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Fracture mechanics testing of irradiated RPV steels by means of sub-sized specimens

Deliverable D6.9
Testing protocols and reporting formats

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[1] **Nature**: **R** -Document, report; **DEM** -Demonstrator, pilot, prototype; **DEC** -Websites, patent fillings, videos, etc.; **OTHER**; **ETHICS** -Ethics requirement; **ORDP** -Open Research Data Pilot; **DATA** -data sets, microdata, etc.
[2] **Dissemination level**: **PU** -Public; **CO** -Confidential, only for members of the consortium (including the Commission Services)

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1 Introduction

This report contains the **general common testing protocols** to be followed by FRACTESUS partners when completing the fracture tests developed within the experimental activities of the project Work Package 3 (WP3): "Fracture mechanics testing and post-test analysis", coordinated by HZDR (Germany). WP3 is divided into four tasks:

- T3.1: "Fracture mechanics testing in the transition region using **0.16 C(T) specimens on unirradiated RPV** steels"
- T3.2: "**Round robin** exercise on a selected **irradiated RPV** steel and additional fracture toughness tests on **irradiated materials using 0.16 C(T) specimens**"
- T3.3: "Fractography"
- T3.4: "Supporting experiments based on **small specimen test techniques (In-kind)**"

In this context, WP6 (coordinated by UC, Spain) has prepared project deliverable D6.9, "Testing protocols and reporting formats".

2 Scope

As a previous step to specimen testing, WP2 (leaded by VTT, Finland) will be in charge of materials selection and fabrication of specimens. This includes **unirradiated materials as well as irradiated** ones, reflecting the range of radiation damage conditions that will be representative of operating reactors in the perspective of long-term operation. All the guidelines collected in this document, although not specified hereinafter, are applicable to unirradiated as well to irradiated materials, with the only difference that **tests carried out on irradiated specimens must be performed in hot cell conditions** without exception.

To qualify and demonstrate the ability of miniature (0.16 C(T)) specimens to determine the fracture toughness, **materials that are well characterized by using large (e.g., 0.5T, 1T, 2T C(T)) specimens have been selected**. WP2 had coordinated the definition of this previous characterization, which should follow well-known widely accepted international standards: **ASTM E-8** [1] **or EN-ISO 6892-1** [2] for tensile testing, **ASTM E-1820** [3] **and ASTM E-1921** [4] for fracture tests, **ASTM E-23** [5] **or EN-ISO 148-1** [6] for Charpy tests.

WP3 will then cover fracture testing and post-test analysis of small specimens of the **un-irradiated as well as irradiated** materials selected by means of **0.16 C(T)** samples. The work includes the **pre-cracking** of the samples, the fracture mechanics **tests according to ASTM E1820** [3] and **ASTM E1921** [4]**, the evaluation of the reference temperature T_0 according to the Master-Curve concept (ASTM E1921** [4]**)** following the single-temperature or multi-temperature methods, and the determination of the J-R curve in the ductile regime in some cases. Each partner performs the complete **analysis based on preferably 16 tests, with a minimum of 12 tests** [7].

Deliverable D.2.1 [8], collects the **list of materials to be used in FRACTESUS**, which is shown in Table 1 extracted from it.

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Material	Type	Cu [wt%]	P [wt%]	Ni [wt%]	Mn [wt%]	Fluence [E19 n/cm²]	T0 [°C]
15Kh2MFAA	BM	0.05	0.01	0.1	0.49	0	-104
15Kh2MFAA	BM	0.05	0.01	0.1	0.49	I_1 = 20 (E > 0.5MeV) (at 270°C)	TBD
15Kh2MFAA	BM	0.05	0.01	0.1	0.49	I_1A (Annealed at 470°C)	TBD
15Kh2MFAA	BM	0.05	0.01	0.1	0.49	I_1A I_2 = 20 (E > 0.5MeV) (at 270°C)	TBD
A533B (JRQ)	BM	0.14	0.018	0.83	1.39	0	-71
A533B LUS (JSPS)	BM	0.24	0.028	0.43	1.52	0	+8
ANP-4	BM	0.05	0.006	0.84	0.85	4 (at 280°C-286°C)	-78
SA 533 B1 (MVE)	BM	0.041	0.005	0.632	1.42	0	-119
SA508 Cl.3	BM	0.04	0.008	0.93	1.37	0	-43
10KhMFT	WM	0.11	0.047	0.14	1.16	IA	-11.6
10KhMFT	WM	0.11	0.047	0.14	1.16	1.6 (I) (at 270°C)	74.4
73W	WM	0.31	0.005	0.6	1.55	0	-64
73W	WM	0.31	0.005	0.6	1.55	1.5 (E>1MeV) (at ~288°C)	34
ANP-5	WM	0.22	0.015	1.11	1.14	0	-38

