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Fracture behavior of nanostructured heavily cold drawn pearlitic steel wires before and after annealing

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ABSTRACT

In situ micro-cantilever fracture testing is used to demonstrate changes in fracture behavior of nanostructured, heavily cold drawn pearlitic steel wires as a function of drawing strain and annealing conditions. It is shown that these steels exhibit a sharp transition in fracture behavior between a drawing strain of 320% and 520% with a drop in fracture toughness from 7.5 to 4 $\mathrm{MPam}^{1/2}$. This is confirmed from the nature of fracture which is stable with some degree of plasticity at drawing strains below 320% and changes to catastrophic cleavage fracture at drawing strains of 420% and above. This transition and associated brittleness is attributed to structural (cementite decomposition and strain induced increase in tetragonality) and microstructural (increasing nanocrystallinity and dislocation density) evolution that these steels undergo at higher drawing strains. On heat treating the 420% strained sample, brittle cleavage fracture continues for low temperature (200 °C) annealing with no visible changes in microstructure, while crack growth is suppressed and large-scale plasticity is recovered for high temperature (500 °C) annealing with accompanying grain coarsening, and re-precipitation of spherodized cementite at grain boundaries.

1. Introduction

Cold drawn pearlitic steel, a nanolaminate composite structure consisting of alternate layers of ferrite and cementite, finds applications spanning from suspension bridge cables, rails and tire reinforcement cords to musical instruments and springs, due to its exceptional mechanical properties and the ability to be drawn into extremely thin wires [1]. The ductile α-Fe phase carries most of the strain while the harder Fe_3C contributes to strengthening and work-hardening by acting as a barrier for dislocation movement. The mechanical performance of such a structure is dominated by the high density of ferrite-cementite interfaces. With increasing drawing strain, the individual colonies of pearlite become increasingly oriented along the drawing direction due to co-deformation with the ferrite matrix, developing a fiber texture along $< 110 >$. On further straining, the hard and brittle cementite phase breaks down and undergoes nano-scale refinement ([2] and references therein). The strength of cementite is known to be 10 times higher than that of the ferrite matrix in undeformed, as-patented pearlite [3]. Because of the widely different properties of ferrite and cementite, a very inhomogeneous stress distribution develops inside the pearlitic steels during wire drawing.

Severe plastic deformation (SPD) processes like high pressure torsion and cold drawing can produce steels with ultra-high tensile strengths, close to that of the constituent cementite [2,4]. Recently Li et al. [5], showed that tensile strengths close to 7 GPa could be achieved in heavily cold drawn pearlitic steel wire at drawing strains above 600%. Not surprisingly, these high strengths were accompanied by significant loss of ductility and linear elastic fracture when pulled in tension. Li et al. carried out correlative atom probe tomography (APT) and transmission electron microscopy (TEM), to establish that these steels undergo fundamental transformation in their structure following decomposition of the cementite phase and supersaturation of the excess carbon in the ferrite matrix at drawing strains above 300% [5]. Djaziri et al. were able to show using synchrotron based X-day diffraction that this supersaturation of carbon in the matrix results in an increase in its tetragonality, leading to formation of strain induced martensite [6].

In real applications like in suspension bridge cables, these cold drawn wires are subsequently subjected to various heat treatments in the process of galvanizing/coating them with anti-corrosive agents. This will expose the highly non-equilibrium cold drawn structure to temperatures high enough to bring about coarsening of their nanostructure and a loss of tensile strength. Thus, thermal stability evaluation

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is of paramount importance. On heat treating wires of drawing strain of 650%, Li et al. observed that the tensile strength is retained for low temperature annealing (< 250 °C) while there is a dramatic drop on annealing these wires at high temperatures (> 250 °C) [7]. In view of the evolving microstructure, the deformation and micro-fracture mechanism of these wires is expected to undergo significant transitions. These have immediate technological implications, when they are required to be exploited for their high strengths.

Fracture behavior needs to be clearly understood as a function of drawing strain and annealing treatments to be able to prevent catastrophic fractures and provide sufficient damage tolerance at high drawing stains, while not compromising on strength. The micro-scale fracture behavior of cold drawn pearlitic steel wire in the longitudinal direction at high drawing strains were not explored earlier due to dimensional constraints of the limited wire diameter. The advent of *in situ* electron microscopy based micro-mechanical fracture testing [8] has changed this. Recently, Hohenwarter et al. [9], compared the damage tolerance of heavily cold drawn wires at two extreme drawing strains of 310% and 650%, both in the longitudinal and transverse directions. They identified and quantified the anisotropy in fracture behavior in the two directions, and observed catastrophic fracture in the longitudinal direction, and micro-cracking induced crack tip stress relaxation in the transverse direction. Their work provides valuable insights on the counter-intuitive contribution of the weak inter-columnar interface along the drawing direction enhancing the damage tolerance of these steel wires in the transverse direction. Our study expands on the work in the longitudinal direction, looking at intermediate drawing strains, and at different annealing conditions, where significant changes in microstructure have been reported [5,6,10,11]. Specifically, the objectives of the present work are to identify and quantify fracture behavior of cold drawn pearlitic steel wires in the drawing direction, in terms of their fracture toughness and fracture surface morphology a) at drawing strains of 320%, 420% and 520%, where a clear transition in microstructure has been observed from nanostructured lamellar pearlite composite, to nano-scaled sub-grained ferrite with supersaturated C [2,5,6], b) before and after low temperature (200 °C) and high temperature (500 °C) annealing for the transition drawing strain of 420% where a significant drop in tensile strength accompanied by grain coarsening has been recorded [10]. In addition to answering fundamental questions of structure-property correlations, quantification of fracture resistance in these wires will have a direct impact on critical applications where SPD steels are prone to fatigue, fracture and wear.

