Microstructural evolution, strengthening mechanisms and strength structure relationship in cold-drawn pearlitic steel wire

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Pearlitic steel wires have a nanoscale structure and a strength which can reach above 6 GPa. In order to investigate strengthening mechanisms and strength structure relationship, the microstructural evolution and structural parameters have been analyzed by transmission electron microscopy and high resolution electron microscopy in wires cold drawn up to a strain of 3.7 and a flow stress of 3.5 GPa. Three strengthening mechanisms, namely boundary strengthening, dislocation strengthening and solid solution hardening have been analyzed and good agreement has been found between the measured flow stress and the value estimated based on an assumption of linear additivity of the three contributions. Extrapolation of strengthening parameters to a larger strain and higher flow stress is also discussed.

1. INTRODUCTION

High carbon cold drawn pearlitic steel wire has the highest strength of all mass-produced steel products with a maximum experimental value of 6.3 GPa (Li et al., 2012). Nowadays 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 tires and springs. In these cold drawn wires the microstructure is alternating ferrite and cementite lamellae aligned along the wire axis. In the initial patented wire before drawing, the microstructure is lamellar pearlite with no preferential alignment to the drawing direction, which has been observed on the longitudinal section (Zhang et al., 2010). During the drawing processes, the structure becomes increasingly aligned to the wire axis (Langford 1977; Zhang et al., 2010; Zhang et al., 2011b), the cementite shows good deformability (Langford, 1977; Zhang et al., 2009b; Zhang et al., 2010; Zhang et al., 2011a; Zhang et al., 2011b) and the deformation of the cementite is strongly related to plastic deformation in the ferrite with coarse slip steps, shear bands and cracks in the cementite plates.
observed parallel to either \{110\} or \{112\} slip plane traces in the ferrite (Zhang et al., 2010; Zhang et al., 2011b). Besides these recent microstructural studies analysis of strengthening mechanisms in drawn pearlitic wires dates back to the 1960s and 1970s, and seminal papers have been published by Embury and Fisher (Embury and Fisher 1966), Langford (Langford 1977) and Gil Sevellano (Gil Sevellano 1974; Gil Sevellano 1979). The high strength was primarily related to the close spacing between the cementite lamellae (or fragments) on the assumption that the cementite acts as a barrier to dislocation glide, as do grain boundaries in polycrystalline iron. They therefore introduced the Hall–Petch relationship and related the flow stress of the wires to the reciprocal square root of the spacing between the cementite lamellae (the interlamellar spacing or ILS). It was found by transmission electron microscopy (TEM) examination that this spacing decreases in accordance with the reduction in wire diameter and a relationship between the flow stress ($\sigma$) and the strain ($\varepsilon$) was formulated (Embury and Fisher 1966):

$$\sigma = \sigma_0 + k_1 \exp(\varepsilon / 4)$$  \hspace{1cm} (1)

where $\sigma_0$ is the friction stress of the ferrite and $k_1$ is a constant which can be derived from the Hall-Petch slope. This equation is a good approximation of the evolution in flow stress with increasing drawing strain as has been shown in many studies (Embury and Fisher 1966; Buono et al., 1997; Danoix et al., 1998; Zelin 2002). An illustration is given in Fig 1. However this figure also shows significant deviation from a linear relationship especially at a large strain which points to other strengthening mechanisms in addition to the barrier resistance of the cementite. This paper will focus on the causes of this deviation by studying the structural evolution with increasing strain and by quantifying structural parameters as the thickness of the cementite and ferrite lamellae and the dislocation density in the ferrite. The quantification of the latter parameters has allowed dislocation strengthening to be estimated which has led to a quantification of the other strengthening parameters, boundary strengthening and solid solution hardening.

![Fig. 1. Summary of mechanical data relating the flow stress to the drawing strain](image)

2. EXPERIMENTAL METHODS

A high-strength near-eutectoid composition steel with a carbon content of 0.8 wt% supplied by NV Bekaert SA (Zwevegem, Belgium) Technology Center Laboratory was used for the study. Samples at five intermediate steps of the drawing process were taken, corresponding to strains of $\varepsilon = 0$ (as-patented wire) to $\varepsilon = 3.68$ (final drawn filament). Details of the samples used are listed in Table 1. The microstructure, dislocation configurations and densities were examined on thin-foil samples made from the longitudinal sections using a JEOL 2000FX TEM at 200 kV.
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and a JEOL 3000 FEG-TEM at 300 kV. The TEM samples were made using a double-jet electropolisher plus ion beam cleaning of the oxide layer produced in electropolishing. The ILS and cementite thickness were measured by TEM, taking care to ensure edge-on conditions to determine the cementite thickness. For each sample data were collected from 30 measurements on randomly chosen areas.

