Hydrogen effects on fatigue strength of a High Strength Low Alloy steel

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ABSTRACT. The results obtained in an experimental study, aimed at characterizing the effects of hydrogen content on fatigue behavior of High Strength Low Alloy steels candidates for advanced hydrogen storage tanks manufacturing, are presented. Tests were conducted, both on hydrogen previously charged and virgin material specimens. Results indicates the hydrogen tend to mainly affect the crack propagation, rather than the crack initiation stage, with nearly no effect on fatigue limit. Such results were also confirmed by SEM observations of fracture surfaces, showing the presence of several “secondary cracks” in hydrogen charged specimens.

SOMMARIO. Vengono presentati i primi dati ottenuti in studio sperimentale volto a caratterizzare gli effetti della presenza di idrogeno sulla resistenza a fatica di acciai High Strength Low Alloy, utilizzabili per la costruzione di recipienti per l’immagazzinamento di idrogeno gassoso. Sono state condotte prove di fatica su provini caricati e non caricati con idrogeno. I risultati ottenuti indicano che l’effetto dell’idrogeno è osservabile soprattutto nella fase di propagazione della frattura, mentre appare modesto su quella di innesco, non influenzando in modo evidente il limite di fatica. I risultati sono stati confermati da osservazioni SEM che, nei provini caricati con idrogeno, hanno evidenziato la presenza di numerose “secondary cracks” sulla superficie di frattura.

KEYWORDS. Hydrogen Embrittlement; HSLA steel; Hydrogen effects on fatigue.

INTRODUCTION

Present work illustrates activities conducted for a preliminary characterization of fatigue behavior of a candidate material for innovative low weight hydrogen storage tanks. The hydrogen interaction with fatigue is a quite complex phenomenon, to date lacking of a detailed characterization both from a phenomenological and an experimental viewpoint. Therefore, current study was mainly aimed at achieving preliminary indications concerning possible hydrogen detrimental effects on fatigue strength.

The few available literature data [1-5] concerning materials pertaining to the same strength category ($\sigma_R \approx 1000 \text{ MPa}$) apparently indicate that hydrogen tends to reduce fatigue endurance, particularly in the short-medium life regime, having a relatively low effect on fatigue limit.

Such results are somewhat in contrast with those concerning high strength steels ($\sigma_R \approx 1500 \text{ MPa}$ and over), for which an extended literature is available, and whose fatigue strength is apparently mainly affected by hydrogen presence in the high cycle regime and as regards fatigue limit. For such materials, a rather strong effect of cyclic load frequency, whose reduction is usually accompanied by a reduction of fatigue life (intended as cycles to failure).

In present work, fatigue tests results from virgin (zero hydrogen content) and nickel plated hydrogen charged (high pressure gaseous permeation technique) specimens were compared.
Moreover, tests were also conducted on nickel plated specimens without hydrogen charging, to verify potential effects of hydrogen absorption during the electrochemical plating process.

**EXPERIMENTAL PROCEDURE**

Fatigue tests were conducted on AISI/SAE 41xx 1%Cr-0.25%Mo steel specimens, pertaining to the “High Strength Low Alloy” or HSLA family. Material was quenched and tempered before tests, to get best combination of strength and ductility.

Tests were conducted on a Rumul resonance axial test frame, making use of a 150 Hz cycle frequency, with R-ratio ($P_{\text{min}}/P_{\text{max}}$) equal to zero.

Tests were stopped after specimen failure or at 10⁷ cycles (runouts).

Tests frame in operating conditions is shown in Fig. 1, while specimen dimensions and shape are illustrated in Fig. 2.

As the hourglass specimen shape implies a limited stress concentration in the minimum section size zone, a linear elastic FEM model was employed to analyse actual stress distribution. Thanks to symmetry, 1/8 of the specimen was included in the model, applying proper boundary conditions. The results are shown, as an example, in Fig. 3. A stress concentration factor, $K_T$, equal to 1.036 was calculated, which was employed to correct nominal stresses in the following analysis.

![Figure 1: Test device setup.](image1)

![Figure 2: Specimen employed for the tests.](image2)
TEST RESULTS

Fatigue tests were first conducted on virgin (zero hydrogen, no nickel plating) material. Subsequent tests were conducted on hydrogen charged specimens, making use of the same test conditions employed for virgin material.

Hydrogen charging was obtained by exposing the nickel-plated specimens to a high pressure hydrogen atmosphere (150 bar), in a small purposely designed autoclave, for a few days, so as to allow the ending of diffusion transient, assumed to be rather short in time, thanks to reduced thickness.

