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Report on the state of-the-art of small specimen use

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Summary

The purpose of this document is to collect and summarize the available practical information of testing mini-CT specimens to assist the project participants to solve the testing and evaluations problems. The information partially come from the participants experience and partially from the relevant literature.

Approval

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Abbreviations and acronyms

Acronym	Description
1T-CT or 1CT	One inch (~25 mm) compact tension
ATP	Atom probe
CG	Clip gauge
CMOD	Crack mouth opening displacement
CRB	Circumferentially cracked round bar
CV	Charpy V notched specimen
Δ_a	Fatigue precrack extension
DCG	Direct crack growth
EDM	Electro discharge machine
FM	Fracture mechanics
LVDT	Linear variable displacement transducer
LUS	Low upper shelf
Mini-CT	0.16 or 10*10*4 mm Compact Tension Specimen
NC	Numerical controlled
NPP	Nuclear Power Plant
RPV	Reactor Pressure Vessel
SANS	Small angle neutron scattering
SPT	Small Punch testing
T_0	Master Curve reference temperature

Summary

The purpose of this document is to collect and summarize the available practical information of testing mini-CT specimens to assist the project participants to solve the testing and evaluations problems. The information partially come from the participants experience and partially from the relevant literature.

Keywords

Mini CT, Fracture Toughness, Pre fatigue, Scatter, Testing temperature

1. Introduction

Large number of irradiated broken CV specimens are available at the laboratories and at the NPP-s. Surveillance specimens irradiated for several years in real environmental conditions (flux, temperature, made from industrial heats etc.) are generally not replaceable. Many unirradiated structures of the NPP-s (and other industrial structures too) are environmentally aged and minimal quantity of archive material of them is available for evaluation of the aged properties required for calculation of residual safe life.

Two ways are available to use the broken Charpy remnants, or for optimal use of mini samples: reconstitution and use of mini CT specimens. In STRUMAT-LTO mainly the mini CT and SPT specimens use planned to extend the information collected from the standard specimens.

The purpose of this document is to collect and summarize the available practical information of testing mini CT specimens to assist the project participants to solve the testing and evaluations problems. The information herein partially comes from the participants experience and partially from the relevant literature. The prescriptions of the Fracture toughness standards (ASTM E-399-20a, ASTM E-1921-20 etc) are not repeated in this document. Furthermore, this document does not deals with the J-R resistance curve measurement on mini-CT specimens.

2. History and literature summary of mini CT-specimen

The first attempts to use mini-CT specimens were made between 2000-2005 [1]. The requirement for evaluation of residual lifetime of the nuclear devices (first the RPV-s) initiated development methods to measure fracture toughness using the remnants of the surveillance Charpy specimens. Valid fracture toughness test requires large size specimens or low temperature testing. The development of the Master Curve allowed to use the data obtained at low temperature for safety and lifetime calculation.

Even though the early research results were satisfactory, few institutes used the mini-CT specimens. The reason was mostly the parallel development of reconstitution technology. Using reconstitution nearly the same amount of information can be obtained from the remnants of Charpy specimens as from mini-CT.

Around 2015 US and Japan institutes started to use again the mini-CT-s. Four laboratory Round -Robin test validated the results obtained on mini-CT-s. However, the production and testing of mini CT-s cut from irradiated specimens caused a lot of uncertainty and initiated new researches. To develop the production and testing technology and validate the results obtained on mini specimens an European framework research project FRACTESUS was initiated. STRUMAT-LTO project uses the existing experience and contributes to the practical use of mini CT and SPT to extend the property database, to solve the question of Mn-Ni effect on irradiation degradation and to develop trend curves for safe long operation life of RPV-s.

3. Geometry

The size of the mini CT specimens cut from the Charpy remnants are limited. The theoretical size of the remnants is 10*10*27 mm, however at the fracture surface there is a 2-4 mm deformed zone. The mechanical properties of this zone deviates from the bulk material, consequently this zone can't be used for FM testing. The undeformed remaining piece is about 22 mm long bar. Two sizes have been used until now. One is the rational reduced geometry of the 1 CT specimen given at the ASTM E-1921-21 standard: the size is 10*9.6*4.15 mm. The other one is a simplified geometry: the size is 10*10*4 mm. The advantage of the last one is the simplification in machining and it's rigidity and the line of load is slightly increased. It also reduces the effect of the notches made for fix the extensometer. This last one (10*10*4?) is used by the US NRC in the testing of the Midland weld [2].

Several variations developed for fixing the extensometers. Either the cut of the crack initiation notch is widened until the load line, or a shallow hole with sharp edges is cut at the specimen surface or two shallow V notch are cut at the load line on the two sides of the specimen. Figures 1,2 and 3. show the most used variations of the specimen geometry*.

The required accuracy of the main dimensions is ±0.1 mm and at the diameter of the pin-hole ±0.04 mm. However, in practice it is better to use pins with diameter 2-0.02 mm and drill the pin hole with positive tolerance (2+0.04 mm).

**(Comment: if the pre-cracking notch is widened for the extensometer, it increases the plastic deformation around the loading pins and rarely used.*

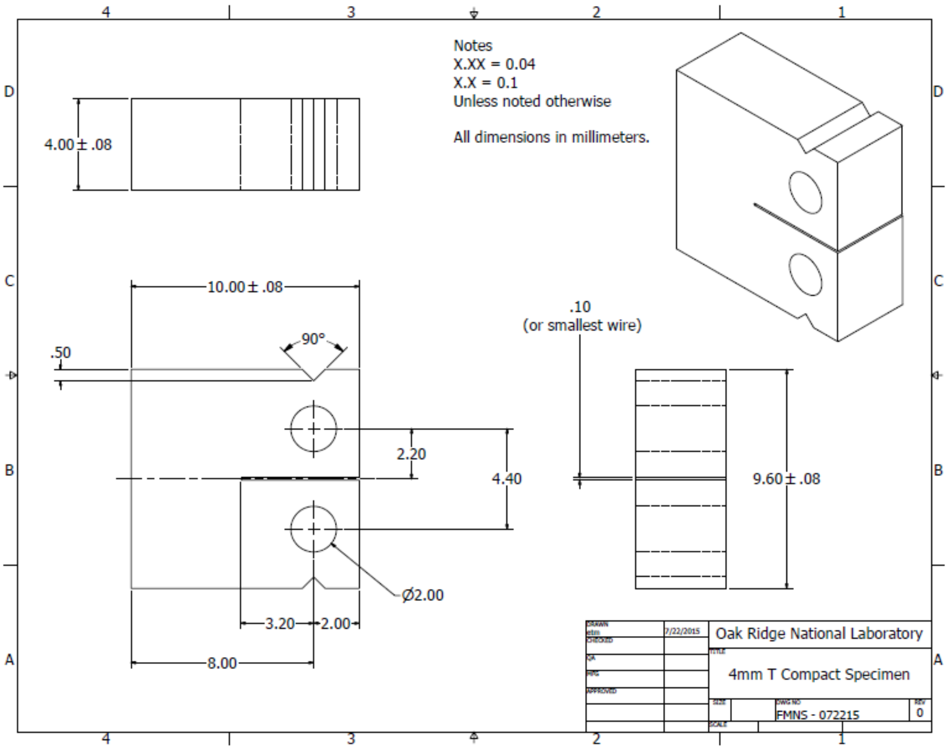
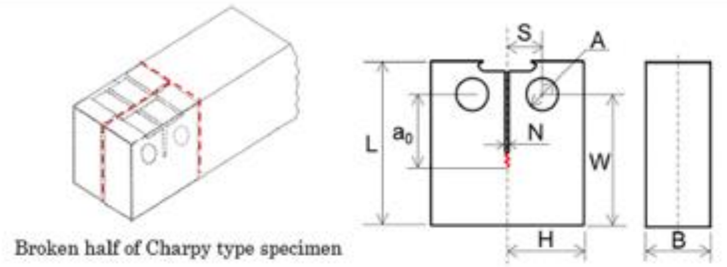


