Ref. Ares(2021)3688978 - 04/06/2021

Call: NFRP-2019-2020
Topic: Innovation for Generation II and III reactors

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

Deliverable D2.1
Test matrix of FRACTESUS

Grant Agreement No: 900014

Contractual delivery date: Month 07

Actual delivery date: Month 09 (04/06/2021)

Lead beneficiary: P3 – VTT

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No 900014.

Grant Agreement No.	900014
Project full title	Fracture mechanics testing of irradiated RPV steels by means of sub-sized specimens (FRACTESUS)
Deliverable number	D2.1
Deliverable title	Test matrix of FRACTESUS
Nature[1]:	R
Dissemination level[2]:	PU
Work package number:	WP2
Work package leader:	VTT
Author(s):	Pentti Arffman
Keywords:	fracture mechanics, miniaturization, test matrix, material selection, specimen design

The research leading to these results has received funding from the Euratom research and training programme 2020-2024 under grant agreement No 900014.

[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)

Table of Contents

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

1 Introduction

The primary objective of FRACTESUS project is the validation of fracture toughness testing of irradiated materials using miniature compact tension (mini-C(T)) specimens. Fracture toughness testing is done by means of brittle fracture initiation transition temperature testing and related master curve determination. This methodology is a direct material toughness evaluation and one of the most modern standardised methods of investigating metallic material properties. Miniature specimens can be manufactured from broken Charpy impact specimens, which has a tremendous potential for surveillance programs and long-term operation of European nuclear power plants.

This report presents the test matrix of FRACTESUS, and as such the roadmap for all testing to be performed within the project. The test matrix aims to provide optimal groundwork for reaching the goals of the project. Such groundwork is laid by considering the materials, participants and specimens. These considerations are laid out in addition to the test matrix.

2 Relevant standards

Currently, ASTM standard E1921 [1] offers the only standardised test method for determination of ductile-to-brittle transition reference temperature T_0 in the Western scientific community. Reference temperature T_0 is defined as the temperature at K_{Jc} = 100 MPa√m of the brittle fracture initiation toughness transition curve, determined using the master curve methodology.

Testing specifications of E1921 refer extensively to the more established ASTM standards E399 [2] and E1820 [3]. As such, these standards should be regarded important sources of information for fracture mechanical testing of compact tension specimens.

The ASTM standard E691 [4] and ISO standard 17043 [5] regarding proficiency testing and interlaboratory studies were utilised in planning of the test matrix and will be utilized in evaluation of results, along with ISO 13528 [6] regarding statistical methods for use in proficiency testing.

3 Participating laboratories

All participants in material testing and investigations are listed in Table 1, along with their abbreviations.

Table 1. Research organisations participating in testing in FRACTESUS project.

Institute	Abbreviation
Studiecentrum voor Kernenergie / Centre d'Étude de l'Énergie Nucléaire	SCK CEN
ÚJV Řež, a.s.	NRI
Teknologian tutkimuskeskus VTT Oy	VTT
Commissariat à l'Énergie Atomique et aux Énergies Alternatives	CEA
Framatome GmbH	FRA-G
Helmholtz-Zentrum Dresden-Rossendorf eV	HZDR
Karlsruher Institut für Technologie	KIT
Bay Zoltan Alkalmazott Kutatási Közhasznú Nonprofit Kft.	BZN

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Magyar Tudomanyos Akademia Energiatudomanyi Kutatokozpont	MTA-EK
Nuclear Research and Consultancy Group	NRG
Slovenska Technicka Univerzita v Bratislave	STUBA
Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas	CIEMAT
Universidad de Cantabria	UC
Paul Scherrer Institut	PSI
The University of Birmingham	UoB
Culham Centre for Fusion Energy (United Kingdom Atomic Energy Authority)	CCFE
Central Research Institute of Electric Power Industry	CRIEPI
Canadian Nuclear Laboratories	CNL

4 Material selection

Selection of materials to be investigated in FRACTESUS followed the objectives of the project. Materials to be studied in the project are listed in Table 2. The value of the materials was considered from the perspective of the nuclear community, and all materials chosen portray steels with relevance to the nuclear power industry.

Selections were made from already available materials in order to study materials with previous results and erase budget and schedule concerns rising from production of new materials. Queries to participating laboratories revealed an assortment of materials, which established the ground for material selection. In addition to simple availability, the participants provided information on the material, including previous results.

A large variance in properties between materials in the test matrix is advantageous, since it means that the range of materials validated for miniaturised testing is larger. Three of the main differences between steels are chemical composition, manufacturing method and later aging/treatment of the material. The focus of the project is in irradiated materials, and as such the effect of the last factor focuses on irradiation, instead of e.g. thermal effects.

The test matrix includes materials from both eastern and western type reactor pressure vessels. Plates, forgings and weld metals are included. The chemical composition, particularly the amount of copper, phosphorus, nickel and manganese, varies from reasonably pure to intentionally enriched low-alloy steels. While most of the materials are unirradiated, included are irradiated, annealed and re-irradiated steels. The reference temperature T_0 of unirradiated materials according to previous tests range from -134°C to +8°C, and the irradiated materials have even higher T_0 values.

The materials used in the interlaboratory exercises are described in Annex 1.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Table 2. List of materials to be used in FRACTESUS.

