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Composite Microstructure of Cold-Drawn Pearlitic Steel and Its Role in Stress Corrosion Behavior

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This paper analyzes the microstructural evolution in a high-strength pearlitic steel subjected to progressive cold drawing in the course of manufacturing to produce prestressing steel wires. It is seen that the material under study possesses a composite microstructure in the form of plated patterns, which evolve toward a markedly oriented arrangement. This occurs at the two basic microstructural levels (the pearlite colonies and the pearlitic lamellae), which become aligned quasi-parallel to the wire axis or cold drawing direction, thus inducing anisotropic stress corrosion behavior of the steels. This paper offers a composites engineering approach to the modeling of this phenomenon. The approach is based on the fundamental idea of materials science: linking the microstructure of the steels (progressively oriented as a consequence of the manufacture process by cumulative cold drawing) with their macroscopic stress corrosion behavior (increasingly anisotropic as the degree of cold drawing increases). The special case of the most heavily drawn steel (strongly anisotropic) is analyzed for conditions of hydrogen-assisted cracking (HAC) and localized anodic dissolution (LAD). In both situations, the material behaves as a fiber-reinforced composite (or as a laminate) at the microstructural level.

Keywords cold drawing, pearlitic steel, microstructural orientation, stress corrosion cracking, hydrogen-assisted cracking, localized anodic dissolution, manufacture-induced anisotropy

1. Introduction

Prestressing steel wires are the main constituent of prestressed concrete structures widely used in civil engineering.[1] The wires are manufactured by cold drawing a previously hot-rolled bar with pearlitic microstructure to increase both the yield strength and the ultimate tensile strength (UTS) of the steel. The manufacture technique consisting of progressive drawing of pearlitic wires through a series of dies with diameters progressively thinner produces important microstructural changes in the material, which could induce anisotropic fracture behavior in air[2,3] and in harsh environments promoting stress corrosion cracking (SCC) of the steel.[4–6]

The aforesaid anisotropy could indicate that, as a consequence of manufacturing, the material behaves as a composite from the microstructural point of view. To clarify this point, this paper offers a composites engineering approach on the basis of materials science, so that the level of plastic deformation or *degree of cold drawing* is considered as the fundamental variable to evaluate the evolution of composite microstructure and anisotropic macroscopic behavior, as well as the relationship between them. The final aim is to provide an interdisciplinary research approach to bridge the gap between materials science and composites engineering.

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2. Materials and Composite Microstructure

2.1 Materials

The materials used in this work were high-strength steels taken from a real manufacturing process. Wires with different degrees of cold drawing were obtained by stopping the manufacturing chain and taking samples from the intermediate stages. The different steels were named with digits 0 to 6, which indicate the number of cold drawing steps undergone, so steel 0 is the hot-rolled bar (base material), which is not cold drawn at all, and steel 6 represents the prestressing steel wire (final commercial product), which has suffered six cold drawing steps with progressive reduction of diameter and increase of both yield strength and UTS. Table 1 shows the chemical composition common to all steels, and Table 2 includes the diameter (Di), the degree of cold drawing (represented by the ratio of the diameter of any steel wire to the initial diameter before cold drawing Di/D_0), the yield strength (σ_{02}) and the UTS of the steels. There is a clear improvement of (traditional) mechanical properties as the cold drawing proceeds. However, the consequences of this manufacture technique from the point of view of the fracture and SCC performance of the steels are not well known and require further research.

2.2 Evolution of Composite Microstructure

Metallographic techniques [7,8] were applied to reveal the pearlitic microstructure of the progressively drawn steels. Attention was paid to the evolution with cold drawing of the two

Table 1 Chemical composition (wt.%) of the steel

C	Mn	Si	P	S	Cr	V	Al
0.80	0.69	0.23	0.012	0.009	0.265	0.060	0.004

Table 2 Diameter (*Di*), degree of cold drawing (*Di/D*$_0$), yield strength (σ_{02}), and UTS of the wires

Steel	0	1	2	3	4	5	6
Di (mm)	12.00	10.80	9.75	8.90	8.15	7.50	7.00
Di/D_0	1.00	0.90	0.81	0.74	0.68	0.62	0.58
σ_{02} (GPa)	0.686	1.100	1.157	1.212	1.239	1.271	1.506
UTS (GPa)	1.175	1.294	1.347	1.509	1.521	1.526	1.762

basic microstructural levels: the *pearlite colonies* (first microstructural level) and the *pearlitic lamellae* (second microstructural level). These two levels can be considered as the components of the composite microstructure of the steels, *i.e.*, the basic units are the pearlitic colonies or the ferrite/cementite plates.