Table 1. *List of materials to be used in FRACTESUS*

3 Testing protocol for 0.16 C(T) specimens

In this section, considerations about the main issues related to fracture tests on 0.16 C(T) specimens are discussed. Fracture tests shall follow **ASTM E-1820** [3] **and ASTM E-1921** [4] standards. The specimens design collected in this document is **in agreement with deliverable D.2.1, Test matrix of FRACTESUS** [8], further details can be found in it.

3.1 Specimen geometry and fatigue precracking

0.16 C(T) specimens of **B=4mm of thickness (Mini-CT)** (then W=2B=8mm) will be employed. The geometry of the Mini-CT specimens (see Figure 1) **must follow the specifications of ASTM E-1921-10E1** [9]. As stated in literature [10], the aforementioned geometry will differ from ASTM E-1921 [4] requirements in the width of the machined slit, which should be 0.01W (0.08 mm). This dimension is very difficult to be fulfilled due to the limitation in the available minimum **wire diameter used in slit machining (~ 0.1mm)**, which makes **the slit width to range between 0.15 mm and 0.2 mm in practice**. Additionally, **side-grooving will not be used in FRACTESUS tests**. Figure 1 shows a possible option of 0.16 C(T) specimen (see Note 1 for details on deviations).

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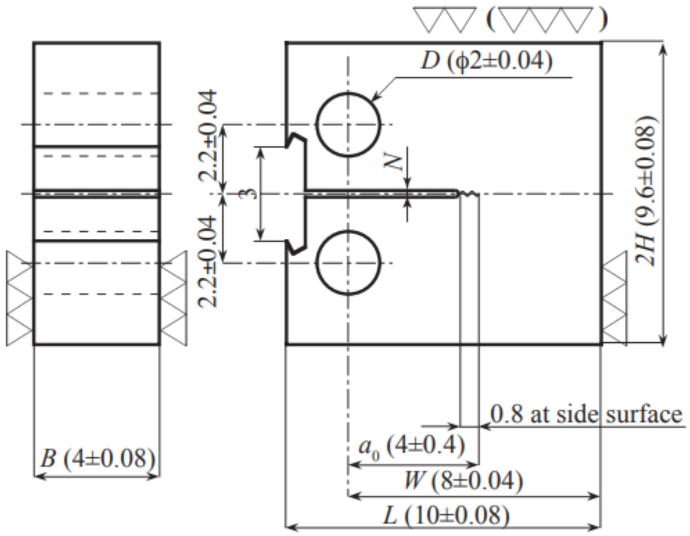

Figure 1. *Example of geometry of 0.16 C(T) specimen to be employed (taken from* [10]*) (see Note 1).*

Note 1:

- *Figure 1 includes dimensions and surface finish.*
- *Differences in fabrication setups results in deviation in the specimens. These differences must be measured and recorded, and may be utilized in further analyses* [7].
- *The specimens that do not strictly fulfill the current standard specifications will also be assessed as they may provide important information. In particular, a number of unnecessary requirements may discard valuable test results. All these effects will be investigated to provide the most appropriate specifications* [7].
- *Table 3 and Table 4 collect all the possible specimen geometries allowed in the project.*

The **notch length resulting from the wire-cut slit should be close to 3.0 mm**, with a subsequent recommended fatigue pre-cracking between 3.6 mm and 4.4 mm, thus leading to a target final pre-crack length of **0.5W±0.05W**, that in any case must fulfill **ASTM E-1921** [4] limitations. The crack length will be controlled by the expressions collected in the aforementioned standards from the load and Crack Mouth Opening Displacement (CMOD), or load-line displacement, amplitude values measured in real time. **Once the test is finished, the real initial crack length will be verified, and corrected if necessary**, by measuring the crack length at 9 points on fracture surface [10], **as indicated in ASTM E-1921** [4]. **Load-line displacement data will be obtained and recorded during the test**.

As specified in D.2.1 [8], **no side-grooving will be used**. On the other hand, although nominal specimen dimensions have been aforementioned, **slight differences in the final geometry employed by each testing laboratory are expected**. Table 3 and Table 4, extracted from [8], collect all the geometrical combinations included in FRACTESUS 0.16T C(T) testing and their nominal dimensions respectively.