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

10KhMFT is extracted from the decommissioned WWER-440 RPV of the Greifswald unit 4, from a circumferential welding seam in the beltline of the vessel. Greifswald unit 4 was in operation for 3207.9 days. The X-butt seam is welded with multilayered submerged arc welding technique. Root and cover layers were welded with unalloyed wire Sv-08A, and the filler material with alloyed wire Sv-10KhMFT, studied here, and flux AN-42.

SA 533 B1 (MVE) was produced by Creusot-Loire Industrie. It is a modern pressure vessel steel used in the replacement head of the RPV of the José Cabrera (Zorita) PWR. Like JRQ, this material was produced in accordance to the specifications of ASTM SA 533 B1, but it contains considerably less impurities.

ANP-4 is a material taken from a forging of 22NiMoCr3-7 grade, manufactured by JSW for an unused RPV shell foreseen for a previous 900 MW German PWR (not built). The material was extensively studied in Germany (reference temperature concept) and abroad and is called reference material P147 BM as well. The reference material 22NiMoCr3-7 (made by JSW) is used in German NPPs Grafenrheinfeld, Grohnde, Phillipsburg 2, Brokdorf and Emsland.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

5 Specimen design

The compact tension specimen is one of the most common specimen types in fracture mechanical testing. The geometry offers efficient use of the test material, utilising most of the specimen volume in the establishment of controlled stress state in the crack tip during loading.

While the standards do not define particular specimen size, the primary dimensions are set relative to others. The specimen width, *W*, shall be twice the thickness, *B*, and initial crack size, a_0, half the width. The ratio of specimen height to width, *H/W*, is defined as 1.2. Total width of the specimen, W_{tot}, is 1.25*W.* ASTM E1921 [1] presents three C(T) specimen designs that have been used successfully. These designs form the basis for specimens utilised in the project. Furthermore, the standard sets tolerances for all dimensions which are followed in the project.

Specimen side-grooving is often carried out in fracture mechanical testing in order to ensure a straight crack front after pre-cracking and an even stress state along the crack front. Wallin et al. [7], however, found side-grooving to have little impact on miniature specimens. Furthermore, side-grooving inherently reduces the measuring capacity of the specimen, something that is already challengingly small with miniature specimens. Therefore, side-grooving will not be performed in the project.

In FRACTESUS, the participating laboratories use their existing specimen designs that fulfil E1921 standard requirements. This makes sure that the participants do not have to spend additional resources to design new tools and measurement setups. Nevertheless, the specimens must be miniature sized. Some deviations from standard requirements are allowed in the test matrix, however.

Considering miniature C(T) specimens with thickness *B*=4mm, the outer dimensions of a standard specimen are 4mm x 9.6mm x 10mm. Since the specimens are often manufactured from standard sized 10mm x 10mm x 55mm Charpy impact specimens, the requirement to reduce the height by 0.4mm imposes additional fabrication and waste, if the orientation is changed from the original Charpy orientation. However, the difference in the crack tip stress state is small, since the additional material lies outside the loading points [8], [9]. In FRACTESUS, both standard specimens and taller, 10mm x 10mm specimens will be used by different participating laboratories.

While miniature C(T) specimen conventionally means a thickness of B = 4mm/0.16”, testing will be conducted with slightly larger specimens by two participants in order to utilise existing setups and maintain project resources. These results will be adjusted to match the others similarly to the size adjustment process described in E1921 for all test results.

If there are differences in the results for one material between different partners, the different geometries must be considered a factor among others. The main differences between specimens of different participants are presented in Table 3. All deviations are recorded and considered possible explanations for any discrepancies in the test results.

Orientation of specimens are kept consistently same among all participants testing a material. This orientation is selected to optimally match previous results.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Table 3. Specimen dimensions of different participating laboratories in the FRACTESUS project. The symbols correspond with the ones in Table 4.

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

5.1 Validation of dimensional measurements

While specimen fabrication in itself will not be validated in the project, dimensional measurement of specimens will be. This establishes a foothold for dimensional accuracy: once the measurement of specimen dimensions is validated, the laboratories can be expected to be able to measure any specimens accurately, and thus notice any discrepancies in fabrication. Furthermore, if the fracture mechanical test results between laboratories deviate from each other, specimen dimensions can reliably be either confirmed or ruled out as a cause.

Validation of dimensional measurements is achieved by means of a dimensional round-robin exercise. The laboratories participating in fracture mechanical testing will be participating in the exercise, apart from CEA. Since CEA will only be participating in testing of irradiated materials and thus will only be using their hot cell facilities, their participation would risk contamination of the specimens. Other participants will be measuring the dimensions without risking contamination. Internal quality protocols are expected to ensure the dimensional accuracy, if irradiated materials are measured in a different manner.

VTT has prepared three (3) mini-C(T) specimens to circulate around to the participants. The corresponding design is illustrated in Figure 1. Each laboratory shall measure these specimens with their corresponding methods. Possible outliers will be investigated.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