Longitudinal (L) and transverse (T) sections were prepared from all steel wires and mounted to undergo four grinding stages, from 320 to 1200 grit, and three polishing passes followed by etching in Nital 2%. The pearlite colonies were observed by optical microscopy, whereas scanning electron microscopy was required to resolve the lamellar structure of the pearlite. Micrographs were taken always axially oriented for the longitudinal cut and radially oriented for the transverse one.

With regard to the first microstructural level, Fig. 1 shows the optical micrographs of two different stages of the cold drawing process where an increasing deformation (slenderizing) is observed in the colonies, which determines their angle in relation to the axis. At the same time, a progressive orientation of the colonies in the cold drawing direction (wire axis) can be seen in the longitudinal metallographic sections, whereas the transverse sections show a randomly oriented appearance at all stages.

In the matter of the second microstructural level, Fig. 2 shows the scanning electron micrographs of two different stages of the cold drawing process where an increasing closeness of packing is observed in the lamellae, with a decrease of the interlamellar spacing.

As in the case of the pearlite colonies, a progressive orientation of the pearlitic lamellar microstructure in the cold drawing direction (wire axis) can be seen in the longitudinal metallographic sections, whereas the transverse sections show a randomly oriented appearance at all stages.

Therefore, both the pearlite colonies and the pearlitic lamellar microstructure (the constituents of the composite microstructure of the steel) tend to align to a direction quasi-parallel to the wire axis as cold drawing proceeds, thus inducing a progressive *strength anisotropy* in the steel, the degree of anisotropy being an increasing function of the level of cold drawing (or strain hardening) in the steels, which can be measured through the reduction of diameter Di/D_0 in Table 2.

3. SCC Behavior

3.1 Experimental Procedure

To relate these microstructural results to the macroscopic SCC behavior, slow strain rate tests were performed on transversely precracked steel wires immersed in aqueous environment and subjected to axial loading in the direction of the wire axis. The relationship between crack depth and diameter of the rod was $a/D = 0.30$ for all the diameters. Specimens were precracked by axial fatigue in air and subjected to five fatigue steps, in such a way that the maximum stress intensity factor in the final stage was always $K_{max} = 0.30\ K_{IC}$ (where K_{IC} is the fracture toughness of the steel). After precracking, samples were placed in a corrosion cell containing aqueous solution of 1 g/L $Ca(OH)_2$ plus 0.1 g/L NaCl (pH = 12.5) to reproduce the alkaline working conditions of prestressing steel surrounded by concrete. The experimental device consisted of a potentiostat and a three-electrode assembly (metallic sample or working electrode, platinum counterelectrode, and saturated calomel electrode as the reference one). The applied displacement rate was constant during the tests and proportional to each wire diameter so that file lowest rate was 1.7×10^{-3} mm/min for the fully drawn rod (steel 6: 7 mm diameter) and the highest was 3.0×10^{-3} mm/min for the hot-rolled bar (steel 0: 12 mm diameter). Tests were performed at constant electrochemical potential with the two values of -1200 mV SCE and -600 mV SCE, the former associated with the *cathodic regime* of cracking for which the environmental mechanism is *hydrogen-assisted cracking* (HAC), and the latter linked with the *anodic regime* of cracking for which the environmental mechanism is *localized anodic dissolution* (LAD), according to previous research on similar steels.[9,10]

3.2 Consequences of cold drawing in SCC behavior

The experimental results showed a fundamental fact in both HAC and LAD: the SCC behavior becomes more anisotropic as the degree of cold drawing increases, so a transverse crack tends to change its propagation direction to approach that of the wire axis, and thus, a mode I growth evolves toward a mixed mode propagation. It may be assumed that the microstructural orientation in drawn steels influences their macroscopic behavior, so the SCC resistance is a directional property that depends on the microstructural orientation in relation to the cold drawing direction (*strength anisotropy* with regard to SCC behavior). This anisotropic SCC behavior of the drawn steels can be evaluated by means of the *fracture profile* (topography of the fracture surface) obtained after the stress corrosion tests.

Figure 3 shows the fracture profile in HAC conditions, where a progressive change in the macroscopic topography as the cold drawing increases was observed in all fracture surfaces. For the lowest (or null) degrees of cold drawing (Fig. 3a), the crack growth develops in mode I in both fatigue precracking and HAC. However, mixed mode crack growth appears from a certain level of cold drawing (Fig. 3b) and is associated with crack deflection, which starts just at the tip of the fatigue precrack; *i.e.*, a deviation in the crack growth path—from its initial fatigue propagation way—appears at the very beginning of the hydrogen embrittlement test. In the last stages of cold drawing, not only crack deflection but also crack branching is observed just after the fatigue precrack tip; *i.e.*, there are two predamage directions (crack *embryos*), only one of which becomes the final fracture path.