Figure 1. Mini CT specimen geometry used by ORNL [3]



Unit : mm

	B	2H	L	W	N	A	a_0
JEAC4216-2015	4±0.1	9.6±0.1	10±0.1	8±0.1	< 0.5, for straight type < 0.25, for narrow type	2±0.04	4±0.4
Steel B	4±0.1	9.6±0.1	10±0.1	8±0.1	< 0.5	2±0.04	4±0.4
3B, 5B	4±0.08	9.6±0.08	10±0.08	8±0.04	< 0.25	2±0.04	4±0.4

Figure 2. Traditional geometry for inside CMOD measurement. The disadvantage is that the measurement is outside of the load line and during evaluation correction is needed [4].

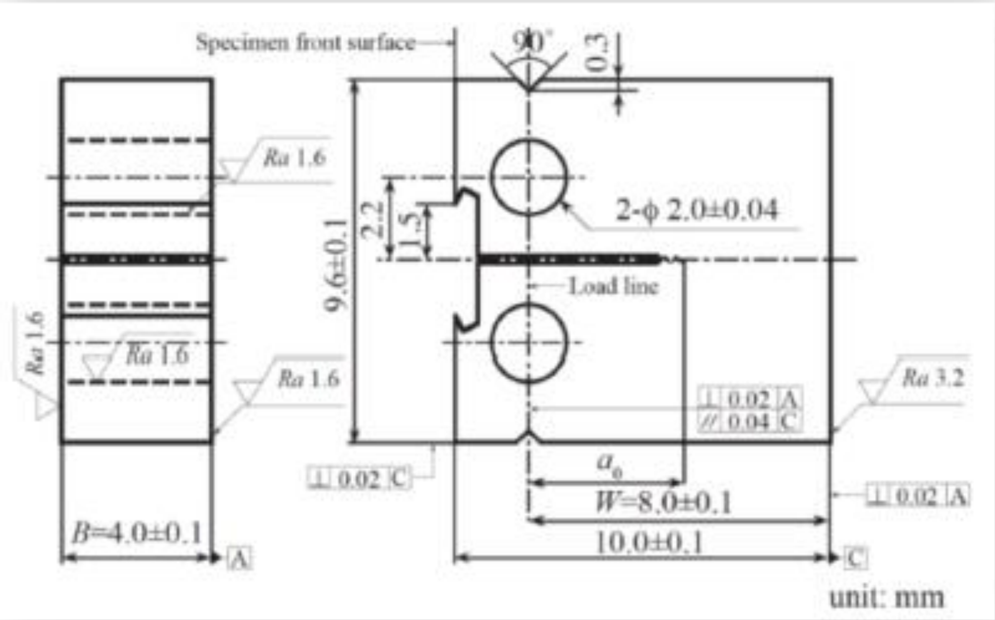


Figure 3. During the US-Japanese Round Robin project the Japanese used two extensometers. There is a surface cut for CMOD extensometer and two small triangular cut notches for load line displacement measurement. The two measurements resulted in practically the same fracture toughness values, Figure from paper [5].

4. Production of irradiated specimens

The first step of the machining is generally to remove the deformed part of the Charpy remnants using any available cutting technology. The fracture surface slice can be kept for fractography. Careful identification of this slices is recommended (e.g. use small coded capsules for storage the slices).

If the original orientation of the Charpy specimen has to be kept, first the remnant should be cut into two 4 mm thick rectangular bars. (Comment: mostly the same orientation of the Charpy specimens used for the mini-CT specimens, but the production of other oriented CT-specimen also available). If the cutting is made by thin wire EDM, the remaining 1-1.8 mm thick plate can be used for metallography, hardness, SANS or ATP testing. In case of half thick Charpy remnants (10*5*55mm- e.g. Lyra-10 specimens in STRUMAT project) the recommended is technology to cut the shape first, and machine the thickness afterwards. Remote controlled (CNC) milling and drilling, or EDM wire machining combined with drilling, or EDM die shrinking machine can be used to produce the mini-CT specimens from irradiated Charpy specimen remnants.

In case of base metal testing the whole remnants can be used and 8 mini-CT-s can be machined from the two remnants of a Charpy specimen. In case of welding specimen, the technology is more difficult. Using macro etching the weldment, the HAZ and the base metal range have to be evaluated, and the mini CT have to be cut from the relevant part only. This limits the number of the specimens possible to cut from the remnants.

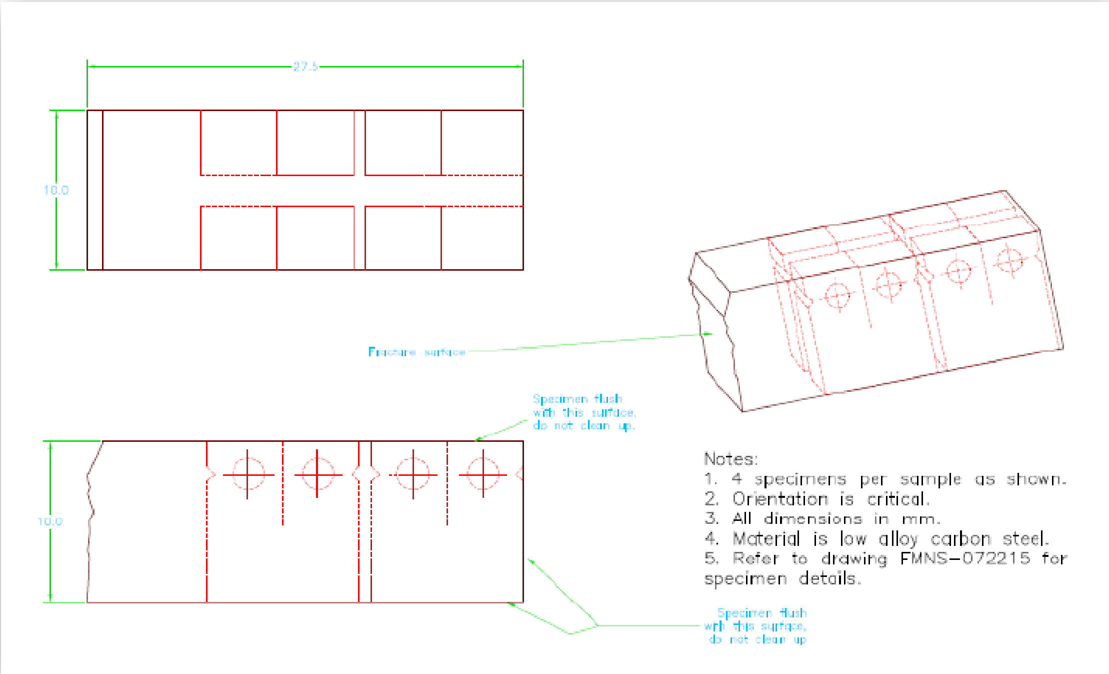


Figure 4. Typical cutting plan for mini CT-s from Charpy remnants. Orientation is the same as the orientation of the Charpy specimens [3].