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Laboratory	W [mm]	Wtot [mm]	a0 [mm]	B [mm]	H [mm]	H* [mm]	ΔFF [mm]	ΔLL [mm]
CCFE	8	10	3	4	9.6	2.2	1.5	4.5
CEA	8	10	2.4	4	9.6	2.2	1	-
CIEMAT	8	10	3.2	4	9.6	2.2	-	4.3
CRIEPI	8	10	3	4	9.6	2.2	1.5	-
FRA-G	8	10	2.7	4	9.6	2.2	1.25	-
HZDR	8	10	3	4	9.6	2.2	1.5	-
KIT	8	11.5	2.5	4	9.6	2.2	2	-
MTA-EK	8	10	3.2	4	9.6	2.75	1	4.5
NRG	8	10	3.2	4	9.6	2.2	-	4.5
NRI	8	10	3	4	9.6	2.2	1.5	-
PSI	9	11.25	2.5	4.5	10.8	2.475	-	-
SCK CEN	8.3	10	2.7	4.2	10	2.3	-	4.5
UC	8	10	3.2	4	9.6	2.2	1.5	-
UoB	8	10	3.2	4	9.6	2.75	-	4.5
VTT CNS	8	10	3.2	4	9.6	2.75	-	4.5
VTT RH1	8	10	3.2	4	9.6	2.2	1	4.5

Table 3. *Specimen dimensions of different participating laboratories in the FRACTESUS project.*

Label	Description	Nominal measure [mm]
W	Specimen width	8.00
Wtot	Full width	10.00
H	Specimen height	9.60
a0	Length of machined notch	3.20
H*	Half span of applied load points	2.75
ΔFF	Half span of front face displacement measurement points	1.25
ΔLL	Half span of load line displacement measurement points	4.50
D1	Diameter of hole #1	2.00
D2	Diameter of hole #2	2.00
B	Specimen thickness	4.00

Table 4. *Dimensions measured in the dimensional round-robin exercise.*

3.2 Testing equipment

For the Mini-CT (0.16 C(T)) fracture tests, the following equipment, with the described requirements each, should be used:

- Dynamic machine:
  - Servohidraulic or electromechanic machines, that allow the application of **cyclic loads** for a proper pre-cracking, will be employed. **Machines must be calibrated and allow displacement control and load control**.

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  - **Load cells employed must be accurate enough** for the loads to be measured during the test. **Load cells must be calibrated.**
  - **A correct alignment of the machine axis** must be ensured, in order to avoid second order effects to the samples. For this purpose, testing **fixtures incorporating kneecaps are always recommended** (see section 3.3) even if the machine alignment is certified.

- Climatic chamber:
  - For low-testing temperatures, a **climatic chamber** must be incorporated to the testing set-up, in such a way that **the Mini-CT being tested** (plus the clevis, the COD, and auxiliary devices, when necessary) **is contained in it** (see Figure 2).
  - The **refrigeration of the chamber** can be obtained either by an **incorporated industrial cold equipment** (compressor, radiator, etc) **or by injecting gaseous elements** (i.e. Nitrogen or Argon).

- Thermocouples:
  - **Temperature in the testing control volume must be monitored by means of a thermocouple** during the fracture testing.
  - ASTM E-1921 [4] recommends monitoring and controlling the testing temperature directly on the specimen. However, in small specimens the dimensions and weight of the thermocouple become relatively large, that might interfere with the mounting of the specimen. In order to avoid this, **thermocouples can be attached to the clevis** beside to the specimen [10] (see Note 2).
  - **The thermocouple must be attached in such a way that a direct and constant contact is assured during the whole tests**. This attachment could be mechanical (screwed, clamped, etc) or glued; if glues or resins are used its durability must be assured during the complete test. Some authors have attached thermocouples to the back surface of the specimens by means of a spring fixture mechanism [9].

    Note 2:
    - *Literature* [10] *assumes temperature differences of 3ºC between clevis and specimen.*

- COD extensometer or LVDT:
  - **Load-line displacement** (LL) **data will be obtained and recorded during the test** by means of a COD extensometer or by using a LVDT. When CMOD measurements are obtained at the specimen front face (FF), they should be corrected to LL values. For this purpose, in order to fulfill all the aforementioned variations in specimen geometry allowed, the following conversion based on the geometrical relation (Landes 1980) [11] will be used; which is also proposed in ASTM E-1921-03 [12] to be R=0.73 when nominal values of a/W=0.5, X/W=0.25 and r=0.3519 are used.

$$\boldsymbol{R = \frac{v_{LL}}{v_{FF}} = \frac{a/W + r \cdot (1 - a/W)}{a/W + r \cdot (1 - a/W) + X/W}}$$

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$r \cdot (W - a)$ being the distance of the rotation point from the crack tip, and X the offset between front face and load line.

