



STUDIECENTRUM VOOR KERNENERGIE
CENTRE D'ÉTUDE DE L'ÉNERGIE NUCLÉAIRE

**SCK•CEN Contribution to the
IAEA Round Robin Exercise
on WWER-440 RPV Weld
Material Irradiation,
Annealing and Re-
Embrittlement**

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RMR

Reactor Materials
Research

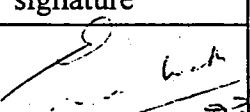
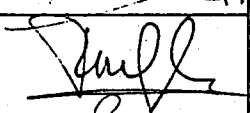
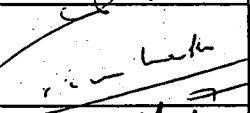
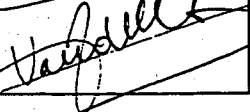
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SCK•CEN Contribution
to the
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Irradiation Embrittlement, Annealing and Re-Embrittlement
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1. Introduction

The objective of SCK•CEN to contribute to this RR on WWER-440 weld material is twofold:

1. to gain experience in the field of WWER-440 steels;
2. to analyse the RR-data according to the models used and developed at SCK•CEN in order to check their validity and applicability.

Here we report information on the testing of the unirradiated material: chemical analysis, Charpy-V impact testing, tensile testing and fracture toughness determination.

On the other hand, we will outline the irradiation strategies that can be followed to complete the program: two irradiation facilities, CHIVAS or MERLIN both making use of the Belgian MTR BR2, could be loaded with the samples to obtain the irradiated (I), irradiated-annealed (IA) and irradiated-annealed-reirradiated (IAR) conditions

2. Test matrix

SCK•CEN purchased part 2 of weld N° 502 representative for WWER-440 weld material and identified as block 502.2. The certification report on the weld manufacturing, heat treatment, chemical composition and mechanical test results can be found in reference [1]. The cutting scheme of the 502.2-block was made according to the procedure stipulated in the terms of reference, IAEA TC Project RER/9/035 WWER-SC-192 [2], and can be found in Annex 1 to this report. The extracted specimens are of Charpy-V impact type (Cv), Pre-cracked Charpy (PCCv) and Tensile specimens (T). The number of specimens extracted and their assigned purpose are given in Table 1. Thirty slices were cut out of the block within the limits defined by program. From each slice four Charpy-type specimens were extracted and, for most slices, one tensile specimen. The nominal dimensions, tolerances and identification of the specimens are given in Annex 1: for Charpy-type specimens the ASTM E23 standard was followed, except for the notch of the PCCV specimens which was made sharper to promote pre-cracking; the tensile specimen corresponds to the 'Gagarinskij'-type with threading on the heads.

Table 1. Samples extracted from block 502.2

WWER-440-block N° 502.2	Cv	PCCv	T
specimen orientation	T-L	T-L	T
# of specimens as fabricated	60	60	24
position	layer 1, 2	layer 3, 4	layer 5
identification	5.01.1-2 through 5.30.1-2	5.01.3-4 through 5.30.3-4	5.01.5 through 5.24.5

3. Chemical Analysis

A thin slice of material next to slice one, mentioned on the cutting diagram in Annex 1, was cut and small steel parts were extracted at the height of each specimen layer in the weld centreline. These samples were numbered from 1 to 5 according to the corresponding layer and where then analysed with the ICP-MS (Inductively Coupled Plasma - Mass Spectrometry) technique in order to reveal their chemical composition. Table 2 gives the chemical content of the major components of material as a function of the position in the weld. The uncertainty is about 3%. These results can be compared with the information found in the weld certification document [2]. The correspondence is very good for V, Ni, Cu and Mo; the SCK•CEN results are systematically lower for Cr and Mn. A second analysis for confirmation is actually performed. The analysis for less abundant elements like C, Ti, P and S remains to be done.

Table 2: Through thickness chemical composition of the weld material

wt%	layer1	layer2	layer3	layer4	layer5
V	0.19	0.19	0.19	0.20	0.19
Cr	1.44	1.50	1.47	1.58	1.55
Mn	0.99	0.96	0.95	0.97	0.96
Ni	0.12	0.12	0.12	0.12	0.12
Cu	0.13	0.13	0.13	0.12	0.12
Mo	0.46	0.46	0.45	0.48	0.47

4. Reference Testing

The number of specimens that need to be tested to determine the baseline impact, tensile and fracture toughness properties of the weld 502-material are respectively, 15 Cv, 15 PCCv and 6T. The other specimens are put aside for the irradiations. Mini Cv samples will be prepared at a later stage from the tested Cv-impact specimens.

The specimen selection for testing is such that a variation over the full layer of the weld material is present.

4.1 Cv-impact testing

The impact tests were carried out on a certified TONI-MFL 300J machine equipped with an instrumented DIN-tup, according to ASTM E23 procedures. Temperature stability is within 2°C. The impact test results can be found in Table 3. The tanh fits of the energy, shear fracture appearance and lateral expansion are shown in Figures 1 to 3. An example of the quality of the instrumented curves is given in Figure 4.

