

## ASPECTS OF THE FATIGUE BEHAVIOUR OF METALLIC MEMBRANES – THE INFLUENCE OF HYDROGEN

### ASPEKTE TË SJELLJES NË LODHJE TË MEMBRANAVE METALIKE - NDIKIMI I HIDROGJENIT

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#### PËRMBLEDHJE

Ky punim paraqet një metodë të re për karakterizimin e sjelljes së materialeve të membranave metalike që u nënshtrohen sforcimeve dinamike, të gjeneruara nga ndryshimet e presionit dhe të temperaturës. Në kushte të tilla pune, karakteristike në veçanti për enët në presion, lodhja oligociklike është shkaku më i mundshëm i shkatërrimit. Metoda e propozuar bazohet në teknikën e disqeve, të përshtatur për të siguruar variacione të programuara të presionit. Epërsi e kësaj teknike është kontrolli i saktë i përbërjes së mjedisit, që lejon vlerësimin e ndikimit të tij dhe në veçanti të hidrogjenit, mbi sjelljen e materialit në lodhje. Nga rezultatet eksperimentale të përfutuara në formën e varësive Presion-N dhe Shigjetë deformimi-N, janë llogaritur lakoret ep-N të materialeve të testuara. Mbi këtë bazë nxirren përfundime mbi jetëgjatësinë e materialeve të ndryshme për membrana, që janë në kontakt ose jo me hidrogjenin.

**Fjalë Kyçe:** lodhje oligociklike, prova e disqeve, modelimi i sjelljes, thyeshmëria prej hidrogjenit, enë nën presion.

#### SUMMARY

This paper presents a new method for the characterisation of the materials behaviour of metallic membranes subjected to dynamic stresses, generated by the pressure and temperature variations. The low cycle fatigue represents in such conditions the most probable cause of failure, especially in the case of pressure vessels. The proposed method is based on the disk testing technique adapted in order to ensure a programmable variation of the pressure. The edge of this technique consists in the accurate control of the environment composition that allows the evaluation of its effect and especially that of the hydrogen on the materials fatigue behaviour. The experimental results obtained in the form of Pressure-N and Deflection-N dependences, are processed in order to calculate the ep-N curves of the material. On this base conclusions have been drawn on the lifetime of different materials for membranes, in presence or in absence of hydrogen.

**Key words:** low-cycle fatigue, disk pressure testing, behaviour modelling, hydrogen embrittlement, pressure vessels.

#### INTRODUCTION

The interest for a larger utilisation of hydrogen as energetic vector has led to search for safer and less expensive solutions, mostly related to pressure vessels. These solutions impose the use of high-strength materials, which, unfortunately, are often the most sensitive ones to the

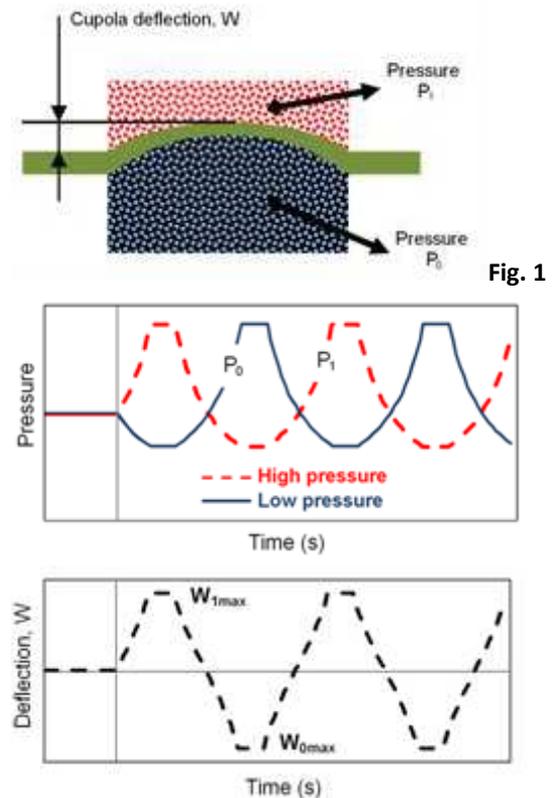
hydrogen embrittlement [1, 2]. In order to characterise this effect and to select the most appropriate materials for such applications, it is successfully used the disk pressure testing, which is included, since 2005, in the ISO standard 11114-4 [3]. Thanks to its simplicity, reliability and sensitivity, this technique allowed the study

of different factors affecting the materials behaviour, which were difficult to be detected by the traditional techniques of tensile testing [4, 5, 6]. Currently many efforts are made to perfect the characterisation of such structures (pipes, pressure vessels/membranes, bellows...), taking into account, besides others, the effects of cyclic loadings caused by variations of pressure and temperature [1, 7]. The tension/compression fatigue testing, commonly used, fails to simulate actual working conditions of membranes, pressure vessels etc.; moreover, it uses relatively massive samples, which are not suitable for assessing the impact of hydrogen, which mainly affects the superficial layers [8]. Therefore, since the disk testing is very sensitive to the environment, efforts were made to use it for the characterisation of materials fatigue behaviour. Cyclic loading of the disk, which takes place during this testing, duplicates what happens in many real structures eg, pressure vessels, during their filling and emptying. In this paper it is presented the improvement made to the experimental techniques of the fatigue disk testing to ensure reliable results using recycled gas. Moreover, it is proposed an original method for the results processing, enabling a more generalized characterisation of the materials behaviour during the low cycle fatigue.

