Development of a methodology for measuring fracture toughness using sub size test specimens

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Abstract. To obtain real data of mechanical properties - fracture toughness - to describe the behaviour of cracks in the material, standard 1T-CT test specimens are usually used that, according to the standard, have the dimensions of 50x62.5 and a thickness of 25 mm. In the case of lack of material obtained for example by 3D printing, SPD methods, or by semi-destructive sampling from a real component to evaluate its residual life, these tests cannot be performed using a standard sample, but can be performed using a designed miniaturized test specimen marked as 0.16T-CT with dimensions 10x10 and thickness 4 mm. This paper contains the development of a methodology for testing fracture toughness using a sub-sized test specimens and a comparison of the obtained results with the results of tests obtained using a standard specimen. The techniques based on miniaturized test specimens for determining fracture toughness have been shown here as a tool to provide a deeper insight into the fracture behaviour of a material and to understand better the behaviour of the material in cases where a limited number of experiments are available.

1 Introduction
Fracture toughness properties provide very important information about the behaviour of material, in which the crack with sharp tip is present. This information is in many cases crucial for deciding whether to use or decommission the component.

However, often the lack of experimental material is an obstacle, but the assessment can be performed using a miniaturized test specimen. An example where a very small volume of experimental material is available are the assessment of the residual life of components in operation, where experimental material can be obtained only by semi-destructive approach, development of new materials, development of nano-materials by extreme deformation methods (SPD-Severe Plastic Deformation), development of special mechanical-thermal treatments using a physical simulator, and recently also in the evaluation of parts produced by additive manufacturing techniques. The article presents the use of sub-dimension test specimens for the fracture toughness evaluation. The experimental program was performed on Cr-Ni-Mo-V ferritic-martensitic refractory low-alloy steels marked as GOST 15CH2NMFA.

2 Test samples
To evaluate fracture toughness, the tests were performed using standard test specimens and sub-dimensioned test specimens.
2.1 Fracture toughness tests on standard test specimens

The fracture toughness tests of standard bodies were performed in accordance with ASTM E 1820-17 – Standard Test Method for Measurement of Fracture Toughness. Six 1T-CT test specimens were tested for the Master curve concept.

The testing procedure consists of the preparation of the test specimens by a fatigue crack propagation of required length using a Rumul Testronic magnetoresonance testing machine. The final oscillated crack length is given by the ratio $a/W = 0.5$ and the value of the stress intensity factor $K_{\text{final}} = 16 \text{ MPa}\sqrt{\text{m}}$. Subsequently, the samples were provided with side notches. This preparation was followed by the testing procedure itself, which took place on a servo-hydraulic testing machine MTS 810. The crack mouth opening displacement (CMOD) of the sample was measured using a clip-on gauge COD extensometer.

2.2 Fracture toughness tests on sub-size test specimens

Recently, much attention has been focused on the geometry of mini-CT (0.16T-CT) test specimens, which can also be made from a broken half of a standard Charpy sample [1-4].

In 2017, a round robin test was performed aimed at verifying the reliability of experimental mini-CT data. The test results confirmed that the mini-CT test specimens offer a very attractive opportunity to derive the reference temperature $T_0$ comparable to the larger fracture toughness samples and the Mini-Charpy (KLST) samples [5]. Mini Charpy and 0.16T CT test specimens were selected for testing. The tests were performed according to the same test standards as the standard test specimens. All test specimens were subjected to the fatigue crack propagation procedure and side notches machining. The tests were performed using a servo-hydraulic testing machine SI-PLAN. A comparison of the test specimens is shown in FIGURE 1a). The drawing of the 0.16T CT test specimen is shown in FIGURE 1b).

![Figure 1 a) Comparison of used test specimens b) Drawing of test specimen 0.16T - CT](image)

3 Fixture for performing fracture toughness tests using 0.16T - CT test specimen

To perform the test using a 0.16T-CT specimen with dimensions of 10x10 mm and a thickness of 4 mm, a device which ensures the exact position of the test specimen in the standard test machine and at the same time allows the measurement of the deformation - opening of the test specimen have to be used.

This problem is solved by a specially designed fixture, which consists of a clamping device for the test specimen gripping during the test and tips attached to the arms of the extensometer to measure
the CMOD. This device has been designed in such a way that it is possible to precisely position the miniaturized test specimen in the load axis of the test machine, and at the same time it allows measuring the crack opening of the test specimen during the test with a COD extensometer. The device must also allow a reliable loading of the test specimen during the test without the effect of friction, which could affect the evaluation of the results of fracture toughness - energy balance of crack growth.

Due to its structural fixture, the designed device enables the realization of not only the own test of static and dynamic fracture toughness, but also its preparation, i.e. crack propagation of the real fatigue crack in the test specimen in accordance with the boundary conditions specified by the standard. Furthermore, the design also eliminates side effects – friction. During the test, it allows sensing the opening of the sample using a COD sensor, and thus obtaining evidence of the behaviour of the inclined real crack in the monitored test sample.

