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APPLICATION OF THE SMALL PUNCH TEST IN COMBINATION WITH THE MASTER CURVE APPROACH FOR THE CHARACTERISATION OF THE DUCTILE TO BRITTLE TRANSITION REGION

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ABSTRACT

The Master Curve approach allows the full characterisation of the ductile to brittle transition region (DBTR) of ferritic steels to be performed with a reduced number of tests. In this paper, the approach has been combined with the application of the small punch (SP) tests. Modified SP specimens have been successfully employed to estimate the fracture toughness values of a pressure vessel steel and three structural steels. In addition, a methodology has been proposed, including a validity criterion for the performed tests. The estimated reference temperatures have been compared to the values obtained by means of full-scale conventional techniques. A unique simple relationship between both methodologies has been established for all the analysed materials. Therefore, this paper confirms the suitability of the small punch testing technique for the characterization of the DBTR of several ferritic steels. It is a promising, simple and cost-effective test, which can be performed with simple equipment.

KEYWORDS
Small Punch, Ductile to brittle transition region, Ferritic steels, Master Curve, Reference temperature, Fracture.

HIGHLIGHTS

- Small punch notched specimens can be applied to estimate the reference temperature.
- A methodology to estimate the reference temperature has been proposed.
- A single correlation has been obtained for the ferritic steels analysed.

1 INTRODUCTION

Damage-tolerant designs or structural integrity assessments require the characterisation of damaged material under working conditions to evaluate the ageing of components during their service life. In the nuclear industry, materials are exposed to irradiation, which can lead to embrittlement, among other effects. In order to guarantee the operation of nuclear components under safe conditions and perform their assessment, an accurate characterisation of these effects is critical.

One of the main parameters to assess the influence of the irradiation embrittlement on ferritic steels is the reference temperature, T_0, employed in the Master Curve (MC) approach [1-4]. It enables the characterisation of the ductile to brittle transition region (DBTR) with a reduced number of tests, thanks to a combination of mechanistic modelling and a statistical approach. Moreover, it addresses the dependence on the thickness of the test sample and the scatter of the results in the DBTR. As a result, this parameter has been incorporated in several standards and codes, such as FKM-Guideline [1], BS 7910 [2], API 579/ASME FFS [3] or ASTM E1921 [4], among others.

The determination of T_0 has usually been performed by means of conventional fracture toughness tests. These require relatively large volumes of material, which is often not feasible for challenging nuclear applications, e.g. for the extension of the operation period of nuclear plants, since the material in the surveillance capsules is becoming scarce [5]. However, the MC approach allows specimens of reduced thickness to be used [1-4]. As a result, large efforts are being done to develop small-scale testing techniques in combination with the MC, in order to further optimise the material [6]. For instance, micro-compact tension specimens of 10x10x4 mm have been successfully applied [7].

Currently, the small punch testing technique is one of the most promising miniature testing techniques. Evidence of this are the current efforts to establish a European standard to cover this methodology [8]. One of its most interesting applications is the estimation of fracture toughness by means of notched specimens of only 10x10x0.5 mm [9, 10], something which is not possible to achieve by means of other miniature testing techniques for such reduced volumes of material.

In this paper, the use of the Master Curve approach in combination with small punch notched specimens is proposed to estimate the reference temperature on a wide range of materials: a pressure vessel steel (HSST plate 13B/A533B plate) and three structural steels (S275JR, S460M and S690Q). A relationship between the reference temperature obtained by means of small punch tests and by means of conventional testing techniques has been established. As a result, a methodology to estimate the reference temperature from small punch tests has been proposed.

2 EXPERIMENTAL DEVICE AND MATERIALS

2.1 Experimental device and specimens

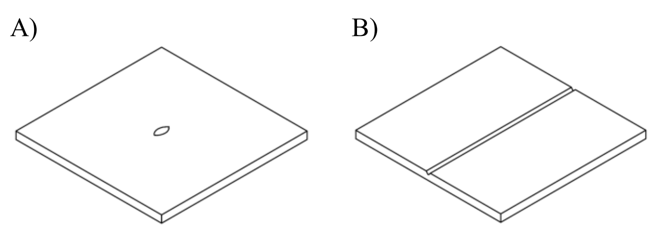
The small punch technique is a simple, cost-effective small sample test technique. It is a relatively recent test [11] that consists of punching a plate of only 0.5 mm-thickness with a 2.5 mm-radius punch passing through a 4 mm-diameter hole until rupture. Given the applicability of the technique and its potential there exists a European Code of Practice CWA 15627:2008 [12] and the technique is currently under standardisation process [8].

The testing procedure for the small punch tests followed in this paper is performed according to the recommendations of CWA 15627 [12]. Since the goal of this paper is the estimation of the reference temperature (or the characterisation of the ductile to brittle transition region), the tests have been performed at low temperatures. To achieve these conditions, the testing rig has been assembled inside an environmental chamber, which kept the temperature constant using a regulated input of liquid nitrogen. A universal machine with a load capacity of 2.5 kN has been employed, applying a crosshead displacement rate of 0.5 mm/min during the tests.

Regarding the specimens, the recommended geometry is 8 mm-diameter discs with 0.5 mm thickness [12]. In this paper, 10x10 mm square specimens of 0.5 mm-thickness have been employed. The use of this geometry eases the orientation of the specimens and enables the direct reutilisation of already tested Charpy specimens. Since the process area of the specimen under the punch remains constant and the clamped area is simply increased, it has already been proved that the use of this geometry does not influence the obtained results [13]. As a result, the use of 10x10 mm specimens will be also included in the future European standard covering the small punch test on metallic materials.

