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D7.3 Materials selection and conditions

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DOCUMENT SUMMARY

The material needs, assessment methods and the required testing conditions for determining the potential detrimental effect of LBE corrosion (in addition to material loss) is outlined in this report. The material mechanical properties, e.g. tensile strength, proof stress and ductility, of the remaining bulk steel is to be studied by comparison of aged material and corrosion exposed material performance for a range of material (cladding tube) relevant test types. The test methods to be applied are; flat plate tensile samples, tube-section tensile samples, full 'tensile' tube samples, ring-tension, ring-compression and small punch test samples as well as internal pressure tube test. The uniaxial 'plate-type' tests are planned to represent the reference material properties. Provisional test matrices to reach the objective of this task are presented for each test type. The comprehensive study will include cold worked 'bulk' material in the form of 1515 Ti rod material and naturally the actual cladding tubes.

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1 Introduction

The PATRICIA Work Package 7 Task 2 (WP 7.2) is designated to assess the integrity of the cladding of the driver fuel of MYRRHA. The main objective of this task is to identify the potential decline of mechanical properties beyond the material loss in exposed claddings. The material performance and mechanical properties of thin walled fuel cladding tubes have previously been evaluated through a number of testing techniques and evaluation methodologies [1] in the EERA JPNM Pilot Project TASTE for the same cladding steel [2] as being tested here. The prior experimental and test technique experience is used as base for the study of cladding corrosion damage impact on material properties. The test program of PATRICIA will also continue to benefit from the ongoing TASTE+ project targeting the validation of the test methodologies in hot cell environment.

In design, it is generally assumed that material loss by corrosion does not have an impact on the remaining material, i.e. the remaining material behaves like the pristine tube steel. However, it is known that corrosion might cause local effects, which could change the mechanical properties of the materials, e.g. ductility. Thus, the test campaign performed within this task is expected to give valuable information about the validity of such a claim for realistic MYRRHA conditions. Since the potential detrimental effects from the corrosion exposure could affect both strength and ductility, a range of different ‘thin walled cladding’ test techniques are being applied to compare the results and detect potential degradation effects.

The methodology used for determining the effect of corrosion[3][4] is explained in detail later in this report. An ‘coefficient of effectiveness’ W is determined by measuring the drop in maximum force between un corroded and corroded samples. This gives access to the loss of load-bearing area that can be compared to the one measured by cross-sectional metallography (expressed as a corrosion depth). Potential ductility change is studied through a ‘ductility factor’ D .

To reach the objective of WP 7.2, the material “states” (aged & corroded) are to be compared for each chosen test technique. The decision to age the virgin (as received) material to the same time-temperature material state as will be caused by the corrosion exposure cycle is based on recent results by [5] showing improved mechanical properties for target corrosion cycle of up to 5000 h at 500°C.

Also, the virgin material state has, to a large extent, already been defined in the TASTE project at both room and elevated temperature.

For thin-walled cladding tubes, there is no standard or test technique that is considered able to fully describe the classical material ‘uniaxial’ bulk material properties. However, there are a range of tests that together can give a comprehensive understanding of the material response. To obtain a sufficient amount of data to better understand the effect of LBE corrosion on the mechanical properties each method is applied to both corroded and un-corroded (aged) with a target of no less than 5 tests per test condition/test method/laboratory to obtain some statistical relevance to any observed change in strength or ductility. The goal is to perform each method in at least two different laboratories to study test technique repeatability.

2 Methods

To study the effect of corrosion the same methodology and sample dimensions as in reference [4] have been applied to enable direct comparisons to the extensive test program performed for corrosion by uranium oxide and fission products. The virgin material tests from the report have been used as base for the planning of the test programmes of this report.

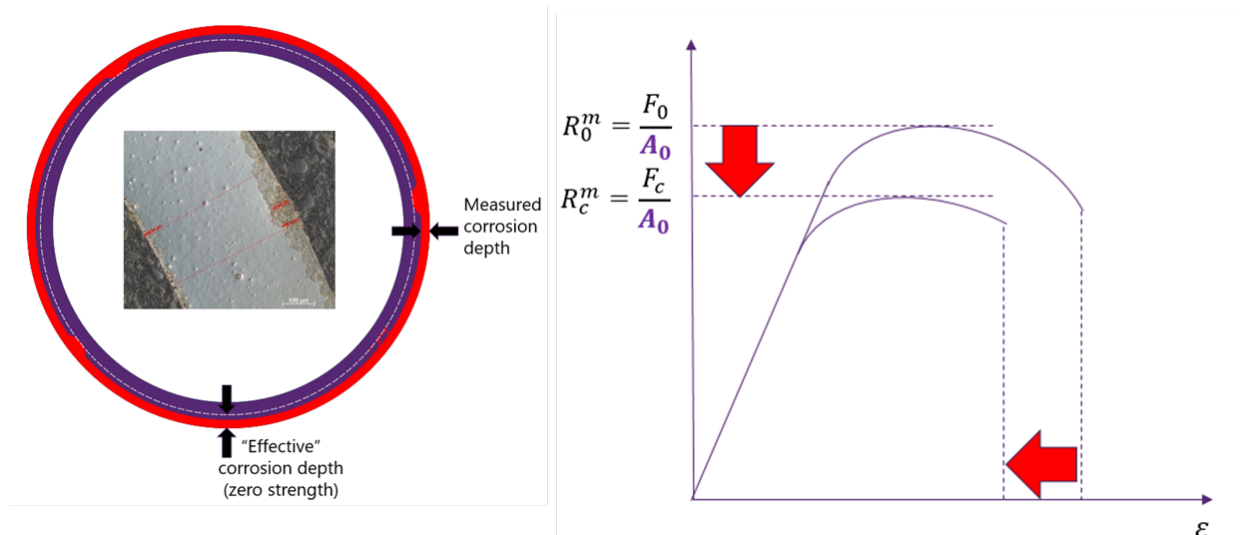


Figure 1. Description of the calculated effective corrosion depth (x) acquired from the force drop (left figure) and the measured corrosion depth from microscopy (c). The R_0^m and R_c^m are the strengths before and after corrosion exposure correspondingly and the arrows indicate the drop in force and ductility.

The sample extraction used for the reference testing is shown in Figure 2 and the technical drawing in Figure 3. The nominal thickness of the sample is $a=0.7$ mm and the width $b=4.0$ mm.