The general trends of the fracture profile for LAD are as shown in Fig. 4. For the slightly drawn steels (Fig. 4a), the fracture surfaces were macroscopically plane and oriented perpendicularly to the loading axis; *i.e.*, they develop in mode I,

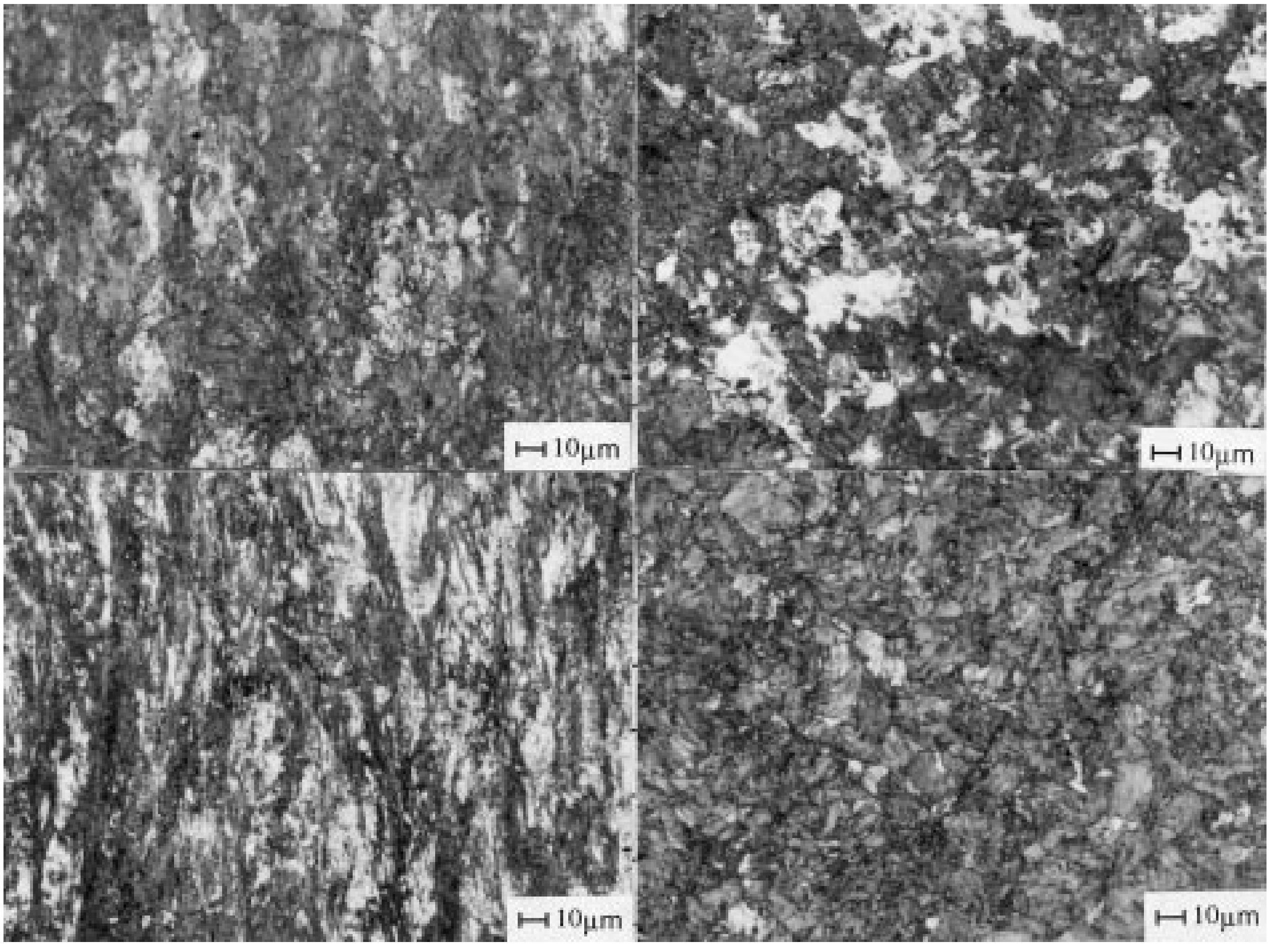


Fig. 1 Optical micrographs of steels 1 (up) and 5 (down), showing the pearlite colonies or first microstructural level. Left side: longitudinal sections. Right side: transverse sections

similarly to the case of HAC. In the steels with intermediate and high levels of cold drawing, the macroscopic fracture profile presents three characteristic zones, as sketched in Fig. 4b. After the fatigue precrack, there is a first propagation in its own plane (mode I cracking) over a disgrace x_I; after this, the crack changes its propagation direction and a mixed mode propagation takes place over a distance x_{II} (measured as the horizontal projection). Finally, the crack path follows the original direction up to final fracture. Again, not only crack deflection but crack branching is observed in the most heavily drawn steels when the mixed mode propagation appears.

