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Critical evaluation of the small punch test as a screening procedure for mechanical properties

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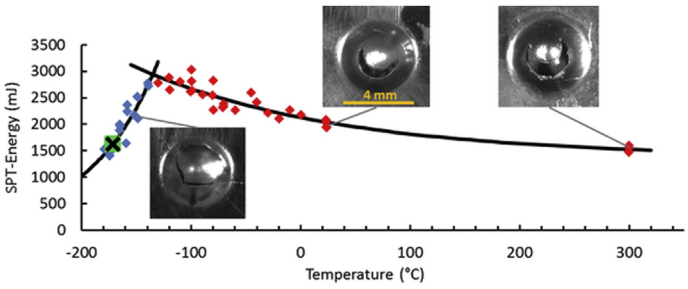
HIGHLIGHTS

- Collective exercise on the small punch test.
- Statistical evaluation of in-house repeatability and inter-laboratory reproducibility.
- Evaluation of neutron irradiation induced hardening and embrittlement of T91 steel by small punch test.
- Small punch test well suited for screening of embrittlement and conditionally suited for hardening.

GRAPHICAL ABSTRACT



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ABSTRACT

Within a collective exercise, a systematic and statistically based analysis of the repeatability and device dependence of small punch test results was performed. An unirradiated ferritic-martensitic steel T91 was selected for this. The test results allowed an evaluation of accuracy and reliability of material properties extracted from load–displacement-based and energy-based parameters. In a second step, neutron irradiated T91 with doses of 2.3 and 4 dpa was investigated in the temperature range −165 °C to 300 °C. The effects of test temperature and irradiation on the load–displacement curves and on the derived parameters were analysed. It was found that the small punch test is well suited for estimation of neutron embrittlement in cases of small amounts of available material or high activity. The preferred procedure for that purpose was specified.

1. Introduction

Small specimen test technologies have long been recognized as a supportive means for the development and monitoring of structural materials for nuclear components [1]. The small punch (SP) test [2–11] received much attraction and widespread use. However, details of the sample preparation, device and sample geometry as well as testing procedure and analysis may differ from laboratory to laboratory. Within the Euratom FP7-Project MATTER (MATerials TEsting and Rules), work package 2 was dedicated to the development of screening test procedures for mechanical properties. Screening procedures are intended to provide approximate data of the properties with as small amounts of material as possible and thus allowing a quick evaluation of ageing mechanisms such as

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Table 1
Composition the T91 heats (wt%).

	C	Si	V	Cr	Mn	Ni	Mo	N	Al	P	S	Ti	Cu	Nb	W
T91-DEM	0.1025	0.22	0.21	8.99	0.38	0.11	0.89	0.0442	0.0146	0.021	0.0004	0.0034	0.06	0.06	0.01
T91-SPI	0.099	0.32	0.24	8.8	0.43	0.24	0.96	0.03	<0.01	0.02	0.004	<0.005	0.05	0.06	<0.01

neutron irradiation induced hardening and embrittlement or liquid metal embrittlement. Amongst others, the SP test was selected as a testing method for screening.

The SP test is well suited to investigate irradiation effects, since the amount of material needed is small and thereby the total activity of specimens is much lower than for classical mechanical and fracture mechanics tests. For example the mass and thus the activity ratio between a SP standard specimen ($\varnothing 8 \times 0.5$ mm^3) and a full-sized Charpy specimen ($10 \times 10 \times 55$ mm^3) is approximately 1:220.

The ductile to brittle transition temperature (DBTT) can be extracted from the SP tests. From a single load–deflection curve one can calculate the energy E_{SP} needed to deform and crack the SP sample. Doing this for different temperatures, the $E_{SP}(T)$ curve can be constructed. Usually this curve is assembled from two fits, the first one describing the brittle and transition regime and the second one the ductile regime. The intersection of the two curves marks the maximum of the fitted $E_{SP}(T)$ course, E_{max}. The SP transition temperature T_{SP} is defined as temperature where $E_{SP} = 0.5^*(E_{max}-E_{min})$ holds [2,3]. A linear correlation with the Charpy transition temperature T_{CVN} is proposed in Refs. [4,5]:

