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To cite this article: M Sánchez et al 2024 J. Phys.: Conf. Ser. 2692 012003

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Fracture characterization of structural steel S690Q by using mini-CT specimens

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Abstract. Mini-CT specimens are an interesting alternative when characterising the fracture behaviour of structural materials and there are issues with regard to, for example, the amount of available material, the irradiation level (in nuclear materials) or material inhomogeneities. Furthermore, in ferritic-pearlitic steels, the characterisation of the fracture behaviour within the ductile-to-brittle transition zone (DBTZ) is of particular interest, given that the material may behave very different, in terms of fracture toughness, when operating at different but relatively close temperatures within this zone. In many occasions, the definition of the DBTZ behaviour is performed through the Master Curve (MC) methodology and, thus, by testing standardised fracture specimens (e.g., CT, SENB) and determining the material Reference Temperature (T₀). The use of mini-CT specimens to define T_0 has been validated in a wide range of steels used in nuclear industry, but its application to structural steels has been scarce. Thus, this work gathers the fracture characterisation results (T_0) obtained in structural steel S690Q, comparing them to those obtained by using conventional standardized SENB specimens. It is shown that, for this particular structural steel, the use of miniaturized specimens provides a T_0 value (-89.3°C) which is comparable to the value obtained from conventional larger specimens (-110°C).

1. Introduction

Reactor pressure vessels (RPVs) are made of ferritic steels. These materials behave ductile at relatively high temperatures and tend to brittle behaviour when reducing the operating temperature. Besides, the transition from ductile to brittle behaviour is shifted towards higher temperatures when these steels are subjected to neutron irradiation. Consequently, RPV steels are subjected to surveillance programs that assess their fracture behaviour throughout their service life, in order to ensure that they are ductile at service temperatures. In this sense, to assess the fracture behaviour of RPV steels within the ductile to brittle transition zone (DBTZ), the Master Curve (MC) methodology has gained wide acceptance in the last decades. If the resulting ductile to brittle transition is proven to occur at low enough temperatures, RPVs can work without the risk of brittle fracture.

The MC is an engineering methodology that allows estimating the fracture behaviour of ferritic steels within the DBTZ [1,2], and it is standardised by ASTM E1921 [3]. The MC is based on the weakest link theory, assuming that the fracture process follows a three parameter Weibull distribution. However, theoretical and empirical considerations have proven that, in ferritic steels, two of the Weibull parameters, the shape parameter (b) and the location parameter (K_{\min}) , may be considered fixed at 4 and 20 MPam^{1/2}, respectively. Thus, the MC does only require a single material parameter,

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 K_0 (the scale parameter), to be defined. Additionally, K_0 only depends on the material reference temperature (T_0), which represents the temperature corresponding to a median fracture toughness ($K_{\text{Jc,med}}$), for a 1T (25.4 mm) thick specimen, of 100 MPa·m^{1/2}. As soon as T_0 is defined for the material being analysed, the MC can be defined for any probability of failure (P_f) by using equation (1):

$$K_{\rm Jc, Pf} = 20 + \left[\ln\left(\frac{l}{l - P_{\rm f}}\right) \right]^{1/4} \cdot \{11 + 77 \cdot \exp[0.019 \cdot (T - T_0)]\}$$
(1)

Furthermore, when T_0 is defined by testing specimens with thicknesses other than 1T (25.4 mm), the MC proposes a correction to convert the actual K_{Jc} value into the corresponding $K_{Jc}(1T)$ equivalent, using the following equation (*B* being the thickness of the tested specimen) (2):

$$K_{\rm Jc(1T)} = 20 + [K_{\rm Jc} - 20] \left(\frac{B}{25.4}\right)^{1/4}$$
 (2)

In this regard, when characterising the DBTZ, the availability of material for fracture testing may be limited. This is the case, for example, in surveillance programme of nuclear power plants (NPPs) operating beyond their initial lifespan (long term operation conditions). However, such NPPs generally have a significant amount of irradiated tested Charpy specimens, on which performing further testing with the remnant material would be very interesting. It is at this point where mini-CT specimens arise as a robust alternative, considering that one tested Charpy specimen allows eight 4 mm thick mini-CT specimens to be obtained and tested. Figure 1 shows one of the common geometries of mini-CT specimens.



