Joint ICTP/IAEA Workshop on Irradiation-induced Embrittlement of Pressure Vessel Steels

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Small Specimen Test Technologies for measuring mechanical properties of reactor pressure vessel steels

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Small Specimen Test Technologies (SSTT) for measuring mechanical properties of RPV steels

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Outline

- Introduction – General concepts
- Miniature tensile specimens
- Instrumented indentation tests
- Reconstitution of Charpy specimens
- Impact tests on sub-size Charpy specimens
- Fracture toughness testing on sub-size specimens
  - transition region (Master Curve)
  - upper shelf regime (miniature C(T) specimens)
- Standardization of SSTT
- Small Punch Testing
Introduction
Why small specimen testing?

- Evaluating mechanical properties is needed for integrity assessments and life predictions.
- Materials are subject to degradation due to high temperatures, aggressive environment and/or neutron irradiation.
- Evaluation of mechanical properties is by definition a destructive technique.
- If specimen size is small enough, it can become “semi-destructive” (easy repair or even no repair is needed).
Small Specimen Test Techniques have been developed, qualified and applied for the mechanical characterization of reactor pressure vessel steels (unirradiated and irradiated).

Mechanical properties addressed:

- Tensile strength (miniature specimens, instrumented indentation)
- Impact toughness (reconstitution of Charpy specimens, KLST sub-size specimens)
- Fracture toughness (transition and upper shelf regimes, various sub-size specimen geometries)
Several options when only broken Charpy specimens are available

- Sub-size tensile samples
- Charpy reconstitution
- Small cracked round bars
- Sub-size Charpy (precracked)
- Miniature C(T)
The specific problem
Miniature flat tensile specimens (2)
Miniature flat tensile specimens (3)
Miniature flat tensile specimens (4) - Basic “facts” -

- Results from miniature specimens are in good agreement with standard sample data, within a few %

- The most critical aspects are:
  - Misalignments and extraneous displacements during gripping and mounting operations have to be carefully avoided (a special “specimen holder” was developed)
  - The significant influence of the test setup compliance has to be accounted for when determining the elastic portion of the test record (only the last part should be considered)
  - Since data scatter tends to increase for decreasing specimen size, a minimum number of 3 tests per temperature is recommended (preferably 5)
Instrumented indentation tests (1)
Instrumented indentation tests (2)

- Favourable comparison with tensile test results

![Graphs showing yield strength and tensile strength estimation](image_url)
Reconstitution of Charpy specimens (1)
Reconstitution of Charpy specimens (2) - Basic “facts” -

- If insert length is greater than 15 mm, no influence of reconstitution can be appreciated.
- For inserts of 10-12 mm length, a decrease in Upper Shelf Energy (Charpy) and upper shelf toughness (PCCv) can be observed.
- No influence of reconstitution in case of toughness tests in the transition regime (Master Curve analysis).
- The shortest inserts (10 mm) allow changing the sample orientation (e.g. from LT to TL).
Impact tests on sub-size specimens
KLST type (1)

Estimation of USE values for full-size specimens

\[ \text{USE}_{fs} = 29.398 \times 10^{0.2378 \times \text{USE}_{ss}} \]

\[ R^2 = 0.962 \]

\[ \sigma = \pm 11.3 \text{ J} \]
Impact tests on sub-size specimens
KLST type (2)

Estimation of transition temperatures

\[ T_{fs} = T_{ss} + 59.3 \degree C \]
\[ T_{fs} = 1.124 T_{ss} + 58.3 \degree C \]
\[ \pm 2 \sigma = \pm 42.2 \degree C \]

\[ T_{fs} = T_{ss} + 65 \degree C \]
Fracture toughness testing
Various geometries investigated (1)

- Ductile-to-brittle transition regime (Master Curve analysis)
  - precracked sub-size Charpy specimens, P-KLST
  - sub-size cracked round bars, CRB
  - miniature Compact Tension specimens, MC(T)

- Fully ductile regime ($J_{lc}$ values, crack resistance curves)
  - precracked sub-size Charpy specimens, P-KLST
  - miniature Compact Tension specimens, MC(T)
Fracture toughness testing
Various geometries investigated (2)
Fracture toughness testing (transition)
Master Curve analysis (1)

- 0.18-SE(B): B = 4.6 mm
- 0.16-C(T): B = 4.15 mm
- 0.20D-CRB: D = 5 mm

Graph showing a curve with temperature (°C) on the x-axis and fracture toughness (MPa m) on the y-axis, highlighting 5% and 95% transition temperatures.
Irrespective of the specimen geometry chosen, reference temperatures measured from small specimens are in good agreement with those measured from larger samples (within statistical uncertainties).

The main issue is the limited test temperature validity domain, determined by:

- the lower limit of **applicability for** the Master Curve method ($T_o - 50 \, ^\circ C$)
- the specimen measuring capacity (inversely proportional to the specimen ligament length)

From this point of view, MC(T) are preferable to P-KLST (longer ligament $\Rightarrow$ larger validity domain).