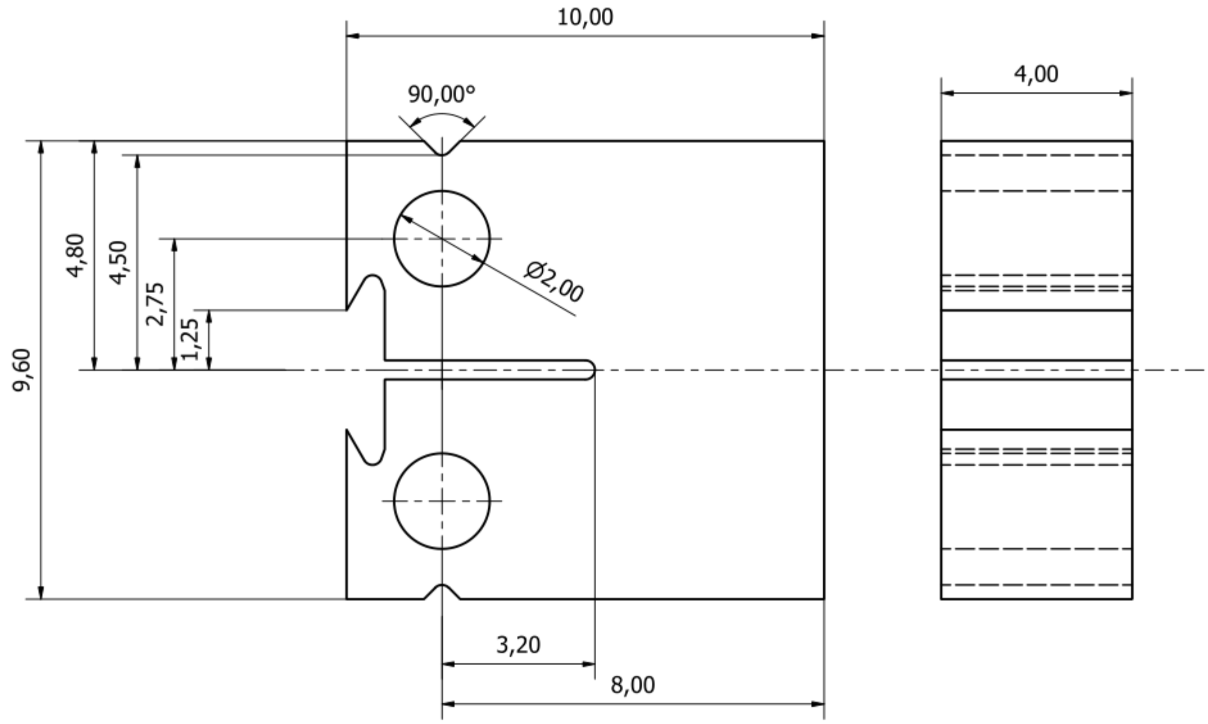

Figure 1. Dimensional round-robin specimen utilised in FRACTESUS.

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

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

6 Testing program

In order to get a higher chance of identifying possible issues and consequently a proper validation, a large portion of testing is done in interlaboratory exercises. By testing the same material in different laboratories in round-robin (RR) exercises, possible issues can be noticed, and corresponding challenges tackled effectively.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

On the other hand, expansion of material database is both scientifically and commercially attractive. Furthermore, utilization of several materials provides a superior foothold for validation, since material differences can be demonstrated to be of no issue. Alternatively, difficulties may be solved, and solutions published in the handbook.

A table of all fracture toughness tests in the project is included in Annex 2.

6.1 Unirradiated interlaboratory exercises

The materials and participating laboratories for unirradiated round-robin (RR) exercises are tabled in Table 5. While the ASTM standard E691 [4] suggests that the number of participants in an interlaboratory proficiency testing exercise should be larger than six, the unirradiated interlaboratory studies are split in several smaller ones. Proficiency testing of participating laboratories encompasses only a portion of the main objective of the project, validation of fracture mechanical testing with miniature compact tension specimens. The objective is pursued more comprehensively by investigating several different materials. On the other hand, some laboratories will be participating in multiple exercises. These laboratories thus provide an opportunity to investigate the proficiency between different test groups.

The nominal amount of tests by each participating laboratory in the exercises is 16. In the first five exercises, the participants are delivered enough material to produce 20 specimens. This additional material allows the fabrication of expected amount of specimens even if unexpected challenges occur.

The sixth exercise consists of only three participants due to material scarcity. Investigating the material is nevertheless valuable due to its high transition temperature and low upper shelf toughness. The participating laboratories for this exercise were chosen so that they are also participating in another round-robin: discrepancies due to the testing party can be identified and any causes for such investigated.

Table 5. Test matrix draft of unirradiated round-robin exercise. N_{test} denotes the nominal number of tests per participant. N_{mat} denotes the nominal amount of material per participant, that is the number of specimens possible to fabricate from delivered material.

RR No.	Material	Orientation	Provider	T0 [°C]	N_{test}	N_{mat}	Testing participant				
							1	2	3	4	5
1	15Kh2MFAA	L-S	HZDR	-104	16	20	SCK CEN	CRIEPI	HZDR	VTT	
2	A533B (JRQ)	T-L	NRI	-71	16	20	NRI	CRIEPI	PSI	CCFE	MTA-EK
3	73W	T-L	SCK CEN	-64	16	20	SCK CEN	CIEMAT	HZDR	VTT	UoB
4	SA508 Cl.3	R-C	CIEMAT	-43	16	20	SCK CEN	CIEMAT	KIT	NRG	UoB
5	ANP-5	T-L	FRA-G	-38	16	20	SCK CEN	FRA-G	HZDR	CCFE	UC
6	A533B LUS (JSPS)	T-L	SCK CEN	+8	16	16	SCK CEN	CRIEPI	UC		

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

6.2 Irradiated round-robin

The irradiated round-robin material and participating laboratories are presented in Table 6 and Table 7, respectively. Contrary to the unirradiated interlaboratory exercises, the irradiated exercise is held with all participants testing the same material. In this fashion, the exercise fulfils ASTM E691 requirement of at least six participants and the proficiency of the laboratories in irradiated materials testing is validated accordingly.

