

Effect of the Strain Rate on the Fracture Behaviour of High Pressure Pre-Charged Samples [†]

Guillermo Álvarez Díaz ^{1,*}, Tomás Eduardo García Suárez ², Cristina. Rodríguez González ¹ and Francisco Javier Belzunce Varela ¹

¹ SIMUMECAMAT Research Group, Edificio Departamental Oeste, University of Oviedo, 7.1.17. Campus Universitario, 33203 Gijón, Asturias, Spain; cristina@uniovi.es (C.R.G.); belzunce@uniovi.es (F.J.B.V.)

² Know-How Innovative Solutions S.L. Espacio Tecnológico Campus, C/Pedro Puig Adam, S/N, 33203 Gijón, Spain; garciatomas@khisgroup.com

* Correspondence: uo224369@uniovi.es; Tel.: +34-659-730-451

[†] Presented at the 2nd International Research Conference on Sustainable Energy, Engineering, Materials and Environment (IRCSEEME), Mieres, Spain, 25–27 September 2018.

Published: 11 December 2018

Abstract: The aim of this work is to study the effect of the displacement rate on the hydrogen embrittlement of two different structural steels grades used in energetic applications. With this purpose, samples were pre-charged with gaseous hydrogen at 19.5 MPa and 450 °C for 21 h. Then, fracture tests of the pre-charged specimens were performed, using different displacement rates. It is showed that the lower is the displacement rate and the largest is the steel strength, the strongest is the reduction of the fracture toughness due to the presence of internal hydrogen.

Keywords: hydrogen embrittlement; fracture toughness; hydrogen embrittlement; displacement rate

1. Introduction

Hydrogen induced cracking is a phenomenon of degradation of the mechanical properties of metallic alloys [1–3] when they are exposed to aggressive hydrogen atmospheres during their in-service life, such as vessels or pipes in the power industry, off-shore platforms and hydrogen powered vehicles [4]. It is known that the hydrogen embrittlement affects structural steels in a different way dependent to their microstructure and strength level.

Several methodologies have been used for decades to analyse hydrogen embrittlement. The most common technique is the slow strain rate tension test (SSRT) [5]. The results of these tests are usually represented as an embrittlement index (EI%), which is related to the reduction of area or to the strength of the specimens tested with and without hydrogen. Submerged fracture toughness tests were also used to analyse this phenomenon, but test conditions are complex due of the aggressive environment. In addition, submerged tests could not represent the real in-service conditions of the component and it is also difficult to know the amount of hydrogen introduced in the material. Thus, in the present work, fracture toughness tests in air at room temperature (RT) using hydrogen pre-charged specimens of two different steels have been performed. The effect of the test displacement rate was also analysed, and the results were justified in base of the steel microstructure.

2. Materials and Methods

2.1. Materials

Two different steels have been used in this study. First, a structural S355 steel, with a ferritic-pearlitic microstructure. The second steel was a quenched and tempered high strength low alloyed

steel, H8, with a tempered martensitic microstructure. The tensile properties at RT of both steels are showed in Table 1.

Table 1. Tensile properties of the steels.

Steels	E (MPa)	σ_y (MPa)	σ_u (MPa)
S355	210,000	386	542
H8	226,000	792	857

2.2. Fracture Toughness Tests

Fracture toughness tests were carried out using SE(B) specimens (Figure 1a) with the following dimensions: $17.5 \times 10 \times 80 \text{ mm}^3$ ($W \times B \times L$). In agreement of the ASTM E1820 standard, specimens were fatigue pre-cracked ($R = 0.1$) until a crack length-to-width relation $a/W \cong 0.5$ and were subsequently side-grooved to obtain a final net-thickness, B_N , of $7.3 \pm 0.05 \text{ mm}$.

After that, some samples were pre-charged with gaseous hydrogen in a high pressure reactor at 19.5 MPa of pressure and 450 °C of temperature during 21 h. After pre-charging, samples were stored in liquid nitrogen, to avoid hydrogen desorption, until the start of the fracture test.

