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Defining a relationship between pearlite morphology and ferrite crystallographic orientation

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ABSTRACT

This study involved fully pearlitic wires of seven different diameters (5.5–1.6 mm). All samples were laboratory annealed to re-austenitize and were then air-cooled to reform the pearlite structure. Morphological alignment of the pearlite, along the wire axis, improved significantly, 32% to 93%, as the wire diameter decreased. This improvement coincided with increases in the <110> ferrite fiber texture, and falls in the axial residual stresses. In all the wires, the majority of the pearlite lamellae appeared to align, in a 2-D analysis, with minimum elastic stiffness (E_{Min} under simple compression) for the ferrite (α). This correlation increased from 80% to 98% with decrease in wire diameter and fall in axial residual stresses. 3-D microstructures by serial sectioning, 3-D rotations seeking E_{Min} and observations on coarse pearlite, indicated that $\{011\}_\alpha$ and $<001>_\alpha$ were, respectively, the pearlite interface (or habit plane) and growth direction.

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1. Introduction

Patented pearlitic wires find wide range of applications [1–4]. Their critical properties are tensile strength and torsional ductility [1,2,5,6]. The tensile strength is controlled by the cementite interlamellar spacing (λ) [1,2,6–12], while the torsional ductility is improved by cementite alignment and appropriate states of crystallographic texture and residual stress [6,13–16]. Combinations of patenting, wire drawing and stress relief appear crucial for successful processing [1–3,6,9,16–18].

The transformation of austenite to pearlite has a rich reservoir of literature. Sorby noticed it in 1886 [19], and commented that the "direction of the alternating plates were determined by the previous crystalline structure". Belaiew in 1922 indicated [20] that the 'the lamellae of cementite will lodge themselves parallel to the crystallographic plane" of ferrite. Mehl recognized [21,22] "ferrite grain originating from the same austenite grain may have different orientations" and even explored austenite-ferrite orientation-relationships (ORs). Mehl also proposed [23] the model for austenite-pearlite transformation as "sidewise nucleation and edgewise growth". It is important to note that the sidewise nucleation model is not accepted today, as pearlitic structure has been reported to be controlled by lamellar branching [24]. The recognition that growth accounts for the larger fraction of pearlite transformation [25] brought models based on diffusion and carbon concentration gradients [26]. There is still, however, an unresolved 'controversy' on the significance of growth ledges reported on the pearlite austenite interface [24,27–35].

However, prior research on pearlite transformation, including modeling of the process, has paid little to no attention to the influence of the volume expansion of the austenite to pearlite transformation. Transformation of close packed fcc austenite to lower density bcc ferrite and orthorhombic cementite involves a volume expansion of 4.76% [36]. It also needs to be noted that bcc ferrite has strong elastic anisotropy in tensile modulus: elastic modulus along <001> being much lower than <011> and especially <111>. This is an important topic that the present study has tried to address. Fully pearlitic wires of different diameters, each re-austenized with the pearlite structure reformed by air-cooling, were characterized by EBSD (electron backscattered diffraction) and subsequent microscopy. This was done at three different thickness locations: near surface (t_0), between centre and surface (t/4) and near centre (t/2) of the respective wires. The objective was to try to establish a possible relationship between the pearlite

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morphological orientation and the ferrite crystallographic orientation. In addition measurements were made, in the annealed wires, of their residual stresses, their ferrite fiber textures, and the alignment of pearlite with the wire axis.

2. Experimental method

This study involved fully pearlitic wires of near eutectoid chemical composition (Table 1). 210 mm diameter industrial billet had been subjected to wire rod rolling, and the resulting 5.5 mm wire rod was manufactured with a finishing temperature of 1323 K. This wire rod was then subjected to multi-pass wire drawing. Samples were obtained at different stages of this drawing operation: the original 5.5 mm wire rod and the same rod after drawing to 3.1, 2.8, 2.5, 2.1, 1.8 and 1.6 mm. All wires were then annealed in a laboratory furnace at 1123 K for 20 min and then air-cooled (at an approximately 20 K/s cooling rate on the surface). At the center, the cooling rates are expected to increase with smaller wire diameters. The seven (5.5 mm−1.6 mm) fully pearlitic wires were then studied as air-cooled from the annealed austenite.