Table 3: Instrumented Charpy-V impact results of CRP-IV specimens

Sp. ID	T (°C)	E (J)	SFA (%)	L.E. (mm)
5.05.1	-60	3	1	0.12
5.02.1	-40	7.2	8	0.24
5.21.1	-30	27.4	11	0.61
5.12.1	-20	23.2	21	0.50
5.15.1	0	42.5	23	0.89
5.11.1	15	68.2	55	1.21
5.25.1	21	87.8	66	1.56
5.22.1	50	104.1	84	1.92
5.01.1	80	111.2	98	1.93
5.04.1	100	115.4	100	1.95
5.14.1	175	117.8	100	2.20
5.23.1	268	124.2	100	2.37

Figure 1: Energy transition curve of WWER-440 weld material

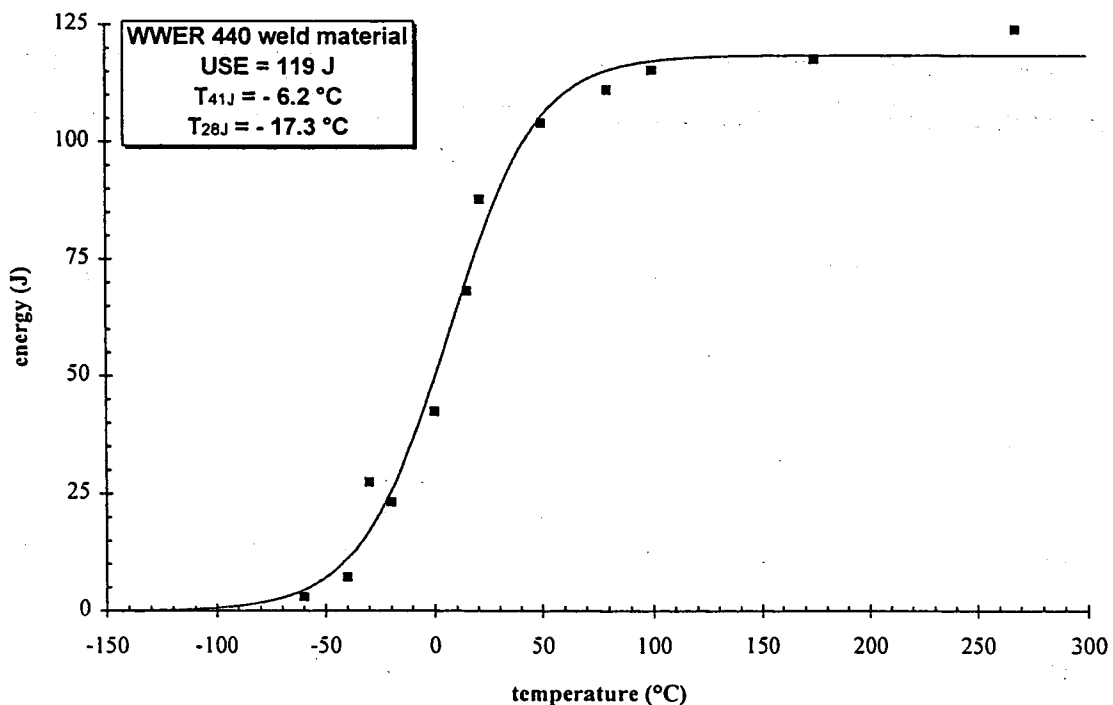


Figure 2: SFA transition curve of WWER-440 weld material

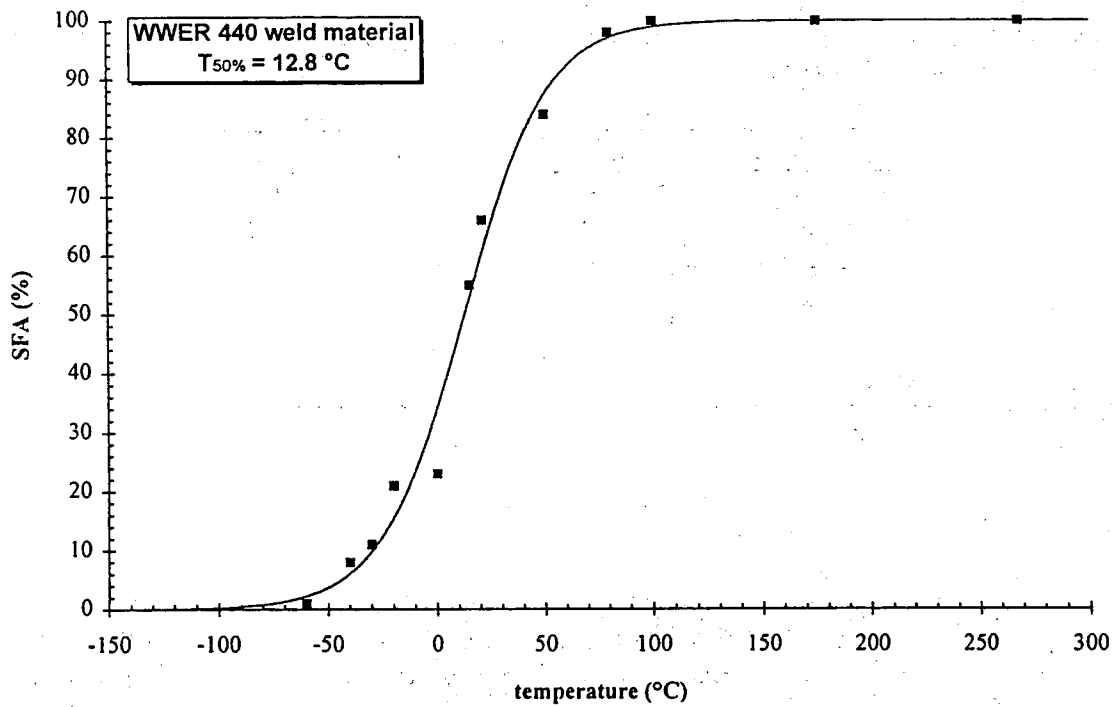
