DIFFERENT METHODOLOGIES TO OBTAIN THE FRACTURE PROPERTIES OF METALLIC MATERIALS USING PRE-NOTCHED SMALL PUNCH TEST SPECIMENS

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ABSTRACT

One important topic in the application of the Small Punch Test (SPT) is to analyse its viability for the determination of the fracture properties of a material. This article describes three different approaches using pre-notched SPT specimens (p-SPT): the first one is based on the concept of crack tip opening displacement (CTOD), the second is based on measuring the area under the load-displacement curve for different initial pre-notch lengths, and the third approach is based on the numerical simulation of the specimen p-SPT for the J-integral determination. To explore the feasibility of this miniature test for obtaining the fracture properties of steels the test values obtained by the different approaches are compared with results obtained from conventional compact specimens C(T). The results indicate that p-SPT specimens can be used, when there is not enough material for conducting conventional fracture test, as an alternative method to estimate a value of the fracture properties under low constraint conditions.

KEYWORDS: Fracture, Small Punch Tests, Notches

1. INTRODUCTION

The Small Punch Test (SPT) is a mechanical testing method in which a small square or disk shaped sample (thickness of 0.5 mm) is clamped in a die and punched until fracture with a spherical indenter. This reduced specimen size means that the SPT can be regarded as an almost non-destructive test that can be used to assess the in-service degradation of materials produced under thermal or irradiation conditions. The result of the test is a load-displacement curve, in which the

most important parameters to be extracted are the yield load, the maximum load, the displacement at failure and the area under the curve.

During the last decades, a great number of works can be found in the literature with the main objective to obtain the elastic plastic material properties, elastic modulus or tensile strength using un-cracked SPT specimens [1-5]. Most studies to date are focused on metallic materials, although SPT applications are recently spreading to other materials [6, 7].

The application of the SP test has gained significant interest in Europe over the last decades, primarily as a result of a coordinated research by the European Pressure Equipment Research Council (EPERC) to develop a CEN Code of Practice for the application and use of the small punch test for creep rupture, tensile and toughness properties [8-9].

Over recent years, there have been several studies dedicated to obtaining the fracture properties of a material using conventional SPT's, which provide empirical equations for the estimation of a fracture initiation parameter based on the measure of the maximum thickness deformation undergone by the specimen at the moment of fracture [10-12]. In a first approach, the absence of a crack or crack-like notch reduces the precision of those methods.

Recently, a number of works [13-17] can be found that use SPT specimens, notched or pre-cracked (denoted here as p-SPT), to validate such miniaturized specimens for obtaining the fracture properties of metallic materials. Different techniques can be applied to generate the crack-like notch, such as micro-machining, laser micro-cutting or Electric Discharge Machining (EDM) [18].

The main objective of this paper is to analyze the feasibility of the pre-cracked SPT's to obtain a representative value of the fracture parameter of metallic materials. In order to obtain the fracture parameter from these specimens three different approaches have been considered here.

The first method is based on the *crack tip opening displacement* (CTOD) concept [19]. The opening of the notch can be considered as a representative parameter of the crack initiation. This technique can be considered as purely experimental and it needs to obtain the crack initiation point by means of interrupted tests. After that, a SEM analysis of the miniature specimen tested is required. The second approach is based on the measurement of the *fracture energy*, using the area under the load displacement curve for different crack sizes [20-22]. And finally, the third approach is based on the

direct numerical simulation of the p-SPT specimen and the *numerical calculation of the J-integral* as a representative parameter of the material fracture [23-26].

In order to validate the methodology proposed in this paper, the fracture properties of the material obtained from p-SPT have been compared with those obtained from conventional fracture tests. The compact tension (CT) specimen has been used for the experimental determination of the fracture toughness of material, following the standard ASTM E1830 [27]. The materials selected for this research exhibit a ductile behavior, in order to allow a comparison of the results from notched specimens (p-SPT) with cracked specimens (CTs).

2. GEOMETRY OF THE p-SPT SPECIMENS

The experimental device used in this research is the proposed in the CEN code of practice. The diameter of the punch was $d_p = 2.5 \text{ mm}$, the diameter of the lower die was $D_d = 4.0 \text{ mm}$ and the accord radius of the lower die was $r = 0.5 \text{ mm}$. The initial geometry of the specimens used in this research was square of $10 \times 10 \text{ mm}$ with a thickness of $t = 0.5 \text{ mm}$. The process zone of this squared geometry is the same that in the disc shape specimen, 8 mm in diameter, proposed in [8], being the only difference between both geometries the area clamped by the upper and lower dies. The tests were conducted at room temperature using a displacement rate for the punch of 0.01 mm/s .

