Hydrogen Induced Damage in Heavily Cold-Drawn Wires of Lean Duplex Stainless Steel

Mihaela Iordachescu, Maricelyde Abreu, Andrés Valiente

Abstract

The paper addresses the sensitivity to hydrogen embrittlement of heavily cold-drawn wires made of the new generation of lower alloyed duplex stainless steels, often referred to as lean duplex grades. It includes comparisons with similar data corresponding to cold-drawn eutectoid and duplex stainless steels. For this purpose, fracture tests under constant load were carried out with wires in the as-received condition and fatigue-precracked, in air and exposed to ammonium thiocyanate solution. Microstructure and fractographic observations were essential means for the cracking analysis. The effect of hydrogen-assisted embrittlement on the damage tolerance of lean duplex steels was assessed regarding two macro-mechanical damage models that provide the upper bounds of damage tolerance and accurately approximate the failure behavior of the eutectoid and duplex stainless steels wires.

Keywords: Cold-drawn lean duplex stainless steel; Embrittlement mechanisms; Damage tolerance;

1. Introduction

Today, high-strength stainless steel wires obtained by cold drawing are potential candidates for prestressing concrete due to their mechanical properties, excellent resistance against corrosion and high damage tolerance when compared to eutectoid prestressing steels, as indicated by Moser et al. (2012a), Valiente and Iordachescu (2012), and De Abreu et al. (2014).

Generally, strain hardening induced by cold drawing reduces the resistance of austenitic stainless steel to pitting
corrosion by transforming the austenite phase into martensite, as resulting from Wu and Nürnberg (2009), Recio et al. (2013), and Grimault et al. (2012). However, recent investigations have shown that this effect is limited in high alloyed, duplex stainless steel wires since most of them are immune to pitting corrosion, according to De Abreu et al. (2014) and Moser et al. (2012b). In these wires, hydrogen uptake increases their potential to axial cracking, as propitiated by the microstructure orientation, De Abreu et al. (2014).

In the last years, a new generation of lower alloyed duplex steels wires is being manufactured often referred to as lean duplex grades. Their sensitivity to corrosion and stress corrosion damage is poorly addressed. Hence, the motivation of present research, which assesses the resistance to stress corrosion damage of one of these high strength lean duplex wires. The paper includes comparisons with similar data corresponding to cold-drawn eutectoid and duplex stainless steels wires. For this purpose, fracture tests were carried out with wires in the as-received condition and fatigue precracked, in air and ammonium thiocyanate solution, under constant load. Microstructure and fracture surface observations have been used for the damage analysis. Finally, the effect of hydrogen-assisted embrittlement on the damage tolerance of lean duplex steels was assessed regarding two mechanical damage models that provide the upper bounds of damage tolerance and accurately approximate the failure behaviour of the eutectoid and duplex stainless steels wires.

### Nomenclature

<table>
<thead>
<tr>
<th>Wire Type</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ES</td>
<td>Eutectoid steel wire</td>
</tr>
<tr>
<td>DSS</td>
<td>Duplex stainless steel wire</td>
</tr>
<tr>
<td>LDS</td>
<td>Lean duplex steel wire</td>
</tr>
</tbody>
</table>

2. Materials and testing design

1.1. Materials characteristics

The materials used in this study are basically 2 high-strength duplex stainless steel cold drawn wires of 4 mm diameter. One of them hereinafter referred as DSS, is high alloyed, and the other, LDS is low alloyed. A third 4 mm diameter high strength wire of eutectoid steel (ES) was added to contrast the research results. Tables 1 and 2 indicate their chemical composition and mechanical properties, respectively. The distinct alloying level of the duplex steel wires is directly reflected by the resistance to pitting corrosion, as evaluated by PREN (Recio et al. 2013). Although both PRENs are high (PREN<sub>DSS</sub> = 37, PREN<sub>LDS</sub> = 27), the difference between the steels is 35%.