## MATERIAL AND METHODS

Several tests were conducted on different materials: low alloy steels, stainless steels, copper and nickel alloys, treated to obtain various microstructures. In this way there are assessed not only the properties of different materials, but also those of the same material in different metallurgical states. The experimental technique includes the testing cell adapted for a bilateral loading, oil/gas separators and compression systems with pneumatic and hydraulic cylinders. The compression systems that act on both sides of the disk are identical, so that the increasing of the pressure on one side, leads automatically to the reduction to the same extent of the pressure on the other side. As a result, it is obtained an alternate up and down

deformation (Fig. 1). The pressure difference  $|P_1 - P_0|$  can be adjusted up to a maximum of 35 MPa. The disk deflection,  $W$ , is continuously measured with an accuracy of 0.01 mm. The system allows different variants of the fatigue tests:  $W_{1max} > 0 > W_{0max}$ ,  $W_{1max} > W_{0max} = 0$  and  $W_{1max} > W_{0max} > 0$ . The lifetime limit is reached when a through-crack of the disk no longer allows the creation of the programmed pressure difference.



**Figure 1.** Pressure and deflection of a disk under fatigue

The speed of pressurisation/depressurisation and the length of stay in the extreme pressures can be adjusted in fairly wide limits; the period varies from several seconds to several minutes. For the tests under hydrogen, the speed must be low enough to saturate the material with hydrogen thanks to its transport by mobile dislocations [9]. To avoid the phenomenon of passivation, it is used very pure hydrogen (> 99.9995%).

An empirical criterion has been proposed by CEA, based on the results of the low cycle fatigue of disks: the embrittlement index, determined by the ratio  $N_{F0}/N_{FH}$ , must be smaller than 5 so that the material can be used under hydrogen ( $N_{F0}$  and  $N_{FH}$  are respectively the lifetimes in the low cycle fatigue of disks in absence and in presence of hydrogen). However, this criterion is insufficient to characterise the fatigue behaviour, because it does not take into account the particularities of the loading and deformation of the material during the test. In our study we intend to make a more generalised interpretation of the results, based on considering exactly these features.

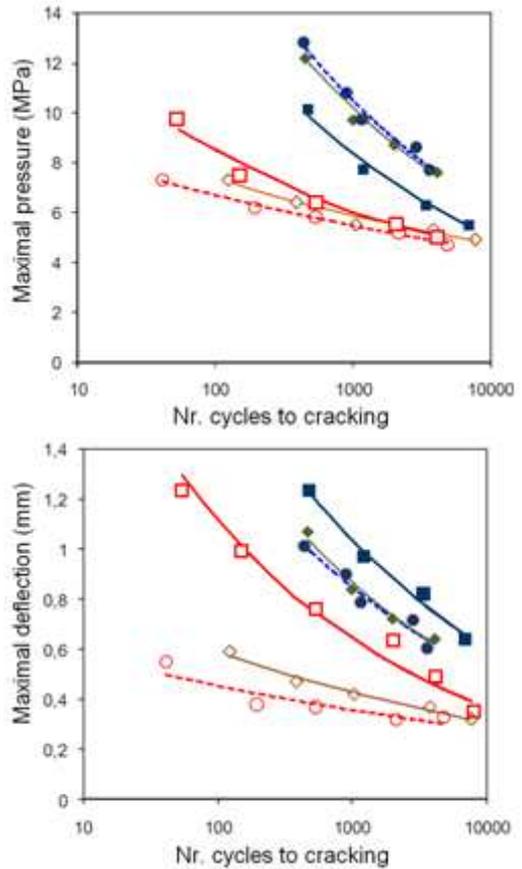
**RESULTS AND DISCUSSIONS**

**Experimental plots**

In Fig. 2 are shown the results of tests performed on a nickel superalloy after different heat treatments conducted to obtain metallurgical states with different sensitivity to hydrogen: low, medium and high. They express dependences of the fatigue life on the pressure amplitude,  $|P_{1max}-P_{0min}|$  and on the maximum deflection,  $W_{1max}$ .

