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Low temperature annealing of cold-drawn pearlitic steel wire

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Abstract. Cold-drawn pearlitic steel wires are nanostructured and the flow stress at room temperature can reach values above 6 GPa. A typical characteristic of the nanostructured metals, is the low ductility and thermal stability. In order to optimize both the processing and application of the wires, the thermal behaviour is of interest. This has been studied by annealing the wires for 1h at temperatures from ambient temperature to 300 ℃ (573 K). It is expected that a raising temperature may lead to structural changes and a reduction in strength. The change in strength is however not expected to be large. For this reason we have applied a very precise technique to measure the tensile properties of the wires from a strain of 10^{-4} to the maximum strain of about 1-2%. The structural changes have also been followed to estimate and relate strength changes to changes in structural parameters and morphology.

1. Introduction

High carbon cold drawn pearlitic steel wires have the highest strength of mass-produced steel products. Nowadays the wires with strength ranging from 2 to 4 GPa have been widely used in a variety of applications including cables for suspension bridges, steel cords for automobile tyres and springs, for their high elastic modulus, reliable and reproducible mechanical properties and low cost. Much effort has been put into improving the tensile strength of such wires to a maximum experimental value of 6~7 GPa (E/33) [1, 2], much higher than the maximum flow stresses of polycrystalline bcc metals which range from E/100 to E/50 [3]. The mechanical properties including strength and ductility can be adjusted by low temperature annealing which is also used to reduce a hazardous macro tensile residual stress introduced during the drawing process. The wires have limited ductility (normally a total elongation around few percent), and the uniform elongation is small at the order of 0.3%~0.5%, which makes it difficult to determine accurately the stress-strain dependency of importance for the various applications of the wires.

The influence of temperature on the mechanical property is of interest for both scientific and technological communities and many investigations have covered the effect of annealing on the mechanical properties of drawn wires [2, 4-6]. However, these investigations focus mainly on temperatures above 200 ℃ and limited work has been done on the quantitative stress and strain relationship due to the difficulty of precise strain measurement as the wire diameters are small of the order of 0.1 mm (wire diameter range: 0.175 - 0.5 mm), also the high sensitivity of total elongation measurements to the extensometer knife/tensile axis alignment. We have therefore developed an in-house testing method for precise strain measurements, but the present study will focus on the changes in the mechanical response of wires annealed up to 300 ℃, and the mechanisms behind.

2. Experimental methods

A high-strength near-eutectoid steel with a carbon content of 0.8 wt.% supplied by NV Bekaert SA (Zwevegem, Belgium) Technology Center Laboratory was used in this study. The specimens were taken at the intermediate step of the overall wire drawing process from the as-patented wire (1.26 mm) to the final drawn wire (0.20 mm). The wire diameter is 0.332 mm with the drawing strain of 2.67. The wires were annealed at 100, 150, 200, 260 and 300 ℃ (373 - 573 K) for 1 hour and tested using in-house developed capstan grips. The tensile test method for wires was according to ISO 6892-1:2009. An Instron electro-mechanic test machine was used with a 1000 N load cell and a Fiedler laser scanner extensometer, as shown in figure 1. Capstan type grips were used for holding the wire (figure 1). The capstan grips were designed in house for static and fatigue testing of high strength steel wires. The wires were wound on the capstan grips and clamped at about 250 degrees from the wires entering point. Thus a part of the tensile load is transferred by friction over a long distance before the stress concentration at the clamp. The capstan diameter was ø90 mm, and the wire runs in a groove on the surface of the grip. The initial center-to-center grip distance was 300 mm.

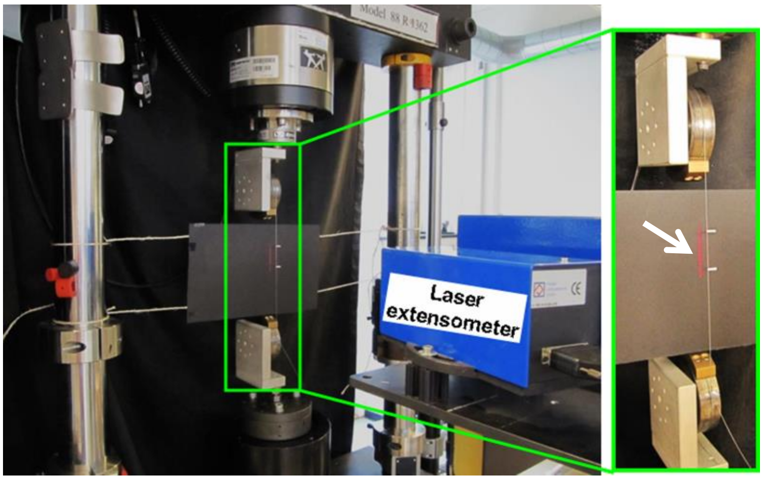

Figure 1. Tensile test setup showing loading capstans, strain labels and laser extensometer for the strain measurement of wire indicated by the white arrow