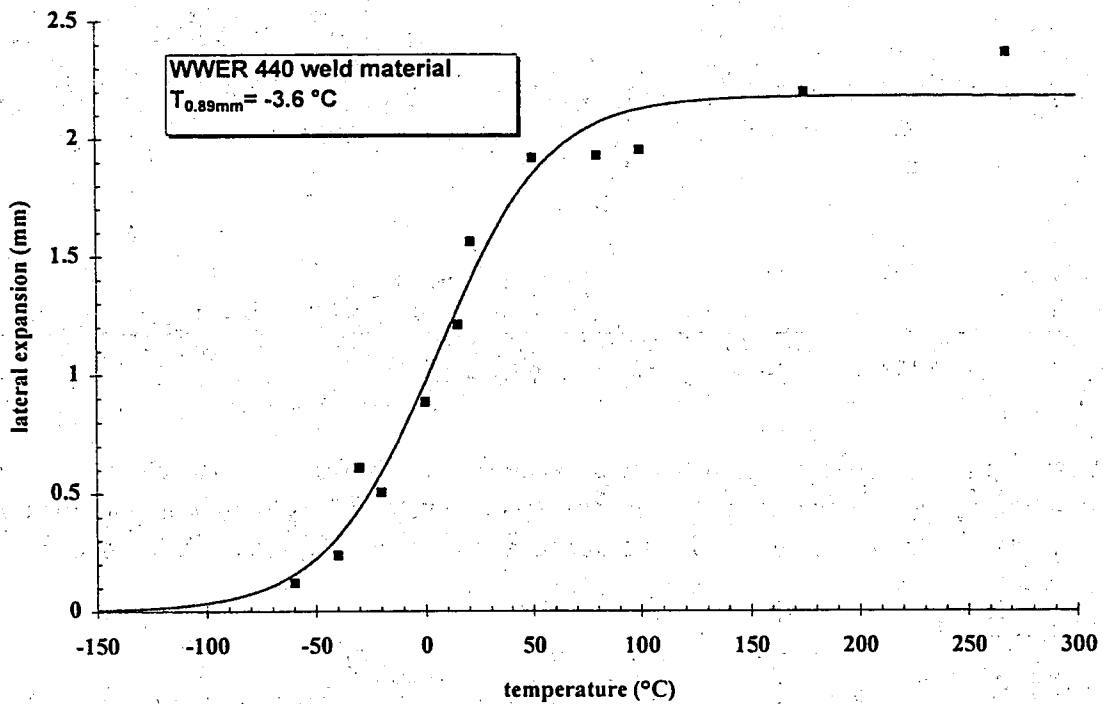


Figure 3: lateral expansion transition curve of WWER-440 weld material



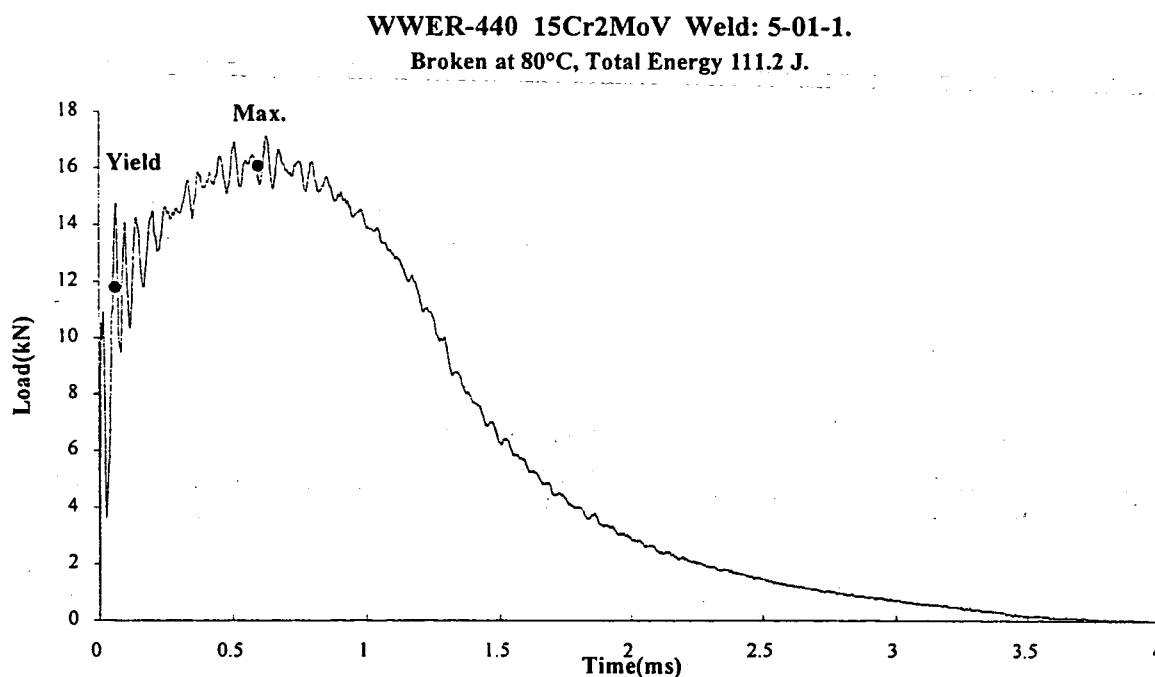
The correspondence of the transition temperatures with the certification exercise [2] is good with a slight deviation for the fracture appearance, as can be seen from Table 4. The fracture

appearance ratio was determined according to the eye-methodology given in ASTM E23. Although this methodology has proven to be quite accurate, a detailed analysis will be made from enlarged photographic reproductions.