- A Clip On Gauges **(COD) extensometer,** with a recommended minimum measuring base of 3.0 mm, must be disposed if it is intended to obtain the displacement amplitude (CMOD) at the specimen front surface [8] and then **convert these data into the load-line displacement**. Given the specimen reduced dimensions and the fact that electrowire cutting will be already used to machine the slit, **knife-edges can be incorporated in the specimen surface during the machining** process (see Note 3).
- A **LVDT comparator placed directly on the load-line of the specimen** will be disposed if it is intended to obtain the load-line displacement amplitude directly.
- The **COD or LVDT must be calibrated** and **certified to work at the temperatures** of the tests being performed.

Note 3:

- *Bolt COD mounting on the specimen could be an alternative, but it is not the preferred option, given the reduced dimensions of the specimen and the bolt holes effect on it. Glued knives on the specimen surface are not recommended to mount the COD either, as results could be distorted.*

- Data acquisition system:
  - During the tests, **data must be registered continuously**, and then transferred to the testing report Excel format (see section 5). **Load and load-line displacement continuous registers will be provided as a result** for further data treatment [4]; other data such as material properties and crack length (measured in the fracture surface) will also be incorporated into the testing report Excel format.
  - For this purpose, a machine-incorporated, or an external, **data acquisition system of at least two channels** (load and COD or LVT) must be used.
  - Data can be also taken during the precracking process for control purposes, but these data will not be used for further analysis.

3.3 Test fixtures

Clevis must follow the requirements of ASTM E-1921 [4] **for the C(T) specimen geometry selected** (W=2B=8mm). Then, the test fixtures necessary to connect the clevis and the testing machine, and pass throw the climatic chamber walls when necessary, will be conveniently defined. **Fixtures incorporating kneecaps are always recommended** (see Figure 2) even if the machine alignment is certified, in order to correct any other misalignment introduced by the fixtures.

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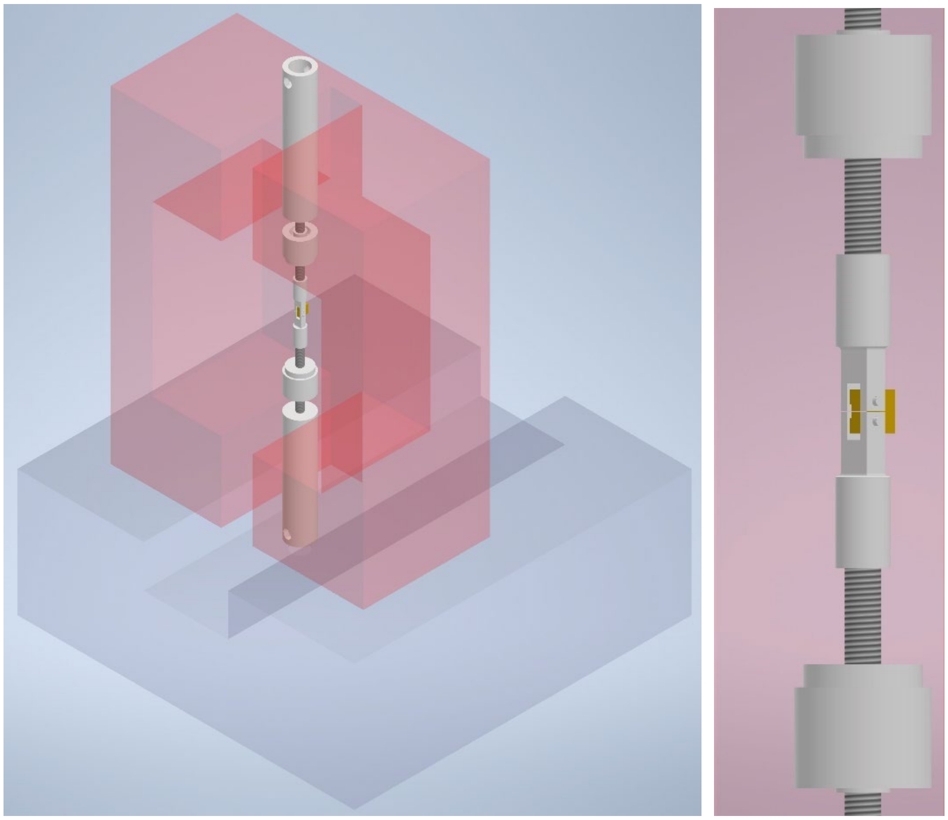