4. Composite Microstructure versus Macroscopic Stress Corrosion Behavior

4.1 Microstructural Orientation and Anisotropic Behavior

Figure 5a shows a plot of the evolution of the orientation angles of the pearlitic colonies and lamellae with cold drawing (angle α between the transverse axis of the wire and the direction marked by the pearlite lamellae in the longitudinal metallographic section; and angle α' between the transverse axis of the wire and the major axis of the pearlite colony, modeled as an ellipsoid). In both cases, there is an increasing trend with cold drawing, *i.e.*, both the pearlite lamellae and colonies become increasingly aligned in the cold drawing direction.

Figure 5b shows the evolution with cold drawing of the macroscopic parameters characteristic of the crack path (fracture profile) in the HAC tests. In the slightly drawn steels, the behavior is isotropic or quasi-isotropic and the macroscopic hydrogen-assisted crack grows in mode I. The steels with an intermediate degree of cold drawing (2 and 3) exhibit a slight crack deflection associated with mixed mode propagation. In the most heavily drawn steels, the crack deflection is more pronounced and the mixed mode takes place suddenly after the fatigue precrack, the deviation angle and the step height reaching their maximum values.

Figure 5c shows the evolution with cold drawing of the macroscopic parameters characteristic of the crack path (fracture profile) in the LAD tests. The behavior is qualitatively similar to that of the HAC tests, *i.e.*, isotropic or quasi-isotropic in the slightly drawn steels and increasingly anisotropic with cold drawing. The important difference is that the material is able to undergo mode I cracking in LAD conditions, even for the heavily drawn steels, although when the crack deflection