To estimate fracture toughness, a modification of the specimens has been introduced. A lateral notch of 4.4 mm and 0.15 mm-radius has been machined, as shown in Fig. 1 (C). The length of the notch has been chosen according to previous experience on materials with similar mechanical behaviours to guarantee the initiation of a crack during the test [5,9,10].

Several approaches have been employed to estimate fracture toughness to date. Some authors have correlated the equivalent fracture strain of ruptured unmodified small punch specimens with fracture toughness [14] or have established methods based on finite element simulations [15]. Other approaches are based on analytical solutions, which require the introduction of a defect on the specimen to initiate a crack; several geometries have been proposed to introduce such defect, such as the use of central notches [16], blind longitudinal notches [17] or circular notches [18], among others, as shown in Fig. 1. (A, B, D). The use of a lateral notch, chosen for this research, enables the orientation of the crack to analyse the direction of the material desired and its geometry can be easily machined by means of electrical discharge machining [9, 10]. In addition, it has shown a good correlation with conventional full scale tests [9, 10].



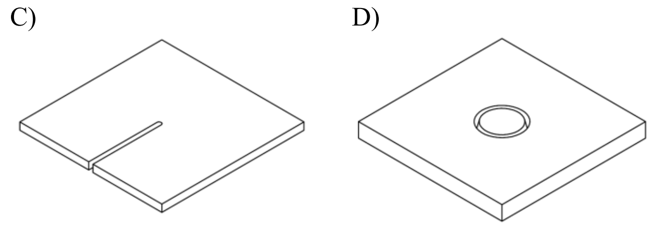


FIGURE 1: SMALL PUNCH SPECIMENS MODIFIED WITH A NOTCH TO ESTIMATE FRACTURE TOUGHNESS [19]: A) CENTRAL NOTCH [16], B) BLIND LONGITUDINAL NOTCH [17], C) LATERAL NOTCH [9, 10], D) CIRCULAR NOTCH [18].

The specimens have been prepared according to the recommendations of CWA 15627 [12]. Firstly, the notch has been machined on 10x10 mm-section prisms. After this first step, the resulting pieces have been cut to 0.55 mm-thick pieces by means of a liquid-cooled cut-off machine and then ground to at least 2000 grit on both sides, until achieving the desired thickness of 0.5 ± 0.005 mm.

In addition, to avoid any possible influence of the notch geometry on the results, a special lower die has been used in the testing rig. During the tests, there can be some clearance between the specimen and the side walls of the lower die. When small punch specimens without a notch are to be tested, this clearance does not introduce any changes to the results. However, with the introduction of the lateral notch, it can lead to misalignments which result in modifications of the notch length, for instance. Consequently, an adjustable lower die has been designed, manufactured and utilised for testing this kind of specimens, which enables the reduction of the clearance to a minimum (Fig. 2). It is a novel design that enables the adjustment of the upper part of the lower die to each specimen individually. Furthermore, the use of this adjustable lower die makes it easier to correct any possible deviations from the desired notch length on the specimen, by simply grinding one of the edges until achieving the desired geometry, something that it is not possible to achieve with other testing rigs.

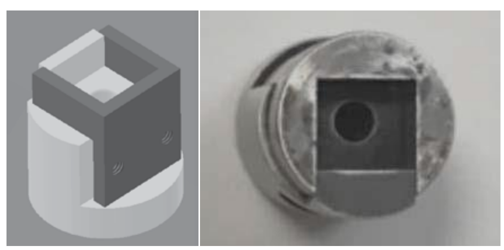


FIGURE 2: ADJUSTABLE LOWER DIE [20].

2.2 Materials

A pressure vessel steel (A533B 13B/A533B plate) and three structural steels (S275JR, S460M and S690Q) have been analysed in this research, covering a wide range of reference temperatures: from 160 to 250 K. Their chemical composition is shown in Table 1 and their mechanical properties are shown in Table 2. The reference temperature values have been obtained from (full-scale) 15 mm-thick SENB specimens for S460M and S690Q, [21], 10x10x55 mm pre-cracked Charpy specimens for A533B [22] and 25 mm-thick CT specimens for S275JR [23].

TABLE 1: CHEMICAL COMPOSITION OF THE STEELS ANALYSED (% WEIGHT) [21-23].

	A533B	S275JR	S460M	S690Q
C	0.12	0.18	0.18	0.15
Si	0.45	0.26	0.24	0.40
Mn	1.49	1.18	1.41	1.42
P	0.012	0.012	0.009	0.005
S	0.001	<0.009	0.005	0.001
Ni	0.016	<0.085	0.56	0.16
Cr	0.062	<0.018	0.18	0.02
Mo	0.001	<0.12	0.49	0.002
V	0.066	<0.020	0.005	0.058

Cu	0.011	<0.06	0.01	0.01
Ti	0.03	<0.022	-	0.003
Al	0.048	0.034	0.021	0.056
Fe	BAL.	BAL.	BAL.	BAL.

TABLE 2: MECHANICAL PROPERTIES OF THE ANALYSED MATERIALS [21-23].

