

# DETERMINATION OF FRACTURE TOUGHNESS IN THE UPPER SHELF REGION USING SMALL SAMPLE TEST TECHNIQUES

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#### **Abstract**

The residual lifetime assessment and the risk of a possible service components failure are critical issues in the safety and reliability analyses of industrial plants. The residual lifetime can be evaluated by the standard mechanical test techniques, such as the tensile test, uniaxial creep test, the Charpy or the fracture toughness test. Fracture mechanics in particular has attained high significance in establishing ultimate load limitations and assessing the integrity of a large number of engineering structures of multifarious types. Standard mechanical tests used to determine the fracture toughness involve extraction of large blocks of material and therefore are not applicable to in-service components.

The development and in-service application of essentially non-destructive, miniature material sample removal systems (e. g. the surface sampling systems, such as Electric Discharge Sampling Equipment - EDSE) provided a practical incentive for development of small specimen test methods to evaluate material properties, e. g. fracture toughness. The EDSE can cut out small slices ('boat sampling') about 3 mm thick and approximately 20 mm wide x 25 mm long from thick-section components leaving behind cavities with round edges that usually do not require repair.

In this study, two different methods will be used for fracture toughness determination: Small Punch Test and multiple-specimen method using sub-size Charpy specimens (3x4x27 mm). Both methods require very little experimental material and specimens can be made directly from the removed 'boat sample' by the EDSE. The applicability and reliability of both methods will be discussed.

Keywords: Small Punch test, facture toughness, Micro-Tensile test, Mini-Charpy test

# 1. INTRODUCTION

There is an increasing demand for the integrity assessment of important components in the course of their service nowadays. Degradation of the properties of materials can lead to a loss of reliability and safety in equipment and structures in service.

Conventional testing methods used for residual lifetime assessment require large amounts of material and often lead to a compromise of the equipment integrity. In order to avoid component damage, an appropriate sampling device has to be used. Non-destructive sampling is enabled by latest equipment using electrical discharge machining processes. This portable device is able to extract a small experimental block ('boat sample') from a real in-service component.

Despite a limited amount of testing material, a few kinds of small size specimens can be prepared and tested. For example, for tensile properties, the Small Punch Test is often used [1-7] as well as the Micro-Tensile test which shows much better applicability and reliability [8-10].

The aim of this paper is to find a reliable test method for fracture toughness determination using small specimens which can be manufactured from the 'boat sample' by the EDSE. Two methods meet this requirement: the Small Punch Test (SPT) and the multiple-specimen method using sub-size Charpy specimens (mini-Charpy specimens).



Some materials have already been investigated using the SPT in the work [10]. The SPT results indicated the usability of this method for fracture toughness determination of selected materials. Therefore, additional assessment was done using the SPT for other material states (four additional materials) to confirm the SPT applicability. Convenience of both the SPT and the mini Charpy method for fracture toughness determination will be discussed.

#### 2. MATERIALS AND METHODS

Two different miniature test techniques were used in order to find a reliable testing method for fracture toughness ( $J_{IC}$ ) determination. For obtaining the reference values  $J_{IC}$ , standard tensile and fracture toughness tests were also performed.

## 2.1. Experimental Materials

Experimental materials were 3 steels. The first one was an experimental low carbon steel, the second one was 34CrNiMo6 steel and the third one is heat-resistant chromium steel P91 which is commonly used at service temperatures up to 600 °C in the power industry. The steels were investigated in different material states, either in the delivered state or after heat treatment. The heat treatment consists of austenitization for 20 minutes, quenching in oil and annealing at a specific temperature which is labeled in **Table 1**.

Altogether, the materials were in twelve different heat treated states. However, eight states had already been investigated in the work [10].

# 2.2. Tensile and Fracture Toughness Tests

Tensile tests according to CSN EN ISO 6892-1 were performed at room temperature on the investigated materials. Round samples of diameter 5 mm and gauge length 25 mm were used (see **Fig. 1**). Prior to testing specimen dimensions were measured. After the test a yield stress  $R_{p0.2}$  was determined as well as tensile strength  $R_m$ . At least three samples were tested for each material.