All specimens should have a clear identity code. The code system should be elaborated before machining of the specimens, and the individual code should follow the specimens in every phase of the production. Engraving (mechanical or laser) is the best technology. However, the use of engraved or print coded capsules, boxes are also good solutions.

5. Pre-fatigue

During pre-fatigue a compliance technology is generally used to evaluate the length of the notch and prefatigued crack. The displacement values of the tensile machine may not be accurate enough to deduce the required size crack. Displacement extensometer or LVDT should be used.

The margin for the a_0 is not proportionally reduced with the specimen size: the notch +pre-crack length margin is ±0.4 mm. However, the CMOD or LLD is directly proportional with the notch length, consequently enhanced sensitivity of the used transducer is required. Due to the small deflection and small cyclic load used at pre-fatigue of mini-CT-s, the compliance pre-fatigue may not produce the required pre-crack lengths. The crack lengths depends on the sensitivity of the servo-hydraulic system and the hardness (spring constant) of the frame of the applied tensile machine. Large load capacity tensile machine may not produce the required size pre-crack when using compliance control.

Optical crack lengths measurement with video microscope is also difficult as the clevises cover large part of specimen.

Electric resistance measurement of the crack growth is a correct solution, see Figure 5, but in practice the use in case of irradiated specimens is difficult, since it requires proper electric contacts production by remote controlled technology [5].

Sensitive LLD or CMOD transducer (extensometer) can be calibrated with a set of differently pre-notched specimens (sharp notch is required, e.g. thin wire EDM notch) and the cyclic wave displacement amplitude, dependent on the applied load and the pre-crack size can be checked by an oscilloscope.

Video or laser extensometer may be applied for deflection measurement, if the response speed is large enough compared with the applied frequency (at least 20-50 measured points are required/cycle).

The frequency for pre-cracking should be selected according to the tensile machine system properties. In practice 10-12 Hz seems to be optimal. The pre-fatigue parameters should be calculated according to the ASTM-E-1921-21 standard. The load must be decreased with the growing crack length according to the rules in the ASTM-E/1921-21. This can be done by compliance-controlled system The required number of cycles for correct pre-fatigue of a steel specimen generally is in the range of 20000-200000 cycles, but this highly depends on the material properties (e.g. grain size etc.).

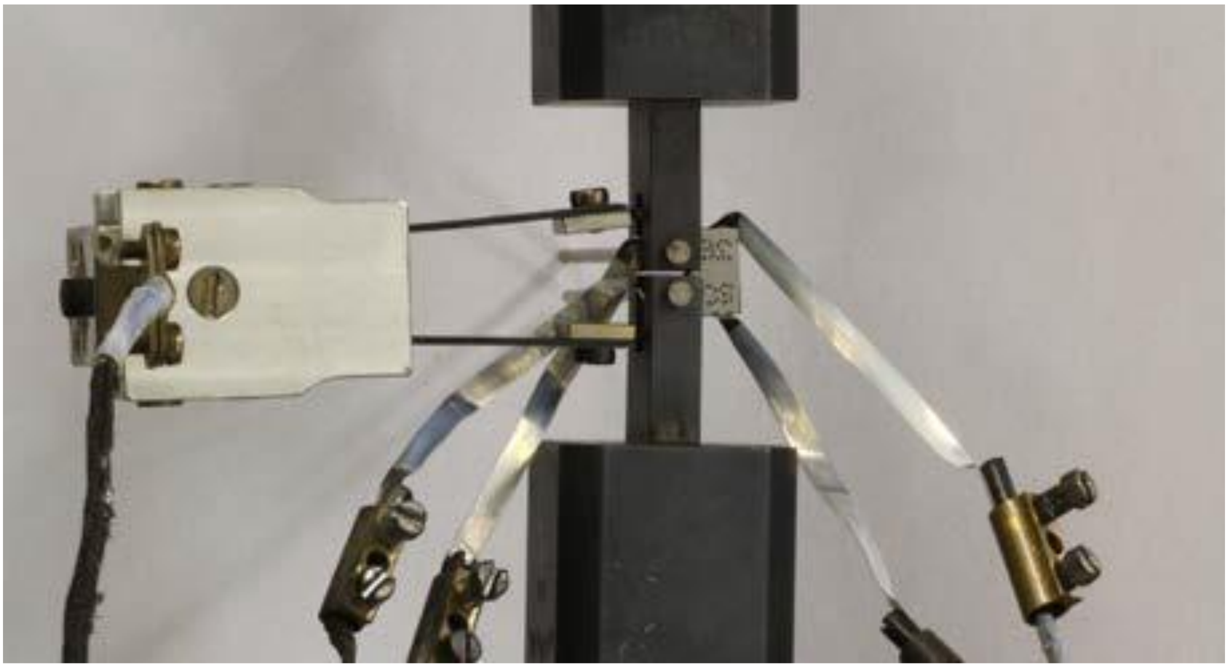


Figure 5. Extensometer and electric resistance crack size measurement on mini CT specimen [5].

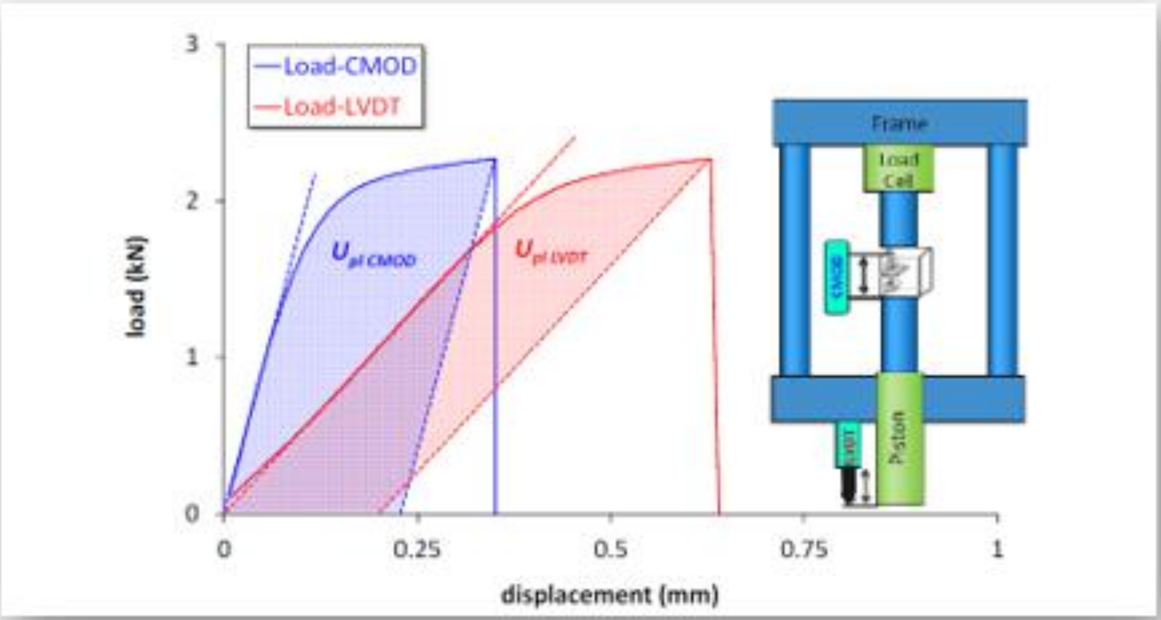


Figure 6. Comparison of CMOD and LVDT measurement [6].