Table 6. Material utilized for irradiated round-robin exercise.

Material	T0 [°C]	Orientation	Fluence [E19 n/cm²]	Number of tests
73W	+34	T-L	1.5 (at ~288°C)	16

Table 7. Participating laboratories in irradiated round-robin exercise.

Testing participant						
1	**2**	**3**	**4**	**5**	**6**	**7**
SCK CEN	VTT	HZDR	MTA-EK	NRI	CEA	NRG

6.3 Further fracture mechanical testing

Some participating laboratories will be conducting experiments in addition to the round-robin exercises. These tests, in terms of materials and testing parties are listed in Table 8.

MTA-EK and BZN will be collaborating in testing of 15Kh2MFAA. It is the same material as used in the interlaboratory exercise RR1, but a different extraction. Therefore, the tests in the unirradiated state cannot be considered a part of RR1 due to possible differences in the material from different extraction locations. There are, however, previous results for brittle fracture initiation toughness from this particular extraction tested with standard Charpy-sized three-point-bend specimens. The results will be re-evaluated before further testing, and mini-C(T) specimens will be fabricated from the tested halves.

Table 8. Test matrix for additional fracture toughness tests outside the round-robin exercises.

Material	Orientation	T0 [°C]	Fluence [E19 n/cm²]	Testing laboratory	No. of tests
15Kh2MFAA	L-S	TBD	0	MTA-EK / BZN	16
15Kh2MFAA	L-S	TBD	I_1 = 20 (E>0.5MeV) (at 270°C)	MTA-EK / BZN	16

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

15Kh2MFAA	L-S	TBD	I_1A (Annealed at 470°C)	MTA-EK / BZN	16
15Kh2MFAA	L-S	TBD	I_1A I_2 = 20 (E>0.5MeV) (at 270°C)	MTA-EK / BZN	16
SA 533 B1	T-L	-119	0	CIEMAT	16
ANP-4	L-S	-78	4 (at 280°C-286°C)	FRA-G	16

6.4 Microstructural investigations

Several participants will be taking part in microstructural investigations of tested specimens. In general, this means investigating the fracture surfaces by means of e.g. scanning electron microscopy (SEM). Initiation sites, their distances from initial crack front as well as their types may be studied, among others.

The actual investigations, however, will depend on the fracture mechanical results. Since validation of fracture mechanical test methods is the main objective of the project, the microstructural investigation will be supporting experiments for this. This means that specimens shall be chosen only after fracture mechanical testing. The expertise of relevant researchers may decide, whether the chemical composition, grain structure, inclusions or something else should be investigated for a particular material.

Table 9. Participants in microstructural investigations.

BZN	CNL	NNL	STUBA
CCFE	FRA-G	NRG	UC
CEA	HZDR	ORNL	UoB
CIEMAT	KIT	PSI	VTT

6.5 Additional investigations

One of the secondary objectives of FRACTESUS is to investigate the applicability of alternative methods for assessment of the mechanical properties of materials. In particular, small punch, microindentation and nanoindentation testing are investigated.

The materials investigated by STUBA and CNL as well as the methodology of CNL will be determined later, to best support the overall objectives of the project once first tests have been analysed.

Table 10. Supporting experiments with small specimen test techniques. (Small punch, microhardness etc.)

Participant	**Material**	**Type**
HZDR	15Kh2MFAA; A533B (JRQ); 10KhMFT; ANP-4	Small punch 15-20 tests per material
STUBA	TBD	Nanoindentation
BZN	15Kh2MFA	Microindentation

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

UC	ANP-5; A533B LUS (JSPS)	Small punch
UoB	SA508 Cl.3; 73W	Nanoindentation
CCFE	ANP-5; A533B (JRQ)	Small punch
CNL	TBD	TBD
CIEMAT	73W; SA508 Cl.3; SA 533 B1 (MVE)	Small punch

7 Conclusion

The test matrix of FRACTESUS provides the foundation for the project to succeed in validation of fracture mechanical testing with miniature sized compact tension specimens.

Materials for the project were chosen to represent real nuclear power plant materials and to vary as much as reasonably possible. A large portion of testing will be done in round-robin exercises, to effectively identify any discrepancies in test results. The materials in the project include Eastern and Western pressure vessel materials. Weldments, plates as well as forgings will be investigated.

Participating laboratories have slightly different specimen designs, which is noted as a possible cause for deviation in test results, and it is planned to be validated in the numerical modelling task. Measurement of specimen dimensions is validated through an interlaboratory exercise.

Additional investigations include microstructural studies with scanning electron microscopy, small punch testing as well as microindentations and nanoindentations.

In total, 656 fracture toughness tests are to be conducted in FRACTESUS. These tests, along with supporting investigations, will provide validation for the miniaturized test techniques and experience for the handbook of guidelines for fracture toughness testing of miniaturized specimens.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

8 References

[1] "ASTM E1921-20, Standard Test Method for Determination of Reference Temperature, To, for Ferritic Steels in the Transition Range." ASTM International, West Conshohocken, PA, pp. 1–20, 2020. doi: 10.1520/E1921-20.2.

[2] "ASTM E399-20a, Standard Test Method for Linear-Elastic Plane-Strain Fracture Toughness of Metallic Materials." ASTM International, West Conshohocken, PA, pp. 1–31, 2020. doi: 10.1520/E0399-20A.

