# IMAGE ANALYSIS OF HYDROGEN-ASSISTED MICRO-DAMAGE IN PROGRESSIVELY DRAWN PEARLITIC STEEL

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# ABSTRACT

This paper analyzes the evolution of hydrogen-assisted damage topography in progressively drawn pearlitic steels. The fractographic analysis revealed changes in the microscopic topographies depending on the fracture propagation mode, with an evolution from pure *tearing topography surface* (TTS) in slightly drawn steels to a kind of *very deformed* TTS (in direction of cold drawing or wire axis) in heavily drawn steels. An image analysis technique was used to analyze the different microscopic fracture modes, thus finding correlations between them. Results showed that the very deformed TTS *really* observed in heavily drawn steels could be *virtually* obtained by deforming –in the drawing direction– the micrograph of slightly drawn steels. This fact provides bases to elucidate the real physical micromechanism of hydrogen-assisted fracture in pearlitic steels.

# **KEYWORDS**

Pearlitic steel, cold drawing degree, hydrogen-assisted micro-damage, fracture micromechanisms

## **INTRODUCTION**

Cold drawn eutectoid steels are high-strength materials used as constituents of prestressed concrete structures in civil engineering. Previous research [1,2] dealt with the fracture of this kind of steel in aggressive environment, showing that heavily drawn prestressing steels exhibit anisotropic fracture behavior associated with changes in the crack propagation direction and transition from mode I to mixed mode propagation approaching the wire axis direction.

In more recent works [3,4] this research line has been developed to analyze steels with intermediate degree of cold drawing, in order to relate –in the materials science manner– the macroscopic behavior of the steels with their microstructure at the two basic microstructural levels of the pearlite colonies [5,6] and the pearlitic lamellar microstructure [7,8], as a function of the cold drawing degree achieved during the manufacturing process to produce the final commercial product.

This paper goes further in the research line and studies –by means of image analysis techniques– the evolution of hydrogen-assisted microdamage topographies at the microscopic levels for the different degrees

of cold drawing, in order to elucidate the physical micromechanisms of fracture of pearlitic steels in hydrogen environments, taking into account the progressively anisotropic fracture behaviour as the strain hardening level produced by cold drawing increases.

## MATERIALS AND MICROSTRUCTURE

The materials used in this work were high-strength steels taken from a real manufacturing process at EMESA TREFILERIA, S.A. 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. Table 1 shows the chemical composition common to all steels, and Table 2 includes the diameter ( $D_i$ ), the yield strength ( $\sigma_Y$ ), the ultimate tensile stress ( $\sigma_R$ ) and the fracture toughness ( $K_{IC}$ ).

CHEMICAL COMPOSITION (wt %) OF THE STEELS							
С	Mn	Si	Р	S	Cr	V	Al
0.80	0.69	0.23	0.012	0.009	0.265	0.060	0.004
TABLE 2         DIAMETER REDUCTION AND MECHANICAL PROPERTIES OF THE STEELS							
Steel	0	1	2	3	4	5	6
$\begin{array}{c} D_{i} \mbox{(mm)} \\ \sigma_{Y} \mbox{(GPa)} \\ \sigma_{R} \mbox{(GPa)} \\ K_{IC} \mbox{(MPam^{1/2})} \end{array}$	12.00 0.686 1.175 60.1	10.80 1.100 1.294 61.2	9.75 1.157 1.347 70.0	8.90 1.212 1.509 74.4	8.15 1.239 1.521 110.1	7.50 1.271 1.526 106.5	7.00 1.506 1.762 107.9

 TABLE 1

 CHEMICAL COMPOSITION (wt %) OF THE STEELS

The microstructural evolution with cold drawing was studied in previous woks [5-8]. Attention was paid to the evolution with cold drawing of the two basic microstructural levels: the pearlite colonies (first level) and the pearlitic lamellae (second level). With regard to the first microstructural level, a progressive elongation and orientation of the pearlitic colonies in the cold drawing direction (wire axis) was observed [5,6]. In the matter of the second microstructural level, the analysis showed an increasing closeness of packing (with decrease of the interlamellar spacing) and a progressive orientation of the pearlitic lamellar microstructure in the cold drawing direction [7,8]. Therefore, both pearlite colonies and pearlitic lamellar microstructure tend to align to a direction quasi-parallel to the wire axis as cold drawing proceeds. Figures 1 and 2 (cf.[8]) show the pearlitic microstructure of steels 0 and 6 respectively, by means of micrographs corresponding to longitudinal metallographic sections of the wires (those sections containing the wire axis). It is seen that, while the microstructure of steel 0 (hot rolled bar which is not cold drawn at all) is randomly oriented, the microstructure of steel 6 (prestressing steel heavily cold drawn) is markedly oriented in the direction of the wire axis (or cold drawing direction) which corresponds to the vertical side of the

micrographs. Figures 3a and 3b plot the quantification of this orientation effect at the levels of colonies and lamellae respectively.



Figure 1: Microstructure of steel 0 (L-section)



Figure 2: Microstructure of steel 6 (L-section)



**Figure 3:** Evolution with cold drawing of the orientation angles of pearlite colonies (a) and lamellae (b), in the longitudinal (L) and transverse (T) metallographic sections. In (a) the angle was measured between the major axis of the colony (modeled as an ellipsoid) and the axial direction (L section) or the radial one (T section). In (b) the angle was measured between the direction of the lamellae in the metallographic cut and the axial direction (L section) or the radial one (T section).