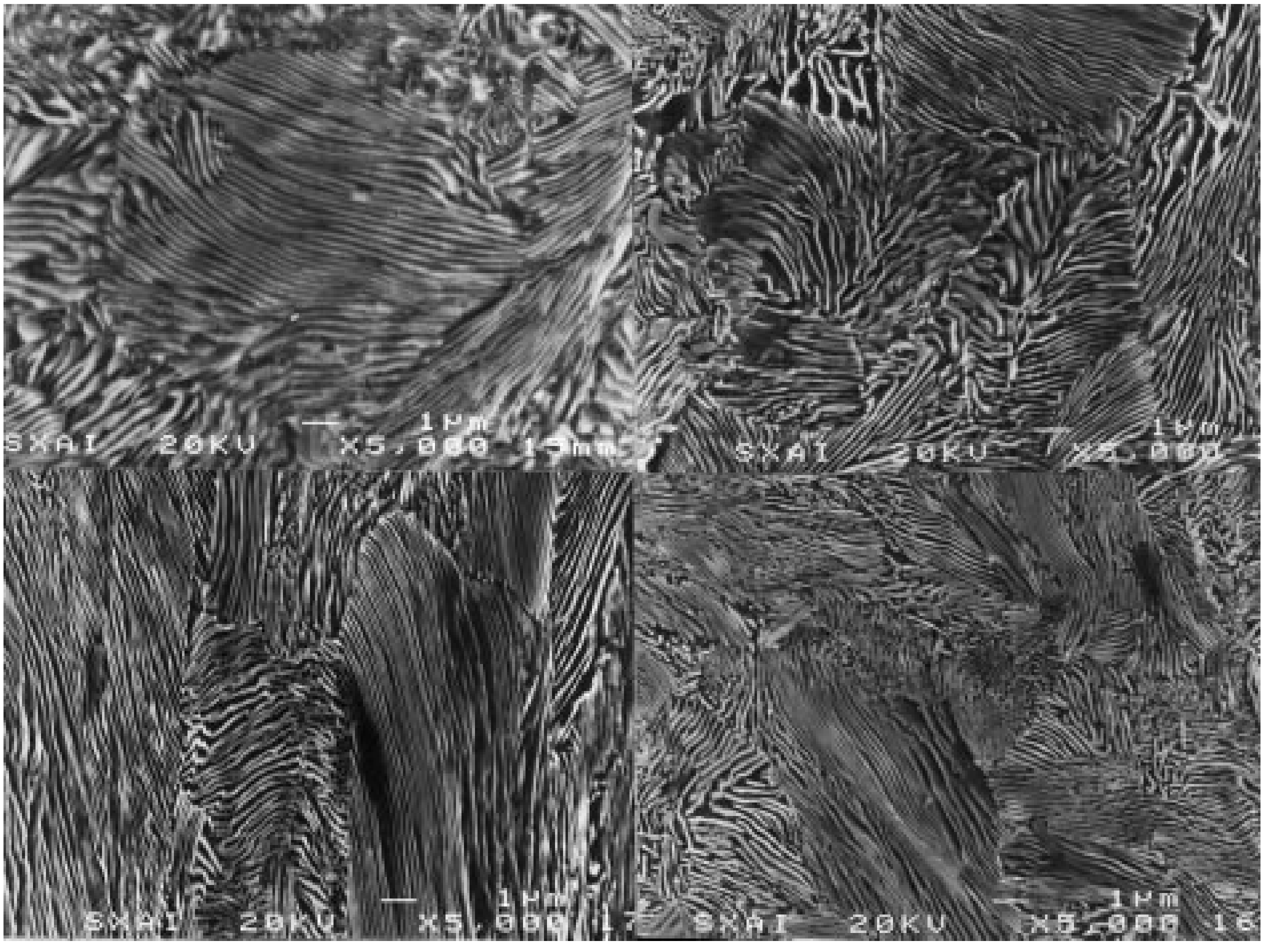


Fig. 2 Scanning electron micrographs of steels 1 (up) and 5 (down), showing the pearlitic lamellar microstructure or second microstructural level. Left side: longitudinal sections. Right side: transverse sections

appears, the mode I propagation distance is a decreasing function of the degree of cold drawing (*cf.* Fig. 5c).

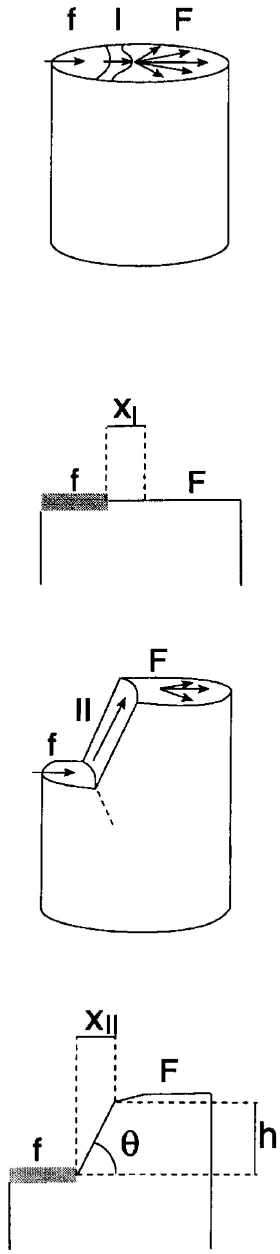
Figure 5 demonstrates that the progressive microstructural orientation (at the two levels of colonies and lamellae) clearly influences the angle and height of the fracture step (increasing with the degree of cold drawing in both HAC and LAD) and the mode I distance in LAD (decreasing with it for heavily draw steels). This change in crack propagation direction can be considered as the signal of the microstructurally induced anisotropy of these materials: from a certain degree of cold drawing, the cracks find propagation directions with lower fracture resistance. This suggests that the macroscopic SCC behavior of the different steels (progressively anisotropic with cold drawing) is a direct consequence of the microstructural evolution toward an oriented arrangement.

4.2 On the Fracture Initiation

Now the question is to find out whether there is a unique fracture initiator or promoter and, in case of existence, to determine which of the microstructural basic units (the pearlite colony and the pearlitic lamellae) plays that role, *i.e.*, if the weakest link for fracture initiation is the bond between pearlite colonies or the ferrite/cementite interface. To clarify this, this section provides a detailed comparison of the evolution with cold drawing of the macroscopic and microscopic parameters, particularly of the deflection angle of the macroscopic crack and the orientation angles of colonies and lamellae.

From observation of Fig. 5, the following statements may be inferred, all of them valid for the entire manufacturing route, *i.e.*, suitable to *all* stages of cold drawing represented by the ratio Di/D_0.