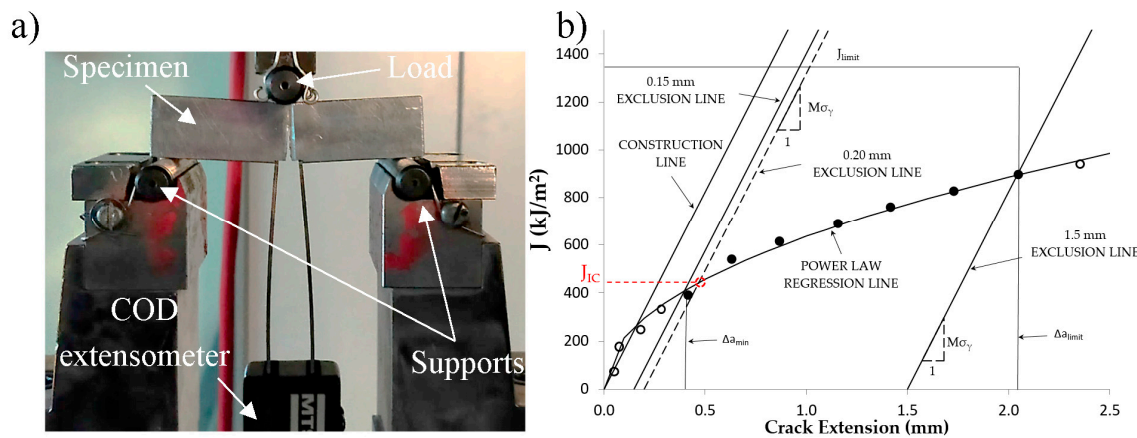


Figure 1. Fracture toughness tests. (a) Loading fixture, (b) J-R curve (without hydrogen).

The single-specimen method, based on the elastic unloading compliance technique was used to determine the J - Δa (J - R) curves of both steels under standard laboratory conditions. Fracture toughness values, J_{IC} , in accordance with the ASTM E1820 standard, were calculated from these curves after a power regression line fitting procedure (Figure 1b). In addition to the standard test displacement rate ($v = 0.1 \text{ mm/min}$), pre-charged specimens were also tested using lower displacement rates (0.01 and 0.001 mm/min). Once the different values of fracture toughness were obtained, the EI(%) embrittlement index was used to quantify hydrogen embrittlement:

$$EI(\%) = \frac{J_{IC} - J_{ICH}(v)}{J_{IC}} \quad (1)$$

where J_{IC} is the fracture toughness of the material without hydrogen and $J_{ICH}(v)$ is the fracture toughness measured with the hydrogen pre-charged samples tested at different displacement rates (v).

3. Results and Discussion

Figure 2 shows the J - R curves obtained under the different test conditions with the two steels. Fracture toughness results for standard (J_{IC}) and pre-charged (J_{ICH}) samples are shown in Table 2. This table also shows the J - R curves power law coefficients and the embrittlement indexes in each tested condition.

