Pipelines offer the most efficient way to transport bulk quantities of gas and petrol from points of production to storage locations or from storage locations to distributed points of end use. Environment such as hydrogen, load frequency and temperature affects fatigue crack growth (FCG) behavior, which look like other forms of corrosion-fatigue. Discussion of the mechanism(s) for FCG in pipeline steels is complicated by the fact that neither the mechanism for FCG, nor the mechanism for hydrogen embrittlement (HE) under monotonic loading in these materials, is completely understood. There are only a few observations that provide indirect evidence concerning the mechanism for heat affected (HA) fatigue crack growth. In presence of high H2S the risk of brittle failure can be increased due to the effect of hydrogen on steel toughness. In this paper the impudence of environments such as, hydrogen, load frequency and low temperature on mechanical properties of two micro-alloyed pipeline materials, API 5L X70 and X80, was studied. The results conducted based on experiments, the mechanisms used to predict FCG rate in hydrogen may differ significantly from those used for statically loaded applications or in fatigue situations. To investigate the effect of hydrogen, load frequency and temperature on pipeline steels, toughness and crack propagation tests of charged and uncharged specimens. An electrochemical hydrogen charging method has been setup. A set of experiments were carried out, by varying the test temperature and the frequency of the load application cycle. The experimental results showed an evident effect of the hydrogen presence on the FCG. The results showed also, that in pipeline applications are susceptible to significantly increased FCG rates in high-pressure hydrogen environments and low temperature as well as low load frequency. In order to operate safe and reliable pipeline networks, a better understanding of the mechanisms responsible for the HA-FCG is required, and more empirical data for a range of pipeline steels and test parameters must be collected.

**Keywords:** Heat Affected, Hydrogen embrittlement, Low temperature, Fatigue crack propagation, Pipeline steels

**INTRODUCTION**

It is an economical way to transport oil and gas through long distance by using pipeline with high pressure and large diameter. In this case, it is usually required the high-grade, high performance pipeline steel to guarantee a safe and...
reliable service at such high pressure, as well as to reduce the cost of construction and operation for a pipeline project. Now X70 pipeline steel has become the most used steel grade in long distance pipeline projects over the world. The projects based on X80 pipeline have been constructed since 1985, which have being operated in safe up to now (Gao Shan and Zheng Lei, 2002). In order to guarantee the safety of the large diameter and high operating pressure, high-performance pipeline steel with high strength, high toughness and ductile fracture propagating arresting, as well as good weld ability.

The study of the mechanical characteristics of the steels, under the influence of hydrogen, is an essential research area due to the importance of these materials for mechanical design of infrastructures, such as pipelines and pressure vessels, in oil and gas industry, in chemical and petrochemical plants or for hydrogen production and transportation. For these reasons, unusual temperatures and environments require the designing of new materials for better operative performances.

One of the most challenging issues, in pipelines design, concerns corrosion and HE of metallic materials, which can lead to disastrous consequences for the environment, the economy and also for the personal health. The effect of hydrogen and low temperature on the fatigue crack propagation is a very important task (Murakami Y and Matsuoka S, 2010; Kanezaki T et al., 2008; Murakami Y, 2002), it depends on materials and microstructures.

Carbon and low alloy steels are commonly used, in oil and gas industry, when general corrosion, due to the presence of H₂S and CO₂, is considered acceptable to stand the design life. However, when sour condition occurs, the onset of Sulphide Stress Cracking (SSC) in the presence of H₂S on susceptible materials must be investigated (EFC, 2002). Furthermore, when the temperatures are very low, as in arctic environment, and are combined with sour conditions, synergistic effects may result on the mechanical characteristics of the used materials.

The acceleration of crack propagation rates in hydrogen environment was found to be more pronounced at high $\Delta K$ (Holbrook J H, 1982; Fukuyama S and Yokogawa K, 1992). This can be seen based on the review by (Somerday B P, 2008). Similar observations were also reported in the tests carried out at R= 0.05 on the specimens pre-exposed to hydrogen environment (Dauskardt, 1986 and Pendse, 1985). They attributed this behavior to the mutually offsetting effects of environmental damage and a local reduction in crack driving force from a crack closure related mechanism, resulting in crack growth rates in low $\Delta K$ ranges and $\Delta K_{th}$ insensitive to hydrogen environment. However, in the work carried out by (Suresh and Ritchie R O, 1982), the FCG rates in dry hydrogen were found to be accelerated not only in the high $\Delta K$ regime, but also near the threshold. They carried out FCG tests in both air and hydrogen environment.