	A533B	**S275JR**	**S460M**	**S690Q**
σ_y **[MPa]**	480	328	484	776
σ_u **[MPa]**	608	519	594	834
T_0 **[K]**	250	247	181	162

3 ESTIMATION OF FRACTURE TOUGHNESS BY MEANS OF NOTCHED SMALL PUNCH SPECIMENS

The estimation of fracture toughness by means of notched small punch specimens is based on the Crack Tip Opening Displacement (CTOD) approach [9, 10]. The CTOD approach, and in the same way the *J* contour integral, can be used as a fracture criterion under elastic-plastic fracture mechanics (EPFM) conditions, i.e. to materials that exhibit time-independent nonlinear behaviour [24]. According to this approach, under EPFM conditions, a crack experiences a certain degree of blunting before fracture. The degree of crack blunting at the moment of the crack initiation, δ, has been proved to be dependent on the material and can be directly used as a measure of fracture toughness [24]. Consequently, it can be directly correlated with the material critical *J* contour integral. Shih [25] established a relationship between both parameters by evaluating the displacements at the crack tip implied by the HRR solution [24], as shown in equation (1). It incorporates a dimensionless constant, d_n, that has a strong dependence on the strain hardening exponent of the material [25].

$$\delta = \frac{d_n J}{s_y} \qquad \textbf{(1)}$$

where s_y represents the yield stress of the material.

When a small punch test is being performed on a specimen with a lateral notch (Fig. 1 (C)), the notch experiences a deformation similar to notch blunting, as shown in Fig. 3. At a certain point, usually at maximum force, a crack initiates from the notch tip. On the Force-Displacement curve obtained during the test, this crack initiation can be identified as an abrupt change on its slope, as shown in Fig. 4 [9, 10].

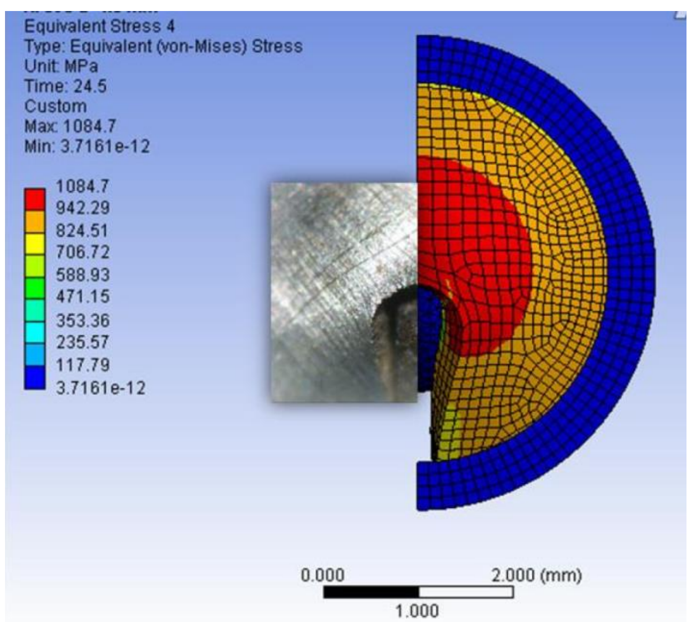


FIGURE 3: IMAGE OF AN INTERRUPTED TEST AND CORRESPONDENT FINITE ELEMENT SIMULATION SHOWING THE DEFORMATION EXPERIENCED BY THE NOTCH DURING THE TEST [13].

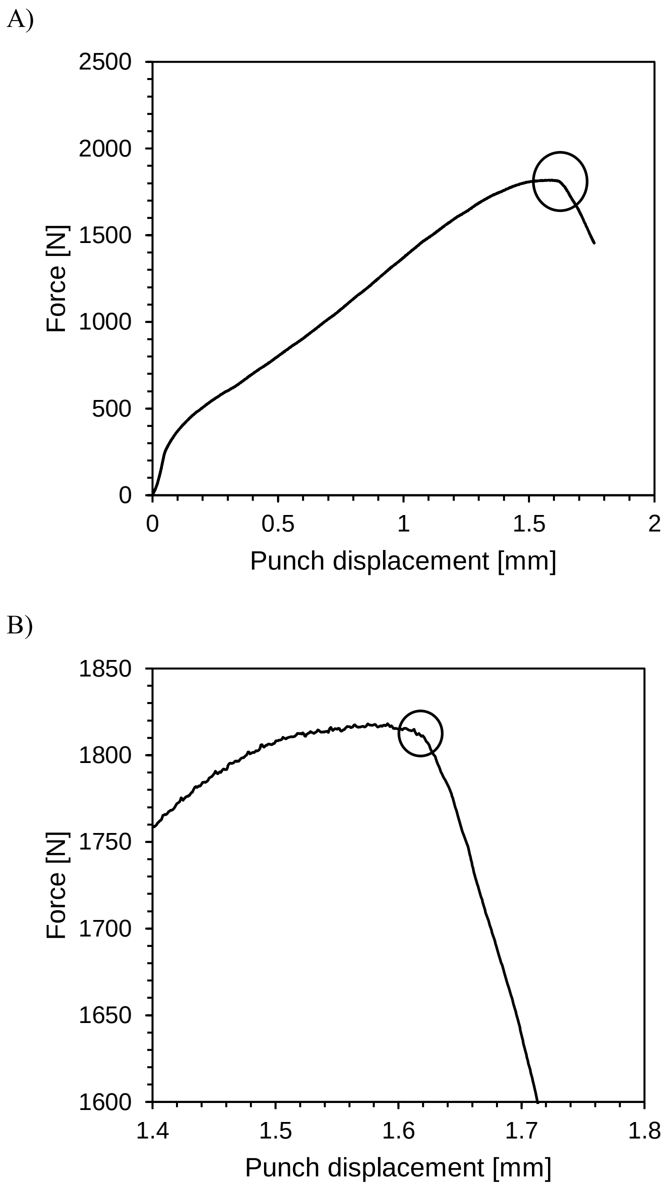


FIGURE 4: FORCE-DISPLACEMENT CURVE OF SMALL PUNCH TEST ON A NOTCHED SPECIMEN, ON WHICH THE CRACK INITIATION HAS BEEN IDENTIFIED: A) WHOLE CURVE, B) DETAIL OF THE ABRUPT CHANGE OF THE SLOPE.