#### Table 1. Chemical composition of studied wires (percentages by weight)

<table>
<thead>
<tr>
<th>Wire Type</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>N</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>DSS</td>
<td>0.03</td>
<td>0.61</td>
<td>1.78</td>
<td>0.03</td>
<td>0.001</td>
<td>0.18</td>
<td>22.80</td>
<td>3.33</td>
<td>4.80</td>
<td>Bal.</td>
</tr>
<tr>
<td>LDS</td>
<td>0.03</td>
<td>1.00</td>
<td>5.00</td>
<td>0.035</td>
<td>0.015</td>
<td>0.11</td>
<td>20.50</td>
<td>0.60</td>
<td>2.25</td>
<td>Bal.</td>
</tr>
<tr>
<td>ES</td>
<td>0.78</td>
<td>0.21</td>
<td>0.67</td>
<td>0.012</td>
<td>0.022</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

#### Table 2. Mechanical properties of studied wires

<table>
<thead>
<tr>
<th>Property</th>
<th>DSS</th>
<th>LDS</th>
<th>ES</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elastic modulus [GPa]</td>
<td>160</td>
<td>180</td>
<td>205</td>
</tr>
<tr>
<td>Yield strength [MPa]</td>
<td>1420</td>
<td>1350</td>
<td>1640</td>
</tr>
<tr>
<td>Tensile strength [MPa]</td>
<td>1660</td>
<td>1820</td>
<td>1740</td>
</tr>
<tr>
<td>Maximum uniform deformation [%]</td>
<td>2.2</td>
<td>2.3</td>
<td>3.2</td>
</tr>
<tr>
<td>Elongation [%]</td>
<td>70</td>
<td>51</td>
<td>50</td>
</tr>
</tbody>
</table>
As illustrated in Fig. 1, the microstructures of DSS and LDS wires in the axial direction, revealed by scanning electron microscopy (SEM), are biphasic and composed by longitudinally elongated grains of austenite (light gray) and ferrite (dark gray). The micrographs show the wires anisotropy induced by cold drawing but no differences between DSS and LDS can be established at this scale.

1.2. Testing design

The method used to determine the sensitivity to hydrogen embrittlement of DSS and LDS wires consisted of conducting stress corrosion tests under constant load in ammonium thiocyanate solution (FIP). Both smooth and fatigued precracked wire samples were tested. ES wires were also tested for comparative purposes.

Additionally, 7 fracture tests of LDS wires precracked by fatigue were made in order to experimentally evaluate their damage tolerance in hydrogen-free condition.

Fatigue precracking was carried out on transversely notched wire samples; cyclic tensile loads between 1 and 7 kN at 5 Hz were used. In order to control the size of the generated crack through flexibility measurements, the notched area was instrumented with a resistive extensometer of 12.5 mm baseline. 2 DSS, 11 LDS, and 2 ES fatigue precracked specimens were prepared.

The constant load FIP test consisted of maintaining the smooth or fatigue precracked samples in aqueous solution of ammonium thiocyanate, at 50°C, loaded at 80% of the specimen’s resistant capacity. The test result is the time elapsed until the specimen breaks or survives when a predetermined number of testing hours have passed. The total number of FIP tests being: 5 of DSS (3 smooth and 2 precacked), 9 of LDS (7 smooth and 2 precacked) and 4 of ES (2 smooth and 2 precacked).

Finally, scanning electron microscopy (SEM) was performed to identify the hydrogen embrittlement and corresponding damage mechanisms of wires.
3. Results and discussion

All FIP tests performed with smooth or fatigue precracked DSS specimens were interrupted after a time not less than 1 week. After a detailed examination of their surface, they were broken in tension. In contrast to the indefinite breaking times of DSS in the FIP tests, LDS showed less resistance to the environment aggressiveness, as they broke after about 100 h. However, the time to failure of these two duplex steel wires widely exceeds the limits required by standards for prestressing steel wires, like ES. The two comparative graphs presented in Fig. 2 provide a clear picture of the time to failure in the FIP test of the analyzed wires. According to the data given in Fig. 2a, the lifetime of LDS smooth specimens exceeds by more than 10 times that of ES specimens in the same condition, but the times to failure of both steel wires are very similar when previously damaged by fatigue cracks. Despite this, their collapse mechanisms substantially differ.

The results indicate that the simultaneous action of constant load and FIP solution do not initiate damage in the DSS wires, but as previously observed by Iordachescu et al. (2015) may increase an existing one (Fig 3a). Hence, the work focuses on the analysis of damage micromechanisms in LDS wires to gain insight into this behavior.

Figs. 3a,b,c are illustrating the macroscopic damage and collapse induced by hydrogen in the fatigue precracked and smooth specimens of DSS and LDS steels when subjected to FIP testing. As shown in Fig. 3a, subcritical longitudinal cracking occurs in the fatigue precracked DSS wires because hydrogen accumulates at the crack tip locally reduces the cohesion of the austenite-ferrite interface. According to Valiente and Iordachescu (2012) a longitudinal crack initiates at the fatigue crack tip, advances between these two phases and arrests when the stresses are not high enough to break the interface, even weakened by hydrogen action. The process is successively repeated resulting in subcritical cracking of specimen parallel to the wire axis. Due to the slow growth of this crack, the FIP test concluded without breaking the specimen, which was subsequently ruptured in air. The transversal compliance provided to the specimen by longitudinal cracking allows the resistant ligament to move towards the loading line and to collapse in simple tension, by plastic instability and necking.