A stronger effect of loading frequency on FCG rates was reported by (Walter R J and Chandler W T, 1976) in SA 105 steel and by (Lindley et al., 1979) in 1%CrMo steel. The FCG rate increased by a factor of ~5 as frequency decreased from 1 to 0.001Hz (Walter R J and Chandler W T, 1976) and about one order as frequency decreased from 10Hz to 0.1Hz (Lindley et al., 1979) in the high $\Delta K$ regime.

The presence of hydrogen has a strong effect also on the fatigue behavior of carbon and low alloy steels and there are several studies on this topic carried out using fracture mechanic approach, i.e., representing the data in terms of $da/dN$ vs. $\Delta K$ curves. These studies are mainly
devoted to measure the fatigue properties of metals and welded joints in different environments, such as seawater (Havn T and Osvoll H, 2002), sweet (CO2) or sour (H2S) condensates (Holtam CM et al., 2010), boiling or pressurized water in nuclear plants (Yan Hui Zhang, 2010), gaseous hydrogen at high pressure (Cotterill PJ and King J E, 1991; Chuang J H et al., 1998; Tsay Y L W et al., 2001). In other cases specimens have been charged with hydrogen using different techniques before fatigue testing in air (Tsay Y L W et al., 2001; Darcis P et al., 2008; Murakami Y, 2006; Tsuchida Y, 2010). A review of the results of fatigue tests in environments of the interest of offshore oil production is given by (Woollin et al., 2004).

Stewart Fassina P, (2011) investigated the influence of temperature on the crack growth behavior of a pressure vessel steel in hydrogen environment and his result was opposite to the above observation. Furthermore, when very low temperatures, e.g., below -40°C, are also present, as in most recent oil and gas fields, a synergistic negative effect may result from the combination of sour conditions and low temperatures on the mechanical behavior of the used materials.

Common characteristic of the above-mentioned environments is the possibility of generating atomic hydrogen which can enter into the metallic material causing embrittlement. This is also demonstrated by fractographic examinations, as a matter of fact many authors have evidenced some features typical of brittle fracture on the fatigue specimen surface (Stewart A T, 1977).

To develop hydrogen energy, it is important to understand the effects of hydrogen on mechanical properties in all applications, production, transportation, storage and fuel cells. Although a considerable amount of work has been carried out on hydrogen related embrittlement, there is comparatively less work on the effect of a hydrogen environment on fatigue performance of steels. It is essential to carry out comprehensive and coordinated research to understand how a component is affected when exposed to a hydrogen environment, how to prevent or minimize the failure probability, and finally to gather critical data to develop design guidance and government regulations to ensure safe operation of infrastructures involving hydrogen environment. (Fuquent-Molano and Ritchie, 1981) investigated the effect of temperature on FCG rates in both air and hydrogen.

Although API 941, (1997) provides recommendations for the allowable maximum hydrogen pressure for a certain elevated temperature. However, no guidance about the effect of temperature on fatigue properties of steels in hydrogen environment is provided in that document. In fact, work on the effect of temperature on FCG is very limited.

To investigate the effect of hydrogen and low temperature on pipeline steels, toughness and crack propagation tests of charged and uncharged specimens were carried out. Toughness tests were carried out as described previously (Havn T and Osvoll H, 2002; Fuquent-Molano and Ritchie, 1981). An electrochemical hydrogen charging method, has been setup, avoiding any critical conditions from the point of view of preparation, safety and disposal. The content of hydrogen was controlled and measured in the specimens.

In order to evaluate these combined effects, the author of the present work, investigated the Charpy and toughness behavior of two microalloyed steels (API 5L X70 and X80) used in oil and gas pipelines, in presence of hydrogen and low temperature. An electrochemical method is prepared for this purpose, which allows to charge with hydrogen in a controlled way.
METHODOLOGY

The (ASTM E647-08, 2012). Standard allows any test specimen configuration to be used, provided that well-established stress intensity correction factor calibrations and that the specimens are of sufficient planar size to remain predominantly elastic during testing. The type of test piece used in this application to conduct the experimental testing are the Single Edge Notched Beam (SENB), which is loaded by the Three-Point Bending (TPB). These specimens were designed according to the ASTM standard E647. The SENB specimen is more flexible in terms of its size, whereas its span can be continuously adjusted to the value that is within its capacity. Thus, the SENB specimens (Figure 1) with a wide range of thickness can be tested using a single fixture. Besides this, the SENB configurations are preferable because of ease of fabrication. The tests also fulfill all the requirements of the ASTM Standard.