By applying finite element simulations, a geometrical relationship between the punch displacement and the degree of blunting of the notch (δ_{SP}) for a given notch length can be obtained [9,10]. In this paper, a visco-plastic continuum mechanics finite element model realised via ANSYS version 18.0 was used to simulate a punch head going into a 10x10 mm sample with 4.4 mm-length notch with notch diameter of 0.30 mm. This simulation provided the curve shown in Fig. 5. Furthermore, since the relationship is material independent, the same estimated curve can be applied on all materials.

As a result, for each particular notch length, once the punch displacement at crack initiation has been obtained during the small punch test, the correspondent δ_{SP} can be identified by simply applying the corresponding δ_{SP} – Punch tip displacement curve, as that shown in Fig. 5. By appling eq. (1), this δ_{SP} value can be easily transformed into a SP measurement of the J-integral, J_{SP}, if the tensile properties of the material have already been obtained, something which can also be achieved by means of small punch tests. However, in this paper, the tensile properties of the materials analysed have been obtained by means of conventional tensile tests [21-23].

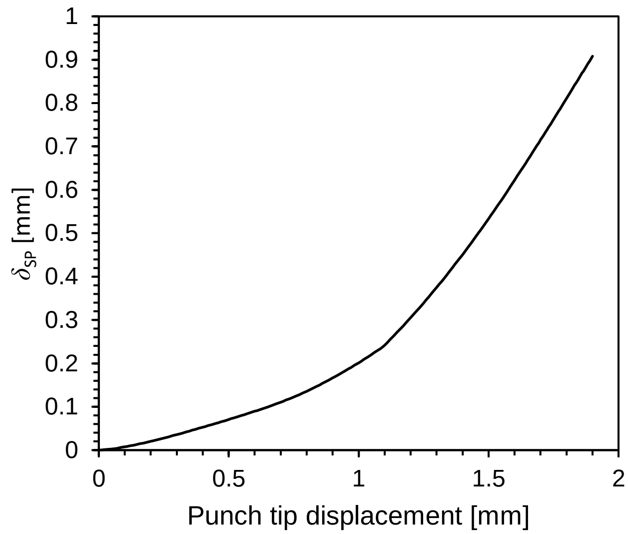


FIGURE 5: δ_{SP}-PUNCH TIP DISPLACEMENT CURVE OBTAINED FOR A 4.4 MM-LENGTH NOTCH BY MEANS OF FINITE ELEMENT SIMULATIONS.

Once J_{SP} has been determined, fracture mechanics relationships, such as eq. (2), can be applied to obtain the estimation of the material fracture toughness in stress intensity factor units, K_J^{SP}, at crack initiation [4, 24, 26].

$$K_{J^{SP}} = \sqrt{\frac{E\,J_{SP}}{1-\nu^2}} \qquad (2)$$

where E represents the Young modulus of the material and ν is the Poisson's ratio.

4 MASTER CURVE APPROACH

The Master Curve is a probabilistic approach that enables the direct estimation of fracture properties in the transition zone of ferritic steels [27]. According to this approach, the dependence of fracture toughness with temperature can be defined by a mathematical model and a single parameter: the reference temperature (T_0). It represents the temperature at which the median of the K_{Jc} distribution from 1T (25 mm thick) size specimens is equal to 100 MPa.m$^{0.5}$ and is the only material dependent parameter required [4].

In addition, this approach considers the (statistical) effects of the thickness of the specimens on the fracture toughness values, as well as the scatter of the results in the DBTR. This reduces significantly the number of tests needed to be performed.

As a result, T_0 can be obtained with reduced volumes of material and with relatively small samples [4]. This has turned the approach into an advantageous solution to characterise the DBTR of ferritic steels, evidenced by its incorporation into numerous codes and standards [1-4].

According to the Master Curve, the value of K_{Jc} for a given thickness (B, in mm) should be converted into the corresponding fracture toughness in a 1T (25 mm thick) specimen. This is performed by using eq. (3):

$$K_{Jc}\,[1T] = 20 + \left[K_{Jc}[B] - 20\right]\cdot\left(\frac{B}{25.4}\right)^{1/4} \qquad (3)$$

For the case being analysed, considering that the thickness of SP specimens is 0.5 mm, this implies:

$$K_{Jc}^{SP}\,[1T] = 20 + \left[K_{Jc}^{SP}[0.5] - 20\right]\cdot\left(\frac{0.5}{25.4}\right)^{1/4} \qquad (4)$$

5 RESULTS AND DISCUSSION

Small punch tests on specimens with a lateral notch of 4.4 mm have been performed at different temperatures, ranging from 158 to 93 K, for the estimation of the reference temperatures of the materials analysed. Figures 6 to 9 show the Force-Displacement curves obtained from the tests. Six tests have been performed per material, according to the recommendations of [4]. From the discontinuity on the slope of the curves, it can be seen that in all cases a crack has initiated during the test, which was also confirmed by means of visual inspection of the specimen after the tests. A detail of a ruptured small punch specimen can be seen in Fig. 10.