[3] "ASTM E1820-20b, Standard Test Method for Measurement of Fracture Toughness." ASTM International, West Conshohocken, PA, pp. 1–54, 2020. doi: 10.1520/E1820-20B.

[4] "ASTM E691-20, Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method." ASTM International, West Conshohocken, PA, 2020. doi: 10.1520/E0691-20.

[5] "Conformity assessment — General requirements for proficiency testing," *ISO/IEC 17043:2010*, vol. 1, no. 1. International Organization for Standardization, pp. 1–46, 2010.

[6] "ISO 13528, Statistical methods for use in proficiency testing by interlaboratory comparison." International Organization for Standardization (ISO), Geneva, 2015.

[7] K. Wallin, M. Yamamoto, and U. Ehrnstén, "Location of initiation sites in fracture toughness testing specimens-the effect of size and side grooves," in *American Society of Mechanical Engineers, Pressure Vessels and Piping Division (Publication) PVP*, 2016, vol. 1B-2016. doi: 10.1115/PVP2016-63078.

[8] M. Lambrecht, R. Chaouadi, I. Uytdenhouwen, and R. Gérard, "Fracture Toughness Characterization in the Transition and Ductile Regime of an A508 Type Weld Metal With the Mini-CT Geometry Before and After Irradiation." Aug. 03, 2020. doi: 10.1115/PVP2020-21515.

[9] R. Chaouadi, E. van Walle, M. Scibetta, and R. Gérard, "On the Use of Miniaturized CT Specimens for Fracture Toughness Characterization of RPV Materials." Jul. 17, 2016. doi: 10.1115/PVP2016-63607.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Annex 1: Information sheets for materials used in the interlaboratory studies

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Material	**15Kh2MFAA**
Type	BM, forging
Testing orientation	L-S
Chemistry [Cu/P/Ni/Mn] [wt%]	0.05/0.01/0.1/0.49
Fluence [E19 n/cm²] (E> 1.0 MeV)	-
T_0 [°C] (as function of thickness layer)	-104

Material description

The material is a WWER-440 reactor pressure vessel (RPV) base metal, 15Kh2MFAA, originating from the forged ring 0.3.1 of the original RPV of the Greifswald NPP Unit 8 (GR-8). The RPV of GR-8 belongs to the 2nd generation of the WWER-440 reactors (model V213) and was produced by SKODA steelworks in the former Czech Slovak Republic end of the 1970s. Unit 8 was not put into operation and cut in segments of 0.4 m × 1 m × 0.15 m for decommissioning. Blocks of this segments were cut into plates over the thickness using a wire travelling electro-erosive discharging machine (EDM). The first plate consists of austenitic stainless steel cladding (10 mm) and the following of base metal 15Kh2MFAA.

Tensile B5, Charpy size 0.4T-SE(B) and 0.5T-C(T) specimens were machined in L-T orientation from the various thickness layers. In addition, 1T- and 0.5T-C(T) and 0.4T-SE(B) specimens in L-S orientation were machined from the range between ¼ to ¾ of the thickness of the forged ring. Tensile (B5 according to DIN 50125), 0.4T-SE(B) and 0.5T-C(T) specimens of 15Kh2MFAA base metal from the forged ring 0.3.1 were irradiated in the LYRA and BAGIRA irradiations rigs in JRC-Petten and KFKI Budapest, respectively. Table 1 summarizes test results in the L-S orientation. An extensive database of test results which were conducted in HZDR and MTA EK laboratories is also available.

Results database overview

specimen type, orientation	**condition**	**T_0 °C**	**σ K**	**$\Sigma r_i n_i$**	**r**	**N**	**K_{Jc-1T} < 2%**	**K_{Jc-1T} > 98%**	**ΔT_0 K**
0.4T-SE(B) L-S	initial	-100	5.4	3.98	25	28	5	3	-
0.5T-C(T) L-S	initial	-108	6.1	2.50	15	15	0	2	-
1T-C(T) L-S	initial	-104	7.9	1.68	7	7	0	0	-

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Material	A533B (JRQ)
Type	BM, plate
Testing orientation	T-L
Fluence [E19 n/cm²]	-
T_0 [°C]	-71 °C [2]

Chemical composition (mass %) of A533B (JRQ) [2]

[wt %]	C	Si	Mn	P	S	Cu	Ni	Cr	Mo	V	Al
min.	0,16	0,24	1,35	0,017	0,003	0,13	0,85	0,12	0,49	0,002	0,012
max.	0,20	0,26	1,43	0,019	0,004	0,14	0,82	0,12	0,51	0,003	0,014

Material description

A533B (JRQ) is a monitoring correlation steel provided by the Kawasaki Steel Corporation [1], introduced the IAEA in 1983. The material was produced by the BOF-LRF process. After rolling, the plates were heat treated normalizing at 900 °C, quenching from 880 °C and tempering at 665 °C for 12 hours, then stress relieving at 620 °C for 40 hours, before being cut into a pair of test plates and a number of test blocks.

JRQ has served in many international and national studies of irradiation embrittlement of reactor pressure vessel steel and continues to be used extensively for its increased response to irradiation. Furthermore, there is plenty of information and many technical publications available concerning the material, e.g. the IAEA TECDOC 1230 [2] and IAEA-TECDOC-1435 [4]. These documents represent a reference collection of available material properties for the JRQ material to aid in its use for both experimental and surveillance programmes.

