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## Effect of hydrogen on fracture toughness properties of a pipeline steel under simulated sour service conditions

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#### ABSTRACT

The effect of hydrogen on the fracture toughness properties of an API X65 pipeline steel is studied under simulated H<sub>2</sub>S in-service conditions. The fracture toughness properties are measured in LT and SL directions (perpendicular and parallel to the pipeline wall thickness, respectively), following ASTM E1820. Due to size restrictions of standard single edge notch bending (SEB) specimens at the direction parallel to the thickness of the pipeline wall, an experimental protocol (see the patent) was developed to carry out the fracture toughness tests, while complying with ASTM standard 1820. This approach is especially useful in situations where hydrogen induced cracking (HIC) and in a broader sense, stepwise cracking takes place, since these cracks initiate and grow primarily in planes parallel to the pipeline rolling plane. Such values of fracture toughness are often different from those commonly measured in planes perpendicular to the rolling plane. Hydrogen might not have the same effect on fracture toughness properties as measured in different directions, due to microstructural features which are inherent from steel manufacturing process. The steady state H<sub>2</sub>S in-service conditions are simulated by electrolytically charging the specimen, for 48 h and then testing (ex-situ) the specimen for evaluating the fracture toughness properties. The steady state H<sub>2</sub>S environment charging was obtained by measuring the hydrogen concentration in the bulk of the specimen through thermal desorption spectroscopy (TDS) at three levels of hydrogen concentration. It was observed that the Ko was moderately decreased with increasing hydrogen concentration in the bulk of the steel, while  $CTOD_0$  showed a significant reduction with increasing hydrogen concentration.

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#### Introduction

The presence of wet H<sub>2</sub>S (sour environment) in hydrocarbons can cause failures in oil carrying pipeline steels, due to the formation of hydrogen induced cracking (HIC) defects [1-3]. These defects are attributed to the atomic hydrogen, produced by the sour corrosion, which in turn enters the bulk of the steel and reacts to form high pressure hydrogen cavities at the interface of nonmetallic inclusions residing in the microstructure and known to be preferential sites of crack initiation. Equilibrium at the surface of the cavity between lattice atomic hydrogen at the surface and molecular hydrogen gas in the cavity is governed by the Sievert's law, which is the main mechanism for hydrogen pressure build-up in the cavity [4]. It was well documented that the steel microstructure [5,6] in addition to the shape, the size and the distribution of nonmetallic inclusions [7] are the main governing factors for the development of internal pressuring hydrogen cavities [8-10]. Advances in steel manufacturing have introduced new steels that are resistant to HIC and in this sense, relevant standards are used to evaluate the HIC resistance of pipeline steels, such as NACE TM0284 [11]. Many works had also demonstrated this fact in the past, such as [12-16]. Some works were focused on studying the effect of hydrogen on tensile properties, ductility and reduction of area, by performing tensile tests on specimens in H<sub>2</sub>S relevant environments [12,14–19], or in acidic environments with the addition of recombination poisons [20-23] or even using carbonatebicarbonate solutions [24]. While other works were realized using impact tests in H<sub>2</sub>S relevant environments [14,15]. Loss of plasticity and reduction of tensile strength are the main effects of hydrogen, which many times increases with increasing hydrogen concentration [14,20], or increasing current density [20,21]. However, most assessment tools [25–30] and standards for pipeline integrity use fracture toughness properties in their evaluation schemes. Relevant fracture toughness data are also critical to the accuracy of finite element predictions of HIC growth rates, as highlighted in Refs. [31-33]. In this sense researchers have performed fracture toughness (FT) tests in order to assess the effect of hydrogen on FT properties [34-36] in order to measure and correlate the hydrogen concentration (CH) in the area around the notch and the crack tip with the FT properties [35,36]. Regardless of CH measured, all of the aforementioned works [34-36] have shown clear reduction of FT properties due to the effect of hydrogen. It is worth pointing out that in the aforementioned works, the FT properties are evaluated with the notch and the crack in the LS direction [34,35] and/or the TL direction [36]. Following this rationale defects which initiate and propagate parallel to the pipeline rolling plane, such as HIC (Hydrogen Induced Cracks) should involve the evaluation of the FT properties in air and in hydrogen relevant environments in the aforementioned direction, i.e. the SL direction. In general, when assessing fracture toughness properties in pipeline steels the properties are evaluated in the directions perpendicular to the pipeline rolling plane, either in directions TL, LT or TS, LS. It is very difficult to evaluate fracture

toughness properties in the SL and ST directions due to the fact that single edge bending (SEB) or compact tension (CT) specimens, following ASTM E1820 [37], cannot be extracted due to restricted pipeline wall thickness. However, it has been shown that fracture toughness properties in thermo mechanical control processed (TMCP) manufactured steels, which is the current norm for pipeline steels, can differ in value for different directions [38,39]. Since HIC damage is developing in cracks parallel to the pipeline rolling plane, it is very interesting to ascertain the fracture toughness properties in the SL direction in inert and in hydrogen related environment. In particular, such data is critically needed to carry out proper engineering critical assessment (ECA) of HIC defects in pipelines. Furthermore, it also observed that in some of the previous works [12,14-16] the measured CH involves the glycerin method [40], the hydrogen evolution method [23,35] or an electrochemical permeation technique [20,23,36]. These methods measure only the diffusible hydrogen, which resides in the reversible trapping sites of the steel. In this study, the CH is measured and evaluated using the TDS (Thermal Desorption Spectroscopy) method, which allows for measuring all of the hydrogen residing in reversible and irreversible trapping sites.

The aim of this study is to assess the effect of crack growth direction, i.e. TL vs. SL, on the fracture toughness of API X65 pipeline steel in air and in hydrogen environment. In particular, the in-service conditions of wet  $H_2S$ , will be simulated by cathodic hydrogen charging of the X65 steel specimens. These conditions represent the steady state hydrogen content in the bulk of the steel after the specimen has been charged with hydrogen and a steady hydrogen concentration has been achieved. In turn, these conditions are correlated with the variation of the fracture toughness properties of the tested API X65 steel.

#### **Experimental procedure**

#### Test sample orientations

The evaluation of the fracture toughness properties  $K_{\rm IH}$  and  $CTOD_0$  in air and under three levels of hydrogen concentrations were performed in two different directions, namely perpendicular to the pipeline rolling plane, (TL direction) and parallel to the pipeline rolling plane (SL direction). The reference directions of notch and crack TL and SL, follow the ASTM E399 standard [41].

#### Material: API X65

API X65 microstructure was analyzed using scanning electron microscope (SEM) Quanta3D FEI<sup>™</sup> and Electron Back Scattered Diffraction (EBSD) from EDAX<sup>™</sup>. Fig. 1 shows the typical microstructure of X65 samples, used in this work. We observe the presence of both phases ferrite and bainite (Fig. 1 (a), (b), (c)). EBSD map shows elongated grains and no texture is observed as the microstructure appears homogeneous. An

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