The strain was measured with a Fiedler P50 parallel scanner laser extensometer. A laser beam driven by a rotating prism, scans continuously along the axis of the sample length. Diffuse scattering of the laser light occurred at the strain labels. The scattered laser light was observed by a receiver. As the laser crosses the strain labels, the intensity of scattered light changes. The time of these changes was measured by a specially developed multi-stop counter. Because the speed of the rotating prism is known, the measured time can be used to calculate the position of the strain labels in the observation area, see figure 2a. Figures 2b and 2c show how the strain is defined. The laser extensometer scanned the distance between two adhesive strain labels attached to the wire (see figure 2b), and the initial gauge length L_0 is 20 mm ±1 mm. Since the extensometer measures the relative change in time between passing the two strain labels, the accuracy of initial gauge length is not critical for measuring relative elongation.

The tensile test setup allowed the proof stress at different offsets (0.05, 0.1 and 0.2%) to be determined accurately and thereby the strain hardening behavior. Other properties of interest were the ultimate tensile strength, the uniform/total elongation and the elastic modulus, see figure 3.

For the observation in a JEOL3000 field emission gun transmission electron microscope at 300 kV, the wires were ground, electro-polished with a double-jet sample-making method and then ion-cleaned in a low angle ion milling and polishing system (Fischione instruments) to remove the oxidation layer introduced during electro-polishing.

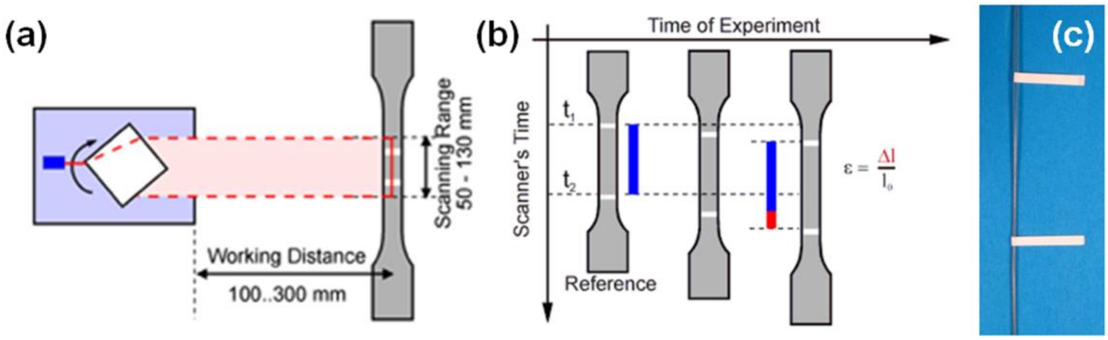

Figure 2. Principle of laser scanning extensometer (a), strain measurement by laser scanning and adhesive labels (b) attached to the wire (c).

3. Results

Nominal stress-strain curves for annealing at temperatures in the range from 20 ℃ to 300 ℃ (293 K - 573 K) are shown in figure 4. These curves can be divided into three groups based on the annealing temperature: (i) RT - 100 ℃ where the temperature effect is small; (ii) 100 – 200 ℃ showing a significant increase in flow stresses but only a small change in UTS and total elongation; (iii) 200 – 300 ℃ showing a decrease in strength with increasing temperature and no change in elongation to 260 ℃ and a significant increase to a total elongation of 5.5% reached at 300 ℃. The observed changes in mechanical properties with increasing annealing temperature are summarized in figure 5. The microstructural evolution with increasing annealing temperature has been followed by transmission electron microscopy focusing on the interlamellar spacing (ILS) between the cementite lamellae and the density and arrangement of dislocations. These parameters are chosen as they determine the contribution to the flow stress from boundary and dislocation strengthening, respectively. In the original wire, the interlamellar spacing: 28 nm, which is the sum of thickness of ferrite (23 nm) and thickness of cementite lamellae (5 nm) [7, 8]. The dislocation structures in the ferrite lamellae are single dislocations and dislocation cell boundaries, with a dislocation density ~ 10^{16} m^{-2} at the drawing strain of 2.67. The dislocation structure is shown in figure 6. The dislocations are spread in the ferrite lamellae, with the two ends of the line located at the ferrite/cementite boundaries [8], i.e., threading dislocations. During the low-temperature annealing process below 200 ℃, the morphology and structural parameters including ferrite thickness and cementite thickness are almost the same as those in the original wire. Further increase of the annealing temperature gradually changes the microstructure and the differences can be observed in the annealed wire at 300 ℃: the slight re-precipitation/spheroidization of cementite, a little change in the interlamellar spacing and a relatively small decrease in dislocation density.