The sub-size CRB results can be corrected for loss-of-constraint using a factor derived from FEM analyses.
Fracture toughness testing
Upper Shelf (fully ductile) regime (1)

**J_{lim} - 1TC(T)**

**"Effective" limit for size-independence**

**J_{lim} - PCCv B=4.2 mm / MC(T)**
Fracture toughness testing
Upper Shelf (fully ductile) regime (2)

\[
J_{1T} = J_{MCT} + 0.0013 \times J_{MCT}^{2.0586}
\]
Miniature specimens (of bend or C(T)-type) clearly underestimate the ductile fracture toughness measured from standard 1TC(T) samples.

An empirical correlation can be established which allows estimating the actual $J_{lc}$ with an uncertainty of 34% at the 95% confidence level.

The role of work hardening in lowering the tearing resistance of small samples has been confirmed.

The use of alternative fracture toughness parameters (CTOD, CTOA, Enrst’s modified J-integral) seems to improve the agreement.
Conclusions
Most critical aspects related to SSTT

- Significance of experimental data
- Transferability of measurements obtained from small specimens to actual components under investigation
- Analytical techniques, which can be:
  - equivalent to the “conventional” ones (e.g. Master Curve analysis)
  - specific to small specimen geometries, i.e. based on correlation approaches (e.g. KLST versus full-size Charpy specimens)
- Accuracy of test methods, accounting for the characteristics of the available instrumentation and the magnitude of the signals involved (force, displacement etc.)
Microstructural considerations dictate that only specimens with cross sectional dimensions sufficient to ensure a representative volume of material is tested should be used.

In order to satisfy this requirement, the size scale and mean separation distance of inhomogeneities that exist in the material must be known.

The cross sectional dimension of the miniature/subsize specimens should be at least 3-5 times greater than the largest inhomogeneity.

Therefore, the recommended SS size depends on the microstructure of the investigated material.
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ISO – International Standards Organisation
- TC164 - Mechanical Tests
  - SC1 (Uniaxial Tests)
  - SC4/P (Pendulum)
  - SC4/F (Fracture)
  - SC5 (Fatigue)
- Meets once a year (September/October)

ASTM (American Society for Testing and Materials)
- Technical Committee E08 (Fracture and Fatigue)
- Technical Committee E28 (Mechanical Tests)
- Meet twice a year (May and November)
Both ASTM E8/E8M and ISO 6892:1998 do not explicitly limit the minimum size of the specimen

Both include “subsize” specimens (an example: round specimen with 2.5 mm diameter)

Any alternative miniature/subsize tensile specimen has to be validated and qualified against standard specimens

If microstructural considerations do not come into play, SS should deliver equivalent results to larger samples

⇒ Standardization is not needed, but robust qualification (unirradiated condition) is required
Subsize Charpy specimens (KLST-type, 3 × 4 × 27 mm) are commonly used in the Fusion community for DBTT measurement and materials’ qualification.

Neither ASTM E 23 nor ISO 148 include subsize specimens as such.

However:

- ASTM E 2248 on miniaturised Charpy specimens has been issued in April 2009 (geometries: 5 × 5 × 27.5 mm and KLST).

⇒ Standardization is already happening; correlations with standard specimens should be validated.
In order to obtain valid fracture toughness measurements in case of fully brittle behaviour, large specimens are required.

Small specimens are generally not applicable to fracture toughness testing in the linear elastic regime.

Furthermore, lower shelf conditions have to be avoided throughout the operation of any structure or component.

⇒ This fracture regime is not relevant for RPV integrity assessments.
Fracture toughness properties in the ductile-to-brittle transition region are of primary importance for assessing the integrity of a structure or component.

- ASTM E 1921 (Master Curve) does not restrict the minimum size of a specimen.
- However, validity requirements related to specimen size have to be fulfilled for the results to be valid.
- The most commonly used are the precracked KLST and the miniature C(T) (thickness 4-5 mm).
- Mini C(T) specimens have a larger validity domain than KLST, and should be given higher priority.

⇒ Standardization is not needed; existing standards (basically E 1921) can and should be used.
Fracture Toughness Testing (fully plastic regime)

- Upper shelf fracture toughness properties consist in the initiation value and the crack resistance curve (R-curve)
- ASTM E 1820 and ISO 12135:2002 do not restrict the minimum size of the specimen
- However, validity requirements related to specimen size are imposed
- SS appear to underestimate the actual fracture toughness of the materials
- Correlations with larger specimens should be established and validated

⇒ Standardization is not necessary, but correlations with larger specimens should be qualified
Small Punch Testing
(a really miniature specimen!)

- It’s the smallest specimen ever (typically, TEM disc with 3 mm diameter and 0.25 thickness)
- Can be used for estimating:
  - tensile properties (using empirical correlations or FEM analyses)
  - DBTT values (using empirical correlations)
  - fracture toughness (using FEM analyses; reliability is doubtful)
  - creep properties
- Correlations are strongly material-dependent and need to be carefully validated
- Standards do not exist nor are in preparation (to my knowledge)
  ⇒ Standardization can be pursued, preferably in the ISO framework
    (Americans are not too keen on this)