Measurement of fine pearlite interlamellar spacing by atomic force microscopy

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Pearlite interlamellar spacing is an important parameter controlling ductility and strain hardening of carbon steels. Fine pearlite is the appropriate initial microstructure for drawing high carbon steel with exponential strain hardening rate, leading to high final tensile strengths. The majority of optical and electron microscopy methods for measuring interlamellar spacing present difficulties when applied to fine microstructures. Atomic force microscopy (AFM) was employed to investigate pearlitic steels lead patented at 510 °C and then cold drawn to 86% reduction in area. Conventional specimen preparation techniques for optical metallography were appropriated to produce high resolution AFM images, on which measurements of minimum interlamellar spacing, in good agreement with spacings estimated using the Embury–Fisher model, were easily performed.

1. Introduction

The high flow stress and, to a certain extent, unexpected ductility of cold drawn eutectoid and hypereutectoid steels constitute an interesting subject for accessing structure–property relationships in metals and alloys. Fine pearlite, having interlamellar spacing between 50 and 200 nm, which is usually obtained by isothermal transformation of austenite around 550 °C, is the appropriate microstructure for cold drawing this material in such a way that tensile strengths of the order of 4000 MPa are achieved after a total of 97% reduction in area [1].

The ductility of fine pearlites has been attributed to several factors, such as the discontinuity of cementite lamellae, which favours the accommodation of large amounts of strain through slip parallel to the lamellae [2]. Transmission electron microscopy (TEM) studies of the plastic deformation of pearlite have shown that the process is characterized by an initial stage in which ferrite and cementite lamellae tend to become aligned along the deformation axis [3]; this stage is followed by a progressive decrease in the interlamellar spacing and finally by bending and fragmentation of the cementite lamellae, the latter occurring by cleavage in coarse pearlites and by necking in fine pearlites [4].

During the deformation of pearlite, a cellular dislocation substructure is developed in ferrite [5, 6], interphase interfaces acting as dislocation sources even at small strains [7]. Average distances between dislocation barriers, or cell walls, decrease in proportion to the reduction in interlamellar spacing. When the strain is caused by cold drawing, the flow stress, σ, generally obeys the Embury–Fisher equation [5],

$$\sigma = \sigma_f \frac{k}{(2S_0)^{1/2}} \exp\frac{\varepsilon}{4} \quad (1)$$

where σ_f is the friction stress in ferrite, k is a constant related to the strength of the substructural barriers, S_0 is the initial pearlite interlamellar spacing and ε is the true strain, given by the expression:

$$\varepsilon = \ln(D_0/D_\varepsilon)^2 \quad (2)$$

where D_0 is the initial wire diameter and D_ε is the diameter after deformation.

Assuming that no new barriers are generated or existing barriers destroyed when the wire is drawn to a diameter D_ε, the mean barrier spacing, and thus the interlamellar spacing, are reduced in proportion to the reduction in wire diameter [6]. This means that the average pearlite interlamellar spacing is related to the deformation by the equation:

$$S_\varepsilon = S_0 \exp(-\varepsilon/2) \quad (3)$$

where S_ε is the interlamellar spacing attained after a wire of initial diameter D_0 and interlamellar spacing S_0 is drawn to a diameter D_ε, corresponding to a true strain ε.

According to the literature [6, 8], Equation 1 gives an accurate description of the strain hardening of drawn eutectoid steels, failing only when strain ageing takes place during, or immediately after, the drawing operation. The occurrence of this phenomenon in steels is associated with dislocation locking by carbon and nitrogen atoms, leading to an additional hardening [8] not included in the Embury–Fisher (EF)

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original model. Although strain ageing is an important mechanism in stabilizing high carbon steel wires for pre-stressed concrete, promoting low stress relaxation rates [9], it is also responsible for a marked decrease in ductility, which can lead to brittle failure of the wires during drawing or in subsequent forming operations [10]. However, the development of strain ageing in high carbon steel can be minimized, when necessary, by decreasing the amount of nitrogen in solid solution and/or preventing excessive heating during drawing [11].