Figure 1. Geometry of mini-CT specimens. Dimensions in mm. Displacements measured on the front face.

The accuracy of the MC obtained when testing mini-CT specimens has been validated in different nuclear grade steels (e.g., [4-9]), in both irradiated and non-irradiated conditions, but to the knowledge of the authors the validation on structural steels used in other sectors such as bridge construction, building, automotive or machinery, is very limited or null. Thus, the purpose of this work is to provide validation of the use of mini-CT specimens to characterise the DBTZ of a common high-strength structural steel (S690Q).

2. Materials and methods

The present research analyses the application of mini-CT specimens in the MC characterisation of high strength structural steel S690Q, which is a quenched and tempered steel supplied (in this case) in 15 mm thick (0.6T) plates. This steel grade and thickness has widespread use in heavily dynamic loaded parts of offshore structures, pressure vessels, pipelines and yellow goods. It presents a bainitic/martensitic microstructure, as shown in Figure 2.

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Figure 2. Microstructure of steel S690Q, showing L (rolling) and S (thickness) directions.

The chemical composition is gathered in Table 1, whereas the tensile properties at room temperature and the Charpy transition temperature (T_{27J}) are shown in Table 2.

Table 1. Chemical composition of steel S690Q (wt.%).

С	Si	Mn	P	S	Cr	Мо	Ni	Al	Cu	Nb	Ti	V
0.15	0.40	1.42	0.006	0.001	0.020	0.002	0.160	0.056	0.010	0.029	0.003	0.058

Table 2. Tensile properties at room temperature (RT) and Charpy transition temperature (T27J). Steel S690Q. *E*: Young's Modulus; s_y : yield stress; s_u : ultimate tensile strength.

$T(^{\circ}C)$	E (GPa)	s_y (MPa)	s_u (MPa)	<i>T</i> _{27J} (°C)
RT	204	775	832	-94

The fracture characterization using conventional 0.6T SENB specimens is described in [10]. Here, sufficient is to say that 13 tests were performed at three different temperatures (-100 °C, -120 °C, -140 °C), providing a reference temperature value of T_0 = -110°C when following the multitemperature method described in ASTM E1921 [3]. The resulting MC and the experimental results (1T fitted, following equation (2)) are shown in Figure 3, revealing good agreement between them.

Concerning the MC characterization using mini-CT specimens, 18 specimens were specifically tested at different temperatures and following ASTM E1921 [3]. Figure 4 shows the experimental setup. The geometry of the specimens coincides with that shown in Figure 1. The mini-CT specimens were extracted from the broken halves of the 0.6T SENB tested specimens described above, ensuring same orientation (LT) and also that the used material had not developed plastic conditions during the original tests.

doi:10.1088/1742-6596/2692/1/012003



Figure 3. Experimental results of K_{Jc} (ASTM E1921) and resulting Master Curve. Steel 690Q, 0.6T SENB specimens.



Figure 4. Experimental setup in the mini-CT testing program of steel S690Q.

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3. Results and discussion

Table 3 gathers the $K_{\rm Jc}$ results obtained in the different tests performed on mini-CT specimens (4 mm thick, 0.16T)), together with the main inputs in the MC analysis, including the conversion of 0.16T results into 1T equivalent values and the measurement of the initial crack lengths (a_0). It is important to note that only 10 of the tests were valid, given that 8 of the tests had a (a/W) value higher than 0.55 (thus, not valid when following ASTM E1921 [3]) or were tested outside the temperature validity range. Additionally, the table gathers the corresponding $K_{\rm Jc,limit}$ used for censoring data, determined as per ASTM E1921 [3] (see equation (3)). The δ_i values refer to the censored/uncensored condition of the test result, with $\delta_i=1$ for uncensored results and $\delta_i=0$ for the censored ones.