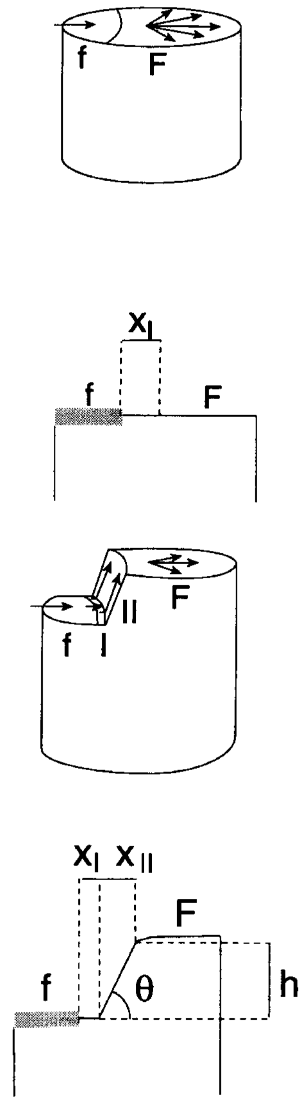
- For any degree of drawing, the microscopic orientation angles (α and α') are above the macroscopic deflection angles (θ_{HAC} and θ_{LAD}). This facts support the assumption that the microstructural pattern (the cause) influences the macroscopic behavior (the effect).
- For any degree of drawing, the microscopic orientation angle of the colonies is higher than that of the lamellae, *i.e.*, $\alpha' > \alpha$ in Fig.5(a), which indicates that the pearlite colonies are oriented first and the pearlitic lamellae do the same on tow.
- For any degree of drawing, the macroscopic fracture angle is higher for HAC than for LAD, *i.e.*, $\theta_{HAC} > \theta_{LAD}$, *cf.* Fig.



(a)

(b)

Fig. 3 Fracture profile for HAC: (**a**) slightly drawn steels; (**b**) heavily drawn steels; f: fatigue crack growth; I: mode I propagation; II: mixed mode propagation; and F: final fracture



(a)

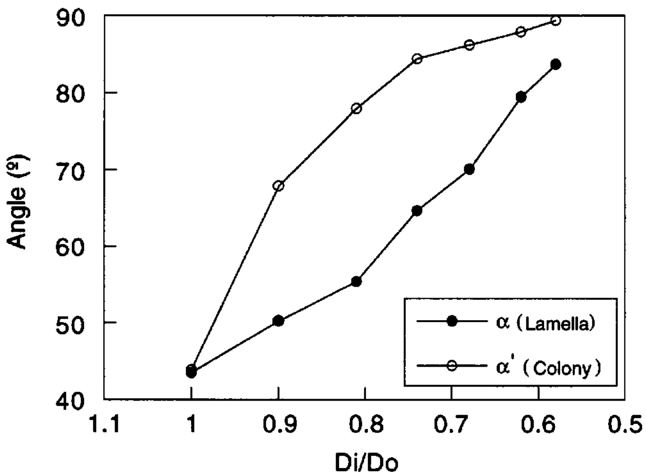
(b)

Fig. 4 Fracture profile for LAD: (**a**) slightly drawn steels; (**b**) heavily drawn steels; f: fatigue crack growth; I: mode I propagation; II: mixed mode propagation; and F: final fracture

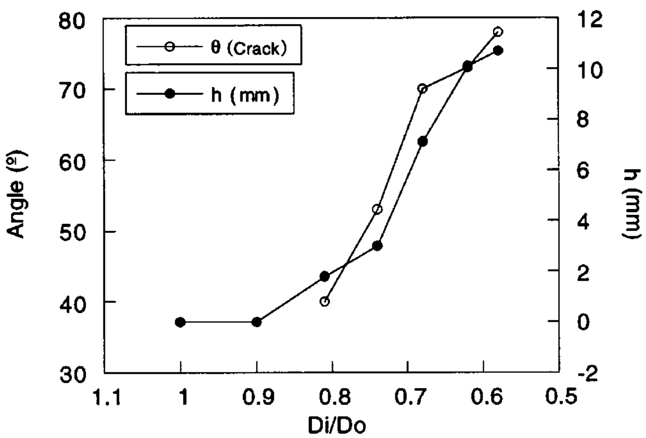
5b and 5c which seem to demonstrate that HAC is more influenced than LAD by the microstructural orientation.

These three statements seem to indicate that the pearlitic lamellae could act as the main promoters of catastrophic fracture in the specimens, the weakest link being the ferrite/cementite interface, and thus the mechanism of final failure would be delamination (or debonding) at the second microstructural level, at least in the case of HAC for which the macroscopic crack deflection angle and the orientation angle of the pearlitic lamellae are really close for the final stages of cold drawing.