Table 1. Summary of wire samples used in this study.

<table>
<thead>
<tr>
<th>Wire diameter (mm)</th>
<th>1.26</th>
<th>0.899</th>
<th>0.591</th>
<th>0.332</th>
<th>0.2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area reduction (%)</td>
<td>0</td>
<td>49</td>
<td>1.51</td>
<td>93</td>
<td>98</td>
</tr>
<tr>
<td>Strain</td>
<td>0.00</td>
<td>0.68</td>
<td>78</td>
<td>2.67</td>
<td>3.68</td>
</tr>
</tbody>
</table>

3. RESULTS AND DISCUSSION

3.1 Microstructural Evolution. The evolution of the cementite morphology as a function of the drawing strain has been reported in a previous paper (Zhang et al., 2009a; Zhang et al., 2010), and it is now used to reveal the microstructural evolution during wire drawing. In agreement with previous findings (Gil Sevillano 1974), it was found that the cementite plates align with an angular spread of 30° with increasing strain to the extent that about 97% are realigned parallel to the drawing direction when the drawing strain reached 1.5. In the longitudinal section the pearlite colonies are classified into three macroscopic orientation classes, based on the angle between the length direction of the cementite plates and the wire axis (0° – 30°, 30° – 60° and 60° – 90°; hereafter called type A, B, and C respectively). The deformation of the cementite is strongly related to plastic deformation in the ferrite. Coarse slip steps, S-bands and cracks across cementite are observed parallel to \{110\}_α,Fe or \{112\}_α,Fe plane traces determined by the largest Schmid factors. Several previous investigations have reported that \{110\}<11-1>Fe₃C, and slip on \{100\}Fe₃C can be activated in wire drawing (Karlsson and Linden 1975a; Karlsson and Linden 1975b). Slip on (12-1)Fe₃C/(1-10)α-Fe was also observed in 10% cold-rolled pearlitic steel sheets (Porter et al., 1978). These results, together with the present data, indicate that slip initially takes place in the ferrite lamellae. This slip is then transferred into the cementite lamellae. Taking into account this slip mechanism for deformation of the cementite (and considering that in the initial state both plate-like and particle-like cementite is present) six types of routes for changes in the orientation and thickness of the cementite plates/particles, can be suggested, as illustrated in Fig. 2. Fig. 2A shows a favorable orientation in relation to the drawing axis since plates/particles so oriented offer a low resistance for dislocation slip in the ferrite phase. Across cementite plates/particles inclined or perpendicular to the wire axis (Fig. 2B and 2C) the applied deformation causes a rigid body rotation, resulting in twisting, and in some cases fracture of the cementite as the strain is increased. At high strains all the cementite becomes aligned close to the drawing direction and the cementite plates are lengthened and thinned in the longitudinal section and transverse section, respectively.
3.2 Structural Heterogeneities. The observation of morphological changes and the detailed analysis show that the structural evolution is not homogeneous during wire drawing. At small and medium strains the cementite reorients, a process which involves shear banding and grain curling (Hosford 1964; Gil Sevillano 1974; Langford 1977; Aernoudt et al., 1993; Zhang et al., 2011b). Such heterogeneities change the structural morphology but may also affect the density and arrangement of dislocations in the ferrite lamellae. However, the characterization of such heterogeneities is for further research, as it will require a detailed characterization of microstructural parameters at different strains and different positions in the wire. The following analysis will therefore be based on the assumption of a fibrous structure where the ferrite and cementite lamellae are parallel to the drawing direction and values for the interlamellar spacing and the dislocation density will be the average values for all structures. This averaging also allows the present data to be compared with data reported in the literature.

3.3 Structural Parameters. The thickness of ferrite (F) and cementite (T) lamellae and the dislocation density in ferrite lamellae are three structural parameters which are chosen in this study for the analysis of strengthening mechanisms and structure-strength relationship. On the assumption that T is reduced in accordance with the wire diameter, T can be related to the true strain, as follows:

\[ T = T_0 e^{-\varepsilon/2} \]  

where \( T_0 \) is the original thickness of about 19 nm. The calculated value for T is plotted in Fig. 3, illustrating good correspondence between the measured and calculated values of T. It is found that this good agreement also exists for the thickness of ferrite lamellae. The thickness of ferrite and cementite lamellae are shown in Table 2. For a strain above 3 the calculated thickness of cementite is 2.9 nm, whereas the measured thickness is about 2.1 nm. This difference points to a decomposition of about 30 wt.% cementite which will enrich the ferrite with about 1.1 at.% carbon.
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Fig. 3. Thickness of cementite lamellae vs. drawing strain. The calculated reduction in thickness of the cementite lamellae is based on the assumption that they deform in accordance with the macroscopic dimensions during wire drawing.