An initial notch was created in the SPT specimens by means of different techniques. In this study, the type of pre-notching used on the specimens was of the through-thickness crack type, from the middle point of one side to a point just passing the center of the specimen. This pre-notching was done using wire electrical discharge machining (WEDM), transverse laser-induced microcracking (TLIM).

The first technique, WEDM, once properly adjusted, yields a pre-notch in the desired position in one step. In the TLIM technique a laser beam is applied in several passes, using a $30 \text{ }\mu\text{m}$ diameter Nd:YAG pulse beam (50W and 1064 nm), working at a frequency of 7500 Hz and a linear displacement rate of 15 mm/s . The laser is applied perpendicularly to the notch direction. The notch shape resulting from this technique can be seen in Figure 1. A tip notch radius of about $200\text{-}250 \text{ }\mu\text{m}$ is obtained for WEDM and $75\text{-}100 \text{ }\mu\text{m}$ for TLIM.

Different notch lengths were machined to a length of a equal to 4.0, 4.5, 5.0 (center of the specimen), 5.5 and 6.0 mm respectively. Taking into account the distance of the notch relative to the punch center, the specimens are referred as: (-1.0), (-0.5), (+0.0), (+0.5) and (+1.0) mm respectively.

Figure 1. p-SPT specimen and the notch obtained by laser micro-cutting.

3. OPTIMUM INITIAL PRE-NOCHT LENGTH FOR p-SPT

The objective of practicing a pre-notch in the SPT specimen is to ensure the crack propagation during the test, which allows the application of the different fracture mechanics approaches to be applied.

In order to determine the optimum pre-notch length range, machined in the SPT specimens, for which a crack propagation on the specimen takes place, Alegre *et al* [13,14] have conducted several tests on specimens p-SPT with different notch lengths from 4 mm to 6 mm, being 5 mm the corresponding position of the load line of the punch. The material used in this research was a 15.5pH stainless steel.

Figure 2 shows the representative curves associated with each notch length tested. A clear difference is observed in the load-displacement curve for different notch sizes, and this difference is associated with the failure mode of the specimen.

Figure 2. Load-displacement curves representatives of different pre-notch length for *p*-SPT specimens.

In this sense, Figure 3 shows the mode of fracture of the specimens for different depths of the initial notch. The first one corresponds to the typical circumferential fracture for ductile materials usually observed in conventional unnotched SPT specimens. In the case of a SPT specimen with a pre-notch length of $a = 4.0 \text{ mm}$, a circumferential fracture without crack propagation in the direction of the notch was obtained. For those cases, with a notch length immediately preceding the line punch ($a = 4.5 \text{ mm}$) to another immediately passing the line punch ($a = 5.5 \text{ mm}$), a crack advance in the longitudinal direction of the initial notch was clearly observed. The crack advance was greater for lower a values, as a consequence of a greater ligament of the specimen to propagate. Finally, for initial notches greater than $a = 6.0 \text{ mm}$ a large deformation of the notch tip was observed allowing the punch to pass without detecting significant crack propagation in the direction of the notch.

Figure 3. Failure mode observed for the different pre-notch SPT specimens.

As a consequence, it is possible to assume an interval for the length of the initial pre-notch, in which the crack extension during the test takes place in the same direction as the initial pre-notch. Such an interval has been found to be for initial pre-notch lengths between 4.5 mm and 5.5 mm, being 5 mm the load line of the punch.

4. FIRST APPROACH: CTOD MEASUREMENT

The first method proposed for the estimation of fracture mechanics parameters is based on the measurement of notch tip opening displacement (similar to CTOD parameter in standard tests) in p-SPT specimens at the time when crack initiation process is taking place. This CTOD-like

parameter, also known as “ δ ”, is strictly a geometrical parameter which can be defined as the opening experienced by the crack –or the notch- during a loading process [15].

The fracture resistance obtained in terms of “ δ ” can be expressed as equivalent “K” or “G” just by applying basic Fracture Mechanics theories [19]:

$$CTOD = \frac{K_I^2}{m \cdot \sigma_y \cdot E} = \frac{G}{m \cdot \sigma_y} \quad (1)$$

where m is a non-dimensional constant which takes approximately a value of $m=1$ for plane-stress conditions and $m=2$ for plane strain.

The control of notch opening value at crack initiation time is particularly relevant. By the similarities with the processes in conventional fracture tests, that instant could be correlated with the moment when, after the blunting of original crack, tearing starts in standard specimens.

With the aim of detecting the crack initiation instant, important for further fracture toughness estimations, two different experimental approaches can be used.

First of them consists of a direct evaluation of notch opening during the tests by installing an endoscope-type-camera in the lower die hole of SPT device. This endoscope allows notch opening during SPTs to be live observed (Figure 4) [15-16].