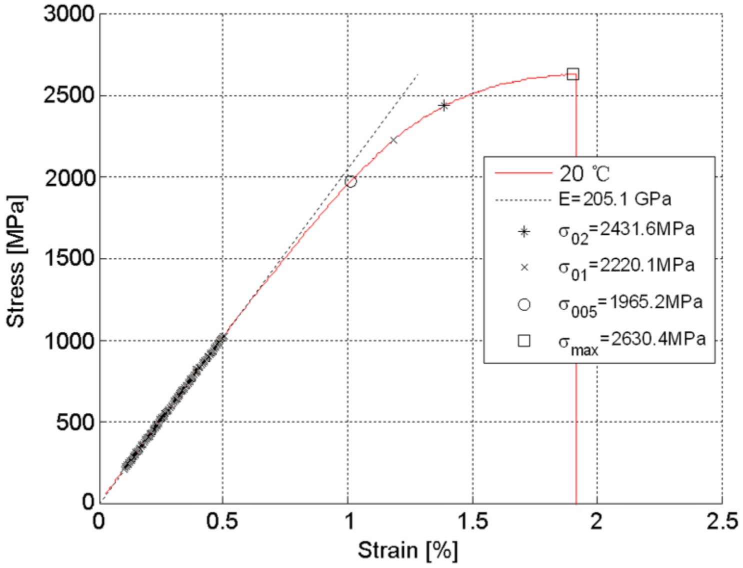

Figure 3. Demonstration of the determination of flow stress at 0.05% (σ_{005}), 0.1% (σ_{01}), 0.2% (σ_{02}) and UTS (σ_{max}).

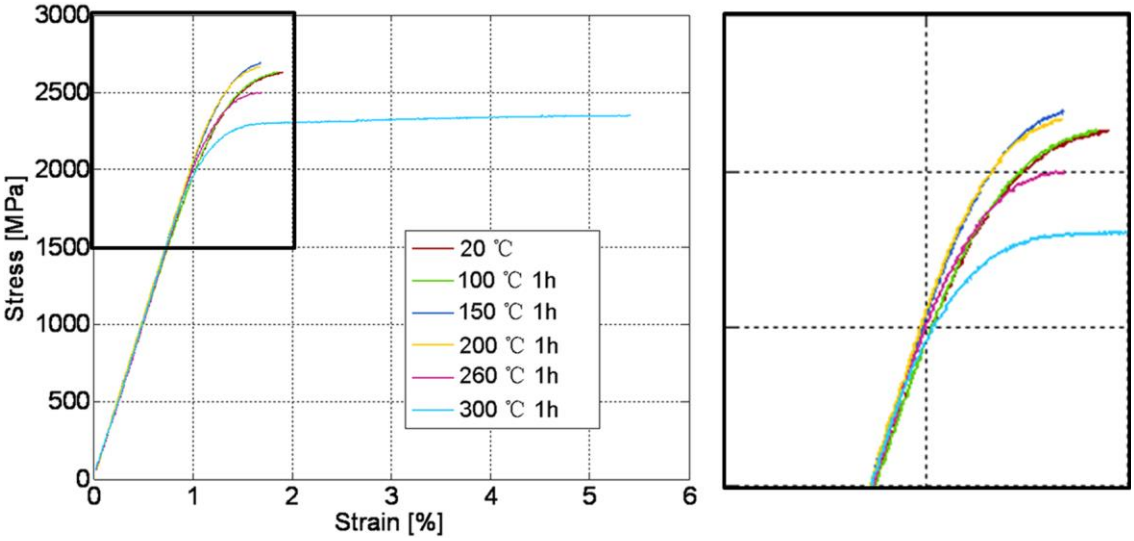

Figure 4. Tensile stress-strain curves of the wires. The area in the black frame is magnified to the right.