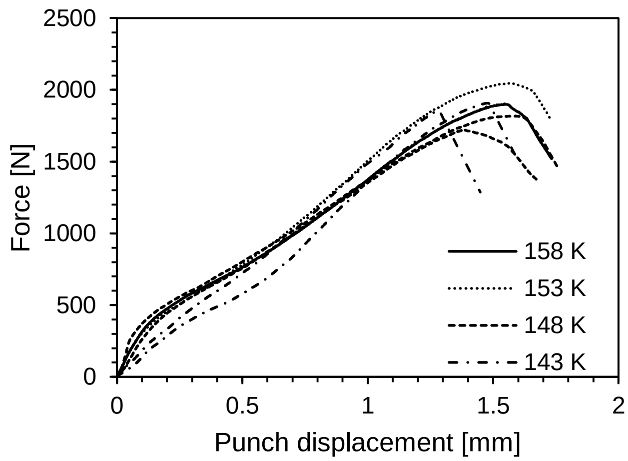


FIGURE 6: FORCE-DISPLACEMENT CURVES OBTAINED FOR A533B PLATE.

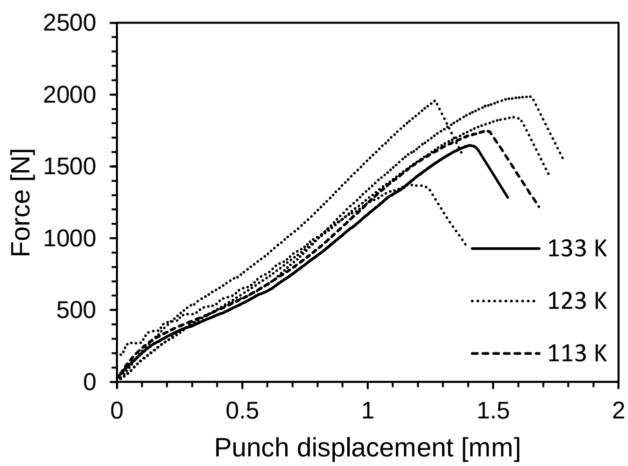


FIGURE 7: FORCE-DISPLACEMENT CURVES OBTAINED FOR S275JR.

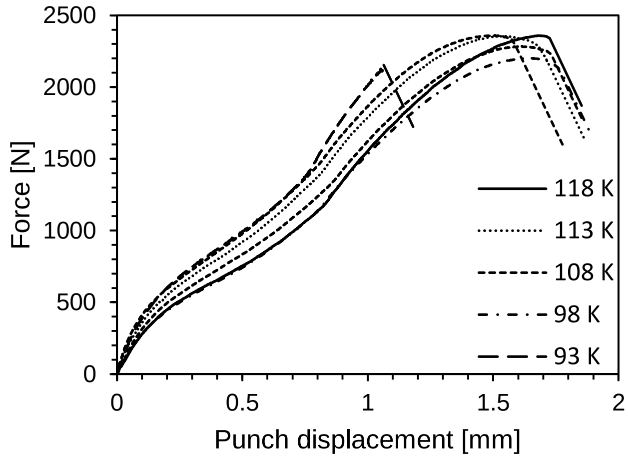


FIGURE 8: FORCE-DISPLACEMENT CURVES OBTAINED FOR S460M.

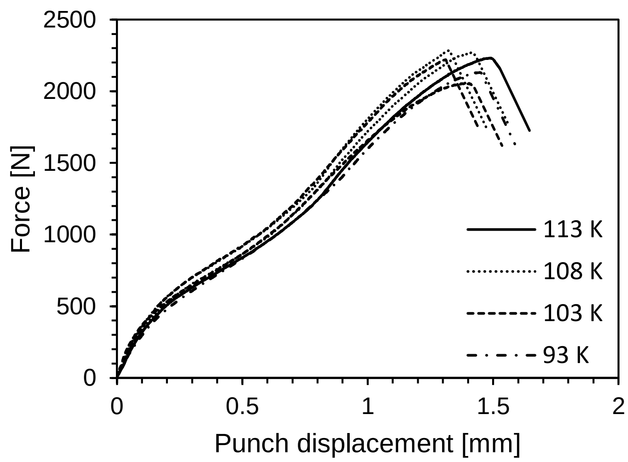


FIGURE 9: FORCE-DISPLACEMENT CURVES OBTAINED FOR S690Q.

