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DISK TESTING UNDER HELIUM APPLIED FOR THE CHARACTERISATION OF THE MATERIALS MECHANICAL BEHAVIOR
APLIKIMI I PROVËS SË DISQEVE NËN HELIUM PËR KARAKTERIZIMIN E SJELLJES MEKANIKE TË MATERIALEVE

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PERMBLEDHJE
Në këtë punim është studiuar sjellja mekanike e fletëve të holla të një çeliku austenitik në kushte ngarkimi dyaksial, duke përdorur provën e modifikuar të disqeve nën presion. Vlerësimet janë kryer në temperatura të ndryshme nga 18 në 650°C, me presim të vazhduar si dhe me mbajtje nën presion konstant. Informacioni i përftuar është krahasuar me atë të provës tradicionale të tërheqjes duke llogaritur sforcimet dhe deformimet të vërteta, si dhe treguesit e përforcimit të materialit. Eshtë konstatuar se diferencat e rezultateve të dy provave theksohen me rritjen e shkallës së deformimit. Në veçanti, prova e disqeve evidenton më qartë dukuritë përforcuese që shoqërojnë deformimin e çelikut dhe që çojnë në evoluimin e sjelljes së tij gjatë shërbimit. Ajo lejon edhe vlerësimin e ndikimit të kombinuar të temperaturës dhe kohës gjatë një procesi shkarrjeje të përshpejtuar.
Fjalë kyçe: prova mekanike, material, plasticitet, shkarje, temperaturë.

SUMMARY
In this work is studied the mechanical behavior of thin sheets of an austenitic steel under biaxial loading, using a modified disk pressure test. Evaluations are performed at different temperatures from 18 to 650°C, by continous pressing or by maintenance at a constant pressure. The data obtained are compared with those of the traditional tensile test by computing the true stresses and strains, as well as the strain hardening parameters. It is observed that the differences in the results of the tests increase with the degree of deformation. In particular, the disk test identifies more clearly hardening phenomena associated with the deformation of the steel, which lead to the evolution of its behavior during the service. It also allows the evaluation of the combined impact of temperature and time during an accelerated creep.
Key words: mechanical testing, material, plasticity, creep, temperature.

NTRODUCTION

Since more than four decades, the disk pressure test has been used as an experimental technique (not as a standardized one), to assess the hydrogen embrittlement. After a long application and after processing thousands of results obtained with different materials, this test was adopted (1990) by the French standard AFNOR for the selection of materials for vessels working under high pressure of hydrogen. In 2005 it was integrated into a more general international standard, ISO 11114-4 [1]. Today we can say that the disk test has passed the stage of 'maturity' and it is a safe reference for characterizing the materials sensitivity to hydrogen. Moreover, the high reproducibility and rialibility of the test results have prompted researchers to consider the possibilities of expanding its range of application [2, 3]. In particular, a great interest is presented by this test because it characterizes the material under biaxial loading, similar to that of pressure vessels. The safety requests of these vessels have been strengthened significantly, and therefore the assessment of the suitability of materials used in such applications is not considered sufficiently reliable if based only on information received from traditional tensile testing [4, 5]. In this paper is presented a modification made to the disk test to enable the characterization of the mechanical behavior of thin sheets of an austenitic steel, subject to biaxial loading.

MATERIAL AND METHODS

The study was performed on an AISI 321 stainless steel, cold-rolled to the thickness of 0.8 mm and heat treated to obtain the austenitic structure at room temperature. Specimens, in the form of flat disks, with a diameter of 58 mm, were cut from a sheet of this steel, and were pressed under helium, while maintaining the original thickness (i.e., without any surface machining). Standard test cell was slightly modified to enable the characterization of the steel behavior during a biaxial loading (Fig.1). For this, the radius *r* was increased from 0.5 to 4 mm to avoid the stress concentration in the embedding zone and to ensure a uniform dome-shaped deformation of the disk.

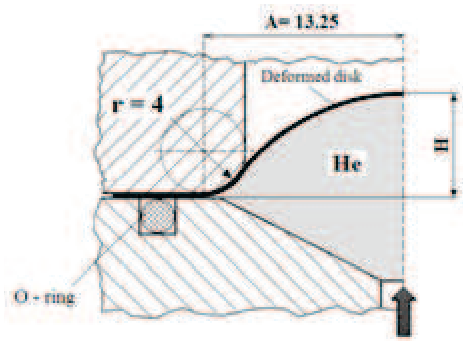

Figure 1. Geometry of the modified disk pressure cell

The main information obtained from the disk tests (with the standard cell, as well as with the modified one), is the curve of the gas pressure versus deformation, which is automatically recorded until the disk rupture. Deformation is measured as the cupola deflection *H*.