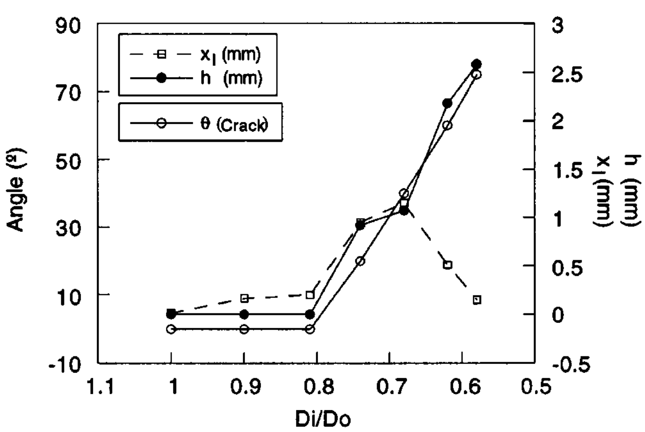
Nevertheless, although the question of the possible existence of a weakest link is a fundamental problem in materials science, from the point of view of the composite modeling of the fracture, it is not a key issue since the two microstructural levels (pearlite colonies and pearlitic lamellae) tend toward a very marked orientation in the direction of cold drawing and they became almost parallel to the wire axis. Thus, any micromechanical model to reproduce this arrangement, as well as its macroscopic consequences, should be constituted by oriented cells, units, or components. The following section of the paper offers a composites engineering approach to modeling the behavior of the final commercial product (that of the highest engineering



(a)



(b)



(c)

Fig. 5 Relationship between microstructure and macroscopic SCC behavior: (**a**) evolution with cold drawing of the orientation angles of colonies and lamellae in the pearlitic microstructure; (**b**) evolution with cold drawing of the macroscopic crack angle and step height in HAC conditions; (**c**) evolution with cold drawing of the macroscopic crack angle, step height, and mode I propagation distance in LAD conditions; and the angles α, α', and θ are measured from the radial direction (transverse to the wire axis).

interest): the prestressing steel wire, which has been heavily drawn in the course of manufacture.

5. Heavily Drawn Steels: A Composites Engineering Approach

5.1 Material Anisotropy

A simple composites approach is used to model the oriented pearlitic microstructure of the fully drawn wire. Two methods may be followed: a one-dimensional (1-D) approach in the form of a fiber-reinforced composite to reproduce the alignment of the microstructure, and a two-dimensional (2-D) approach in the form of a laminate to account for the plated microstructure at the finest microscopical level. Since the microstructure is very markedly oriented (angle of 70 to 90° to the radial direction), the modeling assumes that it is totally oriented and the angle is 90°; *i.e.*, the fibers in the 1-D modeling (or the plates in the 2-D modeling) are completely oriented in the wire axis or cold drawing direction. Since the problem is axisymmetric, the fiber model may be adopted and for the sake of simplicity the prestressing steel (cold drawn) wire can be considered as a *fiber-reinforced* composite.

Such a microstructural arrangement has consequences of both a mechanical and a chemical nature, as follows.

(1) *Strength anisotropy (mechanical anisotropy), i.e.*, fracture toughness K_{IC} as a function of the orientation angle: $K_{IC} = K_{IC}(\theta)$. It may be assumed that $K_{IC}(0°) \gg K_{IC}(90°)$.
(2) *Chemical anisotropy, i.e.*, hydrogen diffusion coefficient D as a function of the orientation angle: $D = D(\theta)$. It may be assumed that $D(90°) \gg D(0°)$.

In both (1) and (2), the orientation angle θ is measured from the radial direction in the wires, so that $\theta = 0°$ is the direction perpendicular to the fibers in the 1-D modeling, whereas $\theta = 90°$ is the direction parallel to the fibers. Therefore, the toughness is lower in the wire axis direction (delamination is easier than breaking the fibers) and the hydrogen diffusion coefficient is higher in the wire axis direction (diffusion parallel to the fibers).

5.2 Micromechanics of HAC

The anisotropy of the material explains the crack deviation from its initial propagation path in mode I. Two mechanisms could be operative, according to a terminology coined by Gerberich *et al.*:[11] hydrogen-enhanced localized plasticity (HELP) or hydrogen-enhanced delamination (HEDE), although it could also be hydrogen-enhanced debonding (or splitting) if the fiber-reinforced model is considered.

With regard to HELP, its importance in the HAC of prestressing steel is probably low, since the lamellar structure of the steel (markedly oriented) probably delays or even blocks the dislocation movement, which otherwise could be operative in an isotropic material.