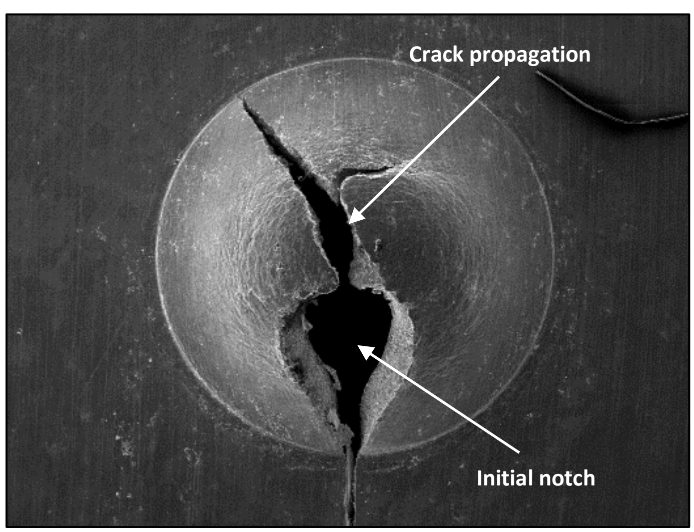


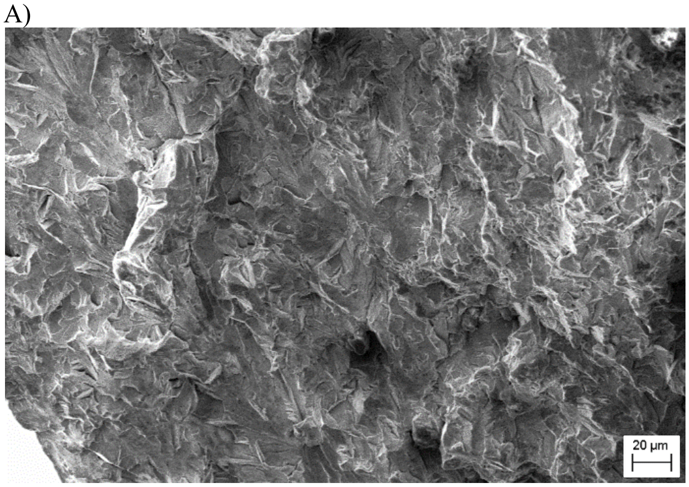
FIGURE 10: DETAIL OF A RUPTURED SMALL PUNCH SPECIMEN OF S690Q. THE DEFORMATION OF THE NOTCH CAN BE SEEN, WHICH HAS LED TO THE INITIATION OF A CRACK.

To guarantee that the Master Curve approach can be applied, the onset of cleavage cracking has been a criterion for the acceptance of the tests as valid for the estimation of the reference temperature. On the Force-Displacement curves, it can be identified as a sudden drop of the load at maximum force or as a discontinuity in its proximity [5]. Stable cracking, exhibiting ductile mechanisms, can be expected in those curves not exhibiting such discontinuity.

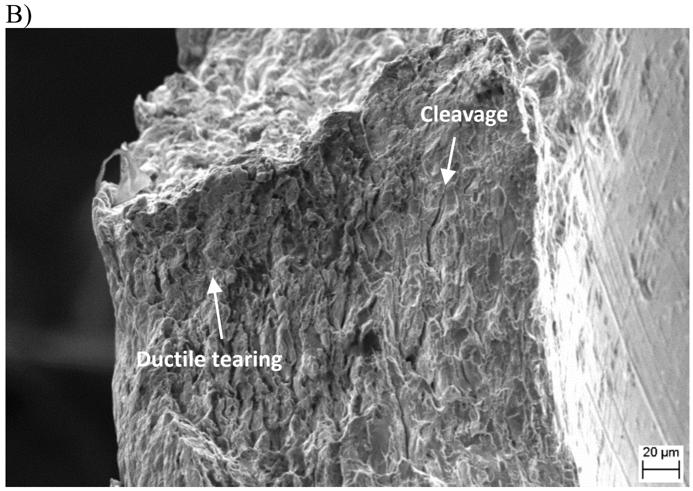
The validity criterion of the small punch tests for the estimation of the reference temperature has been validated with the analysis of the fracture micro-mechanisms present at the crack initiation, as shown in Fig. 11. It can be seen that those specimens suffering a sudden load drop at maximum force exhibit a brittle fracture (Fig. 11 A), while those with a discontinuity on its slope exhibit a mixed mechanism of fracture, with cleavages present on it after some ductile tearing (Fig. 11 B). Finally, tests performed at room temperature which do not show such discontinuity exhibit a fully ductile fracture, as shown in Fig. 11 C.

Consequently, it is recommended to analyse the micro-mechanisms in case of doubt after the performance of the small punch tests, in order to guarantee the validity criterion proposed here and the correct approximation to the master curve approach. This further analysis is not necessary for tests exhibiting a sudden load drop at maximum force, where a brittle fracture is guaranteed.

A)



B)



C)

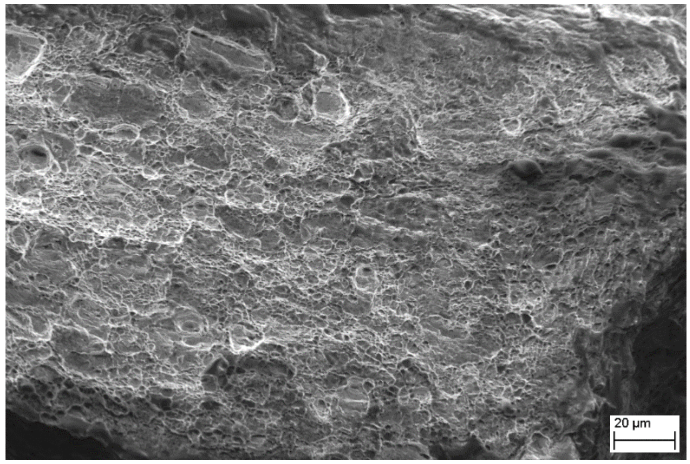


FIGURE 11: A) DETAIL OF A RUPTURED SMALL PUNCH SPECIMEN THAT EXPERIENCED A SUDDEN LOAD DROP AT RUPTURE, B) DETAIL OF A RUPTURED SMALL PUNCH SPECIMEN THAT EXPERIENCED A DISCONUITY ON THE SLOPE AT MAXIMUM FORCE, C) DETAIL OF A RUPTURED SMALL PUNCH SPECIMEN AT ROOM TEMPERATURE.

