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# Effect of hydrogen and inclusions on the tensile properties and fracture behaviour of A350LF2 steels after exposure to wet H<sub>2</sub>S environments

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#### ABSTRACT

The tensile properties and fracture behaviour of A350LF2 steels after exposure to wet  $H_2S$  environments were investigated by corrosion and tensile testing. The results suggest that relative tensile strength and plasticity loss increased as hydrogen concentration increased. Greater tensile property damage occurred in steel that contained more large-size inclusions. When hydrogen was released, the degree of relative strength and plasticity loss decreased. Fractography of the corroded samples exhibited mixed ductile– brittle rupture. Fractured areas with quasi-cleavage features increased with increasing hydrogen concentration. Flakes appeared in the steel with a low sulphur content and low hydrogen concentration.

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#### 1. Introduction

The applications of oilfield equipment and pipeline steels used in the exploration and transportation of oil and gas that contain  $H_2S$  are limited due to the mechanical and corrosion problems induced by  $H_2S$ . Hydrogen damage is one of the major causes of failure in oilfield steels [1–5].

When steels are exposed to a wet H<sub>2</sub>S environment, hydrogen atoms are produced as a result of the cathodic reduction of the proton (H<sup>+</sup>) that accompanies the anodic oxidation of iron. The hydrogen atoms could combine to form molecular gaseous hydrogen. However, the recombination reaction of hydrogen atoms to form molecular hydrogen is retarded on the steel surface due to the presence of  $H_2S$  or hydrogen sulphide ions (HS<sup>-</sup>) [6–8]. As a result, some atomic hydrogen diffuses into the steel. According to the model proposed by Troiano [9] and Oriani[10], the local atomic cohesive force decreased in the presence of hydrogen. In addition, cracking can occur if the critical amount of hydrogen necessary for crack initiation is accumulated [3,11]. On the basis of the aforementioned theory, steel can lose its ductility and strength in a wet H<sub>2</sub>S environment. This phenomenon is known as hydrogen embrittlement (HE) and is in agreement with the observations of Li et al. [12] and Dong et al. [13]. However, another phenomenon has also been observed. A study by Sofronis et al. [14] revealed that the yield stress increased slightly with increased hydrogen concentrations. They also showed a linear relationship between yield stress and hydrogen concentration. According to Delafosse and Magnin [15], the yield stress of an X52 pipeline steel was mildly affected by electrolytic hydrogen, and only the failure strain was reduced. Alhussein et al. [16] investigated the influence of hydrogen on the tensile properties of API 5L X52 pipeline steel and showed that hydrogen had little effect on yield stress and ultimate strength. Thus, no unique, generally accepted theory for the effect of hydrogen on metals has been identified. Additionally, the variations of the tensile properties have not been yet investigated systematically in cases where diffusible hydrogen is released from the steel.

On the other hand, it has been acknowledged [1] that inclusions act as important hydrogen traps and affect hydrogen damage. Thus, the critical role of inclusions in hydrogen damage has been studied extensively during the past 20 years. Many types of inclusions have been verified in steels, such as aluminium- and siliconenriched inclusions, magnesium and calcium oxides, and manganese sulphide inclusions [17]. In addition, the number, shape and size distribution of the inclusions are important parameters that affect hydrogen damage. According to Ciszewski et al. [18], Jin et al. [19] and Xue and Cheng [20], hydrogen-induced cracks are primarily associated with oxides or mixed inclusions; the cracks did not seem to be initiated by manganese sulphide inclusions. Kim et al. [21] showed that oxide inclusions with lengths over 20 µm were harmful to hydrogen induced cracking (HIC) in steels possessing a bainitic ferrite structure. Furthermore, the results presented by Domizzi et al. [22] revealed that there was no clear correlation between the average length of manganese sulphide inclusions or sulphur content and HIC resistance. From the





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Table 1
Chemical composition of the A350LF2 steels (wt.%).

Steel variant	С	Si	Mn	S	Р	Cr	Ni	Cu	Мо	V
A350 I	0.19	0.18	0.81	0.021	0.029	0.10	0.033	0.026	0.006	0.002
A350 II	0.17	0.23	1.2	0.002	0.012	0.019	0.011	0.012	0.005	0.005

aforementioned results, it can be recognized that there is no obvious relationship between hydrogen damage and manganese sulphide inclusions. However, Wilde et al. [23] investigated the role of inclusions in the hydrogen induced blister cracking of pipeline steels in sulphide environments and showed that hydrogen-induced cracks initiated at the manganese sulphide inclusions. Thus, to develop a deep understanding of the role of inclusions in hydrogen damage, it is necessary to further study the relationship between inclusions and hydrogen damage.

Starting from these insights, an experimental study was launched with the objective of researching the effect of hydrogen and inclusions on the tensile properties and fracture behaviour of two variants of A350LF2 steel containing different amounts of sulphur after exposure to wet H<sub>2</sub>S environments.