The hydrogen content at the end of charging was measured via an Hydrogen Analyzer LECO DH603 and found to be about 0.7 p.p.m.. After charging, each specimen was left in air at room temperature for four days, so as to let diffusible hydrogen to flow out, leaving only the hydrogen contained in the so called “irreversible traps”, for a final concentration of about 0.4 p.p.m..

Finally, a few tests were also conducted on nickel-plated (but not charged) specimens, to analyze the effects of the plating process. It was found that the hydrogen content due to nickel-plating only was about 0.2 p.p.m..

In total, 20 tests were conducted on virgin material specimens, 12 tests on nickel-plated not charged (0.2 p.p.m. H) specimens and 15 tests on nickel-plated and hydrogen charged (0.4 p.p.m. H) specimens.

DISCUSSION

The results of fatigue tests on virgin (zero hydrogen, no nickel plating) material are reported in Fig. 4. A rather small scatter can be observed, together with a small slope of the S-N curve, as typical of HSLA steels.

The results of tests on nickel-plated hydrogen charged specimens (0.4 p.p.m. H) are shown in Fig. 5. In this case also, a rather limited scatter can be observed, together with an increase of the S-N curve slope in the limited life region, as compared to virgin material, while fatigue limit did not exhibit significant variations. The S-N slope increase could be interpreted as an effect of an increased crack growth rate in hydrogen charged specimens.

Finally, Fig. 6 illustrates the results of tests conducted on nickel-plated not charged specimens (0.2 p.p.m. H). Experimental trends are quite similar to those observed in hydrogen charged specimens, with increase in S-N slope and nearly no modification of fatigue limit.

Results of fatigue tests were then combined with SEM observations of fracture surface, in order to obtain micrographic confirmation of hydrogen effects.

In Fig. 7, low magnification SEM images of different fracture surfaces are reported. Fig. 7a) was obtained from a virgin specimen test, Fig. 7b) from a hydrogen charged specimen test (0.4 p.p.m. H) and Fig. 7c) from a nickel plated not charged specimen test (0.2 p.p.m.). A nearly equivalent pattern is observed in the three images at this magnification level, with a clearly detectable initiation site and a crack growth zone, before final fracture.
Figure 4: S-N data for virgin material.

Figure 5: S-N data for nickel-plated hydrogen charged material.

Figure 6: S-N data for nickel-plated material.
In Fig. 8, SEM high magnification images of fracture surfaces are reported, from virgin material specimen (8a)), from 0.4 p.p.m. specimen test (8b)) and form 0.2 p.p.m. specimen test (8c)).

Figure 7: SEM low magnification images of fracture surfaces: (a) virgin material, (b) 0.4 p.p.m. hydrogen content material, (c) 0.2 p.p.m. hydrogen content material

Figure 8: SEM high magnification images of fracture surfaces: (a) virgin material, (b) 0.4 p.p.m. hydrogen content material, (c) 0.2 p.p.m. hydrogen content material.
At such magnification level, it is possible to detect morphological differences between fracture surfaces, invisible at low magnification. As a matter of fact, surfaces coming test conducted on specimens containing hydrogen (8b-c)) exhibited several micro-cracks, having mainly a trans-granular morphology ("secondary cracks"). Such micro-cracks apparently originate from fracture surface, having an external front nearly parallel to local fatigue crack front, and then tend to extend in a direction normal to main fracture surface. 

This observations appear to be in agreement with results obtained from S-N curves, which indicated the presence of hydrogen as mainly affecting crack propagation, rather than crack initiation phase. Such results appear rather interesting for applications, as it is well known that the propagation stage occupies, in actual full scale components, a fraction of total fatigue life much more significant than in the case of laboratory specimens, for which due to rather high loads and reduced size, crack growth process tend to be very short.

**CONCLUSIONS**

From above results and discussion, the following main conclusions can be drawn:

- Fatigue tests were conducted on virgin and hydrogen charged HSLA steel (AISI/SAE 41xx ) specimens.
- Test results indicated that the presence of hydrogen tend to affect the crack growth rate, rather than the fatigue limit, increasing the S-N curve slope.
- Such hydrogen damage mechanism was also confirmed by SEM observations, showing, in hydrogen containing specimens, the presence of several secondary cracks, not observed in virgin specimens.
- The effect could potentially be more significant in full scale components, for which crack propagation phase tend to be quite longer than for laboratory specimens.

**REFERENCES**