$$T_{SP} = \alpha \cdot T_{CVN} \tag{1}$$

where absolute temperatures have to be used. The factor α was found to be 0.32 … 0.34 for 2.25Cr–1Mo steels and 1Cr–0.5Mo steels respectively, based on SP specimens of 0.5 mm thickness and a punch ball diameter of 2.4 mm. However, other results [6,7] give rise to the assumption that the factor α depends on both the SP geometry and the material. Kameda provided a rationale for the linear relationship Eq. (1) by means of a kinetic model for ductile–brittle fracture mode transition considering also the strain rate effect [8]:

$$\alpha = \frac{T_{SP}}{T_{CVN}} = \frac{\left\{ \ln\left[\left(\frac{V_S F_S}{V_t F_t}\right) \left(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}}\right)^2 \right] \right\}_{CVN}}{\left\{ \ln\left[\left(\frac{V_S F_S}{V_t F_t}\right) \left(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}}\right)^2 \right] \right\}_{SP}} \tag{2}$$

where V and F are fracture volume and micro-crack density respectively, and the subscripts s and t indicate shear and tension. Based on results for ferritic-martensitic steels, the factor was obtained as $\alpha \approx 0.4$.

The SP test has also been used to estimate hardening, i.e. increase of the yield stress (YS). A linear correlation was proposed for the YS

$$R_{p02} = \beta_{YS} \cdot F_e / h^2 \tag{3}$$

with h being the initial specimen thickness, F_e the SP load at the onset of plastic flow and β_{YS} an empirical factor. Kameda and Mao [9] found $\beta_{YS} = 0.36$ for specimen thicknesses of 0.25 mm and 0.5 mm. In Ref. [10] the correlation was underpinned by finite element simulations of SP tests. Two different correlations were proposed for the estimation of the ultimate tensile strength (UTS) [10,11]:

$$R_m = \beta_{UTS} \cdot F_m / h^2 \tag{4a}$$

$$R_m = \beta_{UTS} \cdot F_m / (h \cdot u_m) \tag{4b}$$

with F_m being the maximum load and u_m the corresponding displacement.

Detailed analyses of stress and strain in the SP disc have been performed by means of analytical elastic–plastic modelling [12,13] and by finite element calculations [10,14,15]. A combination of finite element modelling, SP testing and neural networks was used to identify the parameters of the Gurson–Tvergaard–Needleman model for ductile damage [14] and of the Beremin model for brittle fracture [15].

In this paper, we present the results of a SP test collective exercise with unirradiated T91 steel. This exercise aimed at the analysis of scatter, reproducibility and device dependence of characteristic parameters extracted from SP tests.

Another focus of the paper is put on the investigation of neutron irradiation induced hardening and embrittlement by means of the SP test. The usefulness of the above mentioned correlations for screening of hardening and embrittlement (Eqs. (1)–(4)) is evaluated.

2. Material

All tests reported in this paper are related to the 9Cr ferritic martensitic steel T91 as defined in the ASTM standard A387-Ed99 (Grade 91 Class 2/S50460). Two heats of this material were investigated. The first heat was characterised in the FP6 project EUROTRANS (project domain 4 – DEMETRA). This heat was produced by ARCELOR and is referred to as "T91-DEM" in this paper. It was used for the SP collective exercise. The second heat was produced by UGINE, France. It was characterised within the FP5 project SPIRE. This heat is referred to as "T91-SPI" in this paper. A neutron irradiation experiment at the reactor BR2 in Mol, Belgium, was part of the SPIRE project. The T91-SPI heat was used for the evaluation of irradiation induced hardening and embrittlement. The chemical compositions are listed in Table 1 and heat treatments and product forms in Table 2.

The temperature dependent yield stress and tensile strength are shown in Fig. 1 (T91-DEM) Fig. 2 (T91-SPI). The data were taken from Refs. [16] and [17] respectively. The temperature fits for R_{p02} and R_m of T91-DEM are given by Eq. (5):

$$R_{p02}(\text{MPa}) = 588 - 0.165 \cdot T(\text{K}) + 1248 \cdot \exp[-0.013 \cdot T(\text{K})] \tag{5a}$$

$$R_m(\text{MPa}) = 713 - 0.205 \cdot T(\text{K}) + 995 \cdot \exp[-0.011 \cdot T(\text{K})] \tag{5b}$$

For T91-SPI the temperature fits are given by Eq. (6):

Table 2
Product form and heat treatment.

Heat	Product form	Heat treatment
T91-DEM	Hot rolled plate thickness 15 mm	Normalization 1050 °C/15 min tempering 770 °C/45 min
T91-SPI	Hot rolled plate thickness 15 mm	Normalization 1040 °C/60 min tempering 760 °C/60 min