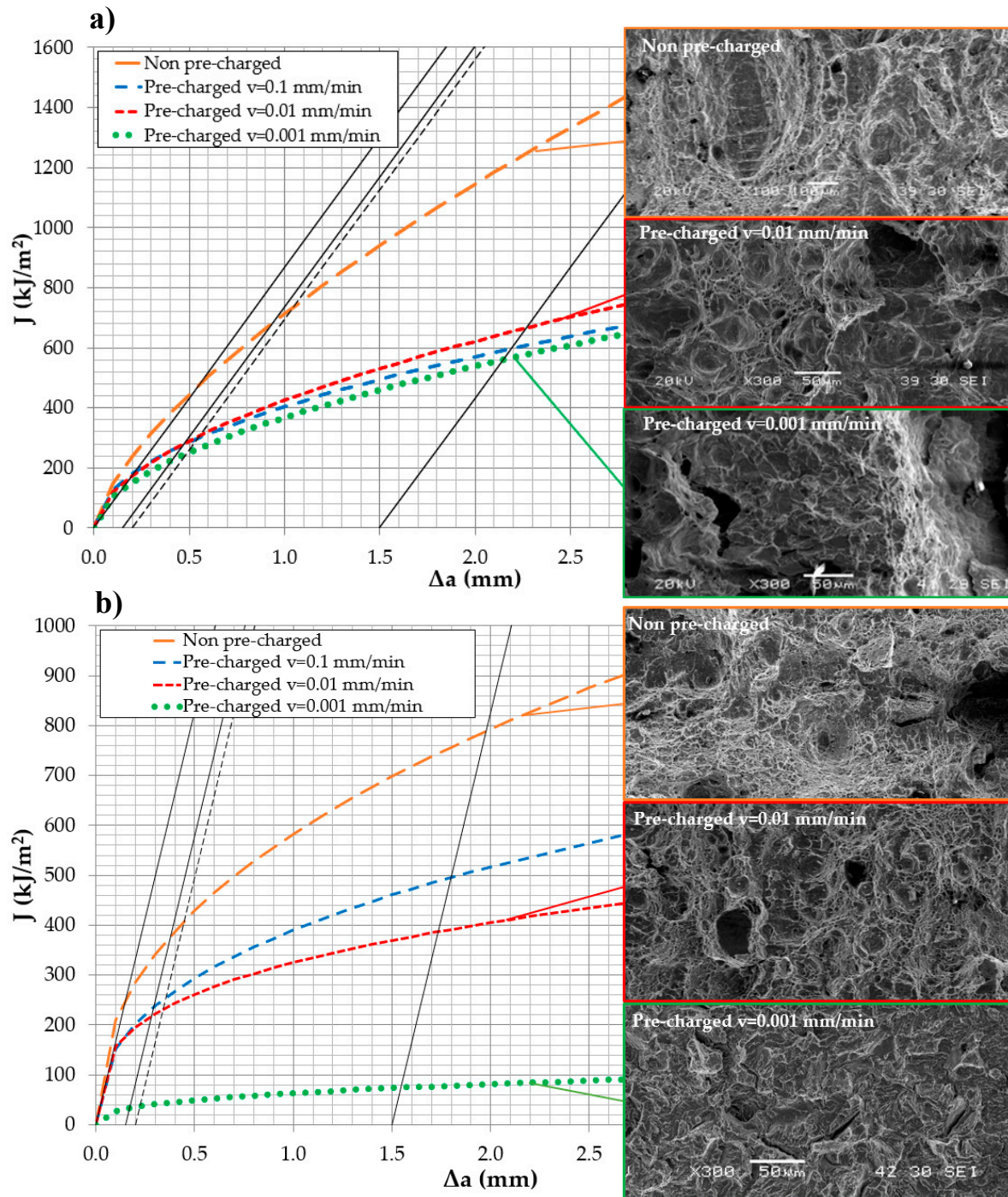


Figure 2. J-R curves and fracture surfaces (a) S355 (b) H8.

Table 2. Fracture toughness results and embrittlement indexes.

Displ. Rate (mm/min)	J _{IC} (kJ/m²)		J _{ICH} (kJ/m²)		EI(%)		
	0.1	0.1	0.01	0.001	0.1	0.01	0.001
S355	750	297	306	247	60	59	67
H8	450	260	230	40	42	49	91

It is worth noting that hydrogen produces a strong decrease in the fracture toughness in both steels, attaining embrittlement indexes between 40 and 90% for the different steels and displacement rates. Furthermore, hydrogen does not only influence the fracture toughness value at the onset of crack growth (J_{IC}), but also affects the slope of the J-R curves, which is much flatter (with a lower tear

modulus) when the steels are tested in the presence of hydrogen (Figure 2). Hydrogen effects are also evident in the fracture surfaces. While both steels show ductile fracture in air (microvoids coalescence, Figure 2a,b orange), typical embrittlement mechanisms (Figure 2a,b blue) are noticed when internal hydrogen is present.