Figure 2. Uniaxial test sample extraction from cold worked bar by EDM

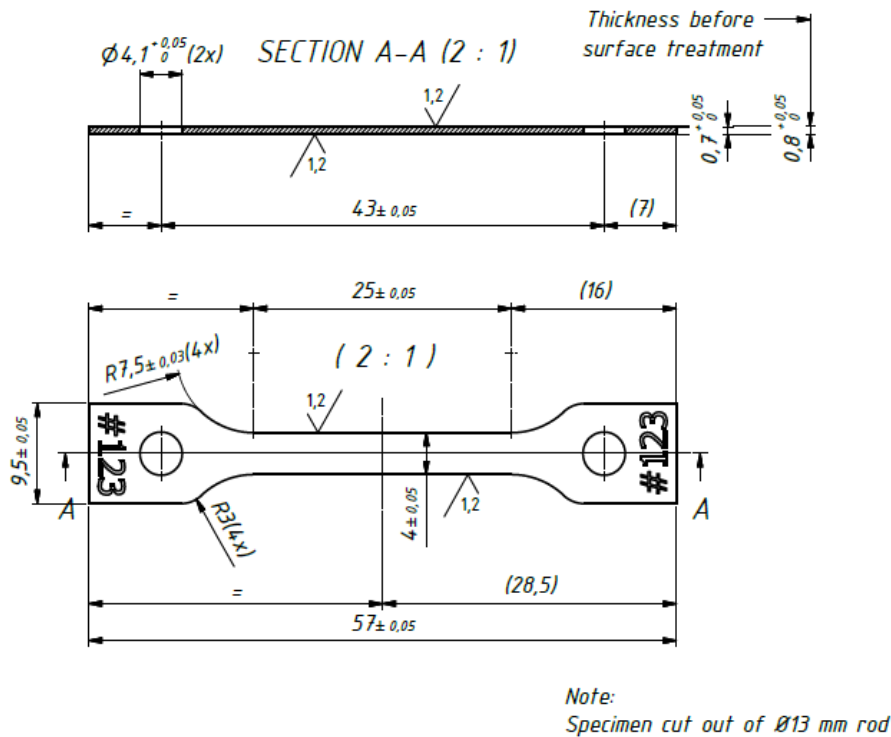


Figure 3. Uniaxial test sample drawing, including dimension tolerances

2.1 The coefficient of effectiveness W

The stress in a uniaxial test sample with the initial effective area A_0 is calculated as in Eq.1-2. For a rectangular test piece with the width b and the thickness a when applying a force F .

$A_0 = a \cdot b$	Eq.1
$\sigma_0 = \frac{F_0}{A_0}$	Eq.2

The 'effective' area A_c of a corroded sample can be attained by comparing the applied (measured) forces from testing corroded and un-corroded samples as given in Eq. 3. From A_c the effective corrosion depth x can be attained as given in Eq. 4. In case of tube sections cut out after corrosion exposure the calculation of x is simpler as given in Eq. 5. In case of a full tube used as a tensile sample the x can be calculated by Eq.6.

$A_c = \frac{F_c}{F_0} A_0 = (a - x) \cdot (b - x)$	Eq.3
$x_{1-2} = \frac{a+b}{4} \pm \frac{1}{2} \cdot \sqrt{\frac{(a+b)^2}{4} + a \cdot b \cdot \left(\frac{F_c}{F_0} - 1\right)}$	Eq.4
$x_1 = a \cdot \left(1 - \frac{F_c}{F_0}\right)$	Eq. 5
$x = R_o \pm \sqrt{R_i^2 + \frac{A_0}{\pi} \cdot \frac{F_c}{F_0}}$	Eq. 6

The acquired ‘effective’ corrosion depth can now be related to the microscopically measured ‘actual’ corrosion depth c . The c can be defined as in [4] using the maximum depth as given in eq. 6 or by an average depth given in Eq. 7. **Note that the limit of ‘detrimental’ effect indicated by W is strongly affected by this definition.** The choice of definition for c will also influence uncertainty calculations regarding the measured min and max values.

$c = c_{max} - \frac{(c_{max} - c_{min})}{4}$	Eq.6
$c = \frac{(c_{max} + c_{min})}{2}$	Eq.7

With all the needed measurements done, the coefficient of effectiveness can be calculated as given in Eq. 8, as effective corrosion depth over measured corrosion depth.

$W = \frac{x}{c}$	Eq.8
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Thus, if the attained calculated x is larger than the measured c the coefficient of effectiveness will be larger than unity ($W > 1$) and indicate a detrimental effect of corrosion. Note that choosing the Eq. 6 definition for c might be a too conservative limit and is likely to result in W values below unity throughout the test program.

2.2 The Ductility factor D

The somewhat easier parameter to study is the ‘ductility factor’ D . The ductility factor is defined in Eq. 9 as the ratio between the ductility of the corroded sample ϵ_c over the ductility at the reference state ϵ_0 . Thus, if the corrosion is affecting the ductility D will be lower than unity ($D < 1$).

$D = \frac{\epsilon_c}{\epsilon_0}$	Eq.9
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3 Materials

3.1 15-15Ti (1.4790) austenitic steel

The PATRICIA test material is the 15-15Ti (X10CrNiMoTiB15-15 or DIN 1.4970) steel. It is a stabilized heat resistant stainless steel especially designed for fuel element claddings. The material for testing is acquired from two product forms originating from the same steel heat, i.e. cladding tubes of 24% cold work (designation 24%CW) [6] and bars ($\varnothing 13$ mm), also cold worked to 19.6% (designation 20%CW). The bar material was produced to enable material property determination by test techniques requiring thicker bulk material than the nominal wall thickness of the tubes (0.45 mm). Note that the bar material specimen is the main choice of reference material due to the possibility to manufacture the exact same type of samples as were used [1] for a range of test types and [4] for the reference plate samples.

Even though the samples were extracted from the cold worked bar material the reference samples will from here on be referred to as ‘plate’ due to their final test sample form. The samples are cut out of the bar by EDM to a thickness of 0.8 mm and then polished to the targeted thickness of 0.7 mm (see Figure 2). All the project test material is made available by SCK·CEN.

The certificate of conformity for the cladding tubes is provided in the SCK CEN report SCK•CEN-R-5686 [4].

Table 1. Chemical composition of the 15-15Ti steel according to [4].

Results chemical analysis (wt. %)	C	Si	Mn	P	S	Cr	Ni	Co	Mo	B	N
Required	0.08 0.12	0.5 0.7	1.0 2.0	<0.015	<0.015	14.5 15.5	14.5 15.5	<0.03	1.0 1.4	0.003 0.008	<0.015
Measured	0.096	0.57	1.86	0.013	<0.001	15.06	15.05	0.02	1.21	0.0025 ¹ 0.0031 ²	0.011
Results chemical analysis (wt. %)	Ti	V	Ta	Cu	Ca	Fe					
Required	0.33 0.55	<0.05	<0.02	<0.05	<0.03	bal					
Measured	0.44	0.034	<0.005	0.026	0.017	bal					

¹Results of first ICP measure at Sandvik; ²Results of second ICP measure at Aubert&Duval after erosion of 75µm external tube layer

The material will be tested in 3 material “states” as described in Table 2.