From the tests performed, K_{Jc}^{SP} [1T] values have been estimated following the aforementioned approach, the results being shown in Table 3. The tensile properties, which have been obtained at room temperature, have been transformed to their equivalent values at each temperature according to the recommendations of BS 7910 [2] before applying them to eq. (1), eq. (2) or to d_n [25].

TABLE 3: RESULTS OBTAINED FROM THE SMALL PUNCH TESTS PERFORMED.

MATERIAL	T [K]	δ_{SP} [mm]	J_{SP} [Kn/M]	K_{Jc}^{SP} [1T] [MPa.m$^{0.5}$]
A533B	158	0.58	429	133
	153	0.71	516	145
	148	0.64	485	141
	148	0.43	319	117
	143	0.37	283	111
	143	0.57	436	135
S275JR	133	0.53	356	123
	123	0.40	272	109
	123	0.35	238	103
	123	0.65	442	136
	123	0.63	429	134
	113	0.41	288	112
S460M	118	0.71	567	152
	113	0.86	698	168
	108	0.88	721	170
	108	0.70	573	153
	98	0.56	498	144
	93	0.19	170	89
S690Q	113	0.42	468	139
	108	0.30	345	121
	108	0.26	296	113
	103	0.52	605	157
	103	0.36	414	132
	93	0.54	647	162

The small punch reference temperature corresponding to each material has been determined according to the standard ASTM E-1921 [4]. In Fig.12 to 15, the small punch Master Curves for 5, 50 and 95% failure probabilities have been represented. The estimations obtained from the small punch tests fit accurately within the predicted curves, confirming the suitability of the methodology. The obtained T_0^{SP} values are shown on Table 4. It can be seen that the reference temperature obtained by means of small punch tests is lower than the values obtained by means of full scale conventional tests. Consequently, a relationship between both reference temperatures needs to be established. To achieve this goal, both values have been compared in Fig. 16.

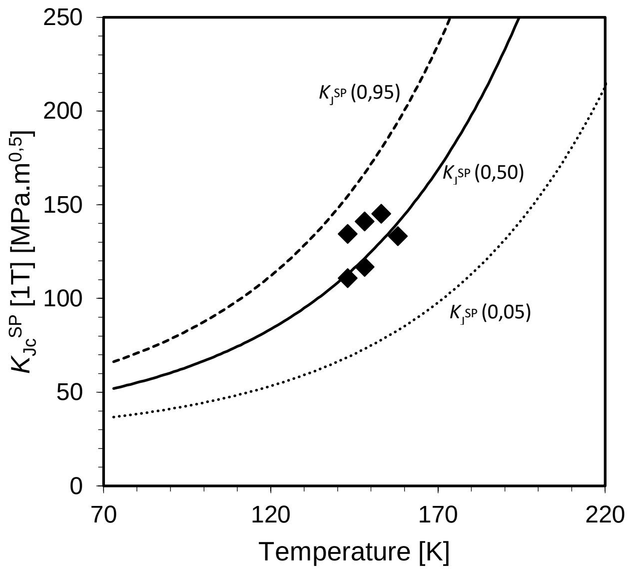


FIGURE 12: SMALL PUNCH MASTER CURVES OF A533B PLATE FOR 5, 50 AND 95% FAILURE PROBABILITIES.

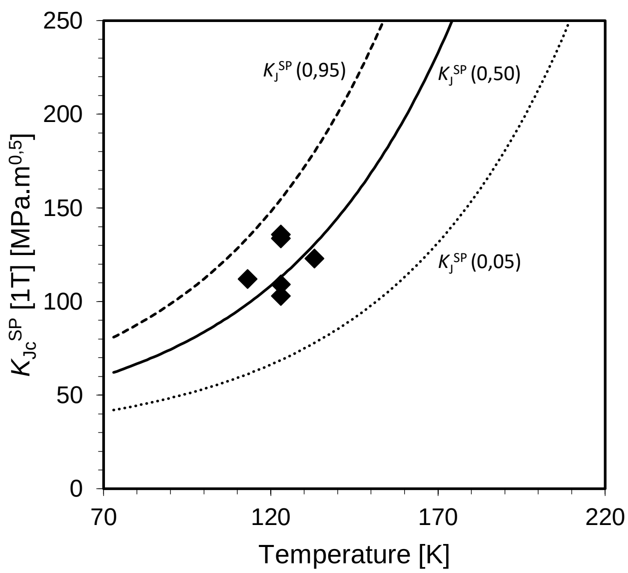


FIGURE 13: SMALL PUNCH MASTER CURVES OF S275JR PLATE FOR 5, 50 AND 95% FAILURE PROBABILITIES.

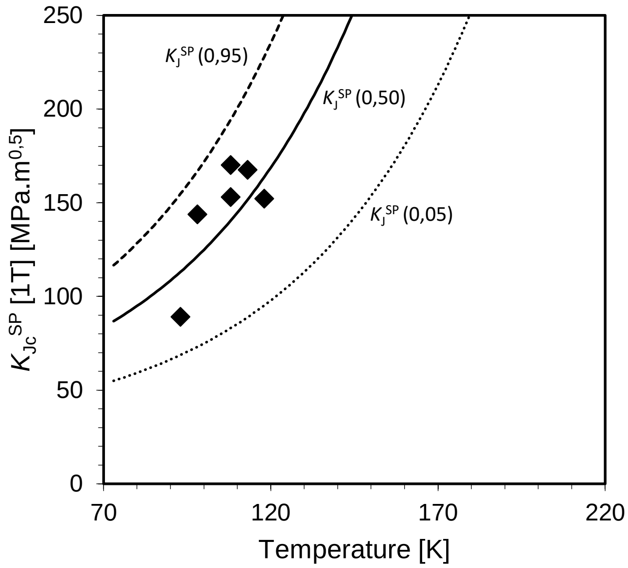


FIGURE 14: SMALL PUNCH MASTER CURVES OF S460M PLATE FOR 5, 50 AND 95% FAILURE PROBABILITIES.