On the other hand, the effect of the test displacement rate in the fracture behaviour of pre-charged specimens of both steels is different. The S355 steel shows an embrittlement index of about 60% for the different displacement rates (Figure 2a), while the H8 steel (Figure 2b) shows a much larger decrease in toughness when the lowest displacement rate is used, reaching an embrittlement index higher than 90% for a displacement rate of 0.001 mm/min. The different behaviour of both hydrogen pre-charged steels with the test displacement rate is also noticed in their fracture surfaces (Figure 2). When both steels are tested at the slowest speed ($v = 0.001$ mm/min), S355 still shows some signs of microvoid coalescence while H8 shows a completely brittle fracture, usually referred to as plasticity-related hydrogen induced cracking (PRHIC) [6].

The different behaviour of both tested steels in presence of internal pre-charged hydrogen is based in their different microstructure and strength [7]. Hydrogen embrittlement is controlled by local stress and local hydrogen concentration and occur when a critical combination of both factors is satisfied in some specific microstructural units (internal interphases). Under the same test and loading conditions, the stronger steel (H8) develops greater hydrostatic stress in the region ahead of the crack, giving rise to higher local hydrogen contents and, consequently, larger embrittling phenomena than the lower strength S355 steel.

4. Conclusions

- Gaseous hydrogen pre-charge is a valid method to introduce hydrogen in the steels in order to study hydrogen embrittlement phenomena at room temperature.
- In general, hydrogen embrittlement increases as steel strength increases and as test displacement rate decreases, but this trend is dependent on the steel, on its microstructure and strength level.

Acknowledgments: The authors would like to thank Know-How Innovative Solutions and Instituto de Desarrollo Económico del Principado de Asturias (IDEPA) for the support received to carry out research project IDE/2016/000283.

References

1. Dong, C.F.; Liu, Z.Y.; Li, X.G.; Cheng, Y.F. Effects of hydrogen-charging on the susceptibility of X100 pipeline steel to hydrogen-induced cracking. *Int. J. Hydrog. Energy* **2009**, *34*, 9879–9884.
2. Park, G.T.; Koh, S.U.; Jung, H.G.; Kim, K.Y. Effect of microstructure on the hydrogen trapping efficiency and hydrogen induced cracking of linepipe steel. *Corros. Sci.* **2008**, *50*, 1865–1871.
3. Sampath, D.; Akid, R.; Morana, R. Estimation of crack initiation stress and local fracture toughness of Ni-alloys 945X (UNS N09946) and 718 (UNS N07718) under hydrogen environment via fracture surface topography analysis. *Eng. Fract. Mech.* **2018**, *191*, 324–343.
4. Dincer, I.; Acar, C. Smart Energy Solutions with Hydrogen Options. *Int. J. Hydrog. Energy*. Available online: https://www.researchgate.net/publication/324392901_Smart_energy_solutions_with_hydrogen_options (accessed on 11 November 2018).
5. Arafin, M.A.; Szpunar, J.A. Effect of bainitic microstructure on the susceptibility of pipeline steels to hydrogen induced cracking. *Mater. Sci. Eng. A* **2011**, *528*, 4927–4940.
6. Takeda, Y.; McMahon, C.J. Strain controlled vs stress controlled hydrogen induced fracture in a quenched and tempered steel. *Metall. Trans. A* **1981**, *12*, 1255–1266.
7. Zafra, A.; Peral, L.B.; Belzunce, J.; Rodríguez, C. Effect of hydrogen on the tensile properties of 42CrMo4 steel quenched and tempered at different temperatures. *Int. J. Hydrog. Energy* **2018**, *43*, 9068–9082.