Table 4: Comparison of the transition temperatures between the qualification exercise and the SCK•CEN results

<i>Cv-impact</i>	T_{41J} (°C)	$T_{50\%}$ (°C)	$T_{0.9mm}$ (°C)
layer 1	-4	3	12
layer 2	-8	-3	-6
layer 3a	-9	0	3
layer 3	-5	-2	5
layer 4	-2	4	8
SCK•CEN (11,12)	-6	13	-4

Figure 4: Example of instrumented curve



The tests on mini Cv specimens still need to be performed. In principle we would prefer to test 3x4x27mm specimens due to test machine limitations and in order to compare the data with our analysis methodologies. This point needs to be discussed at the IAEA-meeting.

4.2 Tensile testing

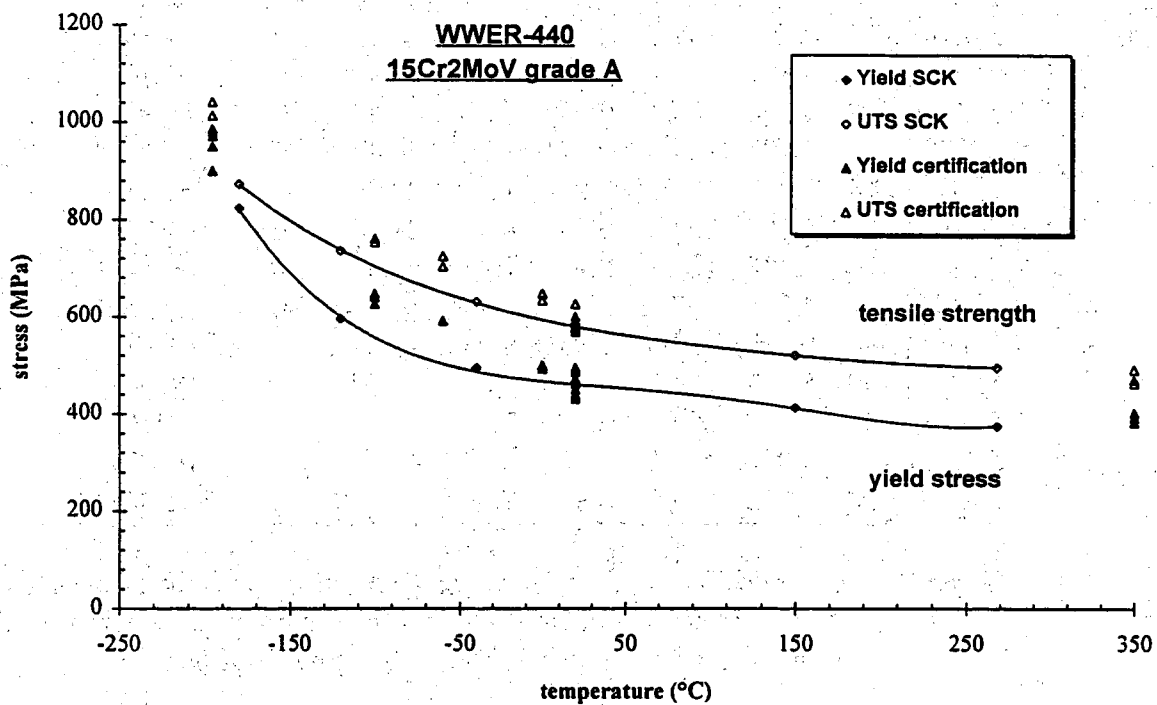
The tensile test were performed on an certified Instron tensile bench in accordance to ASTM E8. The different temperatures are obtained in a PID-controlled environmental chamber with a stability of $\pm 0.5^\circ\text{C}$ at the specimen.

Table 5: Tensile results on block 502.5

Spec. ID	T (°C)	yield (MPa)	UTS (MPa)	ϵ_u (%)	ϵ_t (%)	RA (%)
5.12.5	-180	823.5	872.9	11.82	19.80	56.87
5.11.5	-120	596.0	736.1	12.35	21.39	66.11
5.02.5	-40	495.3	631.0	10.77	19.37	69.13
5.01.5	20	455.0	578.1	8.52	17.07	71.59
5.21.5	150	413.3	521.4	7.38	16.22	72.69
5.22.5	268	375.9	496.5	6.59	15.19	69.41

Figure 5 shows the tensile test results of SCK-CEN (layer 5) in comparison to the certification results (through weld) [2]. The correspondence is very good, taken into account that the certification results reflect the overall distribution of the flow properties throughout the weld.

Figure 5: Tensile properties of layer 5 (SCK-results) compared with the results of the certification exercise. The fits are a guide to the eye and go through the SCK data



4.3 PCCv static 3PB testing

The T_{28J} temperature of the Charpy energy data is -17.3°C . According to the proposed ASTM 'Test Method for the Determination of Reference Temperature, T_0 , for Ferritic Steels in the Transition Range'- Draft 15, the so-called mastercurve approach [3], this leads to a suggested test temperature for fracture toughness testing of -67°C .

