



# Effects of hydrogen assisted stress corrosion on damage tolerance of a high-strength duplex stainless steel wire for prestressing concrete



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## HIGHLIGHTS

- Hydrogen assisted stress corrosion cracking in cold drawn duplex steel wires.
- Effects of hydrogen on damage tolerance of cold drawn duplex steel wires.
- Tensile failure mechanism of damaged cold drawn duplex stainless steel wires.

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## ABSTRACT

The study brings new insights on the hydrogen assisted stress corrosion on damage tolerance of a high-strength duplex stainless steel wire which concerns its potential use as active reinforcement for concrete prestressing. The adopted procedure was to experimentally state the effect of hydrogen on the damage tolerance of cylindrical smooth and precracked wire specimens exposed to stress corrosion cracking using the aggressive medium of the standard test developed by FIP (International Prestressing Federation). Stress corrosion testing, mechanical fracture tests and scanning electron microscopy analysis allowed the damage assessment, and explain the synergy between mechanical loading and environment action on the failure sequence of the wire. In presence of previous damage, hydrogen affects the wire behavior in a qualitative sense, consistently to the fracture anisotropy attributable to cold drawing, but it does not produce quantitative changes since the steel fully preserves its damage tolerance.

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## 1. Introduction

Prestressing concrete is a major finding in civil engineering, based on artificially induced internal stresses to prevent concrete from tensile stresses when external loads act. Since this occurs at the expense of prestressing steel, which is heavily tensile loaded during prestressing concrete, the technique requires steels of extremely high tensile strength and moderate cost. Today, these two conditions are satisfied by applying cold-drawing as manufacturing technique to high-carbon eutectoid steels. Therefore, most of the currently produced prestressing steels with tensile strength ranging from 1600 to 1800 MPa are cold-drawn wires and strands, and to a less extent, quenched and tempered bars.

Prestressing steel is the most critical component of prestressing concrete structures. Often, its failure involves the collapse of the

whole structure. Then, damage resistance and damage tolerance are properties that must be required from prestressing steel to the same extent that damage is propitiated by the service conditions of the structure. In practice, the minimum 3.5% of uniform elongation required to cold-drawn wires for prestressed concrete [1] assures the damage tolerance level intrinsic to ductile steels [2].

In contrast to reinforced concrete, prestressed concrete is free of cracks due to the absence of tensile stresses, but this does not suppress the risk of corrosion damage of prestressing steel by environmental attack. The collapse of prestressing concrete structures originated by corrosion has occasionally occurred for years [3,4]. Once disregarded the cases originated by defective steel or underestimated environmental aggressiveness, the susceptibility to stress corrosion cracking is the feature that governs the corrosion failure of the prestressing steel products [3].

Stress corrosion cracking is a localized damage process resulting from the combined action of tensile stresses and environmental attack. Models of stress corrosion cracking range from the cracking dissolution of the metal to its local embrittlement due to the uptake of damaging species, as dissociated hydrogen. There is a general agreement that hydrogen uptake is the dominant and most

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dangerous stress corrosion cracking mechanism for prestressing steels [5–7].

Protection systems consisting of mechanical or electrochemical barriers to the aggressive media are available, but experience shows that they are doubtfully effective, if not counterproductive [8]. As consequence, two technological testing procedures, FIP and DIBt, were respectively developed by FIP (International Prestressing Federation) and DIBt (German Institute of Structural Engineering), in order to assess the resistance to stress corrosion cracking of prestressing steel [6,9]. Both tests, designed to promote hydrogen uptake into prestressing steel, are standardized [9]. Even though their discriminating power is similar, they differ in the aggressiveness of the employed media and consequently in the test duration [10].

Stainless steel is a structural metallic material with great potential of being used in prestressed concrete due to its excellent corrosion resistance and its ability to be cold worked. Furthermore, stainless steel products are widely used in the construction industry as passive reinforcements of concrete and as mechanical connectors for constructional details [11]. Then, strength and ductility levels typical of prestressing steel can be obtained by deep cold drawing of stainless steel up to 80% reduction in cross sectional area [12–14].

However, cold drawing can give rise to the transformation of austenite into martensite, in both austenitic and duplex stainless steels [15]. Then, strain-induced martensite reduces the corrosion resistance of stainless steel in two ways [16]: by forming with austenite a galvanic corrosion micro-cell that favors pitting corrosion and by providing to hydrogen both a penetration path and a microstructural component easy to embrittle. In austenitic stainless steels the alloying elements such as Ni, Mn and Cu are austenite stabilizers and mitigate the volume fraction of strain-induced martensite [16,17]. In duplex grade steels, strain-induced martensite fraction depends on the volume fractions of austenitic and ferritic phases and the cold drawing level [18]. Their corrosion resistance is considerable improved by additional alloying elements such as Mo and N [18,19].

So far, more attention has been paid to pitting corrosion of cold-drawn austenitic and, to a less extent, to duplex stainless steel, when exposed to the most aggressive environment found in prestressed concrete, given by the carbonated concrete with high concentration of chlorides. However, previously reported results on corrosion resistance of duplex steels wires are highly satisfactory, especially when containing fractions of Mo ranging from 2% to 3%, are enhancing their potential use as prestressing steels [13,14].