Tests were conducted at different temperatures, from 18 to 650°C, with duration of several minutes to 7 hours. The results of tests performed at 18 and 400ºC are shown in Fig. 2. For purposes of comparison, the studied steel was also subjected to the traditional tensile test at room temperature.

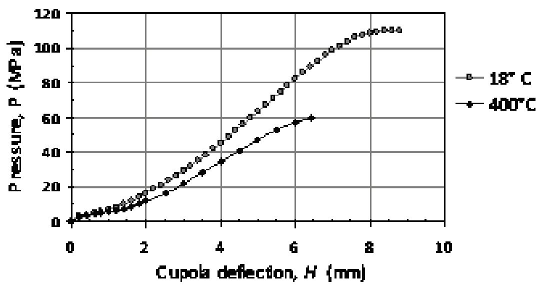

Figure 2 Pressure-deflection curves at 18ºC and 400°C.

RESULTS AND DISCUSSIONS

Calculation model

Gas pressure-deformation relationships provide valuable information about the load-bearing capacity of a pressure vessel with parameters similar to that of the disk deformed during the testing, but not directly reflect the behavioral characteristics of the material. This behavior can be assessed objectively only on the basis of true stresses and strains. For their calculation we have used an analytical model, which assumes that the disk is deformed as a membrane, taking also into account the geometric parameters of the test cell [4]. With this model are calculated the radius *R* of the spherical dome, the engineering strain, *ε*, in the lateral directions (*x* and *y*), and the actual thickness, *e*, for any measured value of the disk deflection, *H*:

$$R = \frac{\sqrt{A^2 + H^2}}{2\sin\left(\operatorname{arctg}\frac{H}{A}\right)} - r \qquad (1)$$

$$\varepsilon = \int_0^H \frac{dH}{R}\left[1-\frac{2r}{A}\left(\frac{1}{1+\frac{H^2}{A^2}}\right)\sin\left(2\,\mathrm{arctg}\frac{H}{A}\right)\right] \quad (2)$$

$$e = e_0 \exp(-2\varepsilon), \quad (3)$$

where:

A - is a geometric parameter of the cell (A = 13.25 mm);

r - is the radius in the embedding zone (r = 4 mm);

e_0 - is the original thickness of the disk (e_0 = 0.80 mm).

Using the above relations and accepting the von Mises yield criterion [6], there are defined formulas to calculate the true stress and strains, σ_T and ε_T:

$$\varepsilon_T = \ln\frac{e}{e_0} = 2\varepsilon \quad (4)$$

$$\sigma_T = \sigma_x = \sigma_y = \frac{RP}{2e} \quad (5)$$

Figure 3 shows the relationship $\sigma_T - \varepsilon_T$, obtained by processing the data of the *P- H* curve with the formulas 1-5.

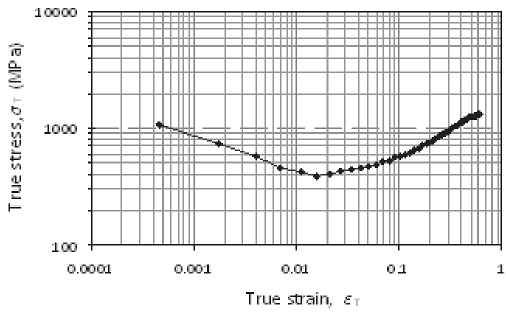

Figure 3 True stress-strain curve obtained by the disk pressure test at 18ºC.

The left branch of the curve presents an anomaly, because it shows the reduction of the stresses acting in the material, whilst its deformation increases. This anomaly can be explained by the border effect of our model and precisely by the transition from the deformation of a flat membrane (requiring a different calculation), to that of a spherical cupola. Observed anomaly (which appears amplified thanks to the logarithmic scale), is limited to small strains, under 2%, whilst for larger ones, up to 50%, the shape of the curve is normal and can be used to study the material behavior up near its rupture limit.