Results database overview

Labs	Test (standard)	Specimen	Number of tests		Reference
			unirradiated	irradiated	
18 countries (IAEA CRP)	Static fracture toughness (ASTM E399, ASTM E813)	SE(B)	383	0	[4]
		1TCT	83	0	
ÚJV Řež, a. s.	Static fracture toughness ČSN [6]	0.5TCT	13	9	[5]
JAEA	Static Fracture toughness	Mini-CT	12	0	[7]
		PCCv	16	8*	
		0,4TCT	15	0	
		1TCT	9	0	

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

	(ASTM E 1921)				
JAERI	Static fracture toughness (ASTM E399, ASTM E813)	PCCv	40	22*	[8]
		1TCT	6	0	

References

1. Manufacturing History and Mechanical Properties of Japanese Materials Provided for the International Atomic Energy Agency, CRP Sub-Committee, Atomic Energy Research Committee, Japan Welding Engineering Society, October 1986
2. INTERNATIONAL ATOMIC ENERGY AGENCY, Reference Manual on the IAEA JRQ Correlation Monitor Steel for Irradiation Damage Studies, IAEA-TECDOC-1230, IAEA, Vienna (2001)
3. M. Brumovsky, M. Marek, L. Zerola, L. Viererbl, V. N. Golovanov, V. V. Lichadeyev, B. M. Raetsky, and A. L. Petelin, "Attenuation of Radiation Damage and Neutron Field in Reactor Pressure Vessel Wall," in Reactor Dosimetry: 12th International Symposium, ed. D. Vehar, D. Gilliam, and J. Adams (West Conshohocken, PA: ASTM International, 2007), 190-195.
4. INTERNATIONAL ATOMIC ENERGY AGENCY, Application of surveillance programme results to reactor pressure vessel integrity assessment - results of a coordinated research project, 2000–2004, IAEA-TECDOC-1435 (2005)
5. L. Bodnar, R. Havel, A. Brožová, "RADIAČNÁ ODOLNOST OCELÍ 533B A 508 - KOORDINOVANÝ PROGRAM MAAE, FÁZA III (IAEA CRP, PHASE III), Celostátní konference, křehký lom materiálů a konstrukcí, 1990
6. Skúšky lomovej húževnatosti kovov pri statickom zaťažení, (ČSN standard for the SFT), Bratislava, 1989.
7. Tobita, Tohru, Nishiyama, Yutaka, & Onizawa, Kunio (2018). Mechanical properties database of reactor pressure vessel steels related to fracture toughness evaluation (JAEA-Data/Code--2018-013). Japan
8. Onizawa, Kunio, Tobita, Tohru, & Suzuki, Masahide . (1997). Investigation on the evaluation of cleavage fracture toughness using PCCv specimens in the ductile-brittle transition range of reactor pressure vessel steels (contract research) (JAERI-Research--97-081). Japan

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Material	**73W**	
Type	Weld metal	
Testing orientation	T-L	
Chemistry [Cu/P/Ni/Mn] (wt%)	0.31/0.005/0.60/1.55	
Main properties	Condition	
	Unirradiated	Irradiated
Fluence [E19 n/cm²]	-	1.5 (@ 288°C)
T_0 [°C]	**-64**	**34**

Material description

Material in the form of welded blocks of 1.22 m length and 218 mm thickness was produced under Fifth Irradiation Series in the Heavy-Section Steel Irradiation (HSSI) Program. The program was aimed to obtain fracture toughness data for two welds with 0.23 wt% and 0.31 wt% of Cu content, labelled 72W and 73W, respectively, in baseline and irradiated condition. There is a large database of test results, which were conducted in ORLN and SCK CEN laboratories.

Capsules of specimens were irradiated in the poolside facility of the Oak Ridge Research Reactor at a nominal temperature of 288°C and an average fluence of about 1.5 x 10^{19} neutrons/cm^2 (>1 MeV) in 1984-1986 years. Unirradiated and irradiated Charpy V-notch impact, tensile, and drop-weight specimens, compact fracture toughness specimens thicknesses of 25.4, 50.8,and 101.6 mm -1T CT, 2T C(T), and 4T C(T) where investigated. Additionally, unirradiated 6T C(T) and 8T C(T) were tested. Results obtained by ORNL are described in [1, 2]. Results of crack arrest tests are presented in [3]. One welded block and parts of broken CT specimens tested by ORNL were used in SCK CEN tensile, PCCv and CVN tests [4-6]. 98°C shift of T_0 was revealed for 73W material after irradiation.

Results database overview

Lab	**Test (standard)**	**Specimen**	**Number of tests**		**Reference**
			unirradiated	**irradiated**	
ORNL	Static fracture toughness (ASTM E399, ASTM E813)	1T-CT	38	29	[1, 2]
		2T-CT	20	18	
		4T-CT	16	8	
		6T-CT	2	0	
		8T-CT	4	0	
ORNL	Drop weight (ASTM E208)	Pellini	6	8	[1, 2]
SCK CEN	Static fracture toughness (ASTM E 1921)	PCCv standard	19	0	[4]
		PCCv reconstituted	27	30	[4, 5]
		Half PCCv (B= 5mm) reconstituted	10	0	[5]

