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# Fatigue crack initiation and growth in a CrMo steel under hydrogen pressure

L. Briottet <sup>a,b,\*</sup>, I. Moro <sup>a,b</sup>, M. Escot <sup>a,b</sup>, J. Furtado <sup>c</sup>, P. Bortot <sup>d</sup>,  
G.M. Tamponi <sup>e</sup>, J. Solin <sup>f</sup>, G. Odemer <sup>g</sup>, C. Blanc <sup>g</sup>, E. Andrieu <sup>g</sup>

<sup>a</sup> Univ. Grenoble Alpes, F-38000 Grenoble, France

<sup>b</sup> CEA, LITEN, DTBH, F-38054 Grenoble, France

<sup>c</sup> Air Liquide – Centre de Recherche Paris-Saclay, 1 chemin de la Porte des Loges, BP 126, 78354 Les Loges-en-Josas, Jouy-en-Josas, France

<sup>d</sup> TenarisDalmine R&D, Piazza Caduti 6 Luglio 1944, 1, 24044 Dalmine, Italy

<sup>e</sup> CSM SPA, Via Di Castel Romano 100, Roma 00128, Italy

<sup>f</sup> VTT Technical Research Centre of Finland, Vuorimiehentie 5, Espoo 02044, Finland

<sup>g</sup> Université de Toulouse, CIRIMAT, UPS/INPT/CNRS, ENSIACET, 4 allée Emile Monso, BP 44362, 31030 Toulouse Cedex 4, France

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## ABSTRACT

Along the hydrogen supply chain, metallic components, such as pressure vessels, compressors and valves, are facing high pressure hydrogen gas. The object of this paper is to address microstructural as well as mechanical aspects of fatigue crack initiation and growth at room temperature in a quenched and tempered (Q&T) low alloy steel under hydrogen pressure in the range 0.5–35 MPa. For such steel, the need to perform tests in-situ under hydrogen pressure is required. The influence of hydrogen gas on the total life in terms of crack initiation and crack propagation is analyzed. The experimental techniques developed to detect crack initiation in a pressure vessel under hydrogen pressure are presented. Thanks to these technical developments the influence of hydrogen gas on the total life duration including crack initiation and crack propagation is analyzed. It is shown that the effect of hydrogen pressure on crack initiation is important. At constant load ratio, the hydrogen pressure effect on fatigue crack growth (FCG) is dependent on the loading amplitude (in terms of  $\Delta K$ ). These results related to cracking behavior are enriched with information on fracture surfaces appearance. The results presented have been achieved within the European project MATHRYCE [1] dedicated to Material Testing and Recommendations for Hydrogen Components under fatigue. They are part of a process necessary to give a scientific background to the development of a design methodology where hydrogen enhanced fatigue damage is taken into account.

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\* Corresponding author. CEA, LITEN, DTBH, F-38054 Grenoble, France.

E-mail address: [laurent.briottet@cea.fr](mailto:laurent.briottet@cea.fr) (L. Briottet).

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## Introduction

Pressure vessels for gaseous hydrogen storage can be submitted to several pressure cycles, therefore taking hydrogen enhanced fatigue damage into account is a key step for a safe design and a large development of hydrogen economy. Several works are currently performed on this subject [1–3]. The European MATHRYCE project aims to propose an easy way to implement vessel design methodology based on lab-scale tests taking into account hydrogen enhanced fatigue damage [1]. Due to the components considered in this study and their associated estimated maximum number of cycles in use, low cycle fatigue (LCF) loading is involved when a defect or a crack is present. Many studies have shown that the fatigue life of LCF samples is strongly reduced when tested under hydrogen pressure. The effect of hydrogen on fatigue crack growth has been intensively addressed. The fatigue crack growth rate of Cr–Mo steels is classically observed to be enhanced by a factor 20–30 at sufficiently high  $\Delta K$ . By contrast, the effect of hydrogen on fatigue crack initiation (FCI) under LCF conditions has not widely been studied since it is believed that for such loading, FCI is not predominant [4]. However, considering a strong decrease of fatigue life time under hydrogen due to accelerated FCG, the life spent in fatigue crack initiation may become important.