The chemical compositions of the selected materials (X70 and X80) and mechanical properties have been evaluated at room temperature and reported in Tables 1 and 2 respectively (Fassina et al, 2011).

A Crack Opening Displacement (COD) technique (Figure 1) was used to record the crack length with the running experiment. The COD parameter, in conjunction to the crack surface analysis, represents a precise technical to evaluation of the influence of micro structural variables in the mechanical behavior of metallic materials. Another factor enhancing the importance of COD is that, nowadays, this parameter is one of the most important for materials selection, which will be submitted to severe environment working conditions.

Table 1: Average Values in Weights % of Main Elements and Chemical Parameters of Pipelines

<table>
<thead>
<tr>
<th>Material</th>
<th>Yield Strength (MPa)</th>
<th>Ultimate Strength (MPa)</th>
<th>Yield/Ultimate Strength</th>
<th>% Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>X70</td>
<td>510</td>
<td>670</td>
<td>0.76</td>
<td>46.5</td>
</tr>
<tr>
<td>X80</td>
<td>630</td>
<td>725</td>
<td>0.87</td>
<td>48.0</td>
</tr>
</tbody>
</table>

Table 2: Average Mechanical Properties of Steel Pipeline Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Mn</th>
<th>Ca</th>
<th>S</th>
<th>P</th>
<th>Mo</th>
<th>Nb+Ti+V</th>
</tr>
</thead>
<tbody>
<tr>
<td>X70</td>
<td>0.09</td>
<td>1.72</td>
<td>0.0022</td>
<td>0.001</td>
<td>0.016</td>
<td>0.006</td>
<td>0.12</td>
</tr>
<tr>
<td>X80</td>
<td>0.16</td>
<td>1.90</td>
<td>0.0022</td>
<td>0.003</td>
<td>0.020</td>
<td>0.006</td>
<td>0.15</td>
</tr>
</tbody>
</table>

The fatigue tests described in the present work are carried out on TPB specimens after hydrogen charging. So the two experimental test phases (hydrogen charging and mechanical testing) are performed in different time periods. While fatigue tests carried out in simulated operating environments guarantee a continuous hydrogen charging of the crack tip during the test. The experimental procedure adopted in our tests allows to evaluate the effect of hydrogen alone on the fatigue
behaviour of the materials and to make careful observations of the fatigue crack, without the disturbance of the effects due to corrosion, such as crack tip blunting, wedging/closure caused by corrosion products and corrosion of the crack surface. Moreover it has also been possible to test materials at very low temperatures, well below the freezing point of water solutions, this experimental apparatus are shown in Figure 2. To conduct fatigue tests in gaseous hydrogen environment, the facilities required include: a servo-hydraulic mechanical testing machine, a chamber containing hydrogen pressure, a crack growth monitoring system for crack growth test, a temperature control system and safety devices. An example showing the set-up for such a test is given in BS EN ISO 11114-4 (ASTM E 647, 2012).

A large number of experimental tests were carried out to characterize the mechanical behaviour and fatigue crack propagation of the uncharged and hydrogen charged steels. TPB specimens of X70 and X80 steels were manufactured based on ASTM 647, with thickness $B=20\text{mm}$. The specimens were loaded by a 100 kN MTS servo-hydraulic loading frame. All tests were carried out accordingly to ASTM 647 and in load control, the cyclic load was applied as a sinusoidal wave with ratio $R=0.1$. Measurement of crack growth was made through the compliance method. The un-charged specimens were fatigue tested by varying the test temperature, in particular the following temperatures were considered: Room Temperature (RT=23°C) and $T=-30°C$.