The device allows to perform a fracture toughness test using a miniaturized test specimen and thus evaluate the parameters of the J-R curve or KIC of the investigated material. This approach is suitable for describing of the real cracks behaviour, even in the absence of material for the production of standard test specimens. It enables a significant refinement of the prediction of crack behaviour in real components and modern 3D printed materials, which do not allow the use of standard test specimens. The essence of the technical solution is a jig for testing the test mini-CT test specimen by eccentric tensile (Compact Tension test), which consists of the upper and lower grips, pins, mini-CT test specimen and extensometer with special tips. Compared to a standard CT test specimen, this is another technical solution that allows to scan the CMOD from the outside, using a standard extensometer equipped with specially designed tips inserted into the outside machined grooves of the mini-CT test specimen. Thanks to the larger initial measured length (L0), this solution ensures greater measurement accuracy for fracture toughness tests (determination of KIC and J-R curves), or higher frequency for fatigue crack propagation velocity tests. The extensometer tip is designed as a self-centering detachable joint with an optimized tip angle abutting the notch of the mini-CT test specimen. The grips are designed to allow maximum opening of the mini-CT test specimen while protecting the extensometer against exceeding its maximum measured length and the resulting destruction. The test specimen is clamped in the grips with clearance by means of pins. The grips are screwed into the rods that are part of the testing machine. The grips are stiff enough not to distort the measurement results. After setting up the test specimen and resetting the force, the test process can be started. After reaching the prescribed test parameters according to the standard, the test is completed, the sample is removed and the device can be used repeatedly. A schematic representation of a device for testing fracture toughness using miniaturized CT test specimens 0.16T CT is shown in FIGURE 2.
Figure 2. Model of the proposed fixture

The assembly diagram of the described device can be seen in FIGURE 3. The fixture for fixing the test specimen comprises an upper 1 and a lower 4 grips with a thread for screwing into rods which are connected to the testing machine. The test specimen 2 is clamped in the jaws by means of special pins with clearance to prevent friction. The pins ensure the correct positioning of the test body in the axis of the machine and pass through the holes in the upper and lower grips 3. Clamping of the test specimen takes place after screwing the upper 1 and lower grips 4 into the rods of the testing machine, the actual clamping of the test specimen 2 is then realized by means of pins through the holes 3 in the upper and lower grips. To ensure friction, there is an H7 / g6 fitting between the pin and the hole in the 0.16T CT test specimen, and there is an H7 / f7 fitting between the hole in the grips and the pin. The special tips 5 of the pressure attachment on the arms of the extensometer 6 are inserted in the groove of the upper and lower grips so that the tips of the extensometer tips fit exactly into the notch of the test 0.16T CT specimen 2. The pressure segment ensures a constant pressure force. This makes the assembly ready for testing.

Figure 3. Description of the proposed fixture

4 Performing tests and evaluation

In order to compare the results of the fracture toughness tests performed using miniaturized and standard specimens, the test specimens were made of 15CH2NMFA material. A total of six 1T-CT, nine 0.16T CT test specimen and fourteen KLST bodies were tested to determine the reference temperature.

The tests were performed at reduced temperatures in a cooling chamber with a supply of liquid nitrogen. The force-CMOD dependence was measured during the test. The CMOD of the samples was measured during the test with a clip-on gauge COD extensometer, see FIGURE 4. A summary of the determination of the reference temperature $T_0$ is given in Tab. 1. The curve with all measured data is shown in FIGURE 5. It is clear from Table 1 that the reference temperature evaluated using KLST samples and 0.16T-CT samples does not meet the validity criterion $\sum r_{ni} > 1$ and these reference temperatures can be considered as the provisional reference temperature $T_{0Q}$. 
Discussion of results

The results obtained here point to the fact that there is currently no general solution available to describe the magnitude of the effect in fracture toughness approaches.

To reliably determine the "size independent" value, the material must be examined and the size effect quantified. The lower transition area is well described by the Master Curve approach, including size effects in the evaluation. It is generally considered that in all cases it is not necessary to obtain size-independent values. This can be, for example, an assessment of the properties of components with a...
small wall thickness, where the normal state of deformation does not in fact prevail. Typical examples of materials made with a small wall thickness that exhibit a high ratio of anisotropy properties are materials and components made by additive manufacturing processes.

6 Conclusion
As can be seen in many cases, the values independent of actual size cannot be achieved for the material, and therefore small size techniques are the only way to characterize the properties. These values apply only to the component and the process under consideration; however, valuable information is provided to enable component design and process optimization. The techniques based on miniaturized test specimens for determining fracture toughness have been shown here as a tool to provide a deeper insight into the fracture behaviour of a material and to understand better the behaviour of the material in cases where a limited number of experiments are available.

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References
[1] Džugan, J.; Procházka R.; Konopík P. Micro-tensile test technique development and application to mechanical property determination. Small Specimen Test Techniques. 6th Volume. STP 1576. Mikhail A. Sokolov and Enrico Lucon. Eds.. s. 12–29. ASTM International. West Conshohocken, PA 2014, DOI 10.1520/STP157620140022.
[2] Konopik P Dzugan J and Rund M 2015 Determination of fracture toughness in the upper shelf region using small sample test techniques .METAL 2015, 6(5) pp 710-715
[3] Gotterbarm M, Seifi M, Melzer D, Džugan J, Salem A, Liu Z and Körner C 2020 Small scale testing of IN718 single crystals manufactured by EB-PBF Addit. Manuf., 36, 101449
[4] Melzer D, Džugan J, Koukolíková M, Rzepa S and Vavřík J 2021 Structural integrity and mechanical properties of the functionally graded material based on 316L/IN718 processed by DED technology, Mater. Sci. Eng. A, 811, 141038
[5] Sokolov M.A., Development of Mini-Compact Tension Test Method for Determining Fracture Toughness Master Curves for Reactor Pressure Vessel Steels, Materials Science and Technology Division, Oak Ridge National Laboratory, 2017