Two series of 7 three point-bend fracture toughness tests on precracked sidegrooved Charpy-type specimens were carried out at -65°C and -50°C. The tests were performed on a certified Instron bench. The temperatures are obtained in a PID-controlled chamber with an stability of $\pm 0.5^\circ\text{C}$ at the specimen. The J integral is calculated from load and CMOD-measurements, that are used to obtain the load-point displacement. K_J is calculated from J using

$$K_J = \sqrt{\frac{JE}{1-\nu^2}}, E = 210 \text{ GPa}, \nu = 0.3$$

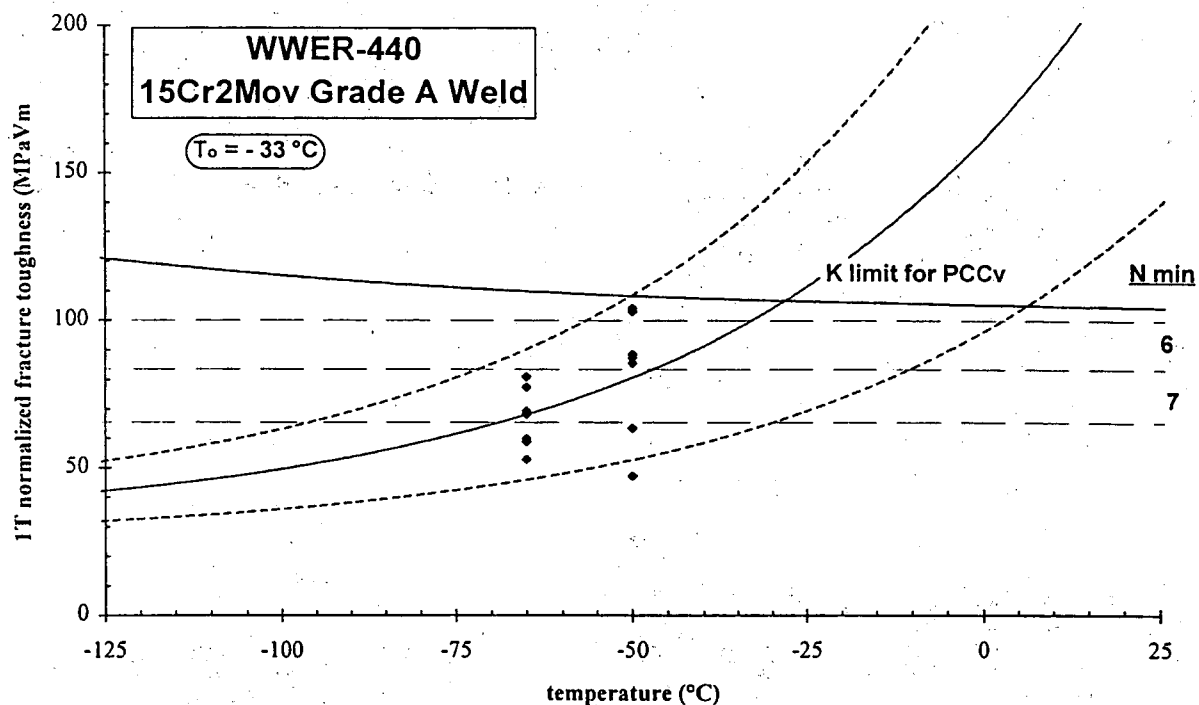
The results of the fracture toughness tests are given in Table 6. All tests are valid according to the master curve procedure [3]. The results were normalised to 1T. The data points for block 502.5, layers 3, are shown in Figure 6 with their master curve leading to a mean $T_0 = -33^\circ\text{C}$. The 5% and 95% bounds and the K-limit for PCCv testing are traced. On the right side of the figure the ASTM recommended number of test specimens is given for a given median K_J -value. As each time 7 valid specimens were tested, this condition is fulfilled for our data.

Table 6: Fracture toughness data for PCCv specimens

Spec. ID.	T (°C)	a_0 (mm)	W (mm)	B (mm)	B_n (mm)	J_c (kJ/m ²)	Δa (mm)	K_{Jc} (MPa√m)
5.01.3	-65	5.741	10.01	9.99	8.00	29.36	0	82.31
5.03.3	-65	4.74	10.00	10.01	8.00	37.15	0	92.59
5.11.3	-65	4.827	10.01	9.98	8.00	40.90	0	97.15
5.12.3	-65	4.994	10.01	9.99	7.99	28.42	0	80.98
5.15.3	-65	4.791	10.00	9.97	8.01	16.43	0	61.58
5.21.3	-65	4.812	10.00	9.98	7.98	20.74	0	69.18
5.22.3	-65	5.202	10.00	9.99	8.00	21.46	0	70.37
5.23.3	-50	4.838	10.01	9.99	7.97	69.33	0	126.49
5.24.3	-50	4.885	10.00	9.99	7.98	12.89	0	54.54
5.02.3	-50	5.117	10.00	9.98	8.05	46.02	0	103.05
5.04.3	-50	5.227	10.00	9.99	8.00	67.77	0	125.06
5.05.3	-50	5.608	10.00	10.00	7.97	48.10	0	105.36
5.13.3	-50	4.896	10.00	9.98	8.00	49.33	0	106.70
5.24.3	-50	4.873	10.00	9.98	7.99	24.49	0	75.18

Legend: a_0 = crack length, W = specimen width, B = specimen thickness, B_n = specimen net thickness, J_c = J-integral at unstable fracture Δa = ductile crack extension, K_{Jc} = fracture toughness

Figure 6: Master curve for block 502.5, layers 3, obtained with PCCv specimens. The master curve is based on all data points. The validity limits are given.



5. Irradiation Possibilities

According to the Terms of Reference a 2 year time span remains for performing the irradiations, the testing and the analysis of the results. Several options remain to perform the irradiations. These will be outlined below.