Figure 2. *Example of proposed experimental set-up.*

3.4 Testing rate and fractography

Tests will be carried out in displacement control, as recommended by ASTM E-1921 [4]. Literature [10] collects testing rates in the **dK/dt range of 0.1 to 2.0 MPa.m$^{1/2}$/sec**, so it must be assured that the testing machine is capable of applying them.

Once the test is finished, **samples will be immediately dried in order eliminate all possible water traces** that can appear by condensation and may generate surface oxidation. **Then, fracture surfaces should be examined by using Scanning Electron Microscopy (SEM)**.

SEM analysis will include fractography observation in order to determine **fracture micromechanisms and crack initiation sites**. If possible, a statistical distribution of crack initiation sites along the crack front will be generated. Preferably, **each participant will characterize its tested specimens**.

4 In-kind small specimens test techniques

As mentioned in the introduction, **task T3.4 includes supporting experiments using small testing techniques**. The materials employed will be the same to those used in 0.16 C(T) specimens, plus additional (if necessary) RPV materials.

Possible techniques included here are the following:

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- **Small Punch (SP) tests**. For this purpose, all the tests will be completed according to the recommendations of the European Standard Draft [13], in agreement with ASTM E-305-20 [14].
- **Depth sensing micro hardness** tests to estimate the true stress-strain curves of irradiated materials (BZN). The reference standard(s) regulations in the material will be followed for this purpose.
- **Small specimen technology (mini-CT, mini-DCT) is applied to a structural material for fusion applications (Eurofer)** to validate the approach of WP3 also for f/m steels (SCK•CEN). The reference standard(s) regulations in the material will be followed for this purpose.

5 Testing report models

Data acquired during the test will be collected in a **testing report Excel format** supplied to the partners, and prepared by WP6, which will consist of **two tabs**:

- **Raw data and calculations tab according to ASTM E-1921 [4]:** this tab will contain the **direct data derived from the test**, which are **load** (N) **and load-line displacement** (mm). **In this sheet the data range available is up to 10^5**, so if longer tests need to be considered the sheet, or the data, should be modified by the user. The tab has two columns to introduce CMOD obtained by a COD at the front face or load-line displacement obtained by a LVDT; leaving the unused column blank the data sheet will directly fill a third column with the load-line displacement corrected value (if necessary) according to the aforementioned Landes proposal (whose parameters can be also introduced). Other data such as material properties and crack length (measured in the fracture surface) will also be incorporated into this tab, which contains also the calculations to obtain the fracture toughness testing result. **The sheet will give as direct results K_{Jc}, $K_{Jc\text{-}Limit}$, $K_{Jc\text{-}\Delta a}$, verify all the possible censures and finally determine if the test is censored or not and calculate the equivalent $K_{Jc\text{-}1T}$ value.**

- **Data summary tab:** this tab **shows the main outputs of the test in one sheet** and has the right size to be directly printed in A4. It directly takes some results from the raw data tab, including the **equivalent $K_{Jc\text{-}1T}$ value, and if it is censored or not**, a **P-COD graph**, **testing temperature, initial crack length** (measured in the fracture surface), **material properties**, etc. There are specific areas also, to manually introduce other result data, such as a picture of the **fracture surface** or **fractography**. **This data summary tab converted to PDF, and no other document, will be uploaded to the repository as each 0.16T C(T) test result carried out in FRACTESUS project**.

Finally, as **T_0 calculation according to ASTM E-1921 [4] will be performed using NASA T0TEM free software** [15], another Excel reporting model will be created for each material in order to be used as an input for the software. This Excel sheet will **include all the tests carried out at all the temperatures** (if several) **per each material** characterized using 0.16T C(T) specimens. **For each test testing temperature, $K_{Jc\text{-}1T}$ value, and if it is censored or not** will be introduced. It has the right size to be directly printed in A4, and when **converted to PDF will be the only document uploaded to the repository as an index of all the 0.16T C(T) tests carried out per each material in FRACTESUS project**.

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