Standard metallography, followed by electro-polishing or sub-micron colloidal silica polish was performed on the longitudinal sections. It is to be noted that a few samples of the 5.5 mm wire rod were subjected to very slow cooling (0.01 K/s), in order to generate a coarser pearlite in which the cementite orientations could also be measured by EBSD. It is to be noted that sub-micron colloidal silica polish was essential for orientation measurements (by EBSD) of ferrite, in all wires, and cementite in the coarser pearlite. In the faster air-cooled pearlites, EBSD of cementite was not possible. Electro-polishing was performed in a Struers™ Lectropol-5, at room temperature, with 16 V DC and an electrolyte of 80:20 (by volume) methanol and perchloric acid. For microstructural observation, the samples were further etched with 4% nitric acid plus methanol ("4% Nital"). Microstructures, for pearlite morphology, were obtained at different cross-sectional locations. These were from near surface (t_0), between centre and surface (t/4) and near centre (t/2) locations in the wires. A FEI™ Quanta-3D FEG (Field Emission Gun) SEM (Scanning Electron Microscopy) was used for recording the microstructures. The same SEM with a TSL-OIM™ EBSD (electron backscattered diffraction) system was used for micro-texture measurements. The procedure was to perform EBSD on the electro-polished surface, followed by Nital etching to record the pearlite morphology of the same area.

Location dependent crystallographic texture and residual stress measurements were made on a Brukers™ D8 Discover system. This system had micro-focus (minimum spot size of 50 μm, with suitable laser tracking) and an area detector (Vantec™). Measurements (with appropriate oscillations) at different cross-sectional locations for crystallographic textures and residual stresses were performed. Four incomplete pole figures were measured. The ODFs (orientation distribution functions) were then calculated by inversion of these pole figures using standard [37] series expansion and the program MTM-FHM™ [38], where FHM stands for fast harmonic method. Location dependent stress measurements were made with standard d-sin$^2\psi$ [6,40–44] method for (110) pole.

Table 1
Chemical composition (in wt % alloying element) of the wire rod and wires used in this study.

C	Mn	S	P	Si	N_2
0.82	0.52	0.01	0.01	0.19	0.003

The residual stresses were also measured at the centre of the wires using a different method: multiple {hkl} GIXRD (grazing incident X-ray diffraction) [39–41,44–49]. Fig. 1a describes the GIXRD geometry and the angular conventions involved. These were,

Φ = rotation around the specimen plane normal
Ψ = inclination between specimen plane normal and the diffraction vector
ω = incident/grazing angle between the incident beam and specimen surface

In GIXRD, reflections for different {hkl} and corresponding Bragg angles (θ_{hkl}) were obtained. This defined ψ_{hkl} as,

$$\Psi_{hkl} = \theta_{hkl} - \omega \tag{1}$$

Residual stress measurements estimate appropriate inter-planar spacings ($d^{hkl}_{\phi\psi}$):

$$\begin{aligned}\varepsilon^{hkl}_{\phi\psi} &= \frac{\left(d^{hkl}_{\phi\psi} - d^{o}\right)}{d^{o}} \\ &= \Big[S_1(\sigma_{11} + \sigma_{22} + \sigma_{33}) \\ &+ \frac{1}{2}S_2\left(\sigma_{11}Cos^2\phi + \sigma_{22}Sin^2\phi + \tau_{12}Sin2\phi\right)Sin^2\psi \\ &+ \frac{1}{2}S_2\sigma_{33}Cos^2\psi + S_2(\tau_{13}Cos\phi + \tau_{23}Sin\phi)Sin2\psi\Big]\end{aligned} \tag{2}$$

σ and τ represent normal and shear components of a stress matrix and the conventions 1, 2 and 3 respectively indicate the RD, TD and ND of the specimen (Fig. 1b). S_1 and S_2 are related to the Poisson's ratio (ν) and Young's modulus (E):

$$S_1 = -\frac{\nu}{E} \tag{3}$$

$$S_2 = \frac{1+\nu}{E} \tag{4}$$

The stress measurements were done by using a multiple {hkl} GIXRD stress measurement technique [39–41,44–49], see Fig. 1c. Standard d-sin$^2\psi$ measurements [6,38–43] involve a range of ψ values. In GIXRD [39–41,44–49], on the other hand, different {hkl} offer a range of d and ψ (Eq. (1)). From the GIXRD data, a linear d-sin$^2\psi$ can be plotted [6,39,40,42,43] with appropriate extrapolation for ψ-splitting [49] (see Fig. 1c) and d shows the corresponding d-sin$^2\psi$ plot. Using appropriate conventions (Fig. 1b), σ_{11} values of the residual stress matrices were measured. A glancing angle (ω) of 5° was used.

It is to be noted that anisotropy of the elastic modulus was considered for all stress analysis. In an anisotropic elastic material, the stress (σ_{ij}) related to the strain tensor (ε_{kl}) through the equation [42].

$$\sigma_{ij} = C_{jkl}\varepsilon_{kl} \tag{5}$$

Here, C_{jkl} is the elastic stiffness matrix.

Similarly, the strain is defined in-terms of stress components through,

$$\varepsilon_{ij} = S_{jkl}\sigma_{kl} \tag{6}$$

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