6. Check of the geometry, Crack size

The check of the geometry of irradiated mini-CT specimens is a difficult and time consuming task. Some laboratory has a measuring microscope built into the hot cell. Others are using video microscope. Extensometers can also be used for the testing of the geometry. In some case gauges or test indicator are used to select the pieces deviating from the required geometry. Using NC technology at the fabrication of the specimens allows the random check of the specimen geometry.

The crack size after pre-fatigue should be checked by video or optical measuring microscope. Measured specimen size and pre-crack size should be filled into the testing template according to the relevant standards (e.g.ASTM-E-399-20a, ASTME-1820-20b or ASTM-E-1921-21).

7. Recommended number of specimens

Evaluation of fracture toughness using mini-CT specimens at a certain temperature requires a minimum of 3 valid tests. In case of mini-CT specimens the testing temperature range for cleavage fracture is narrow so material inhomogeneity also could cause deviations. In this case it is better to use 6 or even more specimens. For Master curve evaluation the ASTM 1921-20 standard recommends at least 8 mini-CT specimens. Most of the RPV materials are inhomogeneous, especially the weld. Simplified bimodal calculation requires at least 10 valid tests, and correct bimodal or multimodal evaluation can be performed, if the number of valid tests (tested within the T0±50°C range) is over 20.

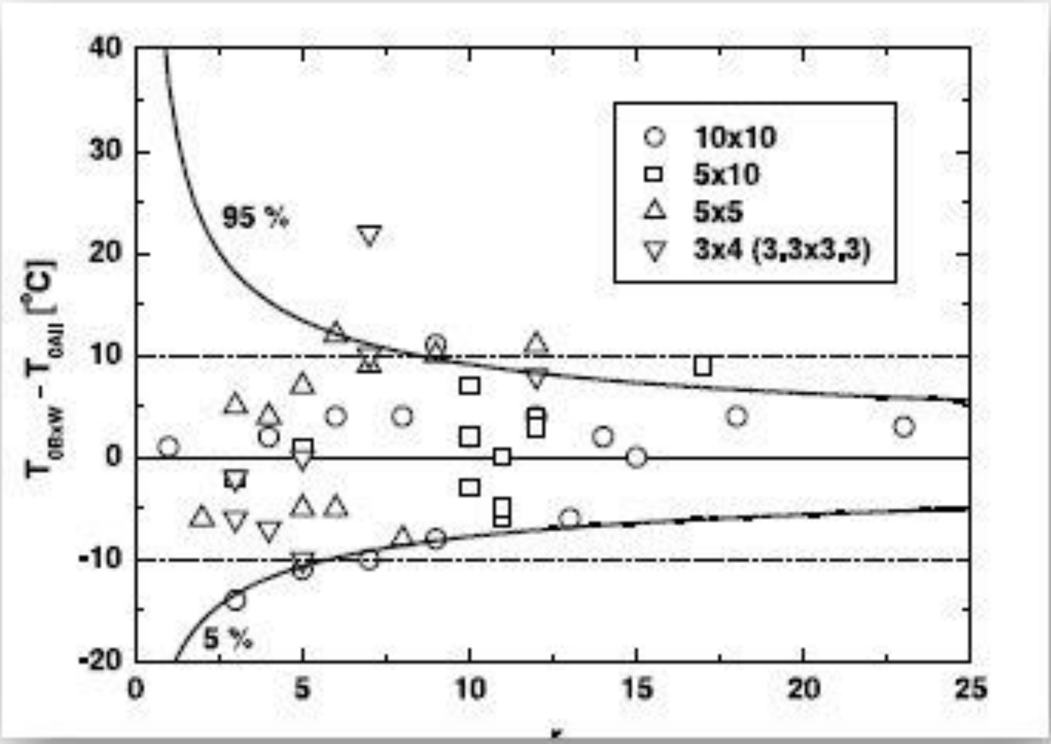
Table 1 shows how many TPB specimens are needed to produce a valid T_0 according to the cross section The ASTM E-1921-21 standard requires only 8 specimens, however in case of mini specimens this may not enough in practice [1].

Specimen type	Number of specimens needed	Amount of material as CVN equivalent
10*10	7	7
5*10	7	3.5
5*5	12	1.5
3*4	28	2.5
3.3*3.3	40	2.5

Table1. Number of different sizes TPB specimens required to produce valid T_0 estimates

The increasing number of tests also reduces the scatter. Figure 7 shows the T_0 scatter in case of TPB specimens as a function of the specimen cross section and size [1].

Figure 7. Effect of number of valid results on the scatter of T_0 offset [1]



8. Testing conditions

The optimal testing temperature for irradiated mini CT specimens is 25-30°C below the expected T_0 temperature. Since the optimal temperature range is narrow, the chance that one limit of the several -required according to the ASTM E-1921-21 standard- is violated is high. Due to the small specimen size the accepted stable crack growth Δ_a is also small (0.25 mm). Small inhomogeneity may cause considerable stable crack propagation before cleavage fracture and may violate the Δ_a criteria. Fractography check of the stable crack growth is recommended, if the load-deflection or CMOD diagram slope partially became negative. Figure 8 shows a typical stable crack growth picture in a mini-CT specimen.

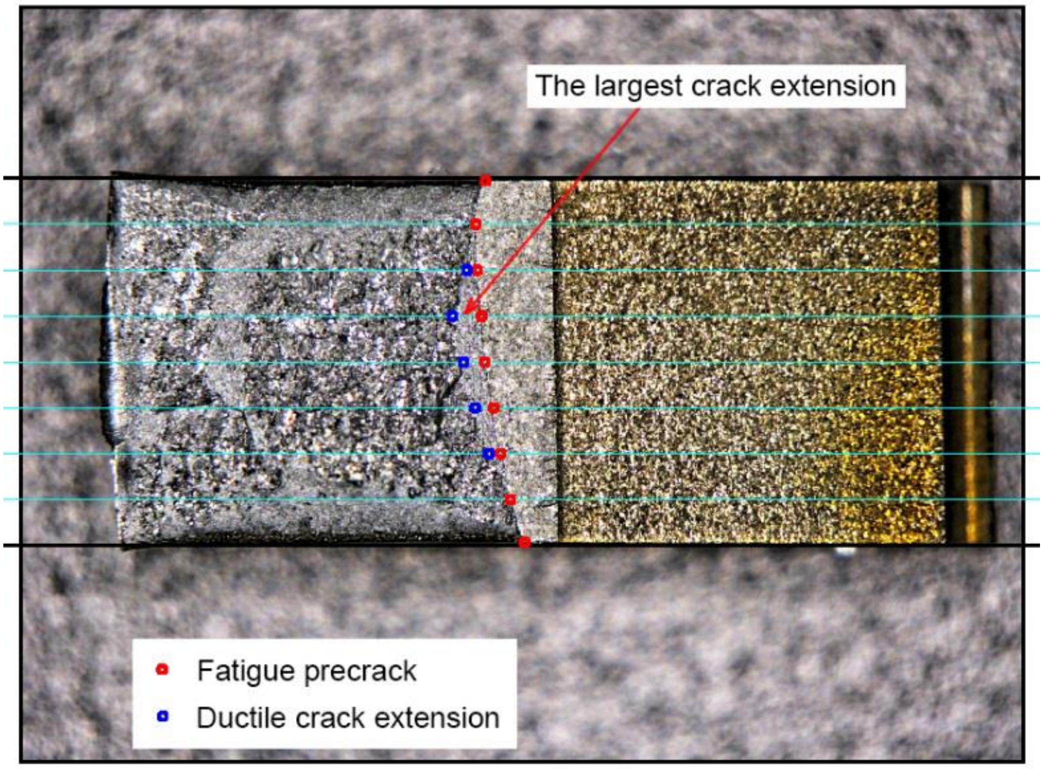


Figure 8. Notch, fatigue pre-crack and ductile crack extension on the fractography picture of a mini-CT specimen [2].