Strain-hardening parameters

It is expected that the deformation of the steel during the test leads to its hardening. To characterize this effect, we have used the Hollomon's equation [7]:

$$\sigma_T = K\varepsilon_T^n, \quad (6)$$

where, *K* and *n* are respectively the strength index and the strain- hardening exponent.

Using the logarithmic expression of this equation:

$$\log\sigma_T = n\log\varepsilon_T + \log K \ , \quad (7)$$

we have processed the results of the test conducted at 18ºC (Fig. 4).

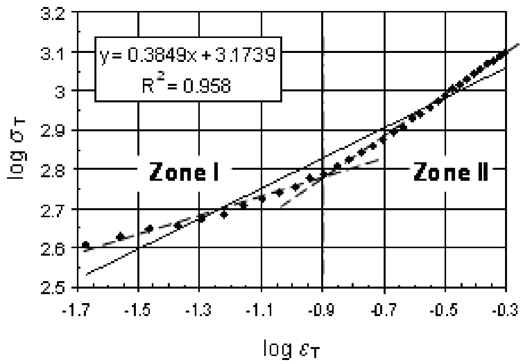

Figure 4 Relationship between *log*σ_T and *log*ε_T (test at 18ºC).

The function logσ_T = f (logε_T) presents obvious deviation from linearity, which means that hardening parameters vary with the deformation. For this reason, the alignment of all data in the interval 0.02 ≤ ε_T ≤ 0.50 with the equation 7, doesn't give a very strong correlation (R^2 = 0.958). A more careful observation shows that this alignment changes its character for ε_T ≈ 13% (log ε_T ≈ - 0.9). Based on this finding, we have distinguished two deformation zones and for each of them we have calculated the parameters of the equation 7. For the first zone (-1.7 ≤ log ε_T ≤ -0.9), we have found $\sigma_T = 966\varepsilon_T^{0.2324}$ with R^2 = 0.978, whilst for the second one (-0.9≤ log ε_T ≤ -

0.39), $\sigma_T = 1810\varepsilon_T^{0.5357}$ with R^2 = 0.998. The comparison of strain-hardening parameters for both zones is illustrated in Fig. 5.

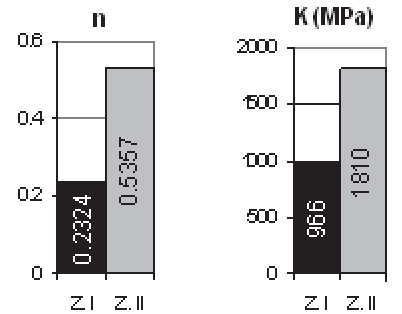

Figure 5 Comparison of the strain-hardening parameters, *n* and *K*, for the zones I and II.

The same analysis, performed with the tensile test results (Fig. 6), identifies almost the same behavior of the material in the first zone, whilst in the second one, the results diverge. So, values of *K* and *n* for the second zone, obtained by the tensile test, are about 25% lower than those of the disk test.

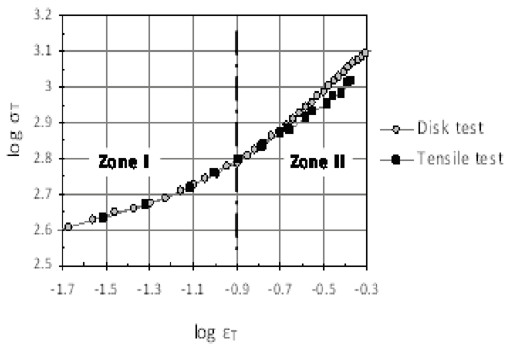

Figure 6. Comparison of $log\sigma_T - log\varepsilon_T$ curves for disk and tensile tests at 18ºC

The differentiation of the steel behavior can not be explained by anything else, except by the effect of a structural transformation, namely a martensitic one, induced by the plastic deformation, when the last one exceeds a certain rate [8, 9]. In the first zone, deformation is insufficient to cause a martensitic transformation, so there is a low strain-hardening, as much as the austenitic structure itself enables. Meanwhile, hardening effect is significantly amplified in the second zone by the creation of martensite. The above reasoning is confirmed by the diffractograms of ruptured disks, which have revealed the presence of martensite. Tensile test, although confirming the dual steel behavior, differentiates less the two zones. Thus, if the disk test shows an increase of the exponent *n* by 2.31 times (in the first zone versus the second one), according to the tensile test, this increase is only 1.85 times. The same conclusion is applied to the index *K*. Different results in the second zone, must be explained by the main cause of the hardening effect (in this case), namely, the martensitic transformation. It seems that the disk test, thanks to the biaxial stress conditions, favors more this transformation, and therefore its effect turns out great.

