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A modified pearlite microstructure to overcome the strength-plasticity trade-off of heavily drawn pearlitic wire

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Abstract

It has long been established that drawn pearlitic steel wires can achieve ultra-high strengths; however, this is usually achieved to the detriment of tensile and torsional ductility. Here, we employed a modified microstructure to overcome these trade-offs through a process involving simple multiple drawing and annealing steps. In this microstructure, the conventional pearlitic cementite plates are replaced by cementite nano-particles bridged by grain boundaries enriched with substitutional elements. After being subject to this modified processing route, 0.92%C steel wires were found to exhibit 2300 MPa tensile strength, 6.4% uniform elongation and 0.73 uniform torsion strain. The wires processed by the new route had higher ultimate strength than wires prepared by the traditional process to the same strain, but with much superior tensile and torsional ductility – indeed they match the ductilities of strain-free pearlitic steel rod.

Keywords: pearlitic steels, microstructural design, lamellar structure, drawing

The natural pearlitic microstructure of high carbon steel has an attractive combination of high strength and relatively low cost [1-5]. Through an isothermal austenitization process, a pearlite microstructure with alternating α-ferrite and cementite lamellae forms in near-eutectoid high carbon steels [6, 7]. The direction of the lamellae can be re-orientated to the drawing direction by cold drawing [8-10], leading to a significant refinement [11-13]. In this manner pearlitic steel wire obtains a nano-lamellar microstructure with high strength though a simple processing route.

Unfortunately, pearlitic steel wires are plagued by low tensile ductility and premature torsion delamination failure [14-19]. Since the wires are prepared by cold drawing, involving a reduction in area greater than 75%, the ferrite in the wire possesses a high density of dislocations, which generally exceeds 10^{15} m^{-2} [20-22]. Furthermore, dislocation slip is restricted by the cementite plates, and by the thickness of the ferrite lamellae, which are typically much thinner than 100 nm following drawing [23]. Thus, heavily cold drawn pearlitic steel wires typically display uniform elongations ≤ 2~3% [16, 19, 20, 24].

In view of the poor ductility and highly anisotropic microstructure, along with the complex straining conditions encountered in practical applications, torsion performance is another useful index for evaluating the plasticity and likely the toughness [25-27]. During torsion, a shear stress is applied along the circumferential direction [26], and it has been found that wires with good torsional ductility do not always display good tensile ductility [16, 28-30]. This lack of correspondence between torsional and tensile ductility is a difficulty when employing annealing to improve the mechanical performance of heavily drawn pearlitic wires. For instance, low-temperature annealing has been found to harden the wire, with the strength increase up to 200 MPa along with modest improvement in tensile elongation [19, 23, 31]. However, when this annealing was carried out on wires drawn to strains $\varepsilon >$ 1.3, the wires were been found to exhibit a marked brittleness under torsion, which many have referred to as “torsion delamination” [16-18, 26, 28, 30, 32-34]. For example, the uniform torsional strain of a 280°C-annealed wire (ε = 2.06) has been found to fall below 0.1 [16], which is just 10% of that recorded in the as-drawn state.

Spheroidization annealing at a higher temperature (e.g., 500 °C) can improve the ductile performance of the wire [28-30], due to the break-up of the cementite lamellae that lead to brittleness. However, spheroidization of the cementite typically leads to the loss of the nano-lamellar structure, along with an inevitable drop in tensile strength [28-31, 35-37]. This dilemma forces the manufacturers of pearlite steel wires to trade-off strength and ductility, in addition to balancing the tensile and torsional ductilities.

In this study, pearlitic steel wires were prepared using a new strategy which involves multiple drawing and annealing steps. ~~In this way~~Using this new processing route, we have engineered a novel microstructure based on nano-particles of cementite between ferrite lamellae. Compared with the conventional pearlitic steel wire drawn to the same strain, a combination of very high strength and excellent tensile and torsional ductility are obtained. In order to identify the origin of the improved mechanical properties, the microstructure is analyzed following the processing.