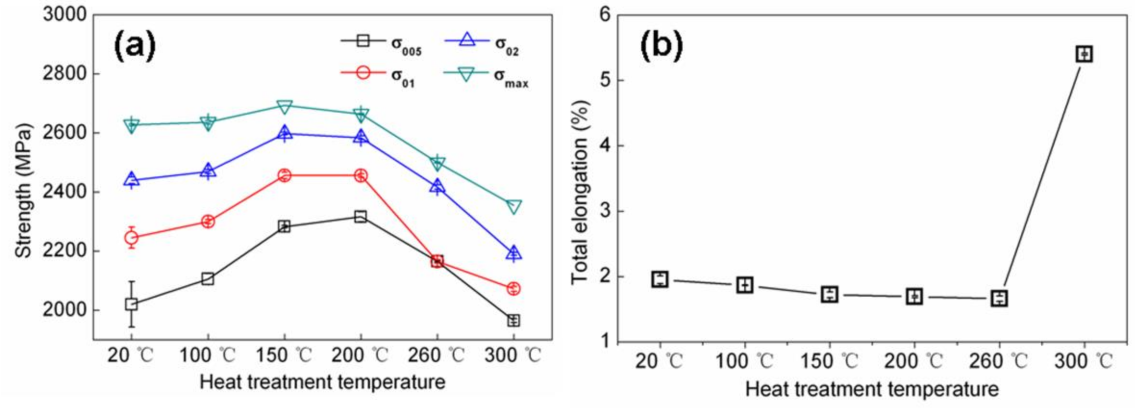

Figure 5. 0.05, 0.1 and 0.2% and maximum stress (a) and total elongation of the wires (b) versus the annealing temperature. The annealing time is 1 hour.

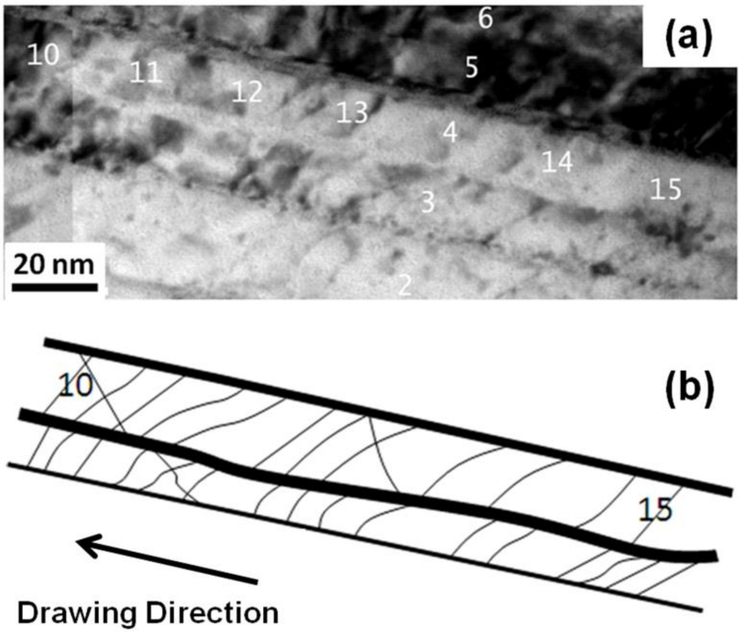

Figure 6. TEM micrograph of the wire of a drawing strain of 2.67 (a) and a sketch of the dislocation structure in the ferrite lamellae (b) showing threading dislocations [8].

4. Discussion

The mechanical data show a significant effect of annealing temperature: a strength increase in the temperature range lower than 200 ℃ and a strength decrease in the temperature range higher than 200 ℃. These changes will be analyzed based on the three strengthening mechanisms we proposed before: boundary strengthening related to the distance between cementite lamellae, dislocation strengthening related to the dislocation density in the ferrite lamellae and solid solution hardening related to the carbon concentration in the ferrite lamellae based on the change of thickness of cementite lamellae [8].

During the low temperature annealing process, C and N atoms dissolved in ferrite will migrate to the dislocations. To analyze their influences on the strength, it is necessary to examine the interaction energies between C and N atoms and the dislocations in steel. From the values reported, 0.78 eV/atom for C [9] and 0.47 eV/atom for N [10], it is reasonable to assume that the effect of dislocation pinning of C atoms is more intense than that of N atoms and in the present study we only consider the dislocation pinning of C atoms.

