



Evolution of carbon distribution and mechanical properties during the static strain ageing of heavily drawn pearlitic steel wires



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ABSTRACT

The static strain ageing of heavily cold-drawn pearlitic steel wires was investigated using both global techniques and local techniques (Atom Probe Tomography (APT)), in order to highlight how the cold-drawn destabilized microstructure returns to a more stable state during post-drawing treatments between 20 °C and 150 °C. The global techniques (thermoelectric power, differential scanning calorimetry) clearly showed that ageing occurs in three successive ageing stages and is due to a redistribution of the carbon atoms coming from the strain-induced cementite dissolution. The first ageing stage was unambiguously attributed to the carbon segregation to the defects, while the second and third stages were interpreted as being due to the precipitation of intermediate carbides (2nd stage) and cementite (3rd stage). The true strain was not found to significantly affect the ageing kinetics and mechanisms but appeared to play a role in the amount of carbon atoms involved in the different ageing stages. APT analyses confirmed that ageing is governed by the carbon depletion of strain-induced supersaturated ferrite. The strengthening mechanisms associated with the different ageing stages were also discussed.

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1. Introduction

In a recent paper [1], the metallurgical mechanisms occurring during the heavy cold-drawing of pearlitic steel wires of eutectoid composition were investigated by combining two types of techniques: i) global techniques using the measurement of a physical property (thermoelectric power, electrical resistivity or internal friction) of the material and ii) a local technique (Atom Probe Tomography (APT)). The combination of these different techniques led to the conclusion that a strain-induced cementite dissolution occurs during the drawing of pearlitic wires and starts to be significant above a true strain of about 1.5, confirming the results of several studies reported in previous literature [2–11]. Consequently, as the drawing proceeds, the carbon content of ferrite gradually increases due to the transfer of carbon atoms from cementite to ferrite.

From a quantitative point of view, the average carbon content in solution in ferrite was assessed using thermoelectric power (TEP) and resistivity measurements for different strain ratios above 1.5. The results of this quantitative evaluation clearly showed that the solubility limit of ferrite is exceeded above a true

strain of 2.5 and that for a strain of 3.8, the carbon content in ferrite reaches about 0.1 wt% leading to a strong solid solution strengthening. APT analyses confirmed these results and the heterogeneity of the microstructure after drawing. Finally, from these analyses, a scenario was put forward to explain the heterogeneous microstructure of pearlitic steels.

In the abovementioned context, the aim of the present paper is: i) to explain how the destabilized microstructure obtained after drawing above a true strain of 1.5 returns to a more stable state during post-drawing treatments, ii) to identify the mechanisms responsible for the static strain ageing of the pearlitic steel wires investigated in the paper [1] after cold-drawing up to different true strains and iii) to highlight the possible influence of the true strain on the ageing kinetics and on the magnitude of the phenomena involved in ageing. In paper [12], a first analysis of the mechanisms involved in the ageing of pearlitic steel investigated in [1] was proposed after drawing at $\epsilon=3.42$. However, no quantification of the phenomena was performed and the influence of the true strain on ageing was not discussed. Moreover, no direct observations by APT were conducted.

These investigations are of great importance considering that ageing significantly modifies the tensile properties of the wires even after a short ageing time (of a few hours) at room temperature, as clearly shown in [12].

With respect to the ageing mechanisms, the results reported in

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literature tend to indicate that static strain ageing occurs in two distinct stages for ageing temperatures ranging between 20 °C and 200 °C [12–15]. Even if no consensus can be found concerning the mechanisms responsible for each stage, it should be noted that all authors support the idea that ageing is due to carbon redistribution and to its interaction with defects.

According to [12–15], the first stage detected between 20 °C and 100 °C is associated with activation energy of 85–90 kJ/mol and could be attributed to the formation of Cottrell atmospheres around the intralamellar dislocations. Other authors [16–17] found a much lower activation energy (of about 30 kJ/mol) and suggested that the first stage could be due to the formation of vacancy-interstitial pairs which could dissociate at higher temperature.

With regard to the second stage detected between 100 °C and 200 °C, it was interpreted as being due either to the thermally activated cementite dissolution [14, 18–19], to a transfer of carbon atoms from the interfacial dislocations to the intralamellar dislocations [3], to a diffusion of the carbon atoms coming from the thermally activated cementite dissolution towards the interfacial dislocations [14] or to an intermediate carbide precipitation [12].

Disagreements between authors on the ageing stages can be partly explained by two experimental facts: i) the cold-drawn microstructure is very heterogeneous and is thus extremely difficult to characterize, especially with local techniques unless specific protocols are developed; ii) the storage conditions of the samples have to be precisely controlled after cold-drawing and before ageing.

In the present study, particular attention was paid to accurately control the storage conditions of the cold-drawn samples and to avoid any unintentional ageing. Furthermore, in order to overcome the difficulties due to the heterogeneity of the drawn pearlite microstructure, global experimental techniques were used as in the case of paper [1]. Thermoelectric power technique (TEP) was thus coupled with electrical resistivity measurements, differential scanning calorimetry (DSC) experiments and tensile tests. Direct observations by APT were also carried out using an original protocol, in order to confirm the results deduced from the global techniques.