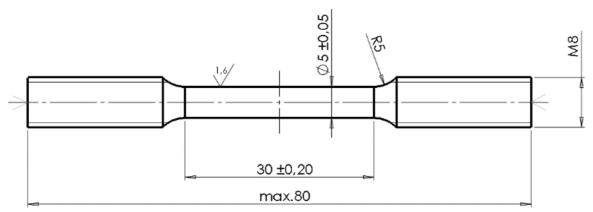


Fig. 1 Specimen for tensile test

The fracture toughness tests were performed on three point bend specimens. The evaluation was done according to ASTM E 1820. Samples were machined then fatigue pre-cracked with the final stress intensity factor of about 20 MPa.m<sup>1/2</sup>. Test pieces were side-grooved after pre-cracking and subsequently tested. Tests were performed according to the multiple specimen method. Crack lengths after tests were measured by digital image processing and fracture toughness values were determined for the materials investigated. At least three samples were tested for brittle material and about ten samples for J-R curves determination in all other cases. Tests were executed at room temperature. Results of the tensile tests and the fracture toughness test are summarized in **Table 1**.



	Heat Treatment	Test Results			
Material		R <sub>p0,2</sub>	R <sub>m</sub>	ε <sub>f</sub>	J <sub>IC</sub>
		[MPa]	[MPa]		kN/m
Experimental steel	as delivered	1145.5	1252.1	1.19	49.4
Experimental steel	annealing at 250 °C/2h	1168.5	1365.6	0.96	39.9
Experimental steel	annealing at 350 °C/2h	1166.6	1285.6	1.10	47.0
Experimental steel	annealing at 440 °C/2h	981.1	1022.1	1.07	253.5
Experimental steel	annealing at 500 °C/2h	812.0	816.0	1.29	382.6
Experimental steel	annealing at 620 °C/2h	661.2	734.0	1.30	517.3
34 CrNiMo6	quenched	1219.6	1965.0	0.13	42.1
34 CrNiMo6	as delivered	932.0	1034.2	0.77	189.9
34 CrNiMo6	annealing at 680 °C/2h	726.8	844.7	0.94	178.4
34 CrNiMo6	annealing at 750° C/2h	528.4	688.3	1.01	263.0
34 CrNiMo6	annealing at 450 °C/2h	1291.9	1373.9	0.23	74.7
P91	as delivered	534.7	696.2	1.16	318.7

Table 1 Results of tensile test and effective fracture strain vs. fracture toughness

# 2.3. Small Punch test (SPT)

Small punch tests were performed on a servohydraulic testing system of 10 kN capacity. Tests were carried out in a testing fixture of the dimensions mentioned in **Fig. 2**.

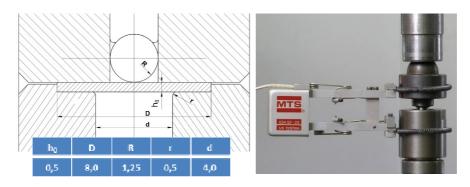


Fig. 2 Small punch test fixture

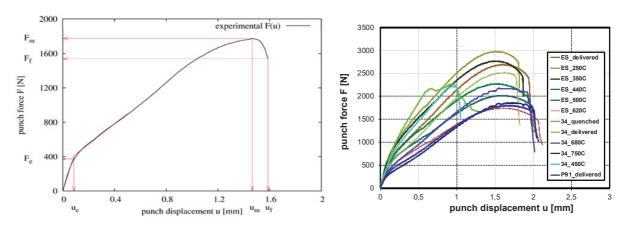


Fig. 3 Left: Scheme of distinctive points determination, right: SPT records



Displacement of the penetrating ball was measured by an extensometer attached to the fixture. Distinctive points from the records were determined according to **Fig. 3-left.** Obtained curves for all materials are shown in **Fig. 3-right**. Tests were performed at room temperature.