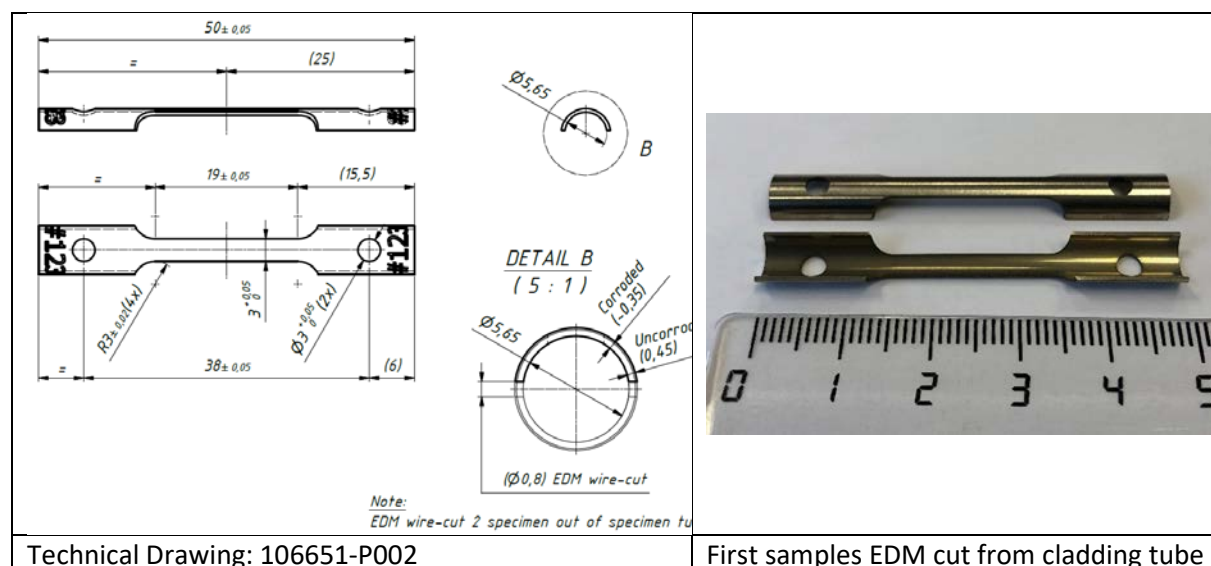


Figure 4. One-legged uniaxial test sample from tube section

Table 2. Test material state identification key, example designation T-C1-001.

Product form	Material state	Identification code	Remark
Tube	Virgin	T-	As-received material [4], test data available from the TASTE project
Plate	Virgin	P-	As received material
Corroded		C(#Nr) ¹	Corrosion exposed to seek maximum corrosion depth (c) at specified test temperature and time.
Aged		A(#Nr) ²	Aged to the same time-temperature state as the corrosion exposure(s)

1) The corrosion depths from each designated corrosion exposure (#Nr) is determined for each exposure batch respectively.

2) Aging exposure (#Nr) representative of specified corrosion exposures.

Note that the material properties are not expected to be very sensitive to ‘small differences’ in the aging time-temperature, thus one aging exposure should be sufficient to represent all corrosion exposures with durations around $5000 \pm 1000\text{h}$ at 500°C . However, according to [5] it is clear that the reference material properties for determining the efficiency coefficient **W** and the ductility factor **D** has to be related to the aged condition. The aging at 500°C of durations in the range of 1000h to 5000h causes microstructural evolution, i.e. formation of TiC nano-precipitates in the cold worked steel, improving both strength and ductility. According to [5] the ultimate tensile strength after exposure at 500°C can be nearly 10% higher than the virgin material and the ductility can be improved by over 45%. This naturally makes the assessment of **W** and **D** more challenging since there is both a strengthening microstructural and a weakening environmental process ongoing during the corrosion exposure. By normalizing the results with the aged material state is expected to negate the above effect. However the extent of the microstructural changes and the time-temperature dependence at ‘low temperatures’ (below 600°C) is not well known and the general data availability for aged 1515Ti is limited.

To find test methodologies that could be more sensitive to the environmental effect of corrosion the PATRICIA task 7.2 studies a range of test methods suitable and complementary for estimating material properties of claddings. The test techniques to be applied for the assessments are identified as described in Table 3. The lab specific designations are given in Table 4 and some examples for fully identifiable test designations in Table 5

Table 3. Testing technique identification key.

Test technique	Identification code	Remark
Uniaxial tensile testing	UT ¹⁾	Specimen according to Figure 2 and Figure 4 for the tube and plate specimen.
Small Punch test	SP	Only for tube material, specimen and test set-up according lab specifications
Ring Tension test	RT	Only for tube material, specimen according to Figure 5.
Ring Compression test	RC	Only for tube material, Tube sections of 6 mm in length
Segmented Cone Mandrel	CM	Only for tube material, Tube sections of 6 mm in length
Internal pressure testing	IP	Raising pressure test for tubes according lab specifications
Internal pressure creep testing	IPC	Constant pressure creep tests according lab specifications

1) Designation not needed in the constructing the test designations due to this being the only test technique using both plate and tube samples, thus T- and P- (see Table 2) markings are sufficient for this end.

The uniaxial tube section samples from the cladding tube is shown in Figure 4, ring tension samples in Figure 5 and small punch samples in Figure 6. The RC samples are just 6 mm long tube sections.

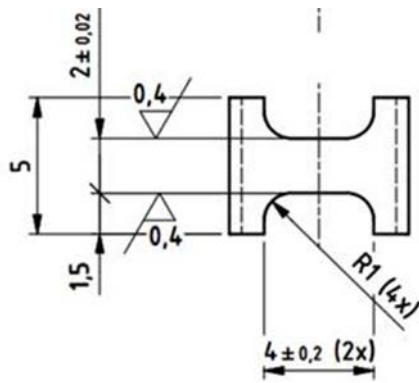


Figure 5. RT sample for testing at RATEN and NRG.