For unirradiated RPV steels the T_0 value can be very low. VTT experienced that the testing temperature -180°C was not low enough to get valid fracture toughness values in the case of one of the STRUMAT model alloy tested by mini CT specimen.

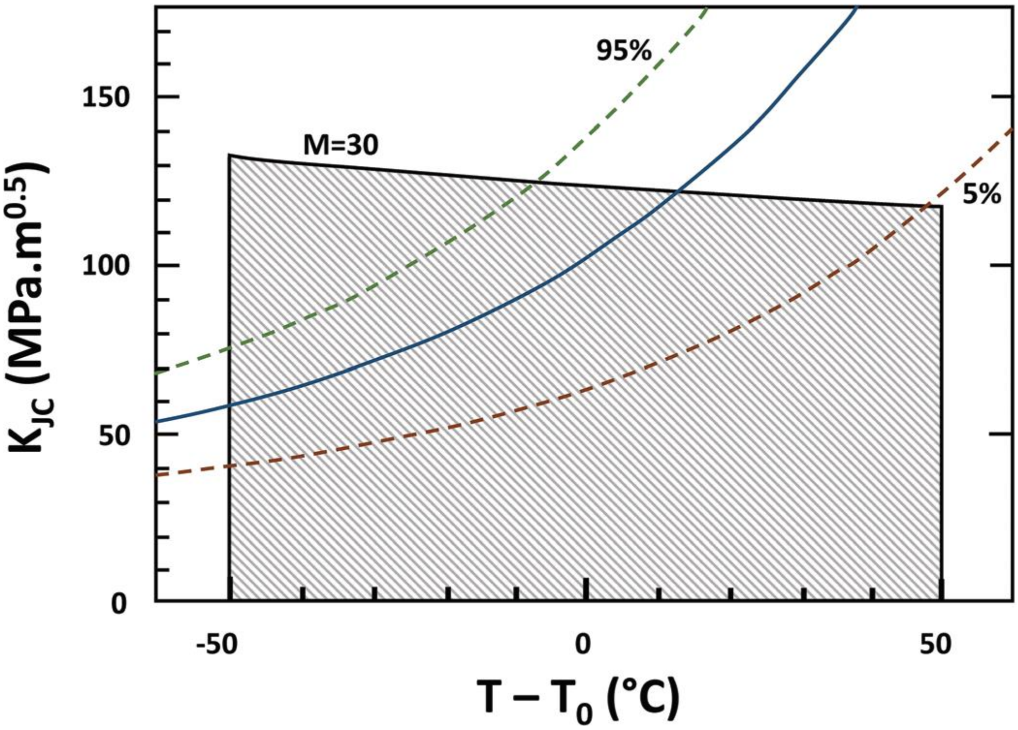


Figure 9. The schematic field of the valid results for Master Curve evaluation [7].

Figure 9 shows the schematic field of the valid results for Master Curve evaluation. Over the validity range the test results are dependent on the specimen size and geometry, and the size correction used in Master Curve is not valid anymore. The tittle of the ASTM-E-1921-21 standard clearly defines that the Master Curve testing is valid for ferritic steels in the transition range.

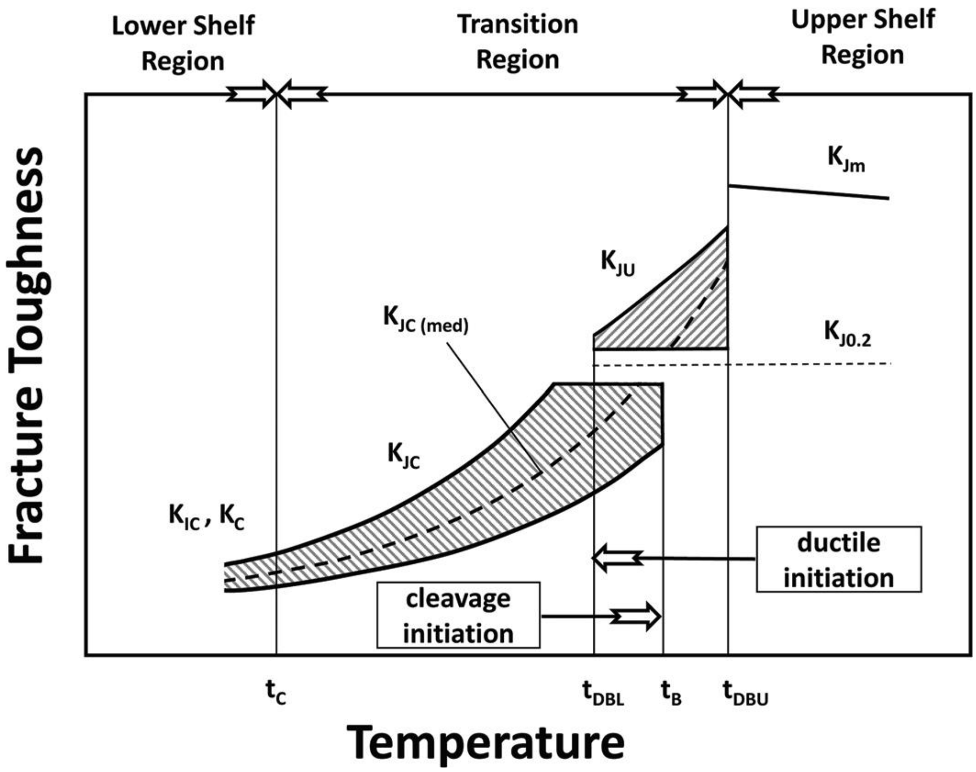


Figure 10. Schematic diagram of fracture toughness behaviour with temperature [7].

Holzmann and Vlach [7] suggested a schematic diagram of fracture toughness behaviour with temperature, see Figure 10, where following fracture toughness parameters are used for an analysis of the fracture behaviour:

- $KJ_{0.2}$—fracture toughness after 0.2 mm of blunting and crack extension.
- KJ_m—value of KJ at the maximum load Fmax for stable fracture behaviour and nonlinear test record.
- KJ_u—post-ductile tearing cleavage fracture toughness; only Jc-tests terminated by cleavage prior to attaining the maximum load Fmax were taken into account.
- KJ_C—fracture toughness for the onset of cleavage fracture after elastic-plastic deformation, but with no prior ductile tearing.
- K_C—the fracture toughness at the onset of brittle fracture; test record linear or with no significant deviation from linearity, but size validity requirements of ASTM E399 are not met.
- K_{IC}—plane strain fracture toughness.

All values of K_J could be obtained by conversion from J-values

The following transition temperatures are denoted in the diagrams:

- t_{DBU}—ductile-brittle upper; the cleavage fracture occurs after certain amount of ductile tearing but prior to attaining the maximum load (the onset of the transition region).
- t_{DBL}—ductile-brittle lower; the end of the region with the above fracture mode.
- t_B—brittle-fracture transition temperature; the onset of the region, where cleavage fracture is initiated ahead of the blunted crack tip but without prior ductile tearing. Due to inherent scatter of material properties, t_B could be within the (t_{DBL}-t_{DBU}) region, where t_C—the lower shelf fracture toughness regime is below this temperature.