The second methodology consists of performing interrupted tests, pausing them close to maximum force, just in the region where crack initiation has been observed, as can be seen in the examples of Figures 5 and 6 [13-14]. The notch tip of interrupted test is analyzed by Scanning Electron Microscopy (SEM) or Optical Microscopy in order to measure notch opening in the lower surface of the specimen.

The crack initiation is considered at the moment in which the maximum load is reached. As can be observed in Figure 5, a great damage in the notch tip occurs just before the maximum load is applied, however the crack initiation is not clearly evident until the maximum load is applied (Figure 6). This approach is very common in fracture mechanics, as the fracture characterization

using standard CT specimens, where the crack initiation is considered when it reaches a conventional finite value (0.2mm).

Figure 4. Evolution of notch tip during a p-SPT.

Figure 5. Crack initiation close to maximum force for a sample with an initial notch $a = 5$ mm (a) general view, (b) enlarged view.

Figure 6. Crack propagation beyond maximum force point.

On the other hand, the research carried out by Lacalle *et al* [15,16] has concluded that, once fixed the initial notch length, the relationship between notch opening and specimen deflection (or punch

displacement) until crack initiation instant, just depends on geometrical considerations, that is, it does not depend on the properties of the analyzed material. As a consequence, a set of master curves for different values of initial notch lengths has been generated by Finite Element simulations (Figure 7). These simulated curves allow the specimen deflection (or punch displacement) to be directly linked with notch opening values at any instant during the tests.

Figure 7. Relationship between Notch opening and Punch Displacement for initial notch lengths in the range of 4 to 5 mm.

Assuming that the crack initiation in p-SPT can be associated with the discontinuity in the slope of the test curve (near the maximum force point) and just entering in the Figure 7 with the Punch Displacement Value registered for that point, a value of “ δ_{SP} ”, characteristic of the fracture process of the specimen, can be estimated.

5. SECOND APPROACH: ENERGY RELEASE RATE

The energy release rate, defined as $G = -\partial U / \partial A$, represents the energy ∂U necessary to produce an increment of the cracked area of value ∂A . The typical procedure to obtain the G value experimentally is using a multi-specimen method. This experimental methodology consists of a

series of test specimens of the same size, geometry and material but with different crack lengths. Each specimen is deformed and a load-displacement curve is obtained. The area under a given curve is equal to the energy absorbed by the specimen (U). When the crack on a specimen initiates and extends from a value of a_1 to $a_2 = a_1 + da$, the energy release rate G can be obtained as:

$$G = -\frac{1}{t} \left(\frac{\partial U}{\partial a} \right)_{\Delta} = -\frac{1}{t} \left(\frac{U_2 - U_1}{a_2 - a_1} \right) \quad (3)$$

where U_1 and U_2 are the energy under the load-displacement curve of two different specimens with crack lengths a_1 and a_2 respectively, and t is the thickness of the specimen.

In order to apply this methodology to SPT pre-notched specimens is necessary to obtain the punch load vs. punch displacement (P - Δ) for a number of specimens with different initial crack lengths. Assuming that the energy stored in the specimen corresponds to the area under the P - Δ , and that from an engineering point of view the crack initiation takes place at the moment when the maximum punch load is applied to the specimen ($P_{\max}$), the energy release rate G can be obtained by testing two specimens with a different initial pre-notch length (Figure 8) and applying the expression (3).

Figure 8. Energy stored under the load-displacement curve for two specimens.

6. THIRD APPROACH: NUMERICAL CALCULATION OF THE J-INTEGRAL

The J -integral is a rate-independent, non-linear fracture mechanics parameter which can be used for fracture analysis under large plastic deformation. Numerical calculations of J -integral for cracked geometries are a common practice in elastic-plastic fracture mechanics analysis, and all major commercial FE codes allow for J computations.

Cuesta et al [28] have performed simulations with symmetric 3D models using p-SPT specimens with different notch lengths. The results obtained in the simulation represent a curve for each path, which defines the value of the J -integral versus punch displacement. Taking into account that the crack initiation will occur at the point of maximum load, it is possible to define a value of critical J -integral value J_c for crack initiation. This methodology is outlined in Figure 9.

Abaqus code has been used to compute the J -integral for pre-notched SPT specimens. The paths of integration for J -integral calculations were defined concentric to the notch tip. Three dimensional solid elements and the reduced integration option were used. The J -integral should be independent of the domain used provided that the crack faces are parallel to each other, but the J -integral estimated from different rings may vary because of the approximate nature of the finite element solution. Since the crack tip causes a stress concentration, the stress and strain gradients are greater as the crack tip is approached. Path dependence in the evaluation of the J -integral may be an indication that the mesh is not sufficiently refined or, if plasticity is included, that the contour integral domain does not completely include the plastic zone.

The geometry was divided into different regions, in order to construct a meshing with hexahedral elements, focused on the calculation of the J -integral.

