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Cementite nano-crystallization in cold drawn pearlitic wires instigated by low temperature annealing

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ABSTRACT

The effects of low temperature annealing on the cementite structure and mechanical properties of cold drawn pearlitic steel wires were investigated. It was found that the thermal annealing at a temperature as low as 483 K could transform cementite in the as-drawn steel wires ($\varepsilon = 2.0$) from amorphous to nano-crystalline state. Against the conventional belief, the low temperature annealing treatment could simultaneously enhance the tensile strength and ductility of the cold drawn wires, for example, by 10% through the annealing treatment at 483 K. The underlying mechanisms responsible for enhanced mechanical properties were discussed, with a focus on the effects of solute carbon diffusion and cementite nanostructure.

High strength pearlitic steel wires have been used for suspension bridges, automobiles and springs [1]. They are typically produced through drawing hot-rolled steel rods. To increase the strength of cold drawn steel wires, the microstructure change during the drawing process has been extensively studied over the past decades.

Pearlite is a two-phased, lamellar structure composed of alternating layers of ferrite and cementite. Although hard cementite phase only represents approximately one-ninth of the total volume in pearlite, its presence is critical to the strength of cold drawn wires [2–3]. Moreover, post-mortem evidence has recently emerged revealing that cementite lamellae also exhibited remarkable ductile behavior during drawing [1,4–5]. Cementite platelets were found to change into numerous nanometer-sized grains as a result of dislocation initiation, propagation and entanglement, before turning into amorphous cementite at a drawing strain of 0.93 [6–7]. Under further drawing, cementite lamellae would start to decompose giving rise to carbon-rich regions between ferrite grains [8–11].

It is known that cold-drawn pearlitic wires are unstable and sensitive to heat treatment [12]. While annealing at relatively low temperatures, the re-distribution of carbon could occur in partially dissolved cementite, thereby affecting the mechanical properties of heavily cold drawn pearlitic wires [13–14]. For example, cementite lamellae could assume a spherical shape when heated up to 400 °C [15], and consequently the torsional properties of the heat-treated wires may decrease [16]. However, structural evolution of cementite and resultant mechanical property change in cold drawn pearlitic wires when subjected to low temperature annealing remains elusive. Such a deficit in understanding has rendered it difficult to develop ultrahigh strength pearlitic wires vital for safety-critical applications.

The present study endeavors to elucidate the structural change of cementite within cold drawn pearlitic wires at low temperature annealing. Furthermore, its impact upon the mechanical properties of the wires was discussed among other factors.

Material used here was hot-rolled rods (Fe-0.83C-0.19Mn-0.20Si-0.01S + P in wt%). After pickling and phosphating [17], the rods (13.5 mm in diameter) were cold drawn to 5.0 mm in diameter ($\varepsilon = 2.0$). The temperature of pearlitic wire in drawing is about 373 K. Heat treatment of cold drawn wires was carried out in oil bath for 60 min at 483 K and 553 K, respectively. Then the wire was cooled in air to room temperature. Thin-foil specimens were sectioned along the wire's longitudinal direction and then examined with a JEOL-2100F TEM operated at the accelerating voltage of 200 kV. Bright field (BF) image and dark field (DF) image was taken at same observation place. The DF Image was generated by sing diffraction spot of cementite. X-ray diffraction (XRD) analysis was also performed with Rigaku D-max2100. Tensile tests were determined at room temperature using CMT5105 universal materials tester machine, operating at a speed of 4 mm/min.

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Four samples were measured for each curve. The tensile tests were performed according to the Chinese National Standard GB/T228-2002.

Fig. 1a shows the microstructure typical of cold drawn pearlitic steel wires (ε = 2.0). The spacing between cementite and ferrite lamellae is about 40 nm and the thickness of cementite plates is about 4 nm. Dislocation clusters can also be seen within ferrite. Fig. 1b shows HRTEM micrograph of cold drawn pearlite nanostructure. The ferrite exhibits distinguishable lattice fringes, which are not apparent in cementite. In addition, discrete bright spots do not appear from the diffraction pattern of the cementite component using the Fast Fourier Transformation (FFT) method. IFFT image of the region area shows a disordered structure of cementite, while the cementite shows a weak short range order structure. It indicates that the single cementite crystal layer has been damaged after heavily cold drawn deformation. Cementite in heavily cold drawn pearlite should be contained nano-crystal and amorphous structure [6].

After annealing respectively at 483 K and 553 K for 60 min, the layered assembly remained for the pearlite. Fig. 1c shows the fine structure of the cementite and ferrite following the annealing at 483 K for 60 min. Lattice fringes can be clearly observed in the cementite, whose discontinuous feature indicates that the cementite may stay in a polycrystalline state. The size of cementite crystal is about 2 nm. FFT pattern of the cementite, as shown in Fig. 1d, was revealed both (210) and (220) crystal planes. The diffraction signal of (210) contains three bright spots positioned close to each other, indicating that lattice defects may exist in the cementite grains. Fig. 1e shows the IFFT image of the cementite with lattice defects, obtained from the marked block in (a). The spacing between cementite atomic layers is 0.206 nm, confirming the existence of the (210) planes. According to Fig. 1b, the lattice defects might be generated during the annealing process when the crystallization of the amorphous cementite took place. Similar results were also obtained after annealing at 553 K for 60 min (not shown here).