The classical metallographic techniques usually applied to the measurement of pearlite interlamellar spacing were reviewed by Ridley [12], who pointed out that the examination of thin foil specimens or shadowed carbon replicas by transmission electron microscopy (TEM) was the most versatile technique to analyse fine pearlites. The parameter most frequently used to characterize pearlitic structures is, according to Ridley, the minimum observed spacing, S_{min}, measured on electron photomicrographs or on the fluorescent screen of the microscope. One method for measuring S_{min} is to search for pearlite colonies of minimum spacing and to count the number of lamellae crossed at right angles by a diametric line of a scribed circle of known size on the fluorescent screen of the microscope. Knowledge of the image magnification enables the determination of a reproducible average minimum interlamellar spacing. Another method involves similar measurements made on TEM photomicrographs of known magnification. Both methods require skilful selection of the observation areas, particularly in the case of deformed pearlites.

Another difficulty with these measurement methods is the preparation of samples for TEM observation, which is not an easy task and requires a great deal of experience in selecting preparation techniques suitable for a particular set of operating conditions of the electron microscope. The whole procedure is time consuming and has turned out to be of limited use for routine purposes. Techniques usually applied in conventional optical microscopy, including the partial resolution method, are strongly limited by the resolving power of optical microscopes and cannot be used in the measurement of interlamellar spacing of fine or deformed pearlites [12].

In this work, the recently developed techniques of atomic force microscopy (AFM), have been applied to measure pearlite interlamellar spacing in eutectoid steels lead patented at low temperature. Measurements have also been made on samples of these steels after cold drawing to 86% reduction in area. The results obtained were compared to values of S_0 and S_ε calculated using Equations 1 and 3, after measuring the yielding stress for 0.2% offset in samples taken after the drawing passes.

2. Materials and methods

Experimental work was carried out using three commercial steels of similar composition (Table I) lead patented at 510 °C. The material was drawn under laboratory conditions in a hydraulic drawbench operating at a constant speed of 16.7 mm s^{-1}. Bars 300 mm long, with 5.5 mm (steels A and B) or 5.0 mm (steel C) in diameter, were submitted to nine drawing passes of approximately 20% reduction in area each, leading to total reductions of 86.24% (steels A and B) and 86.60% (steel C), corresponding to true strains of, respectively, 1.984 and 2.008. In order to avoid strain ageing, the temperature of the wires was kept below 50 °C during drawing by adequate lubrication and use of a cooling system at the die exit. Samples for tensile testing, 150 mm in length, were taken after each drawing pass and stored in a freezer at -10 °C.

TABLE I Chemical composition of the steels (wt %)

Steel	C	Mn	Al	Si	N (p.p.m.)
A	0.82	0.70	0.030	0.23	44
B	0.70	0.45	0.019	0.26	34
C	0.82	0.70	0.023	0.17	95

Tensile tests were carried out using a servo-hydraulic system, operating with a constant ram displacement rate of 0.1 mm s^{-1}. Proof stresses were measured using an electronic extensometer with 2.5 mm gauge length. Atomic force microscopy was performed on metallographic specimens of transverse sections, that is, sections perpendicular to the wire axis, prepared from as-patented wires and from wires drawn to the last pass. Standard preparation techniques for optical microscopy were employed, cold-moulded specimens being ground using wet grinding paper, polished in diamond paste and etched with nital 2%.

The method used for measuring interlamellar spacing took advantage of the high resolution power of the AFM technique, allied to the versatility of the image analysis system of the equipment, a Nanoscope III from Digital Instruments. The topographic features revealed on AFM images, with cementite lamellae forming "hills" above ferrite "valleys", facilitate the choice of pearlite colonies apparently perpendicular to the sectioning plane. This could be done not only for the fine pearlite of the low temperature patented wire, but also when the material was highly deformed by cold drawing, exhibiting microstructures such as the one shown in Fig. 1. After selecting an image of such a colony, a test-line running perpendicularly to the alternating lamellae was applied over the image (Fig. 2a). The image analysis system then provides a topographic profile of the microstructure along the test-line, as shown in Fig. 2b. The system also offers the possibility of measuring distances between markers applied to such a profile. Appropriate positioning of these markers allows the measurement of the distances between the centre of adjacent cementite or ferrite lamellae or of groups of n lamellae.

For each selected pearlite colony, it was generally possible to apply the test-line three to eight times, leading to an average value of the spacing in that colony. At least 40 colonies were analysed on each metallographic specimen.