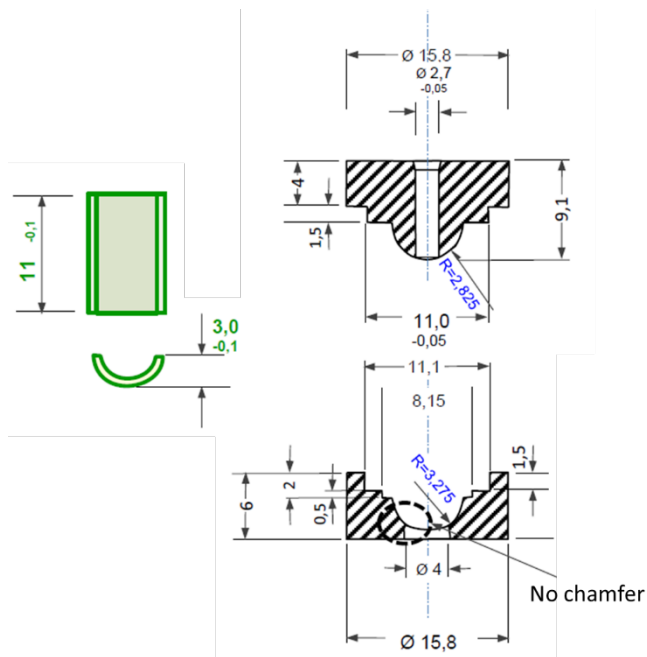


Figure 6. SP sample and test set-up dies for testing curves SP samples (JRC).

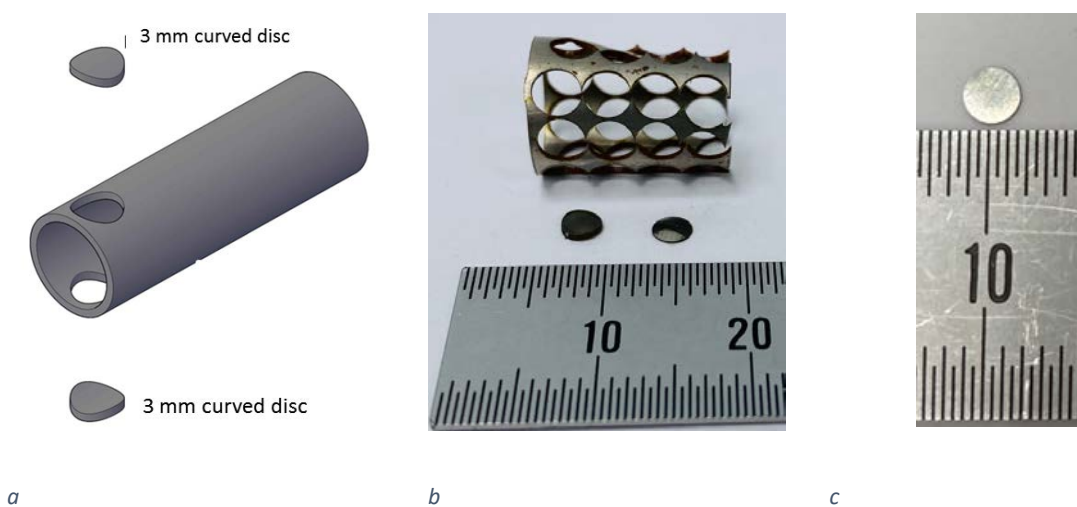


Figure 7. a) & b) SP sample extraction for miniature SP samples from cladding tube (CIEMAT). Note that the final test sample c) is ground flat to the final thickness of 0.25 mm from both sides, removing the external and internal surface layer.

Table 4. Test laboratory identification key

Laboratory	Identification code	Test techniques
CIEMAT	CIE	UT, SP
ENEA	ENE	RC
JRC	JRC	SP, RC
KIT	KIT	UT, IP, IPC
NRG	NRG	RT, SP
RATEN	RAT	RT
VTT	VTT	IP, IPC

Table 5. Examples of full test ID codes.

Laboratory (Table 4)	Test technique (Table 3)	Material (Table 2)	Full ID code (first test)
CIEMAT (CIE)	Uniaxial tensile test	Virgin (tube)	T-CIE-001
CIEMAT (CIE)	Uniaxial tensile test	Virgin (plate)	P-CIE-001
CIEMAT (CIE)	Uniaxial tensile test	Aged (tube), exposure (#1)	TA1-CIE-001
CIEMAT (CIE)	Small Punch	Aged (tube); exposure (#01)	SPA1-CIE-001
JRC (JRC)	Small Punch	Corroded tube, pre-PATRICIA exposure (#0)	SPC0-JRC-001
JRC (JRC)	Ring Compression test	Corroded tube, pre-PATRICIA exposure (#0)	SPC0-JRC-001
JRC (JRC)	Segmented Cone Mandrel	Corroded tube segment, pre-PATRICIA exposure (only external)(#1)	CM0-JRC-001
ENEA (ENE)	Ring Compression test	Corroded, pre-PATRICIA exposure (#00)	RCC0-ENE-001
NRG (NRG)	Ring Tension Test	Aged (tube); exposure (#01)	RTA1-NRG-001
RATEN (RAT)	Ring Tension Test	Corroded, exposure #01	RTC1-RAT-001
KIT (KIT)	Internal pressure test	Corroded, exposure #02	IPC2-KIT-001
VTT (VTT)	Internal pressure test	Corroded, exposure #01	IPC1-VTT-001
VTT (VTT)	Internal pressure <u>creep</u> test	Virgin	IPC-VTT-001
KIT (KIT)	Internal pressure creep test	Corroded in exposure #01	IPCC1-KIT-001

The measure corrosion depths (c) for each test batch can be identified through their designated corrosion exposure number as described below.

4 Corrosion exposures

Prior work at SCK CEN for the MYRRHA design has included rigorous corrosion exposure testing of the 15-15Ti steel [7][8]. The corrosion rate, here given as maximum corrosion depth, can be described as a linear function in time. At 500°C the rate estimate is 0.0257 μm / hour, reaching corrosion depths of 100 μm or more after exposures in the range of ½ year. The planning of the PATRICIA exposures are based on the results of the below described C_0 reference exposures. The calibration and fine-tuning of test program and test matrices for the different test types will be performed according to the preliminary results acquired from testing the C_0 tubes and based on the results from the uniaxial plate sample testing.

The corrosion exposures are performed at the SCK facility as described below.

4.1 The static corrosion test rig at SCK CEN

The static Pb-Li corrosion test rig at SCK CEN is shown in Figure 8. The main components of the apparatus is the stainless-steel autoclave. The autoclave lid is housing the lead-through ports for gas inlet and outlet; thermocouples; the sample holder; the oxygen pump and the Bi/Bi₂O₃ electrochemical oxygen sensor. Autoclave resides the alumina crucible filled with 5 kg of liquid Pb-Bi eutectic. The experimental setup is placed in a glove-box, where a low-oxygen atmosphere is maintained.