#### 2. Experimental procedure

#### 2.1. Test material preparation and microstructure analysis

Two variants of A350LF2 steel flange forgings (designated as A350 I and A350 II in the following text) were used in this study. After hot working, the forgings underwent normalising treatments at 1173 K followed by a tempering heat treatment at 913 K for 3 h and then cooled in air. The A350LF2 steels were not used in any environment prior to the experiments. The tensile strengths of A350 I and A350 II were 588 and 526 MPa, respectively. The chemical composition of the A350LF2 steels is given in Table 1. All the tensile specimens were oriented perpendicular to the forging axis, and the dimensions of the specimens are shown in Fig. 1. Before the experiments, all the faces of the specimens were machined to obtain a finish equivalent to that achieved using 260 grit carbide silicon paper; these specimens were then rinsed with deionised water and degreased with acetone.

For microstructural observations, all specimens were ground with 2000 grit carbide silicon paper and polished with 1.2  $\mu$ m diamond paste. They were then degreased with acetone and etched with nital solution (a mixture of 5% nitric acid and ethanol). The steel microstructures were characterised using an optical microscope (OM). Prior to microstructural characterisation, the distributions of the non-metallic inclusions on the non-etched clean steel surfaces of the ground specimens were observed with a scanning electron microscope (SEM).

#### 2.2. H<sub>2</sub>S corrosion test

Corrosion tests were carried out in a H<sub>2</sub>S corrosion experimental device following the general NACE TM0284-2003 standard [24] (Fig. 2). Before the experiments, the sealed test vessel was purged of air with nitrogen for at least 1 h. Purging began immediately after the test vessel was sealed at a rate of at least 100 ml per minute, per litre of test solution. After purging, H<sub>2</sub>S gas was bubbled through the test solution with a bubbling rate of at least 200 ml per minute, per litre of test solution for the first 60 min. Thereafter, a positive pressure of H<sub>2</sub>S gas was maintained by controlling the flowmeter. The test solution consisted of 5.0 wt% sodium chloride and 0.5 wt% glacial acetic acid dissolved in distilled water, and the pH was adjusted to the desired level through the addition of hydrochloric acid. The conditions of the corrosion experiments are given in Table 2.

#### 2.3. Fracture test

After corrosion testing, tensile tests for some samples were performed immediately on an MTS test machine (MTS810, MTS Corporation, USA) at room temperature (25 °C) in accordance with GB/T 228-2002 (Metallic materials-Tensile testing at ambient temperature). Other samples were subjected to hydrogen concentration measurements prior to tensile testing. The tensile rate was 4 mm/min. The index of relative susceptibilities to hydrogen damage of the various specimens in different corrosion environments was determined by measuring the relative tensile strength loss  $I_{\sigma}$  and the relative plasticity loss  $I_{\delta}$ , which can be expressed as:

$$I_{\sigma}(\%) = \frac{\sigma_0 - \sigma_{\rm H}}{\sigma_0} \times 100\% \tag{1}$$

$$I_{\delta}(\%) = \frac{\delta_0 - \delta_{\rm H}}{\delta_0} \times 100\% \tag{2}$$

where  $\sigma_0$  and  $\delta_0$  are the ultimate tensile strength and elongation to failure in the as-received state (without corrosion), respectively; and  $\sigma_H$  and  $\delta_H$  are the ultimate tensile strength and elongation to failure after corrosion or after hydrogen release, respectively. After performing the tensile tests, the fracture surfaces of the specimens were subjected to a detailed analysis by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS).

#### 2.4. Diffusible hydrogen concentration measurement tests

Diffusible hydrogen concentration in the specimens was measured by the modified JIS Z3113 method [25]. After corrosion, the specimens were removed from the corrosion experimental device, dried and then immediately immersed into a glass tube filled with silicone oil. This transition time was kept to less than 2 min. After releasing all the diffusible hydrogen from the specimens for two days, the total diffusible hydrogen amount was determined from the scale reading of the glass tube. The diffusible hydrogen concentration was calculated according to the following equation [26]:

$$C_0 = \frac{2.44 \times 10^{-8} V}{mT}$$
(3)

where  $C_0$  (wppm) is the diffusible hydrogen concentration, V (ml) is the hydrogen volume released from the specimen at room temperature, m (g) is the weight of the specimen; and T (K) is the temperature.

#### 3. Results

#### 3.1. Microstructure and inclusions

Fig. 3 shows the optical microstructures of the A350LF2 steels. A350 I steel possesses mainly acicular and massive ferrite with an average grain size of  $12-20 \mu m$  (Fig. 3a). In the same figure, some bainite and pearlite patches can also be seen. The microstructure of A350 II steel is very similar to that of A350 I steel except for the average grain size; the average grain size of acicular and massive ferrite in A350 II steel is approximately  $5-12 \mu m$  (Fig. 3b). In addition, SEM observations of the tested steels revealed the characteristics of the inclusions, as shown in Fig. 4.

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