Fatigue precracked LDS (Fig. 3b) breaks in the FIP test because subcritical longitudinal cracking occurs at higher velocities along almost the whole length of the wire specimen immersed into the aggressive solution. The subcritical cracking mechanism of precracked DSS wires is not fully explaining that of LDS, given that in this case hydrogen does not only weakens the austenite-ferrite interface, but it also anodically dissolves the longitudinally elongated grains of ferrite. Fig. 3c and Fig. 3d are longitudinal sections of same smooth LDS specimen from which evidences of hydrogen damage were provided.

As shown in Fig. 3c and Fig. 3d, subcritical cracking across the smooth LDS wires initiates in the FIP test; it occurs once the passivating oxide layer that superficially protects the sample surface is locally broken. Then hydrogen penetration takes place and a subcritical crack develops in a plane perpendicular to the wire axis by the combined action of the applied load and the anodic dissolution of the ferrite phase; the local loss of ferrite weakens the biphasic
microstructure and stress it enough to break the elongated grains of austenite and thus, the crack growth is produced. The specimen fails when the lateral deflection generated by the transverse crack transforms it into a kinked crack that propagates longitudinally. The final collapse mechanism does not differ from that of the fatigue precracked DSS specimens when subjected to an interrupted FIP test and subsequently tensile tested in air up to fracture.

Fig. 4. Failure features of smooth LDS subjected to FIP testing: a) macrofractograph showing the wire rupture; b) elliptical crack model assimilated to the hydrogen damaged area; c) hydrogen induced damage by trans and intergranular decohesion; d) ductile collapse by voids coalescence.

Fig. 5. Comparison of the experimental values of LDS damage tolerance in FIP solution vs. the damage tolerance limits of the theoretical models of combined tension and bending plastic collapse and of plastic collapse in tension of ES and DSS wires in air.

Fig. 4a shows the fracture surface of a smooth LDS specimen that failed during FIP testing, from which two areas of well-differentiated morphologies can be distinguished. The assimilated one to the elliptical crack configuration of
Fig. 4b consists of trans and intergranular decohesion, as detailed in Fig. 4c and, it results from the hydrogen embrittlement of the austenitic - ferritic interface and the anodic dissolution of the ferritic grains. The remaining area is the result of the ductile rupture of the resistant ligament by plastic collapse.

The influence of hydrogen embrittlement on the damage tolerance of LDS steel was assessed regarding two plastic collapse models of cracked wires, which provide the upper bounds of damage tolerance and are in good agreement with the respective failure behavior of the ES and DSS wires (Iordachescu et al. 2014). The predictions of the models are given in Fig. 5 in terms of the quotient between the failure loads in the cracked and smooth condition \( (P_m/P_0) \) versus the relative crack size area \( (A_f/A_0) \). The plot also contains the LDS experimental data obtained from the fracture tests made with fatigue-precracked hydrogen free specimens and as well as from the FIP tests. Then, as resulting from Fig. 5 the theoretical model of plastic collapse in tension accurately approximates the damage tolerance of LDS wires, hydrogen free. Hydrogen uptake slightly decreases the damage tolerance of LDS wires.

### 4. Conclusions

Heavily cold-drawn wires, of lean duplex steel fail by plastic collapse when hydrogen embrittled even containing fatigue cracks. A subcritical cracking process occurs until the applied load equalizes the plastic bearing capacity of the resistant ligament. Then, the wires of this type are highly damage tolerant materials.

In the standard FIP test, the lifetime of this wires is not unlimited, but it exceeds that of cold-drawn eutectoid wires used for prestressing by more than 10 times. The hydrogen-induced damage changes the micromechanisms of failure from void coalescence to trans and intergranular decohesion. The environment-assisted damage progressively extends as a subcritical transversal crack from the wire surface until it reaches the critical size for the plastic tensile collapse of the resistant ligament. However, some previous cracking in axial direction seams to occur, so that collapse can take place in pure tension, with no bending contribution. This feature, which is quite similar to that found in cold-drawn, high alloyed duplex steel wires increases the damage tolerance up to its upper bound behavior.

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### References