Table 2. Thickness of ferrite (F) and cementite (T) lamellae.

<table>
<thead>
<tr>
<th>Strain</th>
<th>0.00</th>
<th>0.68</th>
<th>78</th>
<th>2.67</th>
<th>3.68</th>
</tr>
</thead>
<tbody>
<tr>
<td>F (nm)</td>
<td>70</td>
<td>56</td>
<td>45</td>
<td>23</td>
<td>18</td>
</tr>
<tr>
<td>T (nm)</td>
<td>19</td>
<td>14</td>
<td>10</td>
<td>5</td>
<td>2</td>
</tr>
</tbody>
</table>

Dislocation configurations in the ferrite lamellae parallel to the drawing direction at a strain of 0.68 are shown in Fig. 4. Most of the dislocations are spread in the ferrite lamellae, with the two ends of the line located at the steps in the ferrite/cementite boundaries. Calculation of the dislocation density gives a value of 7E14 m^-2. The dislocation densities in the ferrite lamellae at different strains are shown in Fig. 5. It is interesting that the dislocation density shows a parabolic increase to the strain of 1.5 and a linear increase at higher strain. However saturation in the dislocation density is not observed.

Fig. 4. TEM micrograph of the wire at the strain of 0.68. The dislocation density is 7E14 m^-2. Most of the dislocations are spread in the ferrite lamellae.
3.4 Strengthening mechanisms and structure-strength analysis. The relationship between microstructure and strength will be analyzed on the basis of the structural observations and quantified structural parameters pointing to three strengthening mechanisms: boundary strengthening related to the distance between the cementite lamellae (σ(B)); dislocation strengthening related to the dislocation density in the ferrite lamellae (σ(ρ)); solid solution hardening related to the carbon concentration in the ferrite lamellae (σ(SS)). It is assumed that these strength contributions are linearly additive (Hughes, Hansen 2000).

The contribution of cementite lamellae to the strength is estimated based on the Hall–Petch equation. In applying this equation to pearlite the barrier spacing is taken to be equal to the mean free path of dislocations, which is estimated to be twice the width of the ferrite lamellae (F), i.e. (Gensamer et al., 1942).

$$\sigma_y = \sigma_0 + k(2F)^{-0.5}$$

In previous studies this has been shown to give a satisfactory description of the relationship between wire strength and barrier distance. These barriers have been characterized as tangled walls and cementite fragments (Embry and Fisher 1966), and cementite lamellae (Langford 1977) and k values have been reported of the same order as a k value characterizing the yield stress of polycrystal iron. k = 0.62 (Embry and Fisher 1966) and k = 0.58 – 0.68 (Langford 1977) over a large strain range have been reported and have been corresponded to those for polycrystalline ferrite, indicating similar strength from grain boundaries and cementite lamellae. The introduction of the strengthening mechanisms in addition to boundary strengthening as expressed in Eq. (3) has been suggested (Armstrong et al., 1962) in a modified Hall–Petch equation where $\sigma_o(\varepsilon)$ and $k(\varepsilon)$ are strain-dependent parameters. $\sigma_o(\varepsilon)$ then equals $\sigma_o + \sigma(\rho) + \sigma(SS)$ or $\sigma_o + \sigma(\rho)$ for a structure with no carbon in solution.

$$\sigma(\varepsilon) = \sigma_o(\varepsilon) + k(\varepsilon)(2F)^{-0.5}$$

The contribution of the dislocation density to the wire strength is estimated as forest hardening proportional to the square root of the dislocation density. As an alternative to this mechanism it has been suggested that the flow stress of fine laminated structures as multilayers and drawn wires is controlled by the propagation of single dislocation loops in the volume between
interfaces and the deposition of dislocations at or near the interface (Gil Sevillano 1991; Misra, Kung and Embury 2004; Embury and Hirth 1994). The controlling stress may then be the stress to propagate a dislocation (Orowan stress) and/or the stress to push a dislocation through the interface. Inherent in this alternative mechanism is that dislocations are only stored at or near the interfaces and not in the interior volume. For drawn pearlitic wires the present observations show that this is not the case, as the dislocation density is very high both in the ferrite lamellae and at the ferrite/cementite interface. On the assumption of dislocation forest hardening, Eq. (3) can be expressed as (Hansen 2005):