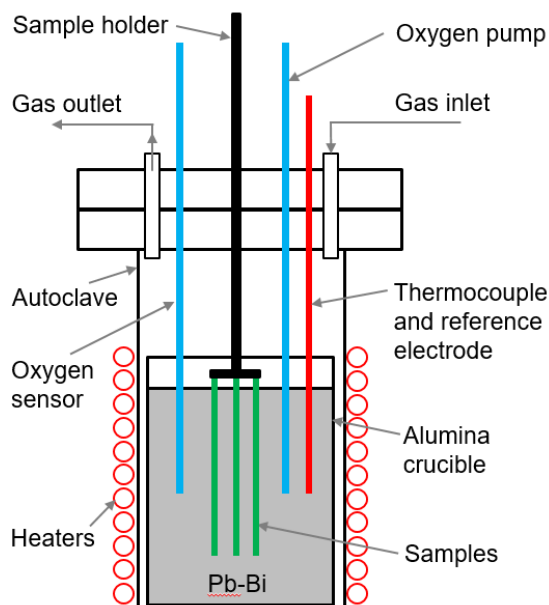


Figure 8. Schematic structure of the Pb-Li static corrosion test rig.

The target conditions for the PATRICIA corrosion exposures are: 500 °C and Pb-Bi providing a dissolution corrosion mode (low oxygen content). The low level of oxygen concentration in the liquid metal is provided by means of continuous purging of Ar+5% H₂ gas mixture over the surface of liquid metal mirror. The static corrosion test and all the preparations are conducted under the supervision of the SCK CEN corrosion team (see Acknowledgment).

4.2 Reference corrosion exposures C₀

This corrosion exposures designated as C₀ are a series of pre-PATRICIA exposures originally intended for testing under the umbrella of the EERA JPNM pilot project TASTE+. The tube sample IDs (not compliant with the here designated identification key) are given in Table 6. The given wall thickness values are from 24 measurements over the full circumference of the tube as shown in Figure 9. These measured remaining wall thicknesses converted to material loss are presented in Figure 10. Note that the average loss of wall thickness (corroded from external and internal side) is about a factor of 2 slower than the (one sided) prediction of maximum corrosion depth. The corresponding maximum loss of wall thickness is very close to the predicted line. A simultaneous maximum corrosion depth on both the inner and outer radiuses is not expected to occur at the same location (see Figure 9).

Table 6. The pre-PATRICIA corrosion exposures with sample ID, time-temperature and measured mean and minimum remaining wall thickness. Note that for corrosion exposures with both internally and externally corroded material corrosion, as is the case for these cladding tubes, a single sided measurement of corrosion depth (c) is not representative for strength reduction assessments and cannot simply be multiplied by a factor of two for calculating remaining wall thickness. The given uncertainty of the average wall thickness relates to the 90% confidence interval.

Designation	Exposure designation	Exposure duration (h)	Average wall thickness (μm)	Minimum wall thickness (μm)
TB-74	C0a	1000	434±4	413
TB-05	C0b	2000	417±4	394
TB-07	C0c	3044	386±7	352
TB-03	C0d	4196	379±6	355
TB-02	C0d	4196	385±8	347

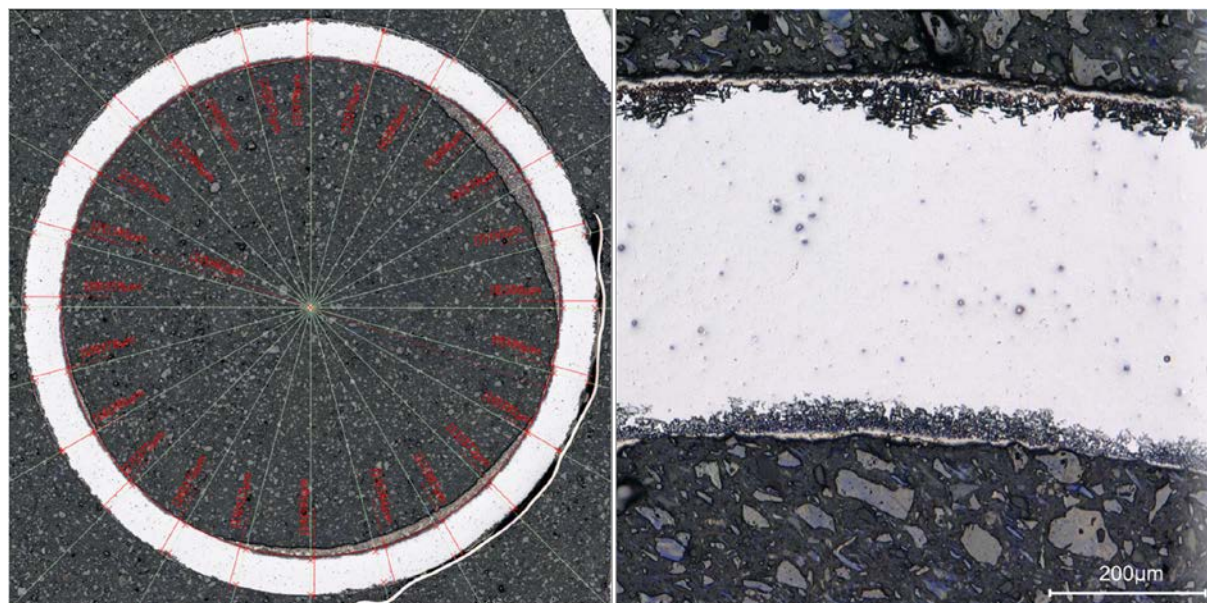


Figure 9. Measurement of remaining wall thickness for sample TB-03.

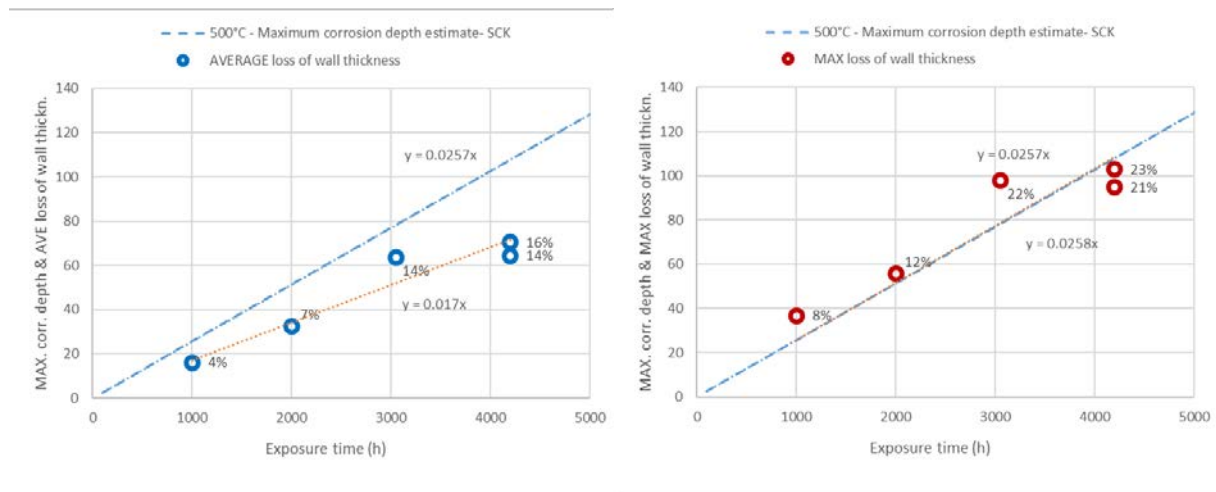


Figure 10. The Table 1 measured loss of wall-thicknesses in relation to the SCK CEN predictive line for maximum corrosion depths.