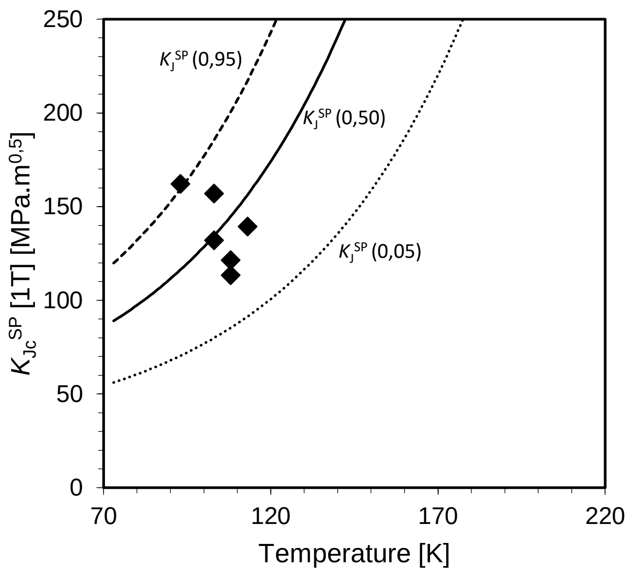


FIGURE 15: SMALL PUNCH MASTER CURVES OF S690Q PLATE FOR 5, 50 AND 95% FAILURE PROBABILITIES.

TABLE 4: SMALL PUNCH REFERENCE TEMPERATURE VALUES OBTAINED FOR EACH MATERIAL.

MATERIAL	A533B	S275JR	S460M	S690Q
T_0^{SP} **[K]**	134	114	84	82

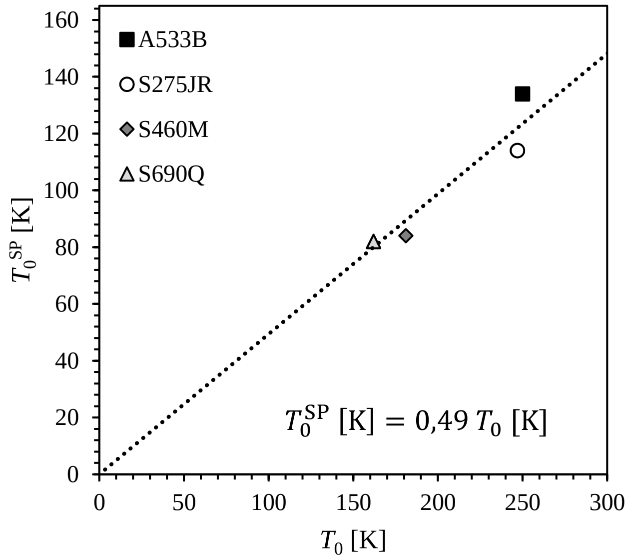


FIGURE 16: RELATIONSHIP BETWEEN THE REFERENCE TERMPERATURE OBTAINED BY MEANS OF SMALL PUNCH TESTS AND BY MEANS OF FULL SCALE CONVENTIONAL TESTING TECHNIQUES.

As seen in Fig. 16, the reference temperature, in K, obtained by means of small punch tests is approximately half of the corresponding value obtained by means of full-scale conventional tests, with accurate results for the four materials analysed. The difference between T_0 and T_0^{SP} might be not related to absolute specimen size, but to the influence of the specimen geometry and loading mode on the cleavage toughness, in a similar way to the differences between C(T) and SE(B) specimens or specimens with deep and shallow cracks [28].

The obtained coefficient is slightly different from the first value obtained in the first approach on materials SA-508M and S275JR [5], where instead of a 0.49 coefficient between T_0^{SP} and T_0, a coefficient of 0.65 was obtained. The reason for this shift could be due to the use of different testing rigs. In this research, special care was given to guarantee that specimen geometry tested was the same in all cases. This was attained by means of the use of a newly designed adjustable lower matrix. In the aforementioned research [5], conventional testing rigs were used, which could have led to some clearance between the specimen and the rig, therefore modifying the tested notch length. Further analysis of the influence of the notch length should be performed in the future to gain further understanding of its possible effects.

6 CONCLUSIONS

Small punch modified specimens, with a lateral notch, in combination with the Master Curve approach have been applied successfully to characterise the ductile to brittle transition region of a pressure vessel steel and three different structural steels. A methodology has been proposed, including a validity criterion for the performed tests. In addition, a single correlation between the reference temperature obtained by means of full-scale conventional tests and small punch tests has been established.

Further testing should be performed on other materials, to confirm the accuracy of the proposed relationship. Special care should be paid to geometry of the specimen and to the correct alignment of the notch during the tests. The effects of the notch geometry should be analysed in order to better understand its influence.

In conclusion, this paper confirms the suitability of the small punch test for the characterisation of the DBTR of ferritic steels. The use of this testing technique in combination with the Master Curve approach allows the evaluation of the DBTR with highly reduced volumes of material. This feature is especially relevant when compared with other sub-size specimens, such as microCT's, since small punch specimens represent only 1/8th of the volume of a microCT specimen. Furthermore, it is a simple and cost-effective test, which can be performed with simple equipment.

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