$$\sigma(\varepsilon) = 60 - M \alpha G b \sqrt{\rho} = k(\varepsilon)(2F)^{-0.5}$$  \hspace{0.5cm} (4)

where M is the orientation factor (1.84) (Van Houtte 1988), a is a constant (0.24) (Hansen 2004), G is the shear modulus of ferrite (77.5 GPa) and b is the Burgers vector (0.248 nm). $k(\varepsilon)$ has been calculated based on Eq. (4) taking $\sigma(\varepsilon)$ as equal to $\sigma(0.2\%)$ for the wire. Values for $k(\varepsilon)$ are given in Fig. 6, showing a fairly constant value for $k(\varepsilon)$ in the strain range 0 – 2.67, where $k(\varepsilon) = 0.31 \pm 0.01$ MPa m$^{-0.5}$, and a higher value at a strain of 3.7, where $k$ is 0.40 MPa m$^{-0.5}$. This change in $k(\varepsilon)$ may point to a contribution by solute solution strengthening due to decomposition of the cementite. A contribution from solid solution hardening is calculated based on Eq. (4) taking $k(\varepsilon) = 0.31$ MPa m$^{-0.5}$. It (Table 4) shows the contribution to the total flow stress is not important up to the strain 2.67. Based on literature data, the contribution from carbon has been estimated to be 400 MPa, in good agreement with the calculated value in Table 3. Addition of strength contributions gives values for $\sigma(\varepsilon)$ in good agreement with the experimental results, as shown in Table 4.

![Fig. 6. $k(\varepsilon)$ vs. strain](image)

### Table 3. Contribution from solid solution hardening.

<table>
<thead>
<tr>
<th>Strain</th>
<th>0.00</th>
<th>0.68</th>
<th>78</th>
<th>2.67</th>
<th>3.68</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma(0.2%)$ (MPa)</td>
<td>880</td>
<td>1286</td>
<td>1614</td>
<td>2365</td>
<td>3395</td>
</tr>
<tr>
<td>$\sigma(SS)$ (MPa)</td>
<td>-80</td>
<td>76</td>
<td>-12</td>
<td>70</td>
<td>478</td>
</tr>
</tbody>
</table>
Table 4. Comparison of measured and calculated flow stress.

<table>
<thead>
<tr>
<th>Strain</th>
<th>Experimental (MPa)</th>
<th>Calculated (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>880</td>
<td>960</td>
</tr>
<tr>
<td>0.68</td>
<td>1286</td>
<td>1210</td>
</tr>
<tr>
<td>78</td>
<td>1614</td>
<td>1626</td>
</tr>
<tr>
<td>2.67</td>
<td>2365</td>
<td>2295</td>
</tr>
<tr>
<td>3.68</td>
<td>3395</td>
<td>3317</td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

The microstructure, strengthening mechanisms and strength–structure relationships have been analyzed in a cold-drawn pearlitic steel with a structural scale in the nanometre range and a flow stress up to about 3.5 GPa. Structural parameters including the interlamellar spacing, dislocation density in the ferrite lamellae and the cementite decomposition, have been analyzed and quantified by transmission electron microscopy and high resolution electron microscopy for wires cold drawn up to a strain of 3.7. It is found that the interlamellar spacing and the thickness of the cementite lamellae are reduced in accordance with the changes in wire diameter up to a strain of 2.5. At a higher strain enhanced thinning of the cementite lamellae points to decomposition of the cementite and carbon enrichment of the ferrite lamellae. Dislocations are stored in the interior of the ferrite lamellae and their density increases to about $2 \times 10^{16} \text{m}^{-2}$. Three strengthening mechanisms, boundary strengthening, dislocation strengthening and solid solution hardening have been analyzed. The individual and combined contributions, of these mechanisms to the wire strength have been estimated. Good agreement has been found between the measured flow stress and values estimated based on an assumption of linear additivity of the three strengthening mechanisms.

In the present study the flow stress has reached 3.5 GPa at a strain of 3.68. However, by drawing to higher strains and finer structure it has been possible to reach a flow stress of 6.3 GPa at the strain of 6. This is at present under investigation if the strengthening mechanisms identified at lower strains can be extrapolated to this higher strain. For boundary strengthening and dislocation strengthening this is a possibility as no saturation of these parameters have been observed at lower strains (See Table 2 and Fig. 4). As to the solid solution strengthening it is difficult to estimate both the concentration and distribution of carbon atom in solution and their strengthening effect. This effect may also be affected by interaction between dislocations and carbon atoms where results from atom probe characterization are now forthcoming.

ACKNOWLEDGEMENTS

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