Our new manufacturing process can be described as a modification of the conventional two-step processing route. The conventional route involves sequential cold drawing operations through dies of decreasing diameter (step 1), followed by post-drawing annealing (PDA, step 2). The PDA could be set as a low temperature annealing (LTA) in order to strengthen the as-drawn wire without considering neither ductility nor torsion performance, or at a higher temperature to modify the cementite morphology and promote dislocation recovery. In our new process, the drawing stage (step 1) is modified by adding low-temperature annealing (LTA) treatments between some of the drawing operations. The detailed parameters of the new and conventional manufacturing processes are shown in the supplementary material (Fig. S1). During the cold drawing processes involved in our modified route, the temperature rise of the wire was controlled to prevent inadvertent LTA, which is known to give rise to brittle torsional performance [16, 18, 32, 38, 39]. The LTA between the final drawing passes is designed to increase the strain hardening of the pearlitic wire during drawing [16, 23, 40]. The additional drawing hardening can offset the decrease in strength during the PDA step. The PDA step for the new process is set ~~to be~~ at 415°C and was carried out for different durations of up to 15 minutes to investigate the effect of holding time.

Steel rods with a diameter of 14.0 mm and chemical composition of Fe-0.92C-0.25Cr-0.9Si-0.7Mn (wt.%) were first heat-treated to obtain a homogeneous pearlitic structure~~, then used in this study~~, see supplementary Fig. S2. Then, ~~U~~using the new, modified two-step process, wires with a diameter of 5.10 mm (a total strain ε = 2.02) were prepared successfully, which are referred to as 'new-processed' wires below. As ~~a~~ benchmarks for comparison, conventional pearlitic steel wires were prepared from the same rod by the simple cold drawing to ε = 2.02, followed ~~and~~ by one of two different types of subsequent annealing treatments: ~~,~~ (i) ~~1st.~~ LTA at 280°C for 60 minutes, or (ii)~~2nd. A~~

annealing at 415°C, w~~hich~~ith the same parameters a~~re~~s ~~the same as~~ the PDA step described above. These wires are referred to as 'conventionally-processed' wires below. Microstructures were examined using either an FEI T20-G2 or a JEOL-2100F transmission electron microscope (TEM), both operated at an accelerating voltage of 200 k~~K~~V. Atom probe tomography (APT) experiments were performed using a local electrode atom probe (LEAP 4000XHR, Cameca Instruments). Tensile tests and torsion tests were conducted at room temperature using a CMT5305 universal tester and CTT500 torsion tester, respectively. The specific experimental details and the test-piece geometries are included in the supplementary information, see Figs S3 and S4.

The microstructure of the new-processed wire following the completion of all the drawing and LTA stages~~eps~~ (i.e., before PDA) is presented in Fig. 1. The microstructure comprises a typical pearlite lamellar structure. High-density dislocations are visible in the ferrite. Fig.1c shows the measurements of the cementite plate spacing (ferrite lamellae thickness) and thickness. Their spacing ranges from 35 nm to 65 nm, whilst their thickness ranges from 3 nm to 7nm.

APT results on the as-drawn wire are displayed in Fig.1(d-e). The atom maps of the wire (Fig.1d) confirms that the lamellar structure consists of carbon-enriched plates (cementite) and low carbon lamellae (ferrite). The iso-concentration surfaces for 8 at.% carbon (red) and 1.5 at.% manganese (orange) are shown in Fig.1e. The two surfaces evidently overlap and almost cover the surface of cementite plate. The 1D atom concentration profiles in Fig.1f show that not only carbon (C) but also manganese (Mn) and chromium (Cr) are segregated to the cementite plates. The atomic concentrations of C, Mn and Cr in the plate are about 22 at.%, 3 at.% and 1.5 at.%, respectively.

The microstructure of the new-processed wires after being subjected to the PDA with different durations was investigated (presented in Fig.S6 and Fig.S7.) In brief, cementite spheroidization and dislocation recovery occurred after the annealing, meanwhile the nano-lamellar ferrite is thermally stable at 415°C. As an example, Fig. 2 shows the characterization of new-processed wires subjected to the PDA at 415˚C for 10 minutes. The cementite particles are no longer continuous along the nano-lamellar boundaries. The microstructure comprises ferrite lamellae and isolated cementite nano-particles (Fig.2a). The selected area diffraction patterns in Fig.2b indicate that neighbouring ferrite lamellae have a small misorientation of ~5.1°. The lamellae spacing ranged from 35 nm to 60 nm (Fig.2c), which is nearly same as the ferrite lamellae thickness in the as-drawn wire. This result suggests that the 415°C-annealing only trigger the cementite spheroidization, but no significant coarsening of ferrite nano-lamellae.