In contrast to pitting corrosion resistance of cold-drawn wire of stainless steel [11,16,18], few data concerning its resistance to hydrogen assisted stress corrosion cracking are available; four austenitic and one duplex steel wire of different Mo content have been FIP tested [16,17]. Only the tests performed with the austenitic steel, Mo free, resulted in catastrophic failure; even though, the wire lifetime was much longer than that expected from any existent eutectoid prestressing steel [16,17].

In this research, the previously assessed results of [2] regarding the damage the tolerance of cold drawn duplex 1.4462 stainless steel wires are extended to damage caused by hydrogen embrittlement as stress cracking corrosion mechanism. The serious risk that this damage process entails to the integrity of prestressing concrete structures, the potential use of cold drawn duplex steel wire as prestressing steel, and the scant available data impelled the investigation.

The FIP testing method is adopted as the basic means of producing the hydrogen assisted damage to the examined duplex steel wire. Subsequent tensile testing and Scanning Electron Microscopy (SEM) analysis were used for the damage assessment. Fatigue

pre-damaged wire specimens were also subjected to the FIP test in order to separate the hydrogen effects in the initiation and propagation stages of the damage process.

## 2. Experimental details

The tested high strength stainless steel is a 1.4462 duplex grade [20], industrially manufactured as a 4 mm diameter wire, by several passes of cold-drawing up to producing about 70% of cross section reduction. Such a cold-drawing process gives rise to a strongly orientated microstructure along the drawing direction and provides a mechanical resistance comparable to that of the eutectoid prestressing steels. The chemical composition of the tested 1.4462 duplex steel is given in Table 1.

Tensile properties of the wire were determined at room temperature [9], on samples of 450 mm length, as benchmark on evaluating its resistance to hydrogen assisted damage. The tensile tests were performed with a 200 kN servohydraulic universal testing machine using a constant crosshead speed of 1 mm/min. Elongations were measured on a gauge length of 12.5 mm with a conventional clip-on extensometer. Table 2 shows the mechanical characteristics of the wire.

Three wire specimens (Fig. 1) were fatigue precracked from sharp notches of 0.5 mm depth, machined with a straight front perpendicular to the longitudinal axis of the wire. Fatigue precracking was performed by applying cyclic tensile loads ranging between 1 and 4 kN. An average number of 10,000 load cycles was applied to each specimen, at a frequency of 5 Hz. To produce cracks of nominally equal size, a conventional clip-on extensometer, of 12.5 mm gauge length, was monitoring the fatigue crack growth through the elastic compliance of the notched zone. One of the precracked specimens was used to determine, by direct measurement, the wire bearing capacity in this damaged condition. The other two precracked specimens and a number of undamaged specimens, of same length, were dedicated to stress corrosion testing.

The damage tolerance of this wire steel had been previously obtained [2] by tensile breaking wire specimens, similarly precracked for a range of crack depths from 0.2 to 0.6 times the wire diameter.

The FIP test method was used to assess the susceptibility to stress corrosion of the prestressing steel wires [3]. It consists of constant tensile loading the wire specimen, immersed in a non-circulating corrosive solution of ammonium thiocyanate, maintained at constant temperature. The load is set to produce a constant stress equal to 0.8 times the tensile strength of the wire. The corrosive solution, of approximate 4.5 pH, contains 200 g of  $\text{NH}_4\text{SCN}$  for each 800 ml of distilled  $\text{H}_2\text{O}$  is kept at 50 °C. The hydrogen embrittlement of the wire steel results from a cathodic stress corrosion mechanism [6]. The test result is the lifetime to fracture of the specimen. Normally, the test is stopped if fracture does not take place within 240 h.

The specimens to be tested were rubbed with cotton dipped in butanone ( $\text{CH}_3\text{COCH}_2\text{CH}_3$ ) and washed with isopropyl alcohol ( $\text{C}_3\text{H}_8\text{O}$ ). Deionized water, of conductivity less than 0.5  $\mu\text{S}/\text{cm}$ , was used for preparing the corrosive solution. The tests were performed in double-walled thermostatic corrosion cells containing about 1 l of the corrosive solution. An exposed length of about 20 cm was delimited at the central part of the wire specimen; an adhesive tape was used to avoid the differential aeration of the cell. The solution temperature was maintained at  $50 \pm 1$  °C through a continuous water flow, pumped in the external chamber of the cell from a thermostatic bath. The specimen was mechanically fastened to the load train with prestressing commercial anchorages and sealed in place. Once vertically anchored the specimen, the inner chamber of the cell was filled with the corrosive solution. The tensile load was immediately applied by hanging dead weights to a lever machine coupled to the loading train. A timer, which automatically stops once the specimen breaks, registered the testing time. The experimental arrangement is depicted in Fig. 2.

Three undamaged wire specimens and two precracked ones were tested according to the FIP procedure with the following variations regarding the applied load: two undamaged specimens were tensile stressed to 80% of the tensile strength of the wire, while the third was only stressed to 60%. The precracked specimens were loaded to 80% of the failure load of an equally cracked wire, obtained by tensile testing, in air, the third precracked specimen.

The test procedure and the equipment used for tensile testing the cracked wire specimens as well as the cylindrical smooth wires, in as received or modified conditions, were the same as required in [9]; the clip-on extensometer, of 12.5 mm gauge length, was attached to the specimen in front of the crack.

**Table 1**

Chemical composition of the tested 1.4462 duplex steel (% weight).

C	Si	Mn	P	S	N	Cr	Mo	Ni	Fe
0.03	0.614	1.779	0.029	0.001	0.178	22.8	3.33	4.8	Bal.

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