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Development of a methodology to study the hydrogen embrittlement of steels by means of the small punch test

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ABSTRACT

Two different methodologies for analysing the deterioration of mechanical properties due to hydrogen embrittlement by means of the small punch test (SPT) have been studied. In the first, specimens were electrochemically pre-charged before testing, while in the second, they were charged at the same time as testing. A novel, simple, easy-to-manage SPT device was developed for the latter purpose. Two different CrMoV steel grades, a base and a weld metal, tempered at different temperatures, were tested. Tensile tests of hydrogen pre-charged specimens as well as hydrogen content measurements were also performed. Greater hydrogen absorption was observed in the higher strength CrMoV weld metal due to its microstructure composed of low tempered bainite. This steel was fully embrittled in both tensile and small punch tests in the presence of hydrogen, and no significant difference between the two SPT methodologies were found in this case. The CrMoV base metal was only embrittled, however, when hydrogen charging was performed at the same time as testing, showing the greater suitability of this small punch test methodology. The fracture pattern of SPT specimens changed completely from ductile to brittle when testing in hydrogen. Typical SPT parameters also exhibited a marked decrease in ductility and fracture toughness, the CrMoV weld metal being more susceptible to hydrogen embrittlement. Finally, the feasibility of the small punch test for ranking the hydrogen embrittlement susceptibility in steels was demonstrated, and the most suitable SPT parameters for analysing the reduction in mechanical properties were defined.

1. Introduction

1.1. Background

Hydrogen embrittlement (HE) is a process by means of which the mechanical properties of metals become degraded [1]. Its study is very important in the case of equipment exposed to aggressive environments, such as vessels or pipes employed in the power industry [2–4], off-shore platforms [5] and hydrogen powered vehicles [6,7]. It is also an issue that must be taken into account during manufacturing processes, as hydrogen could be introduced during welding, acid cleaning or in electrolytic coatings [8,9].

The mechanisms which hydrogen employs to damage steels are still relatively unknown. Roughly speaking, they can be divided in three different types of embrittlement [1]:

1. Hydrogen blistering (HB): hydrogen reacts with the surface of the steel, leading to the formation of blisters which act as crack initiators.
2. Environmental hydrogen embrittlement (EHE), which starts with the adsorption of environmental molecular hydrogen by the surface of the steel, followed by absorption within the lattice after dissociation into the atomic form.
3. Internal hydrogen embrittlement (IHE), produced when hydrogen is introduced into the steel in the course of the manufacturing process.

Different methodologies for testing hydrogen embrittlement have been investigated since the publication of the ASTM Selected Technical Paper in 1974 [10]. The most important test applied by industry is the tensile test at low strain rate, the so-called slow strain rate test (SSRT) [2,6,7,11]. However, several researchers have also employed conventional tensile tests with good results [3,12,13]. Both types of test may be performed inside an aggressive environment which provides hydrogen to the steel during testing [6,11], or immediately after pre-charging the specimen with hydrogen [5,7,8,12,13].

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The small punch test (SPT) is a quasi-non-destructive test which employs a small amount of material, generally small disks of about 10 mm in diameter and 0.5 mm thick. It was developed in the early 1980s with the aim of estimating the grade of deterioration caused by irradiation in nuclear vessel steels [14,15]. Since then, this miniature test has gained importance due to its capability for estimating (or more properly, ranking) the tensile mechanical [15–17], fracture [15,16,18] or creep properties [19–21] of metallic alloys with high accuracy. A European Code of Practice [22] was developed in 2006, confirming the current importance of the SPT, with several research groups worldwide pushing for its standardization [23].

Several studies have been carried out aimed at estimating hydrogen embrittlement (mainly EHE) deterioration by means of the small punch test [5,24–27]. In a pioneering paper published in 1988, Misawa et al. [24] developed a small punch (SP) testing apparatus using miniaturized specimens submerged in high temperature and high pressure aqueous solutions to assess the resistance to stress corrosion cracking (SCC) and the corrosion of candidate structural steels in water-coolant environment under irradiation. More recently, different researchers have sought to estimate the effect of HE by means of the SPT following two strategies: pre-charging the specimens immediately before testing [5,25], or charging the specimens at the same time as testing [26,27]. The first methodology has the drawback that hydrogen can diffuse out during testing. The main problem with the second methodology is that a special testing device must be used, in some cases employing hydrogen gas at high temperature and pressure, thereby increasing the risk associated with the test.