The quality of the experimental results obtained with both gas and three metallurgical states is satisfactory because the dispersions are much lower than those of the usual fatigue tests. It appears that the material with the best fatigue characteristic,  $P-N$ , under helium, is the worst under hydrogen. This confirms the known fact that hydrogen affects more the high-strength materials.

Further analysis of the results shows that the deformation of the disk is asymmetric, so we have a deformation hysteresis. The asymmetry ( $W_1 > W_0$ ) remains more or less the same from the first cycle to the last one, while the deformation evolves over time. This evolution accelerates at the end of the test that corresponds to the macroscopic crack propagation. In all cases, cracking begins at the disk embedding zone.



State	Sensitivity to H	Helium	Hydrogen
1	low	■	□
2	medium	◆	◇
3	high	●	○

**Figure 2.** Fatigue life of disks made of a nickel alloy

Stress	Loading	Centre	Periphery
Radial,	Traction	$0.976 E W^2 / a^2$	$0.476 E W^2 / a^2$
$\sigma_{rad}$	Flexion	$2.86 E W t / a^2$	$4.40 E W t / a^2$
Tangent.,	Traction	$0.976 E W^2 / a^2$	$0.143 E W^2 / a^2$
$\sigma_{tg}$	Flexion	$2.86 E W t / a^2$	$1.32 E W t / a^2$

**Table 1.** Stresses in a thin, elastic, circular membrane, embedded at its periphery

**Calculation model for the elastic zone**

Considering the disk as a thin membrane (with thickness  $t$ ), embedded at the distance  $a$  from the axis, it can be calculated the radial and tangential stresses ( $\sigma_{rad}$  and  $\sigma_{tg}$ ) as functions of the disk deflection (Tab. 1).

The stress  $\sigma_z$  is negligible because it is one hundred times weaker than the radial and tangential stresses (for the thickness and diameter of the disks used). The equivalent stress is calculated by Von Mises formula:

$$\frac{\bar{\sigma}}{\sigma} = \left[ \frac{(\sigma_{rad} - \sigma_{tg})^2 + (\sigma_{tg} - \sigma_z)^2 + (\sigma_z - \sigma_{rad})^2}{2} \right]^{\frac{1}{2}} \tag{1}$$

Calculations show that for small deflections, as those recorded during the fatigue tests ( $W < 1.2$  mm), stresses at the periphery of the disk are larger than those at its centre. This result confirms the experimental observations, indicating the onset of the disk plastification and cracking at its periphery. On the basis of Von Mises stress and the formulas of Tab. 1, it can be calculated the value of the deflection  $Wp$ , for which the plastic deformation starts. This corresponds to the moment when the equivalent stress  $\bar{\sigma}$  overpasses the limit of elasticity of the material.

**Calculation models for the plastic zone and the fatigue**

The evolution of the plastic deformation depending on the actual deflection,  $W$ , is not known, but it can be approximated by a linear relationship, when the values of  $W$  are sufficiently small:

$$\epsilon_p = K(W - W_p) \tag{2}$$

The plastic deformation itself, in the case of low cycle fatigue, is related to the fatigue life by the Manson-Coffin law [10]:

$$\epsilon_p = A_0 N_{F0}^{-a_0} \tag{3}$$

where  $A_0$  and  $a_0$  are constants, while  $N_{F0}$  is the number of cycles to rupture.

If a monotonic rupture test (rupture strain  $\epsilon_{R0}$ ) is considered as a fatigue test within 0.25 cycle ( $N_R = 0.25$ ), the Manson-Coffin law becomes:

$$\epsilon_p = (\epsilon_{R0} / N_R^{-a_0}) N_{F0}^{-a_0} \tag{4}$$

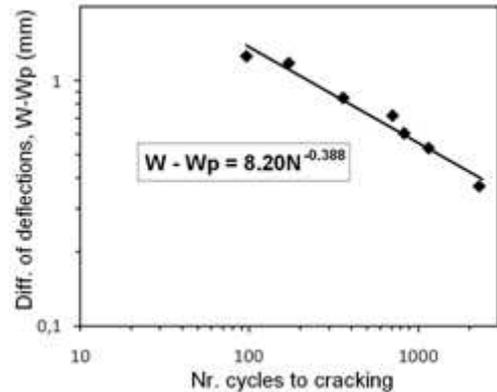
The number of cycles to rupture increases when the imposed plastic deformation is reduced. The processing of our experimental results (Fig. 3), shows that during such evolution the deflection  $W$  is fitted against the cycle number  $N_{F0}$  by the empirical law:

$$W - W_p = CN_{F0}^{-c} \tag{5}$$

The right sides of the equations (3) and (4) have the same form. Assuming that the exponents  $a_0$  and  $c$  of these equations are equal, it turns out that the plastic deformation can be calculated by the expression (2), where  $K$  is:

$$K = (\epsilon_{R0} / N_R^{-a_0}) / C \tag{6}$$

In this way, by processing the  $W-N$  data of the disk tests, it is possible to find the generalised relationships,  $\epsilon - N$ , characterising the materials behaviour in low cycle fatigue.