$$K_{\rm Jc,limit} = \left(\frac{E \cdot b_0 \cdot s_y}{30 \cdot (1 - v^2)}\right)^{1/2} \tag{3}$$

Code	<i>a</i> ₀ (mm)	$T(^{\circ}\mathrm{C})$	$K_{\rm Jc,limit}$ (1T)	K _{Jc} (0.16T)	$K_{\rm Jc}(1T)$	$\delta_{\rm i}$
S690_03	4.4	-130	110.8	77.5	56.2	1
S690_04	4.3	-130	112.2	84.4	60.5	1
S690_05	4.4	-120	109.9	61.3	46.0	1
S690_06	4.4	-110	109.1	76.2	55.4	1
S690_08	4.4	-100	107.3	160.8	108.7	0
S690_11	4.3	-100	107.3	126.2	86.9	1
S690_12	4.4	-100	106.6	127.7	87.8	1
S690_13	4.2	-100	107.3	150.3	102.1	0
S690_14	4.4	-105	107.9	118.2	81.8	1
S690_18	4.2	-105	110.1	140.4	95.8	1

Table 3. Fracture results and different inputs used in the MC analysis when following [3].

Besides, Figure 5 shows an example of load-displacement curves. At this point, it is important to note that the K_{Jc} values obtained using mini-CT specimens ($K_{Jc}(0.16T)$) have to be converted into equivalent 1T (25.4 mm, $K_{Jc}(1T)$) values, following equation (4):

$$K_{\rm Jc}(1T) = 20 + \left[K_{\rm Jc}(0.16T) - 20\right] \left(\frac{4}{25.4}\right)^{1/4}$$
(4)

 T_0 was finally determined following the multi-temperature methodology defined in [3], resulting T_0 = -89.3 °C, in reasonably good agreement with the value derived from 0.6T specimens. The literature shows how mini-CT specimens generally provide values of T_0 in the range of $\pm 20^{\circ}$ C when compared with the T_0 values obtained from conventional fracture specimens (e.g., [11]). Figure 6 shows the resulting MC of steel S690Q, together with the individual experimental results. The equations of the different curves are derived from equation (1). In spite of the reduced number of valid non-censored tests (8 out of 18), the T_0 result obtained is valid following the validity criteria established in ASTM E1921 [3].

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Figure 5. Examples of load-displacement curves obtained on mini-CT specimens.



Figure 6. Steel S690 Master Curve obtained using mini-CT specimens, and comparison with the experimental results.

Finally, the fracture surfaces of the tested mini-CT specimens were analysed using scanning electron microscopy (SEM). The aim was to verify that the basic assumptions of the MC approach were fulfilled: firstly, that fracture is caused by cleavage micromechanisms following the weakest-link theory, implying, among others, that there is a single initiation point; secondly, that the initiation point is generally located around the center line of the fracture section. Figure 7 shows an example of the fracture surfaces observed in the mini-CT specimens, where it can be observed a single centered initiation point. This pattern was observed in all the tested specimens.

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Journal of Physics: Conference Series	2692 (2024) 012003	doi:10.1088/1742-6596/2692/1/012003

4. Conclusions

This paper analyses the application of the Master Curve methodology to the fracture characterization of high strength structural steel S690Q when using mini-CT specimens (0.16T or 4 mm thick).

The experimental program developed with mini-CT specimens has provided a T_0 value of -89.3 °C, in reasonably good agreement with the value derived from conventional (15 mm thick, 0.6T) specimens, indicating the potential of this specimen geometry to characterize T_0 in this particular steel.

Finally, it has been observed that the fracture micromechanisms observed in the tested mini-CT specimens are in accordance with the Master Curve assumptions.



Figure 7. Example of fracture surface observed in mini-CT specimens S690_04. The arrow indicates the location of the (single) initiation point.

Acknowledgments

This research has received funding from the Euratom research and training programme 2019-2020 under grant agreement N° 900014.

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