Fig. 2 shows the X-ray diffraction spectra of cementite obtained from cold drawn steel pearlitic wires subjected to different annealing temperature for 60 min. There are no apparent cementite peaks in the as-drawn wires. It indicates that single cementite crystal layer may have been transformed into nano-crystalline and/or amorphous cementite [6]. With the increase of annealing temperature, both (121) and (221) cementite peaks appeared with increasing intensity, meaning that the cementite in cold drawn pearlite has transformed from amorphous

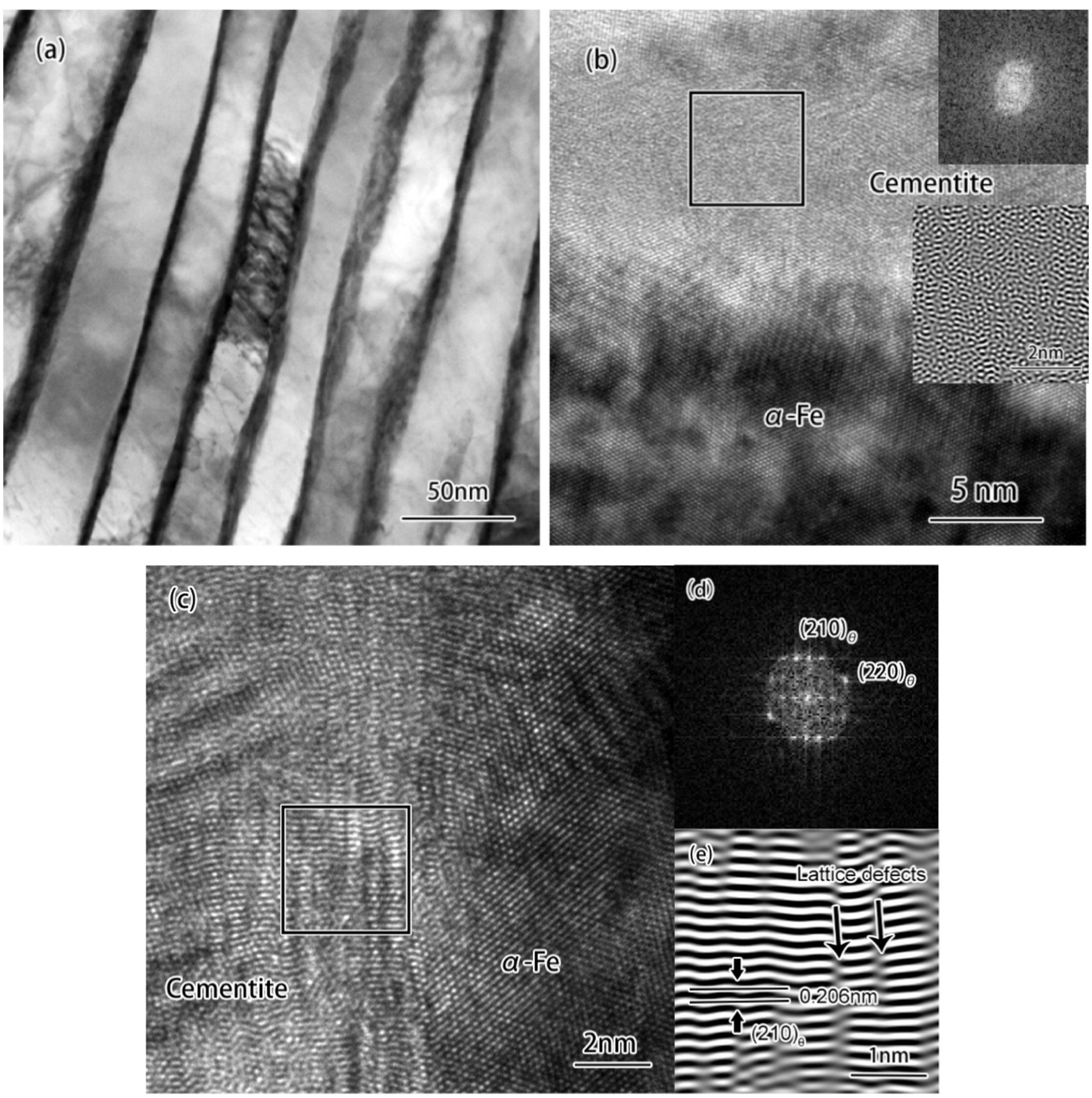

Fig. 1. TEM micrographs of cold drawn pearlitic wires (ε = 2.0). (a) Microstructure of cold drawn pearlitic wires; (b) cementite within pearlitic structure viewed under HRTEM; inset showing the selected area FFT and IFFT images obtained inside the boxed region; (c) HRTEM image of cold drawn pearlite after annealing at 483 K for 60 min; (d) FFT pattern of cementite in the marked block in (c); and (e) IFFT pattern of (210) cementite in the marked block as seen in (c).