The present paper focuses on a low alloy Q&T Cr–Mo steel, classically used to store hydrogen gas up to 45 MPa. Hydrogen enhanced fatigue has been addressed in terms of crack initiation and propagation, in particular to clarify the respective contribution of these two stages of the fracture process with or without hydrogen, analyzing the possible effects of the gas pressure, load-cycle frequency, minimum load/maximum load ratio  $R$ . The developed methodology to detect crack initiation under hydrogen pressure is described and validated. Then, the fatigue crack behavior is discussed together with fracture surface observations.

## Material and methods

### Material

The material used in this study is a 25CrMo4 steel according to EN 10083-3 standard (Table 1). Specimen were machined from a commercially available seamless pressure vessel OD 470 mm x WT 30 mm (Fig. 1). The mechanical properties of this material in the transverse direction are 630 MPa for the yield stress and 785 MPa for the ultimate tensile strength, whereas the total elongation is 19%.

**Table 1 – Chemical composition (wt %) of the product under investigation.**

	C	Si	Mn	Cr	Mo
Standard requirements	0.22–0.29	Max 0.4	0.6–0.9	0.9–1.2	0.15–0.30
Product analysis	0.26	0.24	0.76	0.98	0.20

The material is provided in a Q&T state. Microstructure (Fig. 1) has been analysed by means of Electron Back Scattered Diffraction (EBSD) technique. In particular, packets, which are the regions separated by high-angle ( $>15^\circ$ ) boundaries, were identified through Inverse Pole Figure (IPF) maps obtained by EBSD at mid-wall. Coarse packets were related to tempered bainite, whereas fine packets are likely associated with regions constituted of tempered martensite. The size of the prior austenitic grains has been evaluated around 30–50  $\mu\text{m}$  using EBSD and the ARPGE software [15] to reconstruct the parent grains (Fig. 2).

As small differences in composition, microstructure or non-metallic inclusions can be important when dealing with fatigue resistance under hydrogen environment, a detailed microstructural analysis has thus been performed on the components selected for the project. The observed inclusions are mainly constituted by fine globular sulfides and oxides and by sporadic aluminates. The analysis of these non-metallic inclusions was also done, investigating both the maximum dimension and shape of the inclusions typical for the product. Two methods have been used: the Automatic Inclusion Analysis and the Analysis of Non Metallic Inclusions according to ESIS/ASTM standards (Linear Extreme Value Distribution) [5,6]. The resulting maximum expected inclusion size for the component was predicted to be 120  $\mu\text{m}$ , as per the extreme value analysis. This is based on the largest dimension of the inclusions as observed in metallographic survey. The largest expected non-metallic inclusion is sensibly smaller than detection limits, with adequate probability of detection, of non-destructive examination (NDE) techniques commonly used for industrial structural elements. As an example ISO 11120–1999 [7] prescribes an ultrasonic inspection given where the reference notch depth is set to 5% of the nominal wall thickness with a maximum depth of 1 mm. For conservatism purposes, the minimum defect dimension to be considered in the following of the study will be based on this NDE limit.

### General testing procedure

All the tests were performed at room temperature (RT) on an MTS servo-hydraulic testing system in a pressure vessel filled with gaseous hydrogen up to 35 MPa. The quality of the hydrogen gas was a 6.0 high purity. In order to ensure a good and reproducible environment quality, a specific procedure, including several vacuum plus 6.0 nitrogen filling runs was followed. A final filling of the vessel with 6.0 pure nitrogen is performed at the testing pressure. Finally, hydrogen is introduced and a 30 min dwell at the  $\text{H}_2$  testing pressure was performed before carrying out the mechanical tests under hydrogen. This experimental procedure ensures a reproducible testing atmosphere and a low level of  $\text{H}_2\text{O}$  and  $\text{O}_2$  impurity contents, below a few ppm. Indeed, it is recognized that a higher level of impurity may have an impact on the hydrogen embrittlement sensitivity of metallic materials [23].

### Fatigue crack initiation

The classical effect of hydrogen on steels is to reduce their fatigue life. In particular, it is well known that fatigue crack

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