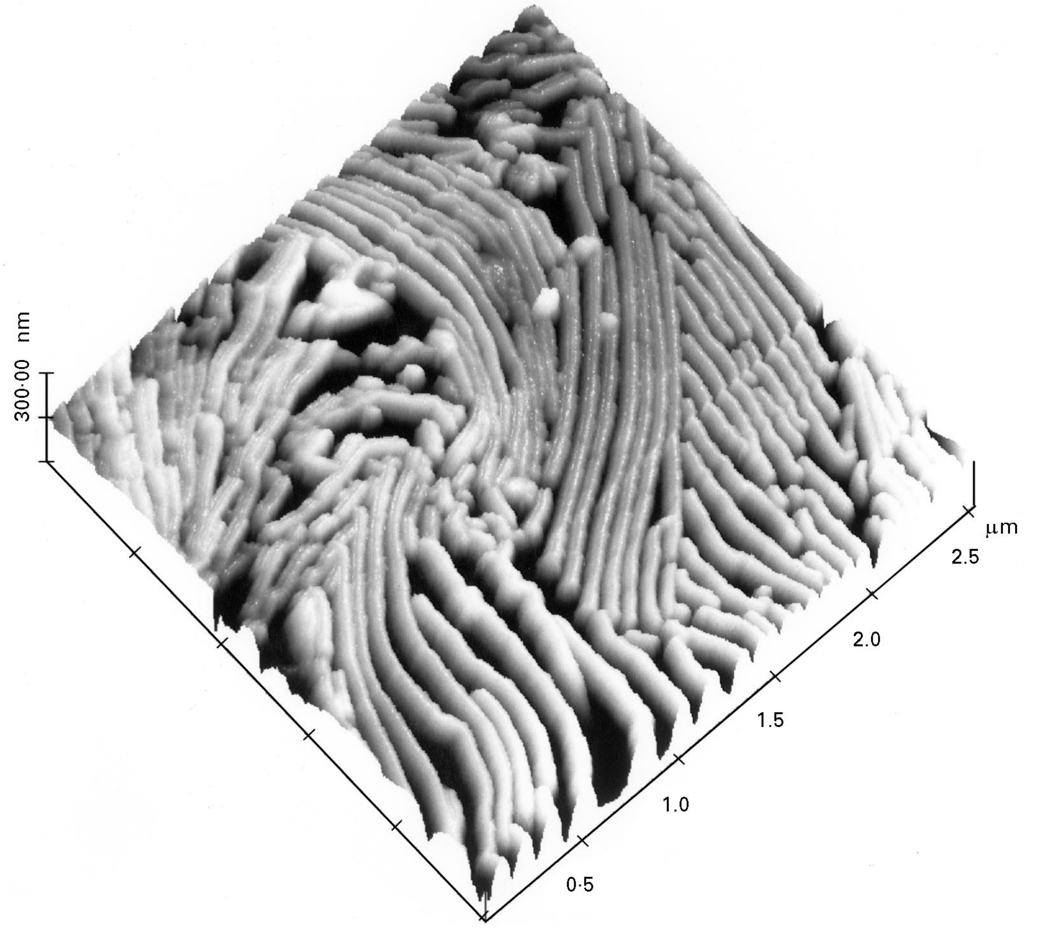

Figure 1 AFM image of pearlite colonies in transverse section of steel C after drawing to the last pass (86% reduction in area).