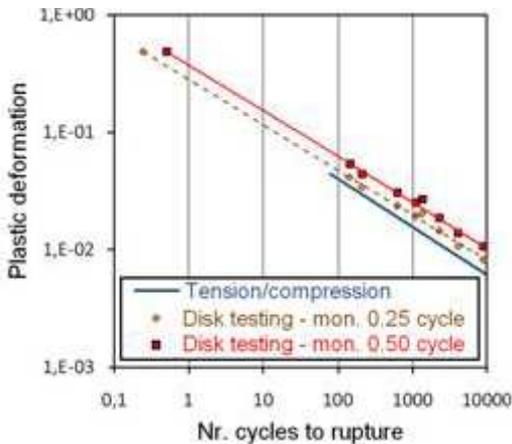


**Figure 3.** Fatigue life versus disk deflection

**Comparison with other data**

The relationships  $\epsilon - N$ , obtained from the disk fatigue testing, are compared with those of traditional fatigue testing (traction/compression), performed on the same materials. The Figure 4 shows this comparison for an austenitic stainless steel (17% Cr, 12% Ni). There is clear evidence that the relationships of the disk testing are very similar to those of tension/compression;

however, the first give fatigue lives always greater than the second ones. This difference is explained by the fact that the steel studied is very sensitive to strain-hardening [10] and therefore, after each traction/compression cycle, the applied stress increases to reach the specified degree of deformation; on the contrary, during the disk testing, for which the set value is the maximum pressure and not the degree of deformation, the last one decreases with the materials hardening. This means that the disk test, 'tires' the material more slowly than the traction/compression. Such a difference is not observed in the case of a low alloyed copper, because this material is less sensitive to strain-hardening.



**Figure 4.** Behavior of an austenitic steel in low cycle fatigue: tension/compression and disk testing

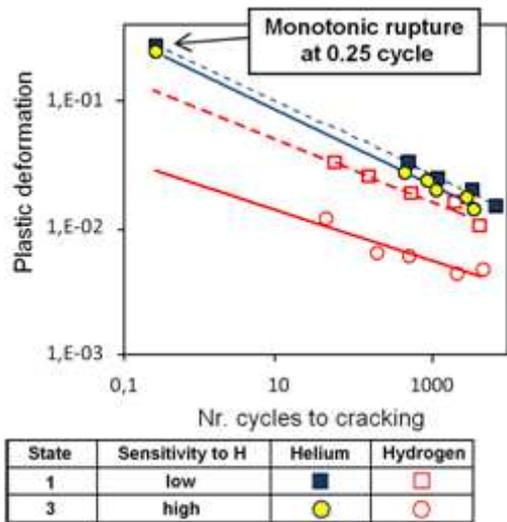
**Influence of the hydrogen**

In Fig. 5 are shown generalised results of the fatigue testing of a nickel alloy. It seems clear that hydrogen reduces the lifetime of the material, especially in the case of its metallurgical state the most sensitive to hydrogen. The analysis of these results can be based again on the Manson-Coffin law, which for the testing under hydrogen can be written:

$$\epsilon_p = A_H N_{FH}^{-a_H} \tag{7}$$

If the material is sensitive to hydrogen, this will result on the values of the coefficients  $A_H$  and  $a_H$ ; both will be lower than the corresponding

coefficients under helium,  $A_0$  and  $a_0$ . The fact that  $a_H < a_0$ , shows that the fatigue laws under helium and under hydrogen tend to be closer for high cycle fatigue, therefore, for small deformations. This observation is explained by the mechanism of hydrogen introduction into the material, based on the presence of dislocations [9]. For small deformations, the number of dislocations created is limited, so they fail to introduce into the material a sufficient amount of hydrogen to cause a significant embrittlement. So, the fatigue relationships, obtained by the disk testing, are consistent with the relevant theory.



**Figure 5.** Resistance to low cycle fatigue: disks made of a nickel alloy

**CONCLUSIONS**

The technique used to characterise the materials behaviour in low cycle fatigue, has many advantages compared to traditional tests because it is conducted with small and simple specimens, using limited volumes of gas and because it avoids the passivation of the material during the test.

The good reproducibility of the results processed by the Manson-Coffin law, allows a reliable prediction of the fatigue life of the equipment working in the presence or absence of hydrogen. In particular, this prediction is valid for membranes, which are subject to local

plastification during the cycles of loading/unloading.

The risk of hydrogen embrittlement is greater in the low cycle fatigue compared to the usual one, but the magnitude of this risk heavily depends on the metallurgical features of the material. The proposed method highlights the effects of these features and on this basis enables a more rational selection of the material itself and of the technology of its processing/treatment.

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