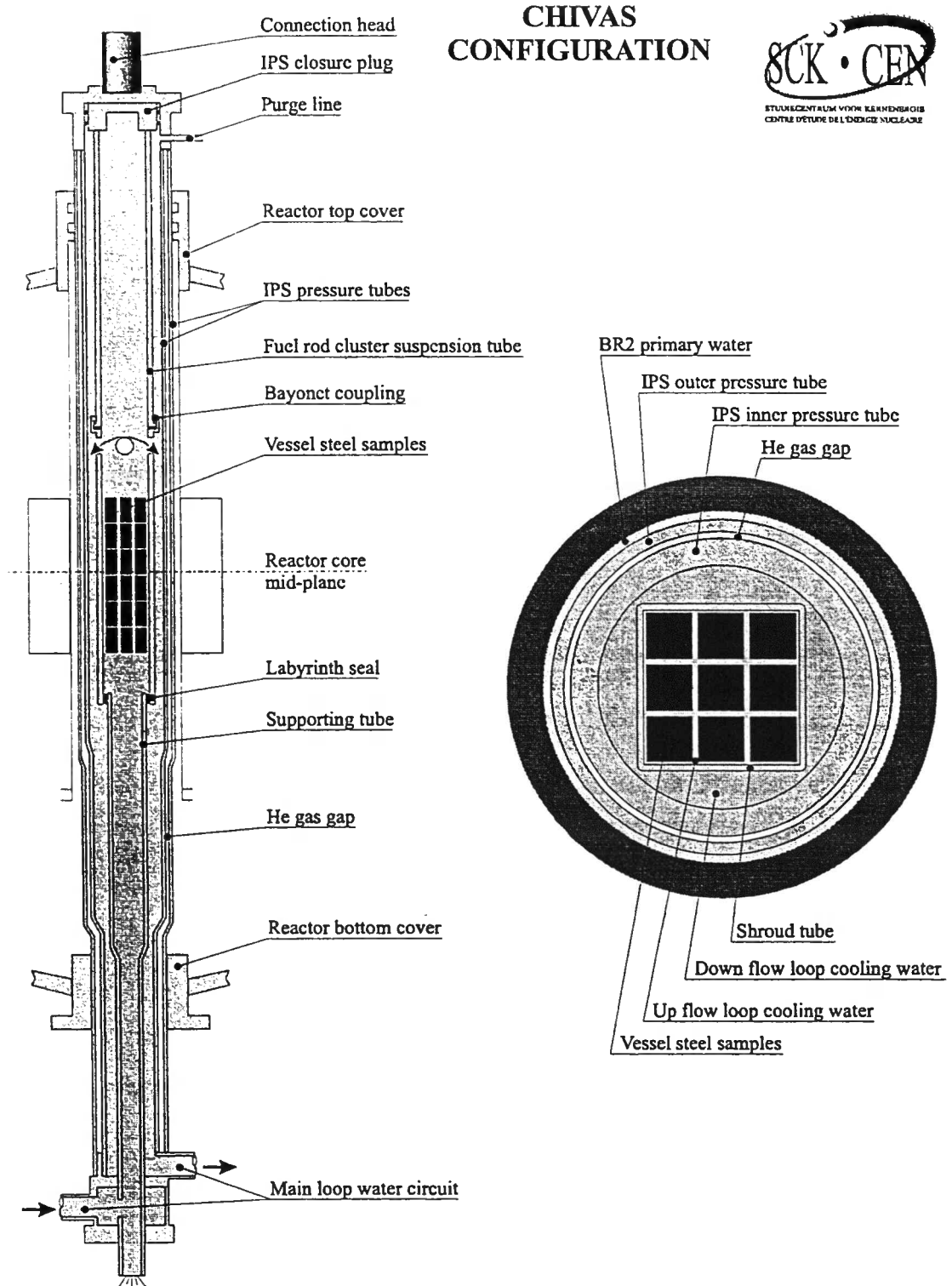
5.1 The CHIVAS-loop

CHIVAS is an irradiation program that is carried out in the CALLISTO-loop of the BR2 Material Test Reactor, located at SCK-CEN. The acronym CALLISTO stands for CApabiLity for Light water Irradiation in Steady state and Transient Operation, and defines a pressurised water loop with three legs that allows to perform accelerated and selective irradiation under PWR-conditions. A schematic representation of one leg is given in Figure 7. Its principal application is the irradiation of PWR-fuels up to high burn-up in well determined environments. The last few years, the loop is more and more used for selective irradiation of reactor pressure vessel steels. The availability of three separate legs allows to irradiated up to 162 Charpy-type specimens at once. The temperature, pressure and water chemistry can be changed as desired.

For the RR on WWER-440 material, two legs of the loop could be used to irradiate the remaining 90 Charpy-V type specimens and the 18 tensile bars — at a temperature of 268°C and a pressure of some 140 bar. A BR2 irradiation up to a fluence ($E > 1$ MeV) of 5×10^{19} n/cm², given an estimated flux of 2.75×10^{13} n/cm²s ($E > 1$ MeV), takes about 21 days. All specimens will be in direct contact with water. After annealing and testing of the irradiated (I)

and irradiated-annealed (IA) condition, a second irradiation to obtain the IAR condition can be performed in one leg of CHIVAS.