These C_0 tubes are to be used for initial sighting testing (on corroded samples) using test techniques requiring small amount of material, e.g. RC, RT and SP. The main difference between these samples and the exposures to be conducted within PATRICIA is that the tubes have been corroded both externally and internally. This feature might cause added uncertainty for testing techniques where friction is a factor that influences the measurands. These tests do not currently have “aged” reference samples to the same time-temperature conditions but the results will eventually be compared to aged material of 1000 h and 4000 hour aging.

The measured remaining wall thicknesses from the C_0 exposures when translating into relative reduction of force needed for tensile failure (due to the reduction of effective area) is shown in Figure 11 assuming the same homogeneous corrosion depth as measured in Table 6.

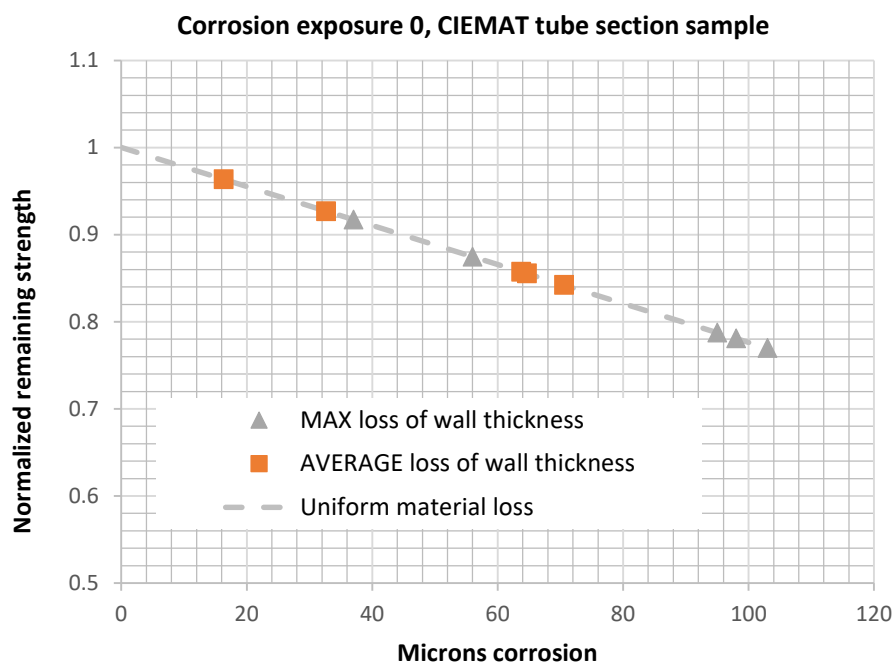


Figure 11. Calculated relative expected reduction in force to attain tensile failure when calculating with the C_0 exposure average remaining wall thickness and the maximum loss of wall thickness (see Table 6 and Figure 10).

From the initial measurements at SCK CEN on the prior-to exposure plate specimen the uncertainty of the effective cross area can be in the range 0.2-2% (95% confidence), in most samples clearly below 1% of the nominal calculated area. The uncertainty to take into consideration is though increasing when combining the initial sample with the post-exposure measurements. The uncertainty of the remaining wall thickness after exposure was shown to be in the range of about 6 microns for the samples given in Table 6. If this is considered a representative indicator for post-corrosion plate samples thicknesses, the combined uncertainty of the effective area (and stress) increases to about 1-3%.

4.3 Corrosion exposures C1-Cx

The first corrosion exposure **C₁** will be conducted on plate samples, targeting a both sided corrosion depth of about 100 µm for the 0.7 mm thick plate samples. The following exposure **C₂** will target the same for tube samples that are plugged to ensure external corrosion only. Sacrificial samples from both exposures will be analyzed for corrosion depth and remaining wall thickness at about 1000 h of exposure and at the final exposure time. Their analyses, and the measured corrosion depths and remaining wall thickness are at least initially considered representative of the whole batch. This is especially the case for the uniaxial plate samples where verification sampling and microscopy would have to be made after testing. In the case of exposed tubes rings can simply be extracted from every tube after exposure and sample manufacturing.

The samples for the main test campaign of RT, RC and SP are extracted from these externally corroded tubes with clean inner diameter. These tubes are also intended for the internal pressure testing at KIT and VTT.

5 Ageing exposures

The aging exposures are performed to acquire material with the same time-temperature history as the corresponding corroded samples. Note that the material properties are not expected to be very sensitive to small differences in the aging time-temperature, thus one aging exposure might be sufficient to represent all corrosion exposures. As mentioned earlier the aging is expected to improve the material properties at the temperature and time frames needed for reaching the target corrosion depths. Thus, the aging is a necessity for applying the methodology described in the 'Method' section of this report.

5.1 Ageing exposures A1-A2

The initial aging exposures will be set to 1000 h (A1) and 5000 h (A2) at 500°C. For virgin materials, these time-temperature exposures are not expected to show any $M_{23}C_6$ precipitation for 15-15Ti [9] but as described in [5] the nano-precipitate TiC can have an impact on both strength and ductility that cannot be ignored for the assessment of corrosion damage. Micro-hardness (HV0.1) testing on the C_0 exposed samples showed negligible change in hardness between virgin material and exposed materials very slightly above the expected virgin material hardness $HV\ 282 \pm 11$ [9] for 20% CW claddings.

6 Test matrix

The test matrix for the different tests, materials and testing labs are based on the target to be able to compare a minimum of 5 test repetitions of each material, test condition and test type. For the more complex internal pressure high temperature tests (burst) the minimum number of tests is two for aged and for corroded tubes at two test temperatures. For the internal pressure creep testing only test to failure are relevant to acquire failure ductility values. Thus two tests at two different test temperatures are targeted.

The initial virgin and aged state properties of the 15-15Ti steel will be conducted by CIEMAT and KIT using the flat plate samples (from then 20%CW bar), full tube samples and EDM cut tube section samples according to the CIEMAT specimen specification.

The sighting tests to be conducted, e.g. ring tension, ring compression and small punch are using the pre-PATRICIA samples will give valuable information to stream line the main test program and make decisions on the applicability of each test method for reaching an answer to the main research question.