In order to clarify which SPT methodology might be the most suitable for estimating the deterioration induced by EHE in structural steels, different small punch tests were performed at room temperature, both pre-charging the specimens before testing and charging them at the same time as testing. A novel, simple, easy-to-manage device was developed for the latter purpose. Two different grades of CrMoV steels employed in the petrochemical industry were analysed: a high strength weld metal, which is very susceptible to HE [1,25]; and a base metal usually employed in equipment submitted to hydrogen atmospheres and hence much less susceptible to this phenomenon. Tensile tests with pre-charged specimens were also conducted in order to compare the results with those obtained by the SPT.

1.2. Small punch test parameters and correlations

Fig. 1 shows the characteristic load (P)–punch displacement (d) curve of a ductile metallic alloy, in which different zones have been defined. Zone I corresponds to the elastic bending of the sample along with the indentation produced by the contact of the head of the punch on the surface of the sample. Zone II describes the progressive spread of plastic bending to the entire sample. From a certain moment onward, plastic bending leads to a membrane regime (general plastic deformation), which predominates in most of the curve, this phase corresponding to Zone III. On approaching the maximum load, the slope of the curve decreases due to the failure micromechanisms that arise, resulting in Zone IV, where a visible crack is finally produced and the load decreases until total failure of the specimen occurs.

According to a previous study [16], the SPT load $P_{y\_t/10}$ (calculated at the offset $t/10$, where t is the initial specimen thickness) seems to be the most suitable parameter for estimating the yield strength of structural steels by means of expression (1), where the coefficient α depends on the material type and testing setup.

$$\sigma_{ys} = \alpha \frac{P_{y\_t/10}}{t^2} \quad (1)$$

$$\sigma_{ut} = \beta_1 \frac{P_m}{t^2} \quad (2)$$

$$\sigma_{ut} = \beta_2 \frac{P_m}{(d_m t)} \quad (3)$$

$$\varepsilon_{qf} = \ln\left(\frac{t}{t_f}\right) \quad (4)$$

With regard to the estimation of the ultimate tensile strength, both expressions (2) and (3) are found in the literature [15–17,23], expression (2) being the most common (P_m is the maximum test load and d_m the displacement at maximum load). Once again, the constants β_1 or β_2 are dependent on the material and test setup.

On the other hand, some authors have related d_m with elongation at failure, but the relationships thus obtained are highly dependent on the tested material [16,17]. Nevertheless, it seems to be a good parameter for comparing the ductility of structural steels (the larger the d_m, the greater the ductility). Another parameter that can be related to the toughness of steel is W_m,

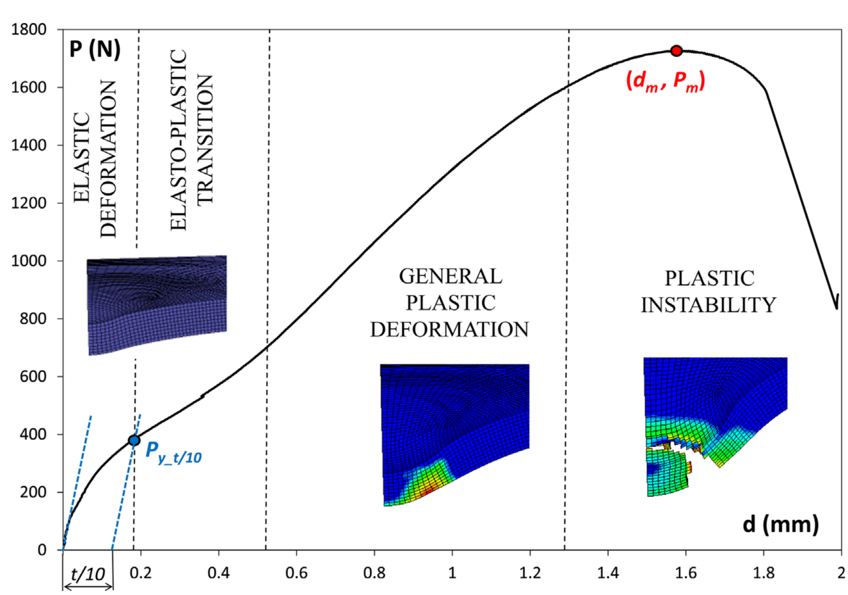

Fig. 1. Typical SPT curve of a structural steel.