Influence of temperature

The results of 10 tests conducted at temperatures of 18-650ºC and processed with the analytical model, are shown in Fig. 7.

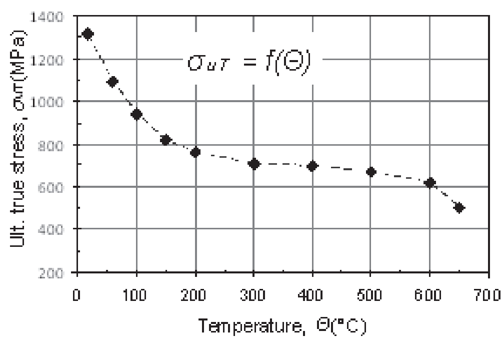

Figure 7. Effect of the temperature on the ultimate true stresses

It is obvious that the increase in temperature reduces the rupture stress of the material, as well as its ductility (see also Fig. 2). Such an evolution cannot be explained simply by the normal 'softening' that materials experience with the temperature rising (because it would be accompanied by the increasing of the ductility), but mainly by the reduction of the martensitic transformation hardening effect. In fact, the rate of this transformation for the AISI 321 steel, gradually decreases with the temperature rising and becomes practically negligible for temperatures above 150ºC [10]. Processed results of the test performed at 400ºC, show that even at this temperature the material behavior remains differentiated in two zones, but this differentiation is much smaller. Specifically, if at 18ºC, the exponent *n* increases by 2.31 times, at 400ºC this increase is only 1.8 times. Since the last one cannot be explained by the effect of martensitic transformation, it appears that within the austenitic structure operates a hardening mechanism more complex than that modeled by Hollomon's equation. The confirmation of this assertion requires further investigation, but we can say that for all practical purposes, the assessment of the hardening rate at 400ºC (Hollomon's equation), remains valid, since the overall correlation in both zones is over 0.98.

Simultaneous influence of temperature and time are studied by keeping the gas pressure in the cell unchanged. These tests, which characterize the material behavior in accelerated creep, are performed at two temperatures: 600 and 620ºC (fig. 8).

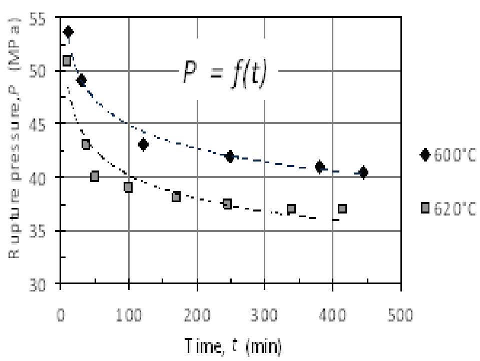

Figure 8 Effect of the test duration at 600 and 620ºC on the rupture pressure.

It seems that the influence of the time in the decreasing of the rupture pressure is reduced with time duration of the test. So, the two first hours of stay at high temperatures cause a decrease in rupture pressure of about 20%, whilst the prolongation of the stay from 5 to 7 hours causes a reduction by only 3-3.5%. The relationships found allow predicting the rate of strength decrease during a not very long stay (in the case of a breakdown, for example) at temperatures close to 600-620ºC.

CONCLUSIONS

1. The disk testing can be successfully used for characterizing the materials behavior under biaxial loading, giving a more direct information (particularly, in the case of pressure vessels) than the tensile test. For true strain rate up to 20%, the results of both tests match well.
2. The disk test is more sensitive to hardening phenomena related to the deformation of the material; it highlights better the hardening effect caused by the martensitic transformation.
3. The calculation model, used in this study, reliably simulates the material behavior in the area between the onset of yielding and the point at which necking begins. It does not cover the elastic region.
4. The rise of temperature up to 650ºC leads to an important reduction of the load-bearing capacity of the investigated steel (more than 2.5 times). The duration of the tests affects the steel in the same direction, but this influence tends to decrease with time.

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