APT results ~~of~~ from the wire are displayed in Fig.2(d-g). Aligned carbon-enriched particles corresponding to the cementite are observed in Fig.2d. Since TEM observations in Fig.2(a-b) indicate that the cementite particles lie at the boundaries between ferrite lamellae, this plane should correspond to the boundary between two ferrite lamellae. Fig.2e shows that the iso-concentration surfaces for C overlap with the cementite particles. Meanwhile, the Mn iso-surfaces show a morphology consistent with the break-up of plates[REV1]. Fig.2f shows the atom concentration profiles of Mn and Cr with Mn-segregation reaching about 3.5 at.% and 2.0 at.%; while the atomic percentage of C is approximately 1 at.%. It should be noted that there are regions between cementite particles that sit inside the Mn iso-surfaces. During the annealing of pearlitic steel wire, the Mn has previously been observed to segregated to the cementite phase [31, 41], as was also seen here. It can be supposed that the Mn atoms near the cementite particles have diffused into the cementite during their initial formation, and have not had chance to diffuse away from their positions during the PDA treatment (unlike C). Thus, in this study, the partial boundary between ferrite lamellae is decorated by ~~manganese~~Mn, ~~chromium~~ Cr and (to a lesser extent) ~~carbon~~C. Fig. 2g shows the atom concentration profiles across a cementite particle, in which the atomic percentages of C, Mn and Cr are nearly same as those for the plates in the as-drawn wire.

Fig. 3a presents the tensile property evolution of the both new-processed wires and conventionally-processed wires subjected to 415°C-annealing. The conventionally-processed wire exhibited some annealing-strengthening after a duration of 4 minutes. The strengthening is proposed to be caused by dislocation decorated by carbon atoms and restoration of heavily deformed cementite [10, 23]. Since LTAs were used during the drawing step in the new process, the annealing-strengthening did not occur for the new-processed wire. In other cases, the new-processed wire shows a 250MPa-level advantage in tensile strength.

Fig.3b presents the typical strain-stress curves of different wires. The ultimate tensile strength (UTS) of the strain-free rod is 1420 MPa, with an average elongation (strain to UTS) of 6.7%. The UTS of new-processed wire and conventionally-processed wire in as-drawn state are 2420 and 2150 MPa, respectively. However, the elongation has fallen to 2.1%. The new-processed wire with PDA at 415˚C for 10 minutes displays a UTS of 2330 MPa on average. It is roughly 900 MPa higher than that of the strain-free rod, and about 250 MPa higher than that of the conventional wire with the same annealing treatment. Despite this, the elongation is 6.4%, i.e., 3 times the plastic strain of the as-drawn wires, which is nearly the same to that of the much softer strain-free rod. Without considering torsion performance[REV2], It was also possible to achieve a UTS level of 2300 MPa in the conventionally-processed wire~~, which has~~ by ~~been~~ anneal~~ed~~ing at 280°C for 60 minutes~~, also exhibited a UTS at the level of 2300 MPa~~. However, the elongation of the wire ~~i~~was less than 4% in this condition.

Fig.3(c-d) shows the tensile strained microstructures in the new-processed wire (415°C-annealed for 10 minutes). After straining to 2%, some parallel dislocation lines are observed in individual ferrite lamella (Fig.3c), with the ends of these dislocation lines located at cementite nano-particles and lamellar boundaries. At higher strains of ~6.4% to failure, some cross-boundary dislocation networks were found distributed in the ferrite lamellae (Fig.3d). By contrast, in the as-drawn wire strained to tensile fracture, the dislocation tangles are constrained within individual lamella (Fig.3e).

As discussed above, while the wire's microstructure is optimized for tensile performance, torsional performance should also be taken into consideration. Here the torsion performance of the new-processed wires subjected to different PDA durations are presented in Fig.4a. In despite of possessing poor ductility, the as-drawn state wire exhibited superior torsion plasticity, with a uniform shear strain (γ_u) of 1.1 and an ultimate torsion strength (τ_{max}) of 2020 MPa. When subjected to the PDA with duration of 4 and 6 minutes, the wires exhibited a torsion delamination behavior. As the annealing duration increasing, the wires exhibited uniform torsion performance with a γ_u of ~0.7.

Torsion stress-strain curves of wires after different processing routes are presented in Fig.4b. The τ_{max} and γ_u of the strain-free rod are 1350 MPa and 0.76, respectively. ~~The new~~New-processed wires with PDA for 4 and 6 minutes show a stress-drop before torsional fracture, which is so-called "torsion delamination". The wire with longer annealing duration, such as 10 and 15 minutes exhibits similar γ_u ~ 0.73 and a significantly improved τ_{max} ~1960 MPa. The wires in both conditions show no stress-drop before torsional fracture, consistent with homogeneous deformation during the torsion test. As ~~presented~~ stated above, the conventionally-processed wire annealed at 280°C for 60 minutes~~,~~ also exhibited a tensile strength at the level of 2300 MPa. However, it exhibited the characteristic torsional delamination during straining which significantly reduced uniform torsional deformability.[REV3]

Fig.4(c-d) shows the microstructure of the wires after torsion testing. In the new-processed wire annealed at 415°C for 10 minutes, high-density dislocation lines are evenly distributed in the ferrite lamellae. Bowing dislocations, which are by-passing cementite nanoparticles, can also be readily observed (Fig.4c).