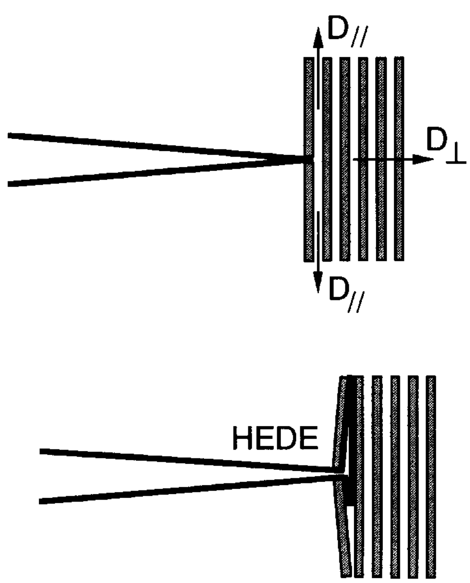
In the matter of HEDE, its importance in HAC of prestressing steel is probably high because of the markedly oriented lamellar structure of the steel. Thus, a micromechanism of fracture by

HAC is proposed in Fig. 6, according to which hydrogen-diffuses mainly in the direction of the plates (Fig. 6a) and can weaken the bonds or interfaces between the ferrite and the cementite lamellae (which are the weakest links even before the hydrogen presence), thus contributing to the hydrogen-induced fracture by delamination or debonding between two similar microstructural units, *e.g.*, at the ferrite/cementite interface (Fig. 6b).

5.3 Micromechanics of LAD

In LAD, the crack does not change its propagation path in spite of the oriented microstructure of the steel. The explanation could lie in the *local strain rate*[12] required to promote the anodic dissolution, which is achieved only at the crack tip in the $\theta = 0$ direction. The crack does finally change its propagation direction after a certain subcritical growth by LAD in mode I because of the presence of very slender *pearlitic pseudocolonies*, with anomalous (too large) local interlamellar spacing and with microcracks that makes them preferential fracture paths with minimum local resistance.[3]

The mechanism of LAD in cold drawn steel could be as follows (Fig. 7): dissolution is produced in mode I along a distance x_{LAD}. The crack continues in mode I along the initial plane and only deviates when it reaches a defect (predamage) in the material: the pearlitic pseudocolonies, which are potential fracture sites. When this happens, final fracture takes place for purely mechanical reasons. Since the pearlitic pseudocolonies are also markedly oriented in the direction of cold drawing or wire axis (in the same way as the standard colonies), the deflection angle (macroscopic) also approaches the orientation angles (microscopic) of the first and second microstructural levels, *i.e.*, the pearlitic colonies and lamellae.



(a)

(b)

Fig. 6 Micromechanism of HAC in heavily drawn steels: (**a**) hydrogen diffusion in longitudinal and transverse directions, (**b**) fracture by HEDE, (hydrogen-enhanced delamination or debonding).

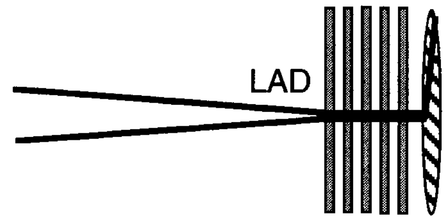


Fig. 7 Micromechanism of LAD in heavily drawn steels by anodic dissolution of the fibers (mode I crack growth) and posterior mechanical fracture of a slender pearlitic pseudocolony.

6. Conclusions

The strong plastic deformations produced during manufacture affect the steel microstructure, which becomes progressively oriented in the wire axis direction as a direct consequence of cold drawing. This happens at the two basic microstructural levels: the pearlite colony and the pearlitic lamellae. The material thus possesses a composite microstructure.

The aforesaid microstructural orientation influences the microscopic and macroscopic aspects of the fracture mode in aggressive environments, showing a general evolution from crack propagation in mode I for slightly drawn steels to mixed mode propagation (with strong mode II component) for heavily drawn steels.

In the two SCC regimes (HAC and LAD), there is a strong correlation between the microstructural orientation angles (at the two levels of the pearlitic colonies and lamellae) and the macroscopic crack deflection angles, which clearly demonstrates the influence of the oriented composite microstructure—and thus of the manufacture process by increasing cold drawing—on the macroscopic SCC behavior of the steel wires.

A composites engineering approach may be used to model the oriented microstructure of cold drawn steels in the form of a fiber-reinforced composite, which exhibits anisotropy of both a mechanical (strength) and a chemical nature, *i.e.*, the values of the fracture toughness and the hydrogen diffusion coefficient depend on the direction.

The micromechanism of HAC in heavily drawn steels (strongly anisotropic) is HEDE or, generally speaking, hydrogen-enhanced debonding between two similar microstructural units, *i.e.*, at the ferrite/cementite interface or at the boundaries between pearlitic colonies.

The mechanism of LAD in heavily drawn steels could be explained by dissolution in mode I along a certain distance: The crack deviates when it reaches a very slender pearlitic pseudocolony with anomalous local interlamellar spacing, which is a potential fracture site and fails for purely mechanical reasons.

Acknowledgments

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