Figure 7: The CHIVAS facility

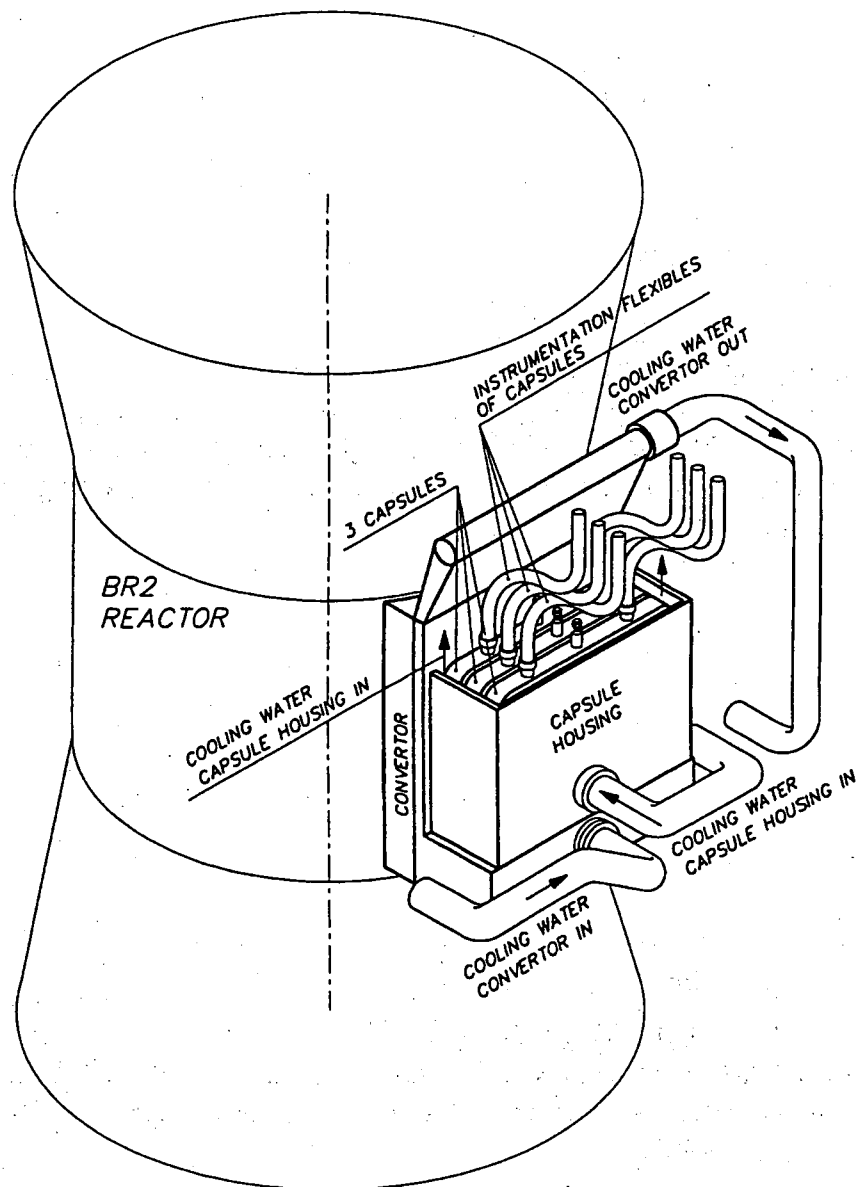


The advantage of using this facility is that it is in use for several years and that it has proven its qualities. The facility is in principle available on a short term basis. The disadvantage is that due to the slightly different radial distance to the reactor core, a fluence variation of a factor two over the specimens exists. This can be avoided by rotating the specimens in order to obtain uniformity. Another barrier to overcome is that some of the irradiated specimens will have to be unloaded, annealed and reloaded into the baskets for the second irradiation.

5.2 The MERLIN-facility [4]

The MERLIN facility (Materials Experimental Research Facility for Large Irradiation volumes of Nuclear steel samples) is a concept that will be constructed in 1999. It will be composed of three irradiation capsules, that contain steel samples, and a neutron converter, which will transform the BR2 thermal neutron outflow into the required fast flux. As shown in Figure 8, the facility will be positioned in the pool, against the BR2 reactor vessel, in order to

Figure 8: Basic layout of the MERLIN facility



provide a large available volume for the samples and enough space for manipulations. It is hence a 'pool-side type facility' (PSF) and can contain up to 900 Charpy-type samples (or a smaller number of larger size samples).

The design is such that it allows rotation and interchange of the capsules to get a uniform fluence distribution, it allows annealing of the capsules without opening a capsule, it allows to irradiate materials at PWR-temperatures.

At flux levels of $1 \text{ E}+13 \text{ n/cm}^2\text{s}$ ($E > 1 \text{ MeV}$), a fluence of $5 \text{ E}+19 \text{ n/cm}^2$ ($E > 1 \text{ MeV}$) is obtained in some 60 days.

The advantage of the MERLIN will be the easy and versatile use of the facility. Moreover, we expect the fluence levels to be more homogeneous (making use of rotation of the cassettes).

The downside, however, is that it is not constructed yet and will only be finalised early 2000. Moreover, it still has to prove its qualities.

6. Conclusions

This report contains the actual status of the SCK-CEN contribution to the IAEA Round Robin Exercise on WWER-440 RPV Weld Material Irradiation, Annealing and Re-Embrittlement. The reference testing of the unirradiated material, except for the mini Cv specimens, was performed and it was shown that good correspondence exists with the results from the certification exercise.

Two possible irradiation facilities that make use of the Belgian MTR reactor BR2, CHIVAS and MERLIN, were presented. Due to uncertainty in time, preference is given to make use of the CHIVAS facility. The irradiations are scheduled for the fall of 1998.