#### **EXPERIMENTAL PROGRAM**

To analyze the hydrogen assisted cracking (HAC) behavior of the different steels, slow strain rate tests were performed on precracked steel wires. Samples were precracked by axial fatigue in the normal laboratory air environment to produce a transverse precrack, so that the maximum stress intensity factor during the last stage of fatigue precracking was  $K_{max} = 0.30K_{IC}$ , and the crack depth was a = 0.30D in all cases, with D as the wire diameter. After precracking, samples were placed in a corrosion cell containing aqueous solution of 1g/l Ca(OH)<sub>2</sub> plus 0.1g/l NaCl (pH=12.5). All tests were conducted under potentiostatic control at -1200 mV vs. SCE at which the environmental mechanism is HAC [1]. The applied displacement rate in axial direction was constant during each test and proportional to each wire diameter:  $1.7 \times 10^{-3} \text{ mm/min}$  for steel 6 and  $3.0 \times 10^{-3} \text{ mm/min}$  for steel 0.

The experimental results showed an important fact: the HAC behaviour becomes more anisotropic as the cold drawing degree increases, as depicted in Figure 4 by means of the *fracture profile* (macroscopic topography of the fracture surface). Thus a transverse crack tends to change its propagation direction to approach the wire axis or cold

drawing direction and an initial mode I growth evolves towards a mixed mode propagation (*strength anisotropy* with regard to HAC).



**Figure 4:** Evolution with cold drawing of fracture surfaces in HAC, from slightly to heavily drawn steels (left to right). Increasingly anisotropic behaviour with cold drawing (f: fatigue; I: mode I propagation; II: mixed mode propagation; F: final fracture by cleavage).

# FRACTOGRAPHIC ANALYSIS

A fractographic analysis by scanning electron microscopy (SEM) was carried out on the fracture surfaces to elucidate the microscopic modes of fracture. Figures 5 and 6 offer respectively the microscopic topographies for hot rolled bar (steel 0 which is not cold drawn at all) and for a heavily drawn prestressing steel (steel 6 which has suffered six steps of cold drawing).

In the hot rolled bar (steel 0; cf. Figure 5) there is a subcritical crack growth in mode I by *tearing topography surface* (TTS, cf. [9-12]) before the unstable cleavage-like propagation associated with mechanical final fracture. This special microscopic fracture mode is undoubtedly associated with hydrogen-assisted micro-damage in steels with pearlitic microstructure [11-12].

In the heavily drawn prestressing steel wire (steel 6; cf. Figure 6) the microscopic fracture mode is a kind of *very deformed* TTS, the deformation axis coincident with the cold drawing direction. This topography is produced as a consequence of the macroscopic crack deflection with mode II propagation, *as if* a previous TTS mode had been strained by shear in its own fracture plane (quasi-parallel to the drawing axis).



Figure 5: Microscopic fracture mode in steel 0.



Figure 6: Microscopic fracture mode in steel 6.

The microscopic fracture modes of Figures 5 and 6 may be classified as *hydrogen damage topographies* (HDT). The appearance of this generic topography is a function of the degree of drawing so that it can be noted as  $HDT^{(i)}$ , where *i* is the number of cold drawing steps undergone by the steel. Thus the topography  $HDT^{(i)}$  evolves form pure TTS (which corresponds *exactly* with the mode described in [9-12]) in steel 0 to  $HDT^{(6)}$  (a kind of TTS *very* 

*deformed* in the wire axis or cold drawing direction) in steel 6. Between them, a wide range of HDT topographies with intermediate degree of deformation was observed.

## DISCUSSION

Form the observation of the fractographs corresponding to  $HDT^{(0)}$  (Figure 5) and  $HDT^{(6)}$  (Figure 6), the following statements may be drawn:

- All the HDT modes for different degrees of cold drawing are associated to the same HAC mechanism (cathodic potentials and decrease of stress intensity factor).
- A generic HDT<sup>(i)</sup> exhibits an *apparent deformation* (in the fractograph) which is an increasing function of the degree of cold drawing (represented by the superindex *i* which indicates the number of cold drawing steps undergone by the steel).
- Such a deformation seems to be oriented in the direction of the larger size of the fractograph, i.e., in a direction quasi-parallel to the wire axis or cold drawing direction.

Thus the question arises about whether or not a geometric relationship does exist between the HDT modes so that one can be *virtually* obtained by deformed a previous one (associated with a lower degree of cold drawing). This virtual transformation is sketched in Figure 7, in which the initial fractograph HDT<sup>(0)</sup> (or pure TTS) in the hot rolled bar is virtually oriented in the direction of HAC in the cold drawn material and deformed by transformations in the radial, hoop and axial directions.

The parameters of the geometric transformations are obtained by the hypothesis of conservation of volume in the Plasticity theory and represent *the virtual deformation* which should be applied to the HDT<sup>(i)</sup> to obtain other HDT<sup>(j)</sup> (j > i). Figure 8 shows the results of this virtual deformation process to obtain HDT<sup>(6)</sup> from HDT<sup>(4)</sup>, where it is seen that this virtual topography really resembles the real one shown in Figure 6, which confirms that the micromechanism of fracture is similar in both cases.



Figure 7: *Virtual* deformation of the fractograph.



Figure 8: Virtual HDT<sup>(6)</sup> by deformation of HDT<sup>(4)</sup>.

# CONCLUSIONS

In eutectoid steels, the microstructural changes produced by the cold drawing process affect the macroscopic behavior with regard to environmentally assisted cracking in general and hydrogen assisted cracking (or hydrogen embrittlement) in particular.

In heavily drawn steels, the microscopic fracture mode is a kind of very deformed tearing topography surface, the deformation axis coincident with the cold drawing direction. This topography is produced as a consequence of the macroscopic crack deflection with mode II propagation.

A proportional relationship was found between the different hydrogen damage topographies (HDT): the very deformed TTS *really* observed in heavily drawn steels could be *virtually* obtained by deforming the micrograph of the pure TTS (associated with the hot rolled material) in the direction of cold drawing.

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