Figure 9. Methodology used for numerical estimation of the J-integral value for crack initiation.

7. RESULTS AND ANALYSIS

In the work carried out by Lacalle *et al* [15,16], several p-SPT tests using four different structural steels grades (S235, S355, S690 and S890) have been performed. Pre-notching was done using wire electrical discharge machining (WEDM). In each case, 3 different initial notch lengths have been used (4.5, 4.8 and 5.0 mm). Following the procedure based on the estimation of the CTOD-like parameter, the values of δ_{SP} corresponding to crack initiation were obtained. These values were expressed in terms of J and then compared with J initiation values obtained for the same materials in conventional tests using C(T) specimens, with a thickness of $B = 20$ mm and side grooved. Figure 10 shows the comparison between the values calculated by both ways. As it can be observed the p-SPT approach results and conventional tests ones are within differences lower than 15%.

The same methodology using p-SPT samples has also been used in other works [29] with the objective of the characterization of ductile-brittle transition region of nuclear vessel steels. For those cases, and given than the plastic region in the vicinity of crack tip is relatively small, the comparison between p-SPT and conventional tests requires transferability analyses that account for thickness effects.

Figure 10. Comparison of initiation “J” values obtained in p-SPTs and in conventional fracture tests.

In the research carried out by Alegre et al [13,14] the three methods presented in this paper have been applied and compared, using for this an 15.5pH stainless steel. In this case, pre-notching was done using transverse laser-induced microcracking (TLIM). Figure 2 presents the load-displacement curves representatives of different pre-notch length, from 4 mm to 6 mm, for p-SPT specimens. Using these curves it is possible to apply the methodology presented, based on the calculation of the fracture energy (second approach). The detailed experimental set up can be found in the reference [14]. As an example of application, in Figure 11 is presented the experimental value of the energy release rate allowable for crack propagation, using as a base two specimens with a pre-notch length of 5.0 mm (SPT16 and SPT17) and two specimens with a pre-notch length of 5.5 mm (SPT11 and SPT12). According to equation (3) the subtraction of the energy stored between two specimens of different crack length (eg. SPT11 - SPT16) allows the energy release rate G to be obtained. At this point, having into account the punch displacement at the initiation that can be considered where the

maximum load in specimens is reached, the value of a fracture energy G can be obtained (in the Figure 11, a mean value of G is estimated).

Figure 11. Energy release rate obtained experimentally from p -SPT curves ($a = 5.0 - 5.5$ mm).

In a similar way can be proceed using the value of J-integral obtained from numerical simulations (third approach). In this case the value of J is directly obtained from a single specimen. In the example presented the pre-notched specimen was an initial value of 5.0 mm. The Figure 12 presents the value of the J-integral calculated for the first ten paths of integration.

Figure 12. Numerical calculation of J-integral for p-SPT with $a=5.0$ mm. 10 first paths are presented.

Finally, for estimating fracture properties using the method of the CTOD, several tests have been interrupted near the maximum load point. The CTOD value at this particular point has been measured, and it has been considered the crack initiation point as has been mentioned in paragraph 4.

In Figure 13, a comparison between the three approaches is presented. The value of the fracture toughness of the material, obtained using CT specimens of different thickness from $B = 20$ mm (Plane strain conditions) to $B = 5$ mm (Plane stress conditions), is also presented. The value of $J = G$ for p-SPT using CTOD approach is obtained using equation (1) for plane stress conditions. In this Figure, the CTOD-mean represents the CTOD calculated using both faces of the specimen thickness, and CTOD-max represent the CTOD calculated using only the maximum CTOD at the external face of the specimen (opposite to the punch contact face). As the crack initiation takes place throughout the entire thickness of the specimen, the CTOD-mean value can be considered as more representative of the global behavior of the specimen.

As a general rule, the multi-specimen approach based on the calculation of the energy release rate (second method) is the technique that provides less scattering on the results. This approach provides an estimation around the value of the CT-5 specimen, corresponding to low constrain conditions.

The main drawback of this method is the necessity to machine several specimens with different pre-notch lengths.

Figure 13. Comparison of the different approaches analyzed in reference [13-14]

8. CONCLUSIONS

In this paper an analysis of the different methods to obtain the fracture parameters of a material based on the use of pre-cracked miniature specimens (p-SPT) has been presented. The results indicate that p-SPT specimens can be used as an alternative method for determining the fracture properties of a material in those cases where there is not enough material to undertake conventional fracture tests.

The multi-specimen method for the determination of the fracture energy is the most promising approach, but the calculation of fracture properties, based on the CTOD approach can be easier to undertake. Results presented here indicate that this geometry allows the value of the material toughness to be obtained, mainly for low constraint conditions. These results are consistent with the geometry of the p-SPT specimens.

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