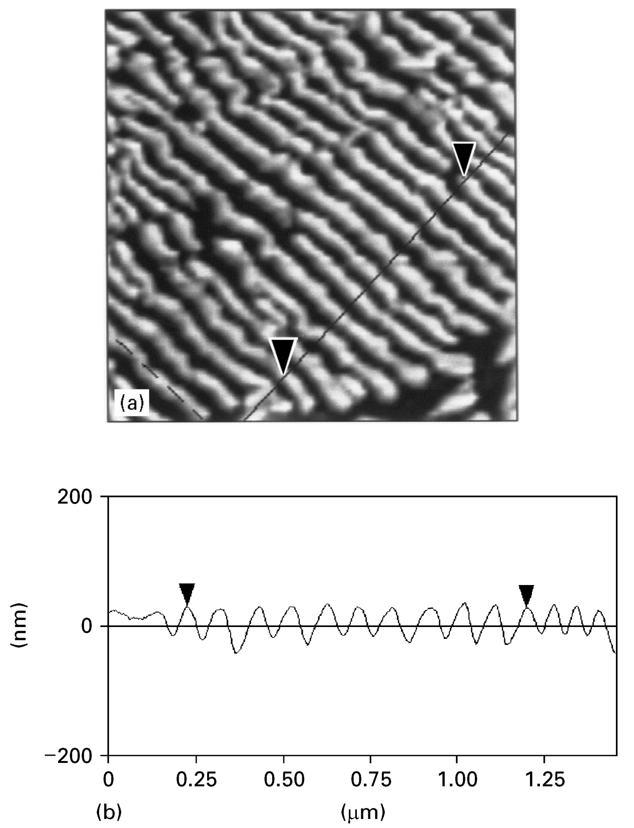

Figure 2 (a) Test-line applied in a direction perpendicular to the lamellae at a pearlite colony imaged by AFM (magnification: 3.6×10^4) and (b) topographic profile corresponding to the region crossed by the test-line.

TABLE II Pearlite interlamellar spacings in as-patented specimens and in specimens patented and drawn to 86% reduction in area

Steel	S_0 (nm)		S_ε(nm)	
	AFM	EF Model	AFM	EF Model
A	81 ± 4	83	35 ± 1	31
B	71 ± 2	91	33 ± 1	34
C	79 ± 3	80	33 ± 2	29

3. Results and discussion

Mean true interlamellar spacings, measured as described above, are presented in Table II, together with values calculated using the Embury–Fisher model, Equations 1 and 3. Values of S_0 refer to initial interlamellar spacings, while those of S_ε were obtained on specimens drawn to the last pass, corresponding to about 86% reduction in area. Standard errors presented with interlamellar spacings obtained by AFM were estimated using Student's t distribution function at the 95% confidence level. The values calculated with the EF model were estimated by linear regression, using the least squares approximation, from the results presented in Fig. 3, which shows the variation of measured 0.2% proof stresses with the deformation parameter, $\exp(\varepsilon/4)$, during drawing. According to the Embury–Fisher equation, Equation 1, the straight

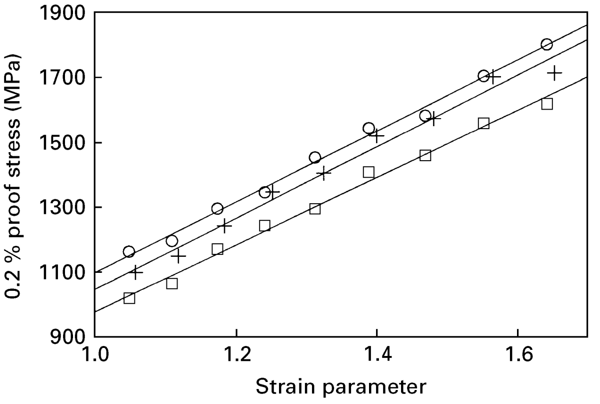

Figure 3 Variation of tensile stress with the strain parameter, exp(ε/4), for drawn wires of steels A (⊖), B (⊟) and C (+).

lines in Fig. 3 have an inclination of $k/(2S_0)^{1/2}$. The constant k, which refers to the strength of substructural barriers, was originally found to be equal to 440 kN m$^{-3/2}$ [5]. This value was later confirmed by other authors [6, 8] and was used in the present work to calculate the interlamellar spacings shown in Table II. Correlation coefficients found for the linear regression were greater than 0.99 for the three steels studied.

Comparison between measured and calculated pearlite interlamellar spacings presented in Table II show a fairly good agreement, except for the values obtained for steel B in the as-patented condition. This discrepancy is related to the fact that this steel contains a small amount of pro-eutectoid ferrite, not observed in the other two steels investigated. The presence of this constituent, which is to be expected in steel B due to its smaller carbon content, is responsible for a decrease in 0.2% proof stress, in comparison to the other two steels. Lower proof stresses, in the scope of the EF model, can only be associated with a larger pearlite interlamellar spacing. Thus, the interlamellar spacing measured in patented specimens of steel B by AFM should be essentially correct, whereas the value calculated using the EF model is probably overestimated.

4. Conclusions

Results presented in this paper show that fine pearlite interlamellar spacing in high carbon steels can be measured by atomic force microscopy using a simple and direct observation technique, applied to specimens conventionally prepared for optical metallography. The technique can be employed in the same straightforward manner to highly deformed pearlitic steels, leading to results in good agreement with values estimated using the Embury–Fisher model.

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