of 6, 6.5, 12, 13, 18, 18.5, 24, 24.5 and 36 Da, according to a standard assignment method [16,17]. To determine the concentration of C atoms with accuracy, specific attention was paid to the peak at 24 Da in the APT mass spectra of ferrite and cementite. In the mass spectrum of ferrite, the peak detected at 24 Da was assigned only to $^{12}\text{C}_2^+$, whereas it was assigned to the combination of $^{12}\text{C}_2^+$ and $^{12}\text{C}_4^{2+}$ for cementite, for which previously reported peak decomposition algorithm [15,18–20]. The C contents of cementite and ferrite in all of the wires were individually determined by clipping each phase, due to the presence of the peak at 24.5 Da in the mass spectra. The C distributions were systematically studied with respect to the cold-drawn pearlitic steels with true strains ranging from 0 to 3.48. In this study, we evaluated the C redistribution in pearlite colonies by measuring the C concentration of cementite particles due to the heterogeneous distribution of C in the ferrite regions as shown in Supplementary Information (Fig. S1).

For the as-patented state ($\varepsilon = 0$), the measured C concentration in the ferrite layers was ~ 0.05 at.%, which was far above the solubility of C interstitials in the ferrite region at the equilibrium condition. This means that the as-patented wire existed in the non-equilibrium regime, which is analogous to the results of previous reports in Refs. [10,18]. The amount of C in cementite for the wires of $\varepsilon = 0$ and 0.63 were estimated to possess nearly identical values of 23.8 ± 0.1 and 23.5 ± 0.3 at.%, respectively. This C value slightly deviates from the exact Fe_3C stoichiometry. This is probably due to the presence of vacancies within cementite, which has been discussed in the literature [12,19,20].

In the atom map created for the cold-drawn wires of $\varepsilon = 1.26$, the C-enriched and C-depleted regions are assigned to cementite and ferrite, respectively (Fig. 1). The APT result describes fairly straight cementite lamellae along the drawing axis (termed A-1 and A-3) as well as a displacement of the deformed cementite lamellar (termed A-2). The A-2 cementite clearly provides the interface steps between the ferrite and cementite phases (marked with a red arrow), meaning that it is the region that further fractured. Thus, the estimated C concentration of the A-1 and A-3 is substantially higher than that of the deformed A-2, although the ferrite/cementite lamellae possess an identical inclination to the APT analysis direction. Considering the dislocation-shuffle mechanism [21], the interface steps could be induced by the shearing of atomic planes along the inclined directions, if the dislocations which nucleated at the cementite/ferrite interfaces can penetrate from the ferrite into the cementite on more than one active slip system [10]. Consequently, we consider that the C-dislocation interaction mainly controls the cementite dissolution.

Fig. 2 shows the atom map and the corresponding concentration profiles of C in the ROI taken from the wires of $\varepsilon = 2.38$. We observe the morphological evolution of the lamellar structure with increasing the drawing strain, especially the fractured cementite lamellae with cylindrical morphology in pearlite colonies. Note that the wire drawing axis was along any directions within the x- and y-planes of the reconstructed map. Other side views of the colony-1 by rotating them with respect to one another reflect the majority of the cylindrical cementite lamellae, which were severely deformed from the cementite plates as shown in Supplementary Information of Fig. S2. We can determine both the arrangements and the longitudinal sections of cementite particles, based on the associated morphological evolution of severely drawn pearlite steels (Fig. S2). Following this concept, two colonies are clearly distinguished in the map according to the alignments or arrangements (marked with black dotted lines) of the cementite lamellae with respect to the analysis direction (termed z). First, the cylindrically fractured cementite lamellae on the left side of the map (termed colony-1) are largely aligned with the z direction (Fig. 2(a)). This implies that the orientation of colony-1 is normal to the drawing direction, based on the previous study [11]. In contrast, the others (colony-2) possess a significant alignment normal to the z-direction, indicating that they are aligned with the drawing direction. It is revealed that colony-2 shown in the map is under the iso-strain state. Notably, the higher amount of cylindrically broken cementite lamellae in colony-1 is visible compared to for colony-2. This would mean that a higher density of ferrite dislocations penetrating through the thin cementite lamella in colony-1 was more activated than those in colony-2. Additionally, the measured C content of the cementite particles found in colony-1 is remarkably lower than those of the particles in colony-2 (Fig. 2(b)). This deviation of the cementite concentration from the two colonies may be falsified, because of APT reconstruction artifacts like local magnification effects [22,23]. This effect depends on how the cementite particles in pearlite wires were inclined to the APT analysis direction, i.e., whether along the z-direction or normal to the z-direction [15]. On the other hand, the distinct difference in the C contents between two colonies can be regarded as real effects related to the C-dislocation interaction. It could have the following reasons: Firstly, the deviation of C contents can be explained by the difference in the amount of the broken cementite lamellae between colony-1 and colony-2. This observation indicates that the cementite decomposition of the two colonies is strongly dependent on their orientation with respect to the applied strains, even at identical strains. Similar observations were reported in the previous studies [11,24]. Thus, Raabe [3,21] proposed that the observed broken cementite lamella in pearlite [25] may indicate the penetration of ferrite dislocations through the thin cementite lamella, where the dislocation-shuffling mechanism may act as a kinetic mechanism for the cementite decomposition. Indeed, we found that the C concentration of the cementite lamellae in the wires of $\varepsilon = 2.38$ strongly depends on the C-dislocation interaction (see Fig. 1). Secondly, from the thermodynamic standpoint, both the solubility and the stabilization of C interstitials in the ferrite of pearlite microstructures were significantly dependent on the strain state [12]. Based on the experimental findings and those in the literature, it is, hence, conceivable that the prevailing C-dislocation interaction can lead to a lowering of C content inside the cementite lamellae in colony-1 compared to that in colony-2, i.e., APT reconstruction artifacts are not likely to be prominent in this study.

The C enhancement in ferrite was also found in APT results recorded for the wires of $\varepsilon = 2.96$. Fig. 3 displays the C maps, the corresponding concentration profile of C in the ROI, and a schematic illustration explaining the details of morphological evolution of cementite plates. Interestingly, the C-enriched boundary in the

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