For the strength increase in the temperature range lower than 200 ℃, it is most notable for the proof stress at small strains, and relatively small in UTS. Since the low-temperature annealing below 200 ℃ for 1h does not bring enough detectable changes in the structural parameters in the TEM to cause the strength increase based on the above three strengthening mechanisms; this strength increase therefore points to the pinning of mobile dislocations/dislocation sources by C atoms/precipitates, as the other possibilities such as removal of mobile dislocations seems less plausible. This is because for steels the starting temperature for the dislocation movement is estimated to be 250 ℃ and the relief of macro residual stress introduced by quenching in steels starts from around 250 ℃ [11]. The pinning of the mobile dislocations/dislocation sources brings more carbon into the ferrite and this has been analyzed by the three dimensional atom probe tomography (3D-APT) [12]. When the dislocations and dislocation boundaries are taken into account, the maximum solubility of carbon in the ferrite also depends on the dislocations/dislocation boundaries in the ferrite lamellae, as it is well known that pipe diffusion of carbon occurs along the edge dislocation line and that dislocation boundaries are easy paths compared with interstitial diffusion in the pure ferrite matrix. Also the rate of dislocation boundary diffusion increases with the misorientation angle. It is reported that in the iron containing 0.011 wt. % carbon in solution, carbide precipitation occurs around 160℃, with the continuous growth at higher temperatures [13]. This is possible in the present case but difficult to detect by the technique we are using because the precipitation location is preferably the original cementite which decompose during the drawing processes and also taken into account the small spacing of ferrite lamellae. This means the thickness of the cementite lamellae may not change but the carbon content in the deformed cementite lamellae may increase. This process may be described as cementite decomposition/precipitation. In the future, the chemical composition change of the cementite lamellae with the annealing temperature will be analyzed by the advanced analytical TEM.

For the strength decrease of the annealed wires at temperatures 200℃ and 300℃, taken into account the three strengthening mechanisms, the key factors appears to be the reduction of carbon content in the ferrite by cementite precipitation/growth/spheroidization and the decrease of dislocation density in the ferrite lamellae, as little change in the distance between cementite lamellae has been observed. The dislocation density decrease may speed up by the increase of dislocation mobility with temperature and by the unpinning of the carbon atoms. In the future, the dislocation density will be quantified in the annealed wires which will contribute to the quantification of other mechanisms for the strength contribution.

Although the modern techniques such as 3D atom probe can provide the detailed information of the carbon atom distribution, the microstructure in the drawn pearlitic steel wire is so heterogeneous [14-25] and the measured carbon concentration varies greatly from one place to another. It is thus very difficult to conclude whether the carbon concentration in ferrite lamellae increases or not during the annealing up to 200 ℃ [4] and how the carbon concentration in ferrite lamellae influences the dislocation density during the annealing up to 300 ℃. For the future quantitative investigation of the carbon atom distribution and its redistribution during the low temperature annealing, the corresponding microstructural observation together with the 3D atom probe characterization of the same areas is demanded. This kind of quantitative investigation also needs several reproducible samples as the 3D atom probe characterization is destructive. To pick up the reproducible samples from the heterogeneous microstructure in the drawn wire is very difficult but becomes possible based on our recent findings that the structural evolution during the drawing process is hierarchical as the structural variations have their cause in a different macroscopic orientation of the cementite in the initial patented structure with respect to the wire axis and that the through-diameter variations subdivide the lamellar structure into two distinctly different types: one (called A_A) has a smaller interlamellar spacing and smaller dislocation density than the other (called A_BC) [8].

5. Conclusion

The changes in mechanical behavior and microstructure have been investigated for pearlitic steel wires annealed for 1h in the temperature range from 20 to 300 ℃ (293 – 573 K). The conclusions are as follows:

- By applying a very precise tensile test setup it is possible to measure the proof stress and the strain hardening from the beginning of plastic deformation to the maximum stress and strain.
- The proof stress at small offsets increases significantly with temperature in the range of 20 - 200 ℃ and decreases at temperatures above 200 ℃. These changes are related to interactions between carbon atoms in solution and the dislocation structure as the changes in strengthening parameters such as the interlamellar spacing and the dislocation density are small.
- The evolution of mechanical properties shows that annealing at 300 ℃ significantly improves the strain hardening behaviour and the total elongation with a decrease in strength of ~ 10%.

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