7 Reference and sighting tests for virgin and existing corroded samples

7.1 Uniaxial tensile tests (UT) on virgin, aged and corroded plate samples

The preliminary test matrix for uniaxial sighting and reference tests for comparison to corrosion exposed tests are given in Table 7. Test for tube with the pre-PATRICIA corrosion exposure (C₀) and test for plate in virgin state are considered to be sighting tests.

Table 7. Preliminary test matrix for uniaxial tensile tests (reference tests) on virgin, aged (A1, A2) and corroded samples (C0, C1, C2). Note that the sample numbers are indicated as xxx only.

Laboratory (Table 4)	Material	Number of tests	ID code	Note
CIE	Virgin (tube)	2	T-CIE-xxx	Sighting (flattening by grip)
CIE	Virgin (plate)	1	P-CIE-xxx	Sighting
CIE	Aged (tube)	5	TA2-CIE-xxx	Reference
CIE	Aged (plate)	5	PA2-CIE-xxx	Reference
CIE	Corroded (tube)	5	TC2-CIE-xxx	Reference
CIE	Corroded (plate)	5	PC2-CIE-xxx	Reference
KIT	Virgin (tube)	2	T-KIT-xxx	Sighting (Full tube)
KIT	Virgin (plate)	1	P-KIT-xxx	Sighting
KIT	Aged (tube)	5	PA2-KIT-xxx	Reference
KIT	Aged (plate)	5	PA2-KIT-xxx	Reference
KIT	Corroded (tube)	5	PA2-KIT-xxx	Reference
KIT	Corroded (plate)	5	PA2-KIT-xxx	Reference

7.2 RT, RC and SP sighting tests on corroded tube samples (C0)

The sighting tests for internally and externally corroded tubes are conducted to evaluate the impact of friction on Ring Tension and Small Punch test techniques. The Ring Compression tests are not affected by friction. The preliminary test matrix for RT test is given in Table 8.

Table 8. Preliminary test matrix for Ring Tension sighting tests on C0 samples. Note that the expected strength reduction is tube specific (see Table 6).

Laboratory (Table 4)	Material	Number of tests	ID code	Note
RAT	Corroded tube C0	2	RTC0-RAT-xxx	Sighting test
NRG	Corroded tube C0	2	RTC0-NRG-xxx	Sighting test

The preliminary test matrix for RC test is given in Table 9.

Table 9. . Preliminary test matrix for Ring Compression sighting tests on C0 samples. Note that the expected strength reduction is tube specific (see Table 6)

Laboratory (Table 4)	Material	Number of tests	ID code	Note
ENEA	Corroded tube C0	2	RCC0-ENE-xxx	Sighting test
JRC	Corroded tube C0	2	RCC0-JRC-xxx	Sighting test

The preliminary test matrix for SP testing is given in Table 10.

Table 10. . Preliminary test matrix for Small Punch sighting tests on C0 samples. Note that the expected strength reduction is tube specific (see Table 6). Note that NRG and CIEMAT will conduct tests on the smaller SP sample requiring sample manufacturing.

Laboratory (Table 4)	Material	Number of tests	ID code	Note
CIEMAT	Corroded tube C0	2	SPC0-CIE-xxx	Miniature SP
JRC	Corroded tube C0	2	SPC0-JRC-xxx	Curved sample

8 Tests on corroded (C1-C2) and aged (A1-A2) materials

These test are the main test campaign of the project task. Note that the number of tests and the test conditions might change after the sighting test and assessments of the C0 exposed samples have been performed.

8.1 Uniaxial tests on corroded and aged samples

The provisional test matrix for uniaxial tensile tests on corroded and aged materials is given in Table 11. The reference tests on virgin and reference aged (A1) material were listed previously in Table 7.

Table 11. Provisional test matrix for uniaxial tests on corroded material (assuming 2 corrosion depth conditions)

Laboratory (Table 4)	Material	Corrosion exposure	Total Number of tests	ID code	Note
CIE	Corroded (tube)	1	5	TC2-CIE-xxx	
CIE	Corroded (plate)	2	5	PC1-CIE-xxx	
KIT	Corroded (tube)	1	5	TC2-KIT-xxx	
KIT	Corroded (plate)	2	5	PC1-KIT-xxx	

8.2 RT tests on corroded and aged tube samples

The provisional test matrix for RT tests on corroded and aged materials is given in Table 12.

Table 12. . Provisional test matrix for RT tests on corroded and aged material.

Laboratory (Table 4)	Material	Exposure	Total Number of tests	ID code	Note
RAT	Corroded (tube)	C2	5	RTC2-RAT-xxx	External corrosion
RAT	Aged (tube)	A2	5	RTA2-RAT-xxx	
NRG	Corroded (tube)	C2	5	RTC2-NRG-xxx	External corrosion
NRG	Aged (tube)	A2	5	RTA2-NRG-xxx	

8.3 RC tests on corroded and aged tube samples

The provisional test matrix for RC tests on corroded and aged materials is given in Table 13.

Table 13. . Provisional test matrix for RC tests on corroded and aged material.

Laboratory (Table 4)	Material	Exposure	Total Number of tests	ID code	Note
ENE	Corroded (tube)	C2	5	RCC2-ENE-xxx	External corrosion
ENE	Aged (tube)	A2	5		
JRC	Corroded (tube)	C2	5	RCC2-JRC-xxx	External corrosion
JRC	Aged (tube)	A2	5	RCA-JRC-xxx	

9 SP tests on corroded and aged tube samples

The provisional test matrix for SP tests on corroded and aged materials is given in Table 14.

Note that the SP test set-up and samples of CIEMAT and JRC differ. The CIEMAT tests will be conducted on miniature samples extracted from mid-wall of the cladding tube. Thus it is possible that any detrimental surface effect from the corrosion exposure might be removed and show little or no differences to tests conducted with the corresponding aged material. In the case of the JRC test set-up on curved, cladding sections (see Figure 6) , the full remaining wall thickness is tested with the maximum multiaxial stresses and strains developing at the corroded external surface.

Table 14. . Provisional test matrix for SP tests on corroded and aged material.

Laboratory (Table 4)	Material	Exposure	Total Number of tests	ID code	Note
CIE	Corroded (tube)	C2	5	SPC2-CIE-xxx	Miniature, mid wall sample
CIE	Aged (tube)	A2	5	SPA2-CIE-xxx	Miniature, mid wall sample
JRC	Corroded (tube)	C2	5	SPC2-JRC-xxx	External corrosion
JRC	Aged (tube)	A2	5	SPA2-JRC-xxx	External corrosion

9.1 IP and IPC tests on corroded and aged tube samples

The provisional test matrix for IP and IPC tests on corroded and aged materials is given in Table 15 and Table 16.