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

		Subsized PCCv reconstituted	10	0	[5]
SCK CEN	Dynamic fracture toughness	PCCv reconstituted	12	14	[6]
ORNL	CVN impact (ASTM E23)	Charpy	83	56	[1, 2]
SCK CEN	CVN impact (ASTM E23)	Charpy	12	0	[4]
		Charpy reconstituted	12	16	[4]
ORNL	Tensile (ASTM E8)	Tensile standard ϕ=5.08 mm, L_0=25.4 mm	15	10	[1, 2]
SCK CEN	Tensile (nonstandard tests)	Reconstituted ϕ=3.0-3.6 mm, L_0=18 mm	19	9	[4]
ORNL	Crack arrest* (ASTM E 1221)	25x76x76 mm^3 weld-embrittled	10	8	[3]
		25x152x152 mm^3 weld-embrittled	11	7	
		33x152x152 mm^3 weld-embrittled	8	3	
		33x152x152 mm^3 duplex	7	0	
		51x203x203 mm^3duplex	1	0	

* fluence 1.9E19 n/cm² (6th Irradiation Series)

References

1. Nanstad, R.K., et al., Irradiation Effects on Fracture Toughness of Two High-Copper Submerged-Arc Welds, HSSI Series 5, in NUREG/CR-5913 ORNL/TM-12156/V1. 1990.
2. Nanstad, R.K., et al., Effects of Irradiation on KIC curves for High-Copper Welds. Effects of Radiation on Materials : 14th International Symposium, 1990. 1046: p. 214-233.
3. Iskander, S.K., W.R. Corwin, and R.K. Nanstad, Effects of Irradiation on Crack-Arrest Toughness of 2 High-Copper Welds. Effects of Radiation on Materials : 15th International Symposium, 1992. 1125: p. 251-269.
4. Chaouadi, R., et al., Fracture toughness test results of precracked Charpy specimens of irradiated 73W weld material. Effects of Radiation on Materials: 19th International Symposium, 2000. 1366: p. 279-294.
5. Chaouadi, R., Fracture toughness measurements in the transition regime using small size samples. Small Specimen Test Techniques, 1998. 1329: p. 214-237.
6. Lucon, E. and R. Chaouadi, Radiation damage assessment by the use of dynamic toughness measurements on pre-cracked Charpy-V specimens. Effects of Radiation on Materials: 20th International Symposium, 2001. 1045: p. 68-78.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Material	**SA 508 Cl. 3**
Type	BM, forging
Testing orientation	R-C
Chemistry [C/Si/P/S/Cr/Mn/Ni/Cu/Mo] (wt%)	0.20/0.24/0.008/0.002/0.15/1.37/0.93/0.04/ 0.52
Fluence [E19 n/cm²]	-
T_0 [°C]	**-60**

Material description

SA 508 Cl. 3 is a forging supplied in 1995 by Forgemaster Steel & Engineering Limited to Equipos Nucleares ESNA. The forging was used in the fabrication of a replacement closure head of the RPV of the José Cabrera (Zorita) PWR. The material was produced with electric furnace processes, it was vacuum stream degassed, killed and produced to fine grain size by controlled aluminium additions.

The mechanical and fracture mechanical properties of the material are studied in the EURATOM project SOTERIA. Fracture toughness results from tests of ½T and 1T C(T) specimens will be available in the deliverable 3.1 of the project, once published. The tests indicate a reference temperature T0 = -60°C.

Results database overview

Lab	**Test (standard)**	**Specimen**	**Number of tests**
CIEMAT	Static fracture toughness (ASTM E 1921)	0.5T-CT	14
		1T-CT	14

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Material	ANP-5
Chemistry [C/Si/P/S/Cr/Mn/Ni/Cu/Mo] (wt%)	0.08/0.15/0.015/0.013/0.74/1.14/1.11/0.22/0.6
Testing orientation	T-L
Fluence [E19 n/cm²]	-
T_0 [°C]	**-38**

Material description

ANP-5, sometimes called P370 WM, is taken from a weld of NiCrMo1 submerged arc welding wire and modified LW320/330 powder. The weld was manufactured by Klöckner Werke AG as stress-relief heat-treated 6 m long test weld seam. It resembles an RPV shell used in a German PWR and has been extensively studied in Germany and abroad. The material was used as bounding material for representative studies for German NPP Obrigheim and Stade. The material is well characterized in the baseline and irradiated condition and was studied in EURATOM projects LONGLIFE and SOTERIA by mechanical tests and microstructural analyses.

There is a large database of test results (Charpy V-notch impact, tensile, drop-weight specimens, PCCV and compact crack arrest specimens) which were conducted in Framatome laboratories.

References

1. H. Hein, E. Keim, H. Schnabel, T. Seibert, A. Gundermann, Final Results from the Crack Initiation and Arrest of Irradiated Steel Materials Project on Fracture Mechanical Assessments of Pre-Irradiated RPV Steels Used in German PWR, J. ASTM Int. 6 (2009), Paper ID JAI101962, Journal of ASTM International (2010), STP 1513 on Effects of Radiation on Nuclear Materials and the Nuclear Fuel Cycle: 24th Volume.