The strength of pearlitic steel wire is principally dominated by ferrite lamellar spacing and the dislocation density in ferrite [8, 42]. The major difference between ~~the~~ pearlitic wires in the as-drawn and PDA states is the inter-laminar cementite plates are broken up, modifying the boundaries. Based on the present observations (Fig.2 and Fig.S8) and related research, the majority of the ferrite-ferrite boundaries should have low-angle misorientations [8, 11, 42]. The effect of such boundaries on the strengthening of nano-lamellar structures is considered to be similar to other boundaries following the Hall-Petch effect [43-45]. ~~Meanwhile~~Importantly, the boundaries in the new-processed wire in present study are decorated by increased concentrations of elements (Mn, Cr and C), which can be referred to as ‘boundary complexion’[46, 47]. Turlo et al. have reported that boundary complexion increased the flow stress required for dislocation propagation [47]. TEM observation in Fig.3 supports the idea that the modified boundary structure in the new-processed wire can more effectively constrain dislocation motion into a single lamella, increasing the strength. Fig. 5a shows dislocation density in the wires with different annealing condition. The new-processed wire and conventionally-processed wire possess a dislocation density of ~3.2×10^{15} and ~1.3×10^{15} m^{-2}, respectively, in the as-drawn state. Notably, after the PDA for 10 minutes, the dislocation density in the new-processed wire is still at the level of 10^{15} m^{-2}. Thus, the new-processed microstructure consisting of the modified boundary and ferrite nano-lamellae[REV4] possessing high-dislocation density ensures the high strength of the wire.

Dislocation recovery was confirmed to occur during the PDA for the new-processed wire [REV5](Fig. S7 and Fig. S8), and this is believed to increase the tensile ductility [22, 48]. However, torsion performance of annealed pearlitic steel wire has been demonstrated to be dominated by the cementite morphology [16, 30]. During torsional testing of annealed pearlitic steel wire where the cementite morphology is plate-like, i.e., the 280°C-annealed conventionally-processed wire in present study, the cementite plates are barriers which dislocation could not bypassed [16, 30]. As a result, partial cementite plates are fractured and accompanied by dislocation tangles (Fig. 4d), which has been observed to be associated with delamination crack in torsional pearlitic wire [16, 18]. In the new-processed wires after the PDA at 415°C, the cementite plate has been modified (Fig. 2). ~~Despite~~ Although the cementite nano-particles and decorating elements on the boundary are believed to provide some resistance to dislocation motion, the dislocation in ferrite can bypass the ferrite-ferrite boundary to avoid stress concentration or severe local deformation (Fig. 4), thereby delaying crack initiation. A high enough proportion of ferrite-ferrite boundary in the modified microstructure is critical for ensuring the torsion performance of the new-processed wire. As presented in Fig. 5b, when duration of the PDA at 415°C is longer than 10 minutes, the ferrite-ferrite boundaries~~y~~ account ~~higher~~ for more than 45% of all ferrite nano-lamellar boundary. In this case, the new-processed wire exhibited a uniform torsion behavior as presented in Fig.4a. Thus, the PDA at 415°C played a crucial role in improving the wires' plastic performance by two aspects: dislocation recovery and modified cementite morphology.

In summary, a new strategy combining simple drawing and annealing operations is proposed to produce pearlitic wire with a superior combination of ultra-high strength and ductility (both tensile and torsional). This process produces modified ferrite boundaries, which are decorated with cementite nano-particles and enriched with substitutional elements and carbon in place of the cementite plates between ferrite nano-lamellae. The prepared wires are 900 MPa stronger than the strain-free pearlitic rod material without sacrificing either tensile ductility or torsional toughness. The prepared wires possess a 2300 MPa tensile strength, 6.2% uniform elongation and 0.73 uniform torsion strain, which ~~shows~~ is significantly more tensile and torsional ductility than conventionally-processed wires annealed at low-temperature ~~annealed wires at similar~~in order to achieve similar levels of strength.

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