At low temperature testing the icing of the specimen, extensometer and the clevis may affect the measurement. Especially the laser and video extensometer use are blocked by the icing of the temperature cabinet window. Inductive or strain gauge type extensometers generally are not compensated for testing below -100°C and may give false results. The icing on the clevis and the specimen destroys the connection between the specimen and extensometer. Deep temperature testing (required by using mini-CT specimen) may causes difficulty for the tough unirradiated RPV steels. In case of irradiated specimens, the T_0 is increased, and it is probably near to the room temperature, the measurement is easier.

Measurement of LVDT can solve the problem (see figure 5) if the testing frame is rigid enough [6]. It is suggested to make calibration tests at room temperature using CMOD or LLD extensometer connected directly with the specimen and also measure the LVDT. If the two measurements give very similar toughness values or LLD and LVDT gauge measure the same displacement than the LVDT transducer can be used.

Reference [12] compared the different clip gauge attachment configures. At mini-CT specimens most laboratory use the mount outside in line with the load as it shown in Figure 11.

Figure 13 and 14 compare the traditional and outside mounting. Outside mounting has two advantages: testing in the load line and the specimen is more rigid in the region of the loading pins. Disadvantage is that when plastic deformation occurs around the pins it affects the measured values [6].

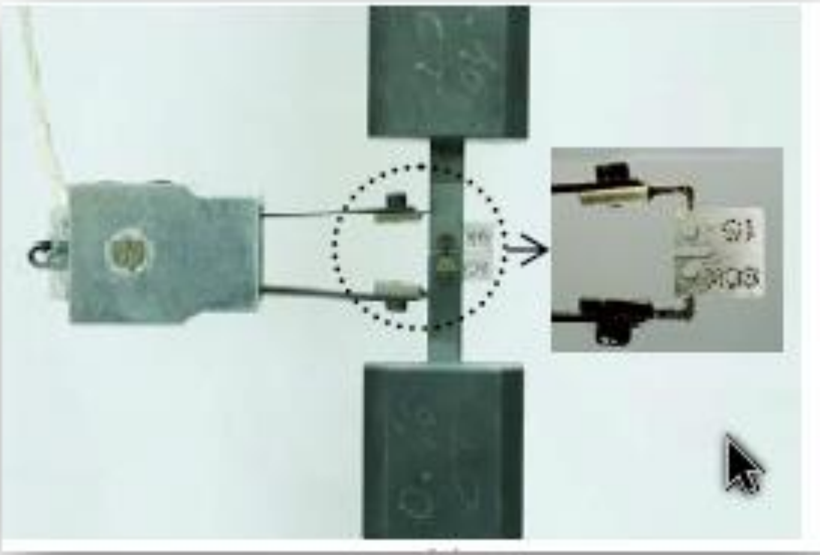


Figure 11. Outside mounted extensometer.

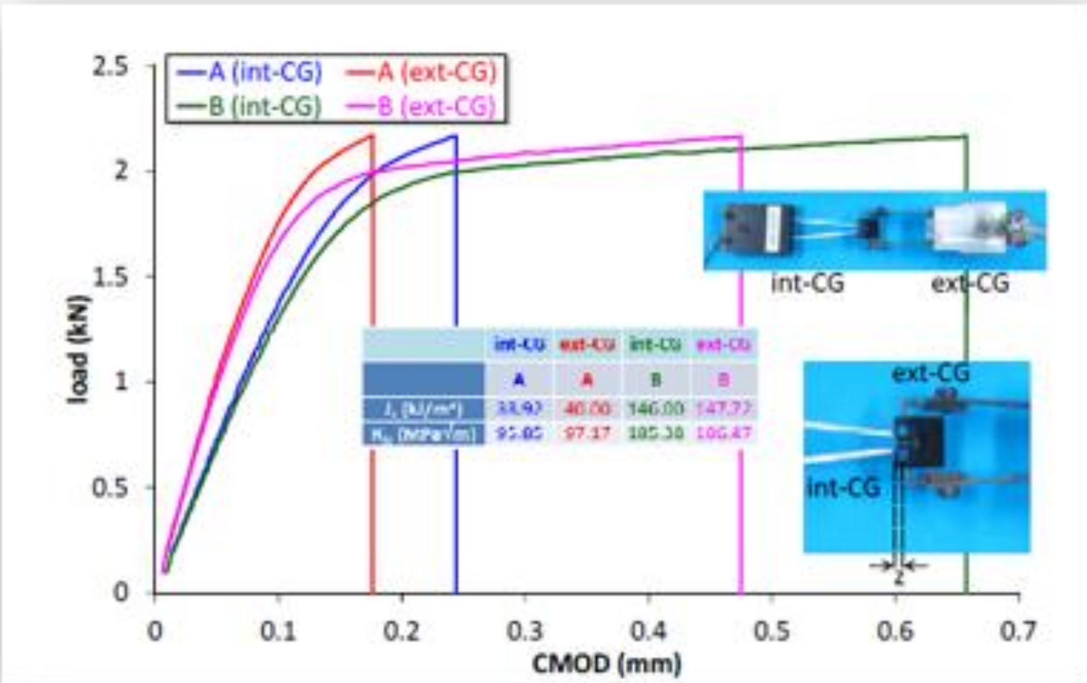


Figure 12. Comparison of the load-CMOD test record with two clip gauge attachment configurations in CG where the clip gauge is mounted on the specimen front face and the out CG where the clip gauge is mounted outside the specimen along the load-line [6].

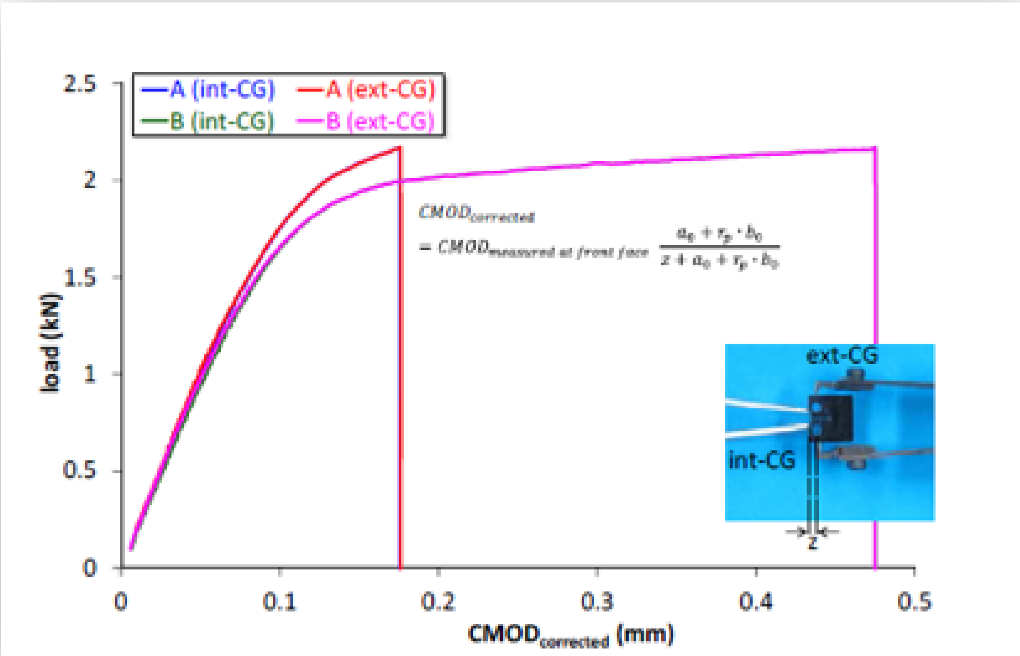


Figure 13. Load CMOD test records after correction for rotation with two clip gauge attachment configurations [6].

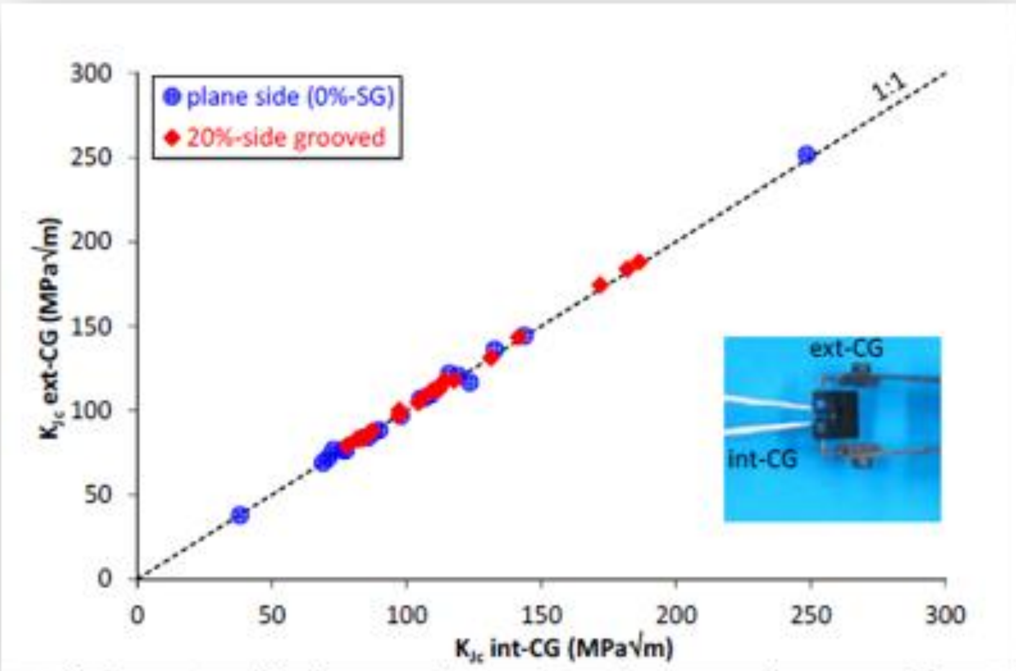


Figure 14. Comparison of the fracture toughness values with respect to clip gauge configuration [6].

R.Chaoudi et al. [6] compared the side grooved mini CT with smooth mini CT results, They found that the side groove effect on the mini CT specimen is negligible (see figure 15) .

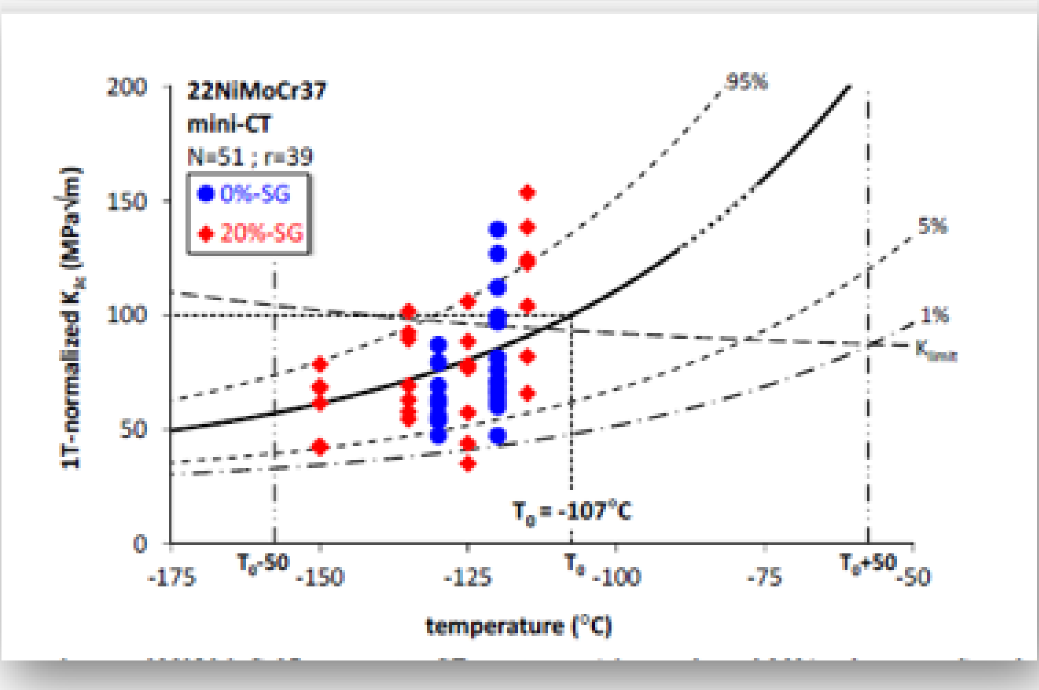


Figure 15. Master Curve on smooth and 20% side grooved mini CT specimens [6].

9. Validation of use of mini specimens

Several publications introduce results proving the fracture toughness measured on unirradiated or irradiated mini CT specimens provides realistic values.

Table 2 and Figure 16 show the results obtained on Midland Beltline weld. Both the irradiated and unirradiated material, tested using large (2 CT, 1CT), medium size (0.5 CT, PCVN) and mini CT specimens provided the same results within ±°C margin. All test results had been re-analysed according to the ASTM-E-1921-21 standard.[5].

Specimen type	Number of specimens N/r	T_o °C	σ_s °C
Unirradiated			
All	84/59	-60	4.6
2T C(T)	14/10	-61	6.0
1T C(T)	32/26	-56	5.3
0.5T C(T)	14/8	-65	7.5
PCVN	19/13	-61	6.4
Irradiated			
All	47/39	29	4.9
1T C(T)	14/11	28	6.7
0.5T C(T)	17/16	26	6.0
PCV N	16/14	33	6.3

Table 2. T_0 values measured on Midland Beltline weld [5] (N= number of specimens tested, r =valid results).

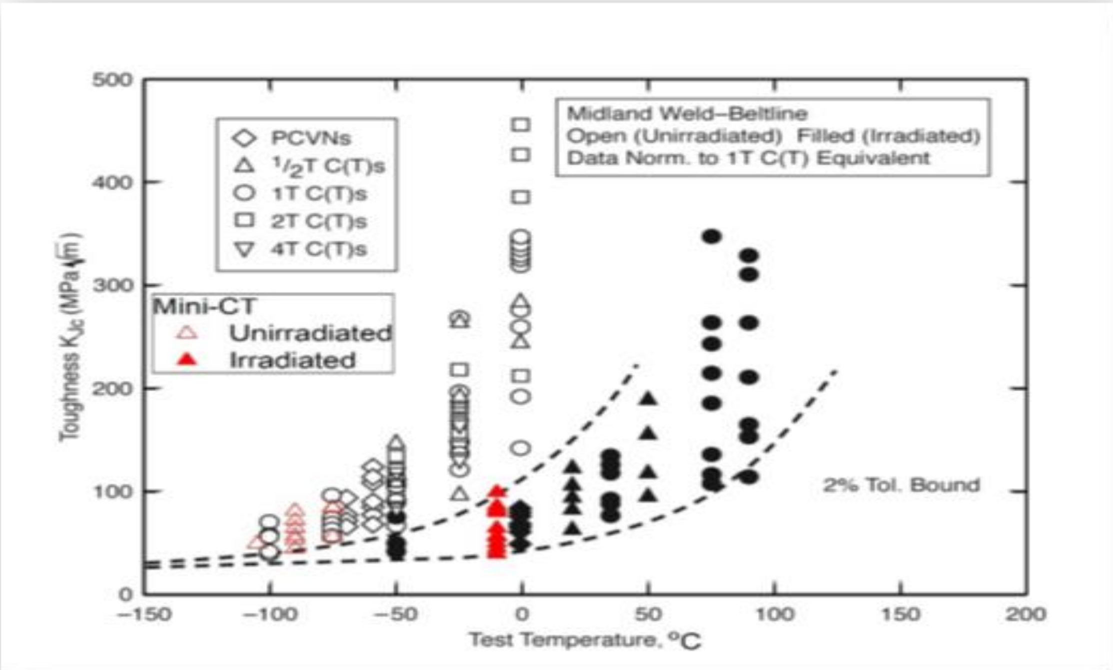


Figure 16. The results of the Midland weld beltline testing using different size CT and TPB specimens [5].

Similar work was performed on 22NiMoCr37 steel at the SCK/CEN [6]. The results verified the use of mini CT specimens, see figure 17. [6]

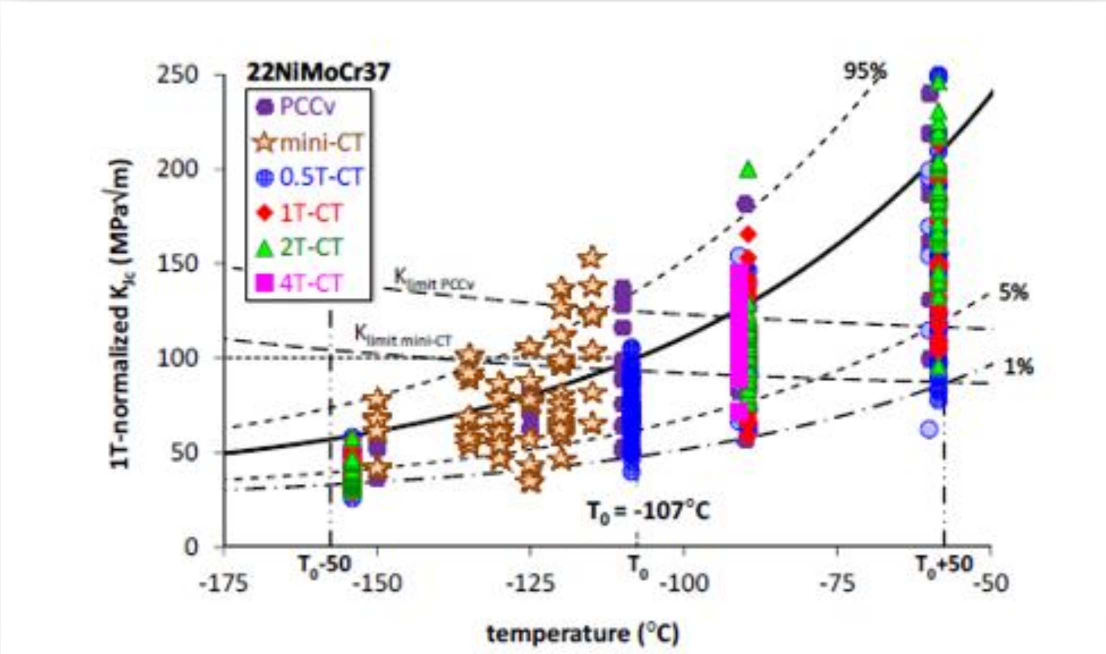


Figure 17. Fracture toughness distribution of 22NiMoCr37 in the temperature range of interest [6].

An irradiated low upper shelf Linde 80 weld metal has been tested by four laboratories as a part of an inter laboratory assessment of use of mini CT specimens for Master Curve determination. This material can exhibit DCG at low temperatures near to the temperature of cleavage fracture toughness initiation. The results compared with the results obtained on larger CT specimens. The evaluation indicated good agreement between the mini CT and the larger specimens, however the testing temperature and specimen number selection are more difficult for mini CT-s in the case of LUS material [5]

10. Conclusions

The large experimental programs on the mini-CT specimens have shown that the usage of mini-CT specimens is appropriate for fracture toughness characterization in the transition regime and application for the master curve. The mini CT size is particularly attractive when specimens are fabricated from half broken Charpy specimens, in particular in the irradiated condition where material is scarce. Moreover, the clip gauge is mounted on the outside of specimen and measures directly the load line displacement, avoiding therefore correction for rotation. Mini CT reliably determine the transition temperature T_0, according to the Master Curve approach. Minimum of 12 mini-CT specimens (equivalent to a volume of 1 and a half Charpy specimen) are required to get acceptable value. The test temperature range of this small specimen is drastically reduced. Ideally, the tests should be done in the region between T_0-50°C and T_0-30°C where the probability of exceeding the maximum allowable toughness is significantly low. Because T_0 is not a priori known, the minimum number of specimens should be increased to 16 (volume equivalent to 2 Charpy specimens) to guarantee a reliable valid T_0-value. Finally, the clip gage use can be avoided by relying on the LVDT measuring the total displacement, which results in a small bias of about 2°C on the Master Curve transition temperature T_0.

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12. Annex 1. Useful correlations at FM and Master Curve testing.

Several correlations exist among for the different mechanical properties. Since the different mechanical properties are measured on different geometry specimen and load conditions these correlations didn't provide exact values, but can be used as first guess of the expected results for preparation of a new measurement.

A1. Correlation between the Charpy Energy and fracture toughness Barson and Rolfe 1970

$$\frac{K_{IC}^2}{E} = 2.2 \times 10^{-4} \quad (CVN)^{3/2}$$

A2. Equations for calculation of approximate values of ultimate tensile strength *UTS(from Rockwell and Brinnel hardness (ASTM E-370)

$$UTS = 0.0047*HRB^3 - 0.9544*HRB^2 + 72.368HRB - 1570\ MPa, \quad (1)$$

$$UTS = 0.0133*HRC^3 - 0.6203*HRC^2 + 24.86*HRC + 401.73\ MPa, \quad (2)$$

A.3 Equation to calculate Yield strength from Vickers hardness [9]

$$\sigma_y^{0.2\%} = 3.62VHN - 228\ (MPa),$$

A.4. Relationship between irradiation induced ΔT_{41J} and ΔT_0 [9]

$$\text{Weld Metals: } \Delta T_0 = 1.0 \times \Delta T_{41J}\ (\pm 26\ °C),$$

$$\text{Base Metals: } \Delta T_0 = 1.16 \times \Delta T_{41J}\ (\pm 36\ °C),$$

A.5. Relation between the $\Delta\sigma_{ys}$ and ΔT_0 (note the low correlation) [9]

$$\Delta T_0 = 0.70 \times \Delta\sigma_{ys}\ (°C)\ (r^2 = 0.66),$$