Various procedures for fracture toughness evaluation from the SPT for ductile fracture [1-8] have been published. In the case of upper shelf behavior, the following relation is widely used:

$$J_{IC}=k.\varepsilon_{\Gamma}J_{0} \tag{1}$$

$$\varepsilon_f = \ln \left( h_0 / h_f \right) = \beta \cdot (u_f / h_0)^{\times} \tag{2}$$

where  $\varepsilon_f$  is fracture strain, h<sub>0</sub> initial sample thickness, h<sub>f</sub> samples thickness in the crack region, u<sub>f</sub> is displacement at sample fracture and  $\beta$ , x, k and J<sub>0</sub> are empirically determined constants.

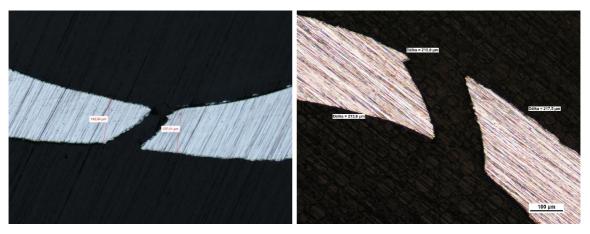
In the case of brittle fracture, the following relation is recommended [3]:

$$K_{IC} = C. [\sigma_{fSP}]^{2/3}$$
 (3)

$$\sigma_{\rm fSP}$$
=130 ( $P_{\rm max} / h_{\rm o}^2$ ) - 320 (4)

where  $P_{max}$  is force at unstable crack propagation and  $h_0$  is the initial sample thickness. C is empirically determined constant. The determination of fracture behaviour using SPT for the brittle and ductile states is usually done separately. However, for the investigated steels a linear correlation between fracture toughness  $J_{IC}$  and fracture strain  $\epsilon_f$  was applicable for the whole scale of fracture behaviour.

There are two possibilities of  $\varepsilon_f$  determination as can be seen from Eq. 2. The first one is the determination of fracture strain by measuring the sample thickness after a fracture in the crack region according to **Fig. 4**. The second possibility is its determination from test records with the use of displacement at fracture ( $u_f$ ). In this study, fracture strain was determined by measuring the sample thickness after a fracture.



a) Experimental steel - annealed at 500°C

b) 34 CrNiMo6 - as delivered

**Fig. 4** Determination of  $\varepsilon_f$  on the basis of optical measurements

Results of fracture strain from SPT and standard fracture toughness test (see **Table 1**) are depicted as  $\epsilon_f$  vs.  $J_{IC}$  in **Fig. 5**. On the left, data published in the work [10] are depicted and the reliability coefficient R was 0.9689. However, when additional assessment of four other material states was considered (see picture on the right), the reliability coefficient R was only 0.2992.



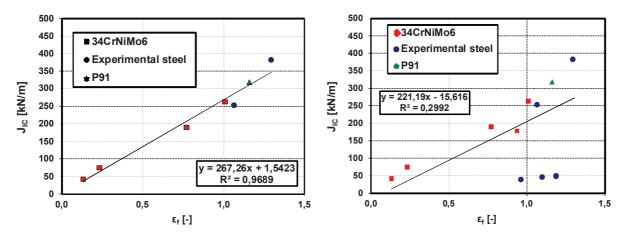


Fig. 5 Fracture strain vs. fracture toughness, left: published data in [10], right: added data

# 2.4. Mini-Charpy test for fracture toughness determination

Material 34CrNiMo6 in the delivered state was tested using mini-Charpy specimens for fracture toughness determination. The evaluation was done according to ASTM E 1820. Samples were machined then fatigue pre-cracked with the final stress intensity factor of about 20 MPa.m $^{1/2}$ . Test pieces were side-grooved after pre-cracking and subsequently tested. Tests were performed according to the multiple specimen method. Crack lengths after tests were measured by digital image processing and the fracture toughness value was determined for the material investigated. The J-R curve was determined and the fracture toughness value, defined as the intersection of the 0.2 mm offset construction line with the J-R curve, was calculated. Due to small specimens size, the dimension criteria for size independent fracture toughness given by ASTM E1820 standards were not fulfilled. Therefore, this test is denoted as  $J_Q$  in the results table. Test results obtained from mini-Charpy specimens and compared with standard test results are shown in **Table 2**. J-R curves of both geometries are depicted in **Fig. 6**. The deviation between both fracture toughness values is only 1.7 %.