7. Acknowledgements

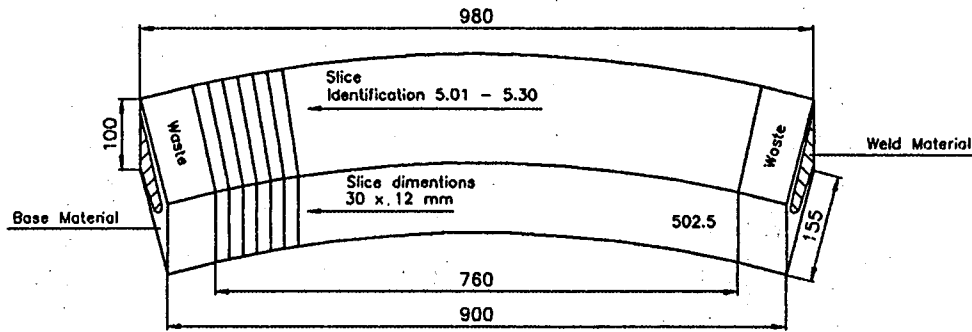
The authors are indebted to the technical staff of the LHMA department, more particular to R. Mertens, A. Pellettieri, R. Vosch for the specimen machining and testing, and to F. Verstrepen for partial analysis.

8. References

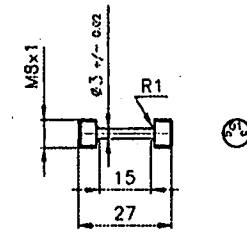
- [1] Investigation Results Report, Weld N° 502 Certification, MOHT-OTJIG RM and EDO 'Gidropress', 1997
- [2] IAEA Round Robin Exercise on WWER-440 RPV Weld Material Irradiation, Annealing and Re-Embrittlement: Terms of Reference, IAEA TC Project RER/9/035 WWER-SC-192, November 1996
- [3] 'Test Method for the Determination of Reference Temperature, T_0 , for Ferritic Steels in the Transition Range'- ASTM, Draft 15, 1997
- [4] Pouleur Y., De Raedt Ch., Malambu E., Minsart G., Vermunt J., in 'Optimisation of a Fuel Converter for the MERLIN Materials Testing Facility,' 2nd International Topical Meeting on Research Reactor Fuel Management, ENS RRFM'98, Bruges, Belgium, March 29-31, 1998.

Mat 15Cr2MoV grade A

ANNEX 1

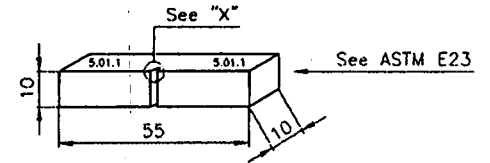


Tensile*

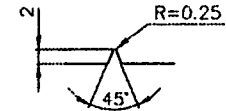


* prepared with 2 centerholes

Charpy-V

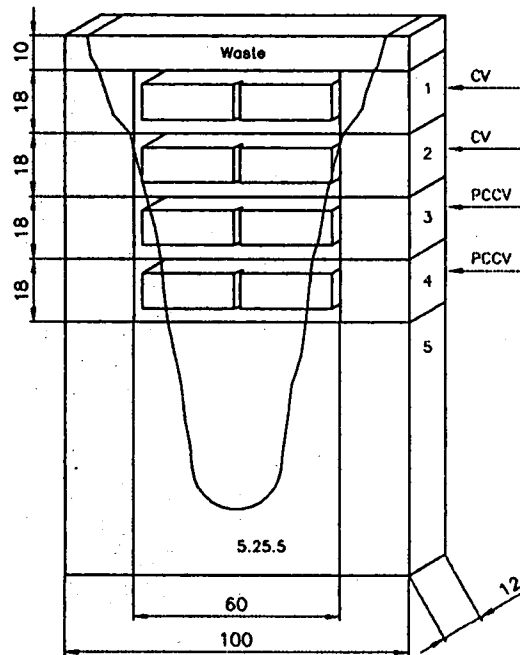
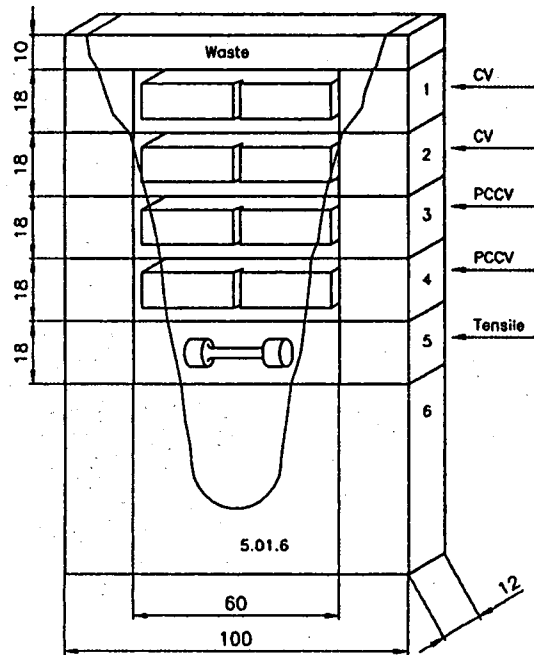


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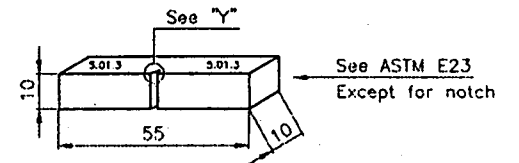


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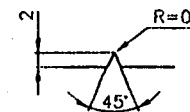
Slice 25 - 30



Pre-