**RESULTS AND DISCUSSION**

The detrimental effect of gaseous hydrogen on FCG rates depend on many factors such as $\Delta K$ magnitude, hydrogen pressure, loading frequency, stress ratio, temperature, gas composition, microstructure etc. The effect of hydrogen gas is more pronounced on FCG rates than fatigue endurance of steels. The latter hardly exhibits the detrimental effect in the long life regime (>10⁶ cycles) in smooth specimens. The results can be explained considering that the crack growth rate is due to the addition of two contributions: a frequency dependent phenomenon i.e., the load effect and a time dependent one, i.e., the hydrogen effect. Because the hydrogen effect is more severe on crack growth rate and it is difficult to rule out the presence of defects in components, the new ASME Code rules for boiler and pressure vessels recommend using the fracture mechanics approach for designing hydrogen infrastructures in high pressure hydrogen (Rana M D et al., 2007). The new rules require determining the fatigue crack growth rates and fracture resistance properties of the materials to be used in the construction of pressure vessels in high pressure hydrogen gas.

Hydrogen charged specimens were heated from liquid nitrogen to the test temperature as fast as possible by an ethanol bath kept at the test temperature, in order to minimize the loss of hydrogen from the material before fatigue testing.

The effect of hydrogen is evident both in X70 and X80 specimens. The hydrogen charge causes, in fact, embrittlement and crack growth acceleration in all the considered conditions. Figures 3 (a and b) show the crack length with num-

![Figure 2: COD Experimental Apparatus](image)
number of cycles curve obtained for X70 and X80 respectively, by the uncharged specimens at two different temperatures. In the same Figures the crack length verses life cycles curves for the hydrogenated specimens were presented at $T = 23$ and $-30^\circ C$. The number of life cycles for uncharged X70 steels (Figure 3a) are $4.6 \times 10^5$ and $6.4 \times 10^5$ cycles under the 23 and $-30^\circ C$ respectively. For the charged case the reduction in life cycles are 16% and 25% for the two temperatures cases (RT and $-30^\circ C$).

For the case of X80 steel (Figure 3b), it is so clear that the life of uncharged specimens are 48% greater at low temperature than RT and for charged are 18 to 27% lower than the charged one. The load frequency for these cases are 20 Hz. The number of cycles for the charged specimens are $1.2 \times 10^6$ and $2.5 \times 10^5$ under RT and $-30^\circ C$ respectively.

If these comparison between X70 and X80, these factors (hydrogen and temperature) can give different values, so for uncharged; the life of X80 are 60% cycle more than of the X70, while for the effect of temperature the life of X80 is 30% cycles more than that X70. At $T = -30^\circ C$ the hydrogen influence on crack growth rate is reduced, if compared with tests carried out at room temperature.

**Figure 3: Fatigue Life at Different Temperatures for Steel Pipelines (a) X70 and (b) X80**

![Fatigue Life at Different Temperatures for Steel Pipelines](image)

Figures 4 (a and b) show the crack length as a function of number of life cycles. Figure 4a shows the fatigue life results in X70 for the hydrogen charged specimens, at RT and $T=-30^\circ C$, by varying the load frequency. In particular the considered frequencies are: $f = 1$ Hz and $f = 10$ Hz.

It is evident the effect of the hydrogen and load frequency on the crack rate, the number of loading cycles to failure of a hydrogen charged X70 specimen, tested at RT and $f = 1$ Hz, is equal to cycles, the 14000 cycles, the corresponding value for a 10 Hz and 20 Hz are 45000 and 75000 cycles respectively.

At low temperature (-$30^\circ C$), the effect of hydrogen is lower with respect to the case at RT ($23^\circ C$), for tests carried out on charged specimens. The number of cycles to failure under 10 Hz load frequency is twice of that case of 1 Hz, while for the load frequency 20 Hz, it was a multiple of a factor ~5. At $T = -30^\circ C$ and $f = 10$ Hz, it was observed that the crack growth rate approaches the uncharged curve one at load frequency 20 Hz. The curve at $T = -30^\circ C$ and $f = 1$ Hz shows a trend similar to the one of the uncharged specimens, on the contrary, the crack rate is higher with respect to the rate without hydrogen.
Figure 4b shows the case study of X80 steels under the same environment of charged specimens with different load frequencies and temperatures. The number of cycles at 10 Hz is twice that at 1 Hz, while at 20 Hz is five times that of 1 Hz.

At high frequency the crack propagation rate per loading cycle, due to the load effect, is high and the time per cycle (period) is low, therefore hydrogen has no enough time to diffuse toward the fatigue crack tip and the hydrogen contribution to the crack propagation is low. On the contrary, at low frequency hydrogen can diffuse in a large quantity to the plastic zone ahead of the crack tip contributing to a large amount to the crack tip embrittlement.