2. Material and experimental procedure

2.1. Material

The material used in this study is a pearlitic steel wire of eutectoid composition: Fe-0.91C-0.32Mn-0.22Si-0.22Cr in wt% or Fe-4.10C-0.32Mn-0.43Si-0.22Cr in at%. The wire rod, 5.5 mm in diameter, was cold-drawn to 0.26 mm up to the true strain $\epsilon=3.81$ (associated with a wire rod of 0.26 mm in diameter). As-drawn samples were taken every two dies, immediately immersed in liquid nitrogen and kept at -80 °C in order to avoid any unintentional ageing. The samples drawn above a true strain of 1.5 were then artificially aged in oil baths at temperatures between 20 °C and 150 °C to simulate the post-drawing treatments performed on pearlitic steels.

2.2. Thermoelectric power (TEP)

The principle of the TEP technique [20] is to measure the voltage (ΔV), arising from the Seebeck effect, between two junctions of the studied sample with pure metal blocks. The temperature of these is T and $T + \Delta T$ with $T = 15$ °C and $\Delta T = 10$ °C. The apparatus used in this study gives the relative TEP ($S = \Delta V / \Delta T$) of the steel with respect to the TEP of pure copper at room temperature. However, knowing the TEP of pure iron with respect to the TEP of

pure copper, one can define the relative TEP (noted S) of the sample with respect to the TEP of pure iron.

The value of S is affected by the defects present in the lattice of the iron matrix and is the sum of various contributions: $S = \Delta S_{ss} + \Delta S_d + \Delta S_{pp}$, where ΔS_{ss} , ΔS_d and ΔS_{pp} are due to elements in Solid Solution (ss), to dislocations (d) and to precipitates (pp).

The contribution of the elements in solid solution on the diffusional component of TEP of pure iron is given by the Gorter-Nordheim law [21], which can be expressed as follows:

$$\rho \cdot S = \sum_i \rho_i s_i = \sum_i s_i \alpha_i [i_{ss}] \quad (1)$$

where $\rho = \rho_0 + \sum_i \rho_i$ is the resistivity of the steel (given by the Matthiessen's rule), ρ_0 is the resistivity of the pure metal, $\rho_i = \alpha_i \cdot [i_{ss}]$ is the increase in resistivity due to the solute atoms "i". $[i_{ss}]$ represents the concentration of solute element "i" in wt%; α_i is its specific resistivity and s_i is its specific TEP.

When the concentration of the alloying elements in solution is low or when the variations of the solute content are weak, the resistivity (ρ) may be considered constant, so that the Gorter-Nordheim law becomes:

$$S = \sum_i \frac{s_i \alpha_i [i_{ss}]}{\rho} = \sum_i P_i \cdot [i_{ss}] \quad (2)$$

where P_i is the coefficient reflecting the influence of solute element 'i' on the TEP.

As already explained in [1], the carbon atoms in solid solution in iron lead to a considerable decrease of the TEP. Consequently, if the concentration of carbon atoms in solid solution decreases due to precipitation or to segregation on defects (dislocations, interfaces), the TEP is expected to increase. With regard to the carbon atoms segregated to the abovementioned defects, the works of [22,23] clearly showed that these atoms are not detected by TEP. Hence, TEP is only sensitive to the decrease in the carbon content in solution when these atoms diffuse towards dislocations or interfaces.

As far as the dislocations are concerned, they tend to decrease the TEP, as was shown in [1]. Lastly, precipitates are considered to have no significant effect on TEP (ΔS_p nearly equal to 0, unless they are small and coherent) [24].

The experimental protocol, which was used to follow the static ageing kinetics using TEP measurements is summarized in Fig. 1. Measurements were performed: i) on as-drawn samples in order to assess their initial TEP value, $S(t_0)$, and ii) on aged samples after isothermal ageing for different periods of time t at different temperatures T (with 20 °C $\leq T \leq 150$ °C) to evaluate their TEP value after ageing noted $S(T,t)$. Thus, the TEP variation induced by the ageing for a time t at a temperature T , $\Delta S(T,t) = S(T,t) - S(t_0)$, could be assessed.

2.3. Electrical resistivity

Electrical resistivity was determined by measuring the voltage drop across both a standard resistance (1Ω) and the specimen connected in series and supplied by a source of continuous current (0.5 A). The four-point method, two for the application of the current and two for reading the potential drop, was used to avoid measuring the contact resistance. The resistance of the sample was calculated by: $R = \Delta V / I$ where ΔV is the voltage drop in the sample and I is the instantaneous current, which was calculated from the voltage drop measured in the standard resistance. Knowing the specimen's dimensions, the electrical resistivity could be assessed. Specimens were 350 mm in length with variable diameters, depending on the considered strain. In order to compare the

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