2. H. Hein, E. Keim, H. Schnabel, J. Barthelmes, Ch. Eiselt, F. Obermeier, J. Ganswind, M. Widera, Final Results from the CARINA Project on Crack Initiation and Arrest of Irradiated German RPV Steels for Neutron Fluences in the Upper Bound, 26th ASTM Symposium on the Effects of Radiation on Nuclear Materials, Indianapolis,12-13 June 2013

3. M. Serrano, F. Bergner, B. Radiguet, J. Lin, C. Grovernor, M. Hernandez-Mayoral, E. Meslin, K. Wilford, W. Van Renterghem, J. Kocik, S. Ortner, H.W. Viehrig, R. Chaouadi, M. Brumovsky, H. Hein, F. Gillemot, P. Todeschini, T. Planman, J Ruoden and E. Altstadt, Correlation between microstructure features and mechanical properties of irradiated LONGLIFE RPV steels, Fontevraud 8 - Contribution of Materials Investigations and Operating Experience to LWRs' Safety, Performance and Reliability, France, Avignon – 2014, September 14 |18.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Material	A533B LUS (JSPS)
Institution offering material	SCK CEN
Testing orientation	T-L
Chemistry [C/Si/P/S/Cr/Mn/Ni/Cu/Mo] (wt%)	0.24/0.41/0.028/0.023/0.08/1.52/0.43/0.19/0.49
Fluence [E19 n/cm²]	-
T_0 [°C]	**+8**

Material description

The A533B LUS material was used in a round robin organized by the Japan Society for the Promotion of Science (JSPS). This material is characterized by low USE (72J) and a positive T_0 value in unirradiated state resulting from high P and S content. The objective is to exhibit properties as expected after long service in the real RPV conditions. Japanese round robin was conducted mostly on 0.5T-, 1-CT and some larger 2T- and 4T- CT specimens with T-L orientation in -25, 0 and +25°C. Total number of 118 specimens was tested [1-3]. Additionally, material taken from broken large CT specimens was used for PCCv and 0.5T-CT specimens manufacturing, also with T-L orientation, which were tested at SCK CEN (in total 46 specimens). 15°C shift between T_0 determined on CT and PCCv specimen was observed [1-3].

Results database overview

Lab	Test (standard)	Specimen	Number of tests	Reference
JSPS	Static fracture toughness (ASTM E1921)	0.5T-CT	53	[1-3]
		1T-CT	52	
		2T-CT	10	
		4T-CT	3	
SCK CEN	Static fracture toughness (E1921)	PCCv	46	[1-3]
		0.5T-CT	24	
SCK CEN	CVN impact	Charpy	19	[1-3]
SCK CEN	Tensile	Diameter 3.6 mm, gauge section length 18 mm	17	[1-3]

References

1. Chaouadi, R., et al., On the use of the master curve based on the precracked charpy specimen. PVP. 393.

2. Lucon, E. and M. Scibetta, Comparison between precracked Charpy-V and C(T) specimens for To determination of the JSPS RPV steel. 2000, SCK CEN R-3378.

3. Chaouadi, R. and M. Scibetta, On the use of the master curve concept and the Charpy size specimen to characterize fracture toughness in the transition regime: Part II: RPV steel with low upper shelf - A533B (JSPS). 1999, SCK CEN R-3326.

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

Annex 2: Table of all fracture toughness tests

The table lists all fracture toughness tests to be performed in the FRACTESUS project.

Material	Fluence [E19 n/cm²]	Orientation	T0 [°C]	Testing laboratory	Specimen type	No. of tests
73W	0	T-L	-64	SCK CEN	Mini-C(T)	16
				CIEMAT	Mini-C(T)	16
				HZDR	Mini-C(T)	16
				VTT	Mini-C(T)	16
				UoB	Mini-C(T)	16
73W	1.5	T-L	34	SCK CEN	Mini-C(T)	16
				VTT	Mini-C(T)	16
				HZDR	Mini-C(T)	16
				MTA-EK	Mini-C(T)	16
				NRI	Mini-C(T)	16
				CEA	Mini-C(T)	16
				NRG	Mini-C(T)	16
15Kh2MFAA	0	L-S	-104	SCK CEN	Mini-C(T)	16
				CRIEPI	Mini-C(T)	16
				HZDR	Mini-C(T)	16
				VTT	Mini-C(T)	16
				MTA-EK	Mini-C(T)	16
15Kh2MFAA	0	L-S	TBD	MTA-EK	Mini-C(T)	16
15Kh2MFAA	I: 20 (E>0.5MeV)		TBD	MTA-EK	Mini-C(T)	16
15Kh2MFAA	IA		TBD	MTA-EK	Mini-C(T)	16
15Kh2MFAA	IAI: 20 (E>0.5MeV)		TBD	MTA-EK	Mini-C(T)	16
SA508 Cl.3	0	R-C	-43	SCK CEN	Mini-C(T)	16
				CIEMAT	Mini-C(T)	16
				KIT	Mini-C(T)	16
				NRG	Mini-C(T)	16
				UoB	Mini-C(T)	16
SA 533 B1 (MVE)	0		-119	CIEMAT	Mini-C(T)	16
A533B (JRQ)	0	T-L	-71	NRI	Mini-C(T)	16
				CRIEPI	Mini-C(T)	16
				PSI	Mini-C(T)	16
				CCFE	Mini-C(T)	16

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.

A533B LUS (JSPS)	0	T-L	8	SCK CEN	Mini-C(T)	16
				CRIEPI	Mini-C(T)	16
				UC	Mini-C(T)	16
ANP-4	4		-78	FRA-G	Mini-C(T)	16
ANP-5	0	T-L	-38	SCK CEN	Mini-C(T)	16
				FRA-G	Mini-C(T)	16
				HZDR	Mini-C(T)	16
				CCFE	Mini-C(T)	16
				UC	Mini-C(T)	16
TOTAL ALL MATERIALS					Mini-C(T)	**656**

This project has received funding from the Euratom research and training programme 2020-2024 under grant agreement No. 900014.