Table 2 Fracture toughness results

Material	Sample size mm	J <sub>IC</sub> ,J <sub>Q</sub>	Error
34CrNiMo6	15x30	189.9	7.5
state-as delivered	3x4	193.2	1.7

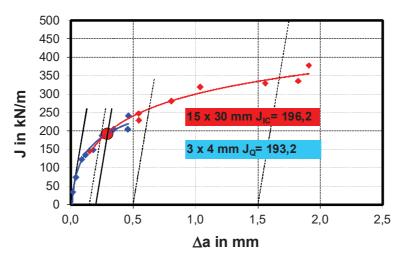


Fig. 6 J-R curves of 34CrNiMo6, state as delivered



## 3. CONCLUSION

Two small specimens test techniques were investigated in order to assess fracture toughness: Small Punch Test (SPT) and multiple-specimen method using sub-size Charpy specimens (3x4x27 mm). Evaluation using SPT is based on empirical correlations. The SPT correlations are considered valid only for specific types of materials. Due to very promising results from the previous work, other material states, obtained by heat treatment of the same materials, were measured to confirm the correlation relation obtained for these materials. However, these results do not fit in the previous correlation. Therefore, the SPT cannot be considered as a reliable method for fracture toughness determination of various materials.

On the other hand, the second technique using mini Charpy specimens does not require any correlations due to maintaining the same load as in the case of standard fracture toughness tests.

Though only one material has been tested so far using mini Charpy specimens, the result indicated that this method is more convenient for fracture toughness determination of various materials in the upper shelf region.

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## **REFERENCES**

- [1] WANG, Z., X. et al. Small punch testing for assessing the fracture properties of the reactor vessel steel with different thicknesses. Nuclear Engineering and Design, Vol. 238, No. 12, December 2008, pp. 3186-3193
- [2] MATOCHA, K., PURMENSKY, J. Non destructive evaluation of mechanical characteristics of in-service components materials by small punch tests, In conference: NDT for Safety, Prague, ČVUT, 2007, Prague, pp. 185-189.
- [3] Small punch test method for metallic materials, Part B: A code of practice for small punch testing for tensile and fracture behaviour, CWA 15627.
- [4] SHINDO, Z., ZAMAGUCHI, Z., HORIGUCHI, K. Small punch testing for determining the cryogenic fracture properties of 304 and 316 austenitic stainless steels in a high magnetic field. Cryogenics, Vol. 44, No. 11, 2004, pp. 789-792.
- [5] BULLOCH, J. H. A study concerning material fracture toughness and some small punch tests data for low alloy steels. Engineering failure analysis, Vol. 11, 2004, pp. 635 653.
- [6] Ha, J. S., Fleury, E. Small Punch Tests to Estimate the Mechanical Properties of Steels for Steam Power Plant: II. Fracture Toughness. International Journal of Pressure Vessels and Piping, Vol. 75, 1998, pp. 707-713.
- [7] Konopík, P., Džugan, J. Determination of Tensile Properties of Low Carbon Steel and Alloyed Steel 34CrNiMo6 by Small Punch Test and Micro-Tensile Test. In the 2nd International conference, 2012, (ISBN 978-80-260-0079-2), pp319-328.
- [8] DŽUGAN, J., KONOPÍK, P. Evaluation of fracture toughness properties for low carbon steel in the brittle state by small punch test technique. Hutnické listy, Vol. LXIII, 2010, pp119-122.
- [9] Konopík, P., Džugan, J., Procházka R. Evaluation of Local Mechanical Properties of Steel Weld by Miniature Testing Techniques. Materials Science & Technology, Vol. 53, 2013, pp. 27-31.
- [10] Konopík P., Džugan J., Procházka R: Determination of Fracture Toughness and Tensile Properties of Structural Steels by Small Punch Test and Micro-Tensile Test. In METAL 2013: 22<sup>nd</sup> International Conference on Metallurgy and Materials. Ostrava, TANGER, 2013, pp. 722-777.