2. Experimental procedure

2.1. Material

Hypereutectoid steel wires of nominal composition Fe-0.98 wt%C (Fe–0.98C–0.31Mn–0.20Si–0.20Cr–0.01Cu–0.006P–0.007 S in wt%) supplied by Suzuki Metal Industry Co., Ltd. of drawing strains of 320%, 420% and 520% were used for the present study. Corresponding average lamellar thickness/grain sizes were 18, 15 and 10 nm respectively [2]. A second series of tests were carried out on annealed wires (200 °C and 500 °C for 30 min) of 420% drawing strain to evaluate the role of recovery and grain coarsening on the fracture behavior of these steels in comparison to the as-drawn state. Corresponding mean subgrain sizes were 19 and 130 nm respectively [10]. Table 1 shows the drawing strains, diameters and heat treatment conditions of the corresponding wires used.

In addition, magnetron sputtered Fe thin films of 2.2 ± 0.06 µm thickness on SiO_2 substrates were also used to evaluate the fracture behavior of pure unalloyed nanocrystalline α-Fe. The average in-plane grain size of these thin films was 200 nm ± 33 nm and these were columnar in nature with the grains oriented primarily along the < 110 >. These were used to compare the fracture toughness of pearlitic steels that contain C at the grain boundaries in either elemental or compound forms, to nanocrystalline Fe without any C in them. These comparisons are motivated by first principle calculations that indicate that C improves the interlamellar cohesion of ferrite, and its absence could enhance the brittle behavior [12].

2.2. Micro-scale fracture testing and characterization

The wires being in the range of tens to hundreds of microns in diameter, fracture toughness determination in the longitudinal drawing direction requires micro-mechanical testing. The validity of micro-scale fracture test geometries in determining the fracture toughness of brittle and semi-brittle systems has already been established [13–15]. Focused ion beam machined and notched (Zeiss AURIGA®) micro-cantilevers were tested in bending *in situ* in the scanning electron microscopy (SEM) (ASMEC UNAT-2 in JEOL JSM 2000) till fracture. Beam dimensions for all drawing strains were chosen to be in the same order of magnitude ~8 × 2 × 2 μm^3 with a crack length to width (a/W) ratio ~0.3 (Table 1). All beams were Ga^+ FIB machined at 4 nA and polished at 240 pA and eventually notched from the top at 10pA current at 30 kV. All micro-beams for a given drawing strain were machined on wires drawn from the same batch, but owing to the limited wire diameter, each wire piece could support 3–4 microbeams only (Fig. 1a-b). The loading was initially monotonic for all cases (Fig. 1c) but lead to different responses as a function of drawing strain or annealing conditions. For example, high drawing strains ($\geq$ 420%) showed a purely elastic-brittle behavior and lower drawing strains (320%) showed significant deviation from linear elasticity (Fig. 1d). In such cases, cyclic loading with a series of load-partial unload-load sequence was carried out (see supplementary information). The latter was to enable J-integral calculations by recording the changing compliance during the unloading sequences of the load (P)-deflection (d) curve. Linear elastic fracture mechanics (LEFM) was used to determine K_C for higher drawing strains ($\geq$ 420%) (Eqs. (1) and (2)) [14], while it was used to determine only the lower limit of fracture toughness for lower drawing strain (320%). Fracture energy G and plastic zone size r_p was evaluated according to Eqs. (3) and (4) respectively. Results were averaged over 5 or more micro-cantilevers for each case except for the annealed condition. Post-mortem high resolution imaging of the fracture surface and crack morphology was carried out in the Zeiss AURIGA® SEM.

$$K_c = \frac{P_{crit}L}{BW^{3/2}}f\left(\frac{a}{W}\right) \tag{1}$$

$$f\left(\frac{a}{W}\right) = 1.46 + 24.36\left(\frac{a}{W}\right) - 47.21\left(\frac{a}{W}\right)^2 + 75.18\left(\frac{a}{W}\right)^3 \tag{2}$$

$$G = J = \frac{K_C{}^2}{E}(1-\nu^2) \tag{3}$$

$$r_p = \frac{1}{2\pi}\left(\frac{K_C}{\sigma_y}\right)^2 \tag{4}$$

where K_c is the stress intensity factor, a is the crack length, σ is the applied stress, $P_{\rm crit}$ is the fracture load, and L, B, and W correspond to the cantilever length, breadth and width respectively, G is the fracture energy, J is the J-integral, E is the elastic modulus, ν is the Poisson's ratio, σ_y is the tensile yield strength. J-integral measurements were carried out by repeated load-unload cycles in the case of the 320% as-drawn samples, which showed some plasticity during the cantilever fracture experiments. The procedure is detailed below.

The elasto-plastic fracture toughness in terms of J_C values for the 320% drawing strain sample, was determined by the procedure as established by Wurster et al. [16] on two micro-cantilever specimens. Quantification of fracture toughness in the classical sense in more ductile systems at small length scales remains a challenge [8]. This is a problem for pearlitic wires of lower drawing strains. ASTM like standards of plane strain and small scale yielding are impossible to be