Figures 5 (a and b) show the curves $\frac{da}{dN}$ as a function of $\Delta K$: these curves are very similar, with the same slope ($m \approx 2.99$) and slightly differing in the C values. Propagation tests on uncharged specimens were carried out at a load frequency ($f$) equal to 20 Hz. In these conditions, as widely shown in literature, frequency is not a parameter affecting growth rate. It should be pointed out that variations between crack growth rate at different temperatures are very small and that crack growth rate, at same $\Delta K$, decrease while decreasing temperature, as expected. Indeed, cyclic plastic zone is reduced while decreasing temperature because yield strength is increased, at the same time.

Figure 5 show also that, the crack growth rate vs. stress intensity factor range curves are very well reproducible and not scattered. Fatigue behavior of steel in the “as received” (uncharged) conditions follows the Paris’ law; the curves, in fact, have the same slope ($m \approx 3.9$) and the C parameter values slightly decreasing with temperature. This behavior can be explained taking into account that the plastic zone at the crack tip at the same applied $K$ value is reduced when the temperature decreases, because the yield strength increases. The decrease of the slope is controlled by the size of the cyclic plastic zone. When, in fact, the cyclic plastic zone reaches a certain length, comparable with the grain size, new slip planes and grain boundaries take part to the process, constraining the movement of the dislocations. For this reason, there is a reduction of Paris exponent, which means an increased strength of the material and a higher constrain to dislocation movements.
At the same frequency and $\Delta K$ value, the fatigue crack propagation rate is always higher at RT than at $T=-30^\circ C$, i.e., the crack rate increases when the temperature increases, and fatigue crack propagation rate very much increases when the frequency decreases and in our tests there is approximately factor of four for X70 steels and three for X80 in the crack growth rates at 1 and 10 Hz.

The fatigue behavior is mainly controlled by the mechanical properties of the material (as if it were hydrogen free) since there is not enough time for hydrogen to reach the crack tip because of the high crack growth rate. Again by observing the previous Figures, the curves of hydrogen charged specimen, at $f=1$ Hz and RT shows a strong enhancement of the embrittlement effect because of the long load application times and temperature that facilitate hydrogen diffusion at crack tip.

Fatigue crack growth rates in hydrogen gas generally increase as the load cycle frequency decreases. This trend is illustrated in Figure 6, which displays $da/dN$ vs $\Delta K$ relationships for SA 105 steel in 100 MPa hydrogen gas over a range of load cycle frequencies from 0.001 to 1 Hz (Yan Hui Zhang, 2010). As frequency decreases from 1 to 0.001 Hz, the crack growth rate increases by about a factor of five. Fatigue crack growth rate data in air, nitrogen, or helium are included for comparison.

When the hydrogen effect is predominant the crack growth rate, measured as $da/dN$, is somehow strongly dependent on the cycle period ($=1/f$). It is interesting to observe that in our experimental conditions the effect of hydrogen can be lower than the one measured by (Murakami Y and Matsuoka S, 2010) who have found a maximum increasing between charged and uncharged specimens. Control over these variables may allow carbon steels to be applied safely in hydrogen gas environments. For example, limiting the magnitude and frequency of load cycling can improve the compatibility of carbon steels with hydrogen gas.
CONCLUSION

Crack growth rate is the sum of two contributions: one named “mechanical” that depends on applied loads and a second due to environment effect (hydrogen and temperature). When crack growth rate increases, the “mechanical” contribution prevails because hydrogen atoms do not have enough time to accumulate at the crack tip: as a consequence crack growth rate is no longer hydrogen dependent.

The influence of hydrogen, load frequency and temperature on the fatigue properties of two micro-alloyed steel pipeline materials API 5L X70 and X80 steel pipes was investigated by fatigue crack propagation tests. Hydrogen effect was clearly observed and influenced by temperature and load frequency: low temperature reduce the mobility of hydrogen in the lattice (i.e. reduce diffusion coefficient), reducing the embrittlement effect; low load frequencies allow the hydrogen to migrate at the crack tip, as a consequence hydrogen embrittlement effect on crack growth rate are enhanced.

In the presence of hydrogen, the crack growth rate increases significantly respect to uncharged specimens.

To improve fatigue performance of steels in hydrogen, it is important to have project manage-


of the Inter. Conf. on the Mechanisms of Environment Sensitive Cracking in Materials, University of Surry, 4-7, pp.400-411.