Table 15. . Provisional test matrix for IP (raising pressure) tests on corroded and aged material. Assuming 1 temperature chosen according to lab specific pressure limits (to support the IPC testing)

Laboratory (Table 4)	Material	Exposure	Total Number of tests	ID code	Note
VTT	Corroded (tube)	C2	2	IPC2-VTT-xxx	Lab spec. Temp
VTT	Aged (tube)	A2	2	IPA2-VTT-xxx	Lab spec. Temp
KIT	Corroded (tube)	C2	2	IPC2-KIT-xxx	Lab spec. Temp
KIT	Aged (tube)	A2	2	IPA2-KIT-xxx	Lab spec. Temp

Table 16. . Provisional test matrix for IPC tests on corroded and aged material. Assuming 2 (or 3) pressure levels at lab specific test temperature.

Laboratory (Table 4)	Material	Exposure	Total Number of tests	ID code	Note
VTT	Corroded (tube)	C2	3	IPCC2-VTT-xxx	2-3 pressures at lab Temp.
VTT	Aged (tube)	A2	3	IPCA2-VTT-xxx	2-3 pressures at lab Temp.
KIT	Corroded (tube)	C2	3	IPCC2-KIT-xxx	2-3 pressures at lab Temp.
KIT	Aged (tube)	A2	3	IPCA2-KIT-xxx	2-3 pressures at lab Temp.

10 Additional testing

Additional uniaxial testing for strain aging effect (CIEMAT, KIT)

The additional tests recommended by CIEMAT to identify the potential impact of strain aging [10]- [14] are given in Table 17.

Table 17. . Provisional test matrix for detection of stain aging by uniaxial tensile testing at 3 startup strain rates of $3.3 \cdot 10^{-5}$ /s , $3.3 \cdot 10^{-4}$ /s and $3.3 \cdot 10^{-3}$. (proof strength setting)

Laboratory (Table 4)	Material	Number of tests	ID code	Note
CIE	A2	3	TA-CIE-xxx	550°C / 3 rates
CIE	A2	3	TA-CIE-xxx	600°C / 3 rates
CIE	A2+C2	3+3	TA/PA-CIE-xxx	Detected DSA range
KIT	A2	3	PA-KIT-xxx	700°C / 3 rates
KIT	A2	3	PA-KIT-xxx	650°C / 3 rates
KIT	A2+C2	3	PA-KIT-xxx	Detected DSA range

Additional RC tests at elevated temperatures (ENEA)

ENEA is planning to in addition to the room temperature RC testing also perform tests at elevated temperatures, e.g. 200 °C or at cryogenic temperatures towards where the Ductile to Brittle Transition Temperature shift should be detectable. These tests are targeting conditions where a drop in the uniform and total elongation could be expected. The provisional test matrix is given in Table 18.

Table 18. Provisional test matrix for RC tests at elevated or cryogenic temperature.

Laboratory (Table 4)	Material	Exposure	Total Number of tests	ID code	Note
ENEA	Corroded	C2	4	RCC2-ENEA-xxx	4 test at chosen elevated temperature
ENEA	Aged	A2	4	RCA-ENEA-xxx	4 test at chosen elevated temperature
ENEA	Corroded	C2	4	RCC2-ENEA-xxx	4 test at chosen temperature below DBTT
ENEA	Aged	A2	4	RCA-ENEA-xxx	4 test at chosen temperature below DBTT

Segmented Cone Mandrel tests (JRC)

Additional test on ring segments have been proposed by JRC. These test will be conducted in one laboratory only. The provisional test matrix is given in Table 19.

Table 19. . Provisional test matrix for SCM tests on corroded and aged material. Tests at lab specific test temperature and elevated temperature (e.g. 500°C).

Laboratory (Table 4)	Material	Exposure	Total Number of tests	ID code	Note
JRC	Corroded (tube) Only the outside is corroded	C2	4	SCM-JRC-xxx	2 test at room and elevated temperature
JRC	Aged	A2	4	SCM-JRC-xxx	2 test at room and elevated temperature

11 Specimen manufacturing

11.1 Uniaxial plate type samples from bar (20%CW)

To date 14 flat plate samples have already been manufactured by SCK CEN and have been measured through automated dimensional measurement for each specimen separately. The measurements verify that each sample has to be measured and the results analyzed on sample specific dimensions to avoid additional uncertainties. The preliminary test matrix given in Table 7 shows a minimum need of 22 plate samples. The corrosion exposure facility can expose 18 plate samples in a batch. A number of 22 additional samples are currently being manufactured at the SCK CEN workshop. Thus, the first

complete batch of 18 samples for exposure will cover the RT tests (5+5) and 2 sacrificial samples for corrosion depth measurement removed at 1000 h of exposure and at the end of exposure. The remaining 6 corroded plate samples are intended for the additional test program (see Table 17).

Note that the plate sample thicknesses have to be re-measured before the corrosion exposure start due to the need of surfaces polishing to remove all oxide layers (from air exposure) that could impair the LBE wetting and thus the corrosion rate.

11.2 Uniaxial tube section specimen (CIEMAT design)

In all 20 samples for uniaxial and corrosion exposures will be manufactured. 10 for virgin and aged material testing and 10 from corrosion exposed tubes.

11.3 Ring Tension samples

In all 20 samples for RT testing will be manufactured. 10 from virgin and aged material and 10 from corrosion exposed tubes. An additional 6 samples will be extracted from the pre-Patricia corroded test tubes.

11.4 Ring Compression samples

In all 20 samples for RC testing will be manufactured. 10 from virgin and aged material and 10 from corrosion exposed tubes. An additional 6 samples will be extracted from the pre-Patricia corroded test tubes.

11.5 Small Punch samples

Approximately 20 samples for SP testing will be manufactured at SCK and CIEMAT. The preliminary sample estimate is 10 from aged material test and 10 from corrosion exposed tubes. The miniature SP samples are manufactured at CIEMAT and the JRC compatible curved samples at SCKCEN. An additional 6 samples will be extracted from the pre-Patricia corroded test tubes.

12 Test result data management

The data will for the initial phase of the project be stored in a format defined by the testing laboratory. The data is collected and moved into a data format that will be defined by the consortium. The initial intention is to download all data into the European Commission Mat-DB data base.

13 Conclusion

This report defines the assessment method chosen for the determination of potential detrimental effect of LBE corrosion, in addition to the expected material loss. The material mechanical properties tensile strength, proof stress and ductility are used as base in the comparative assessment by reference cold worked plate sample testing and a range of test methods for cladding tubes at room and elevated temperature. There is a need to expand the tests to include aged materials as a base line due to recent publications showing material property improvements in the temperature-time range imposed on the test material when corrosion exposed. This report presents the preliminary test matrices for the chosen test methods for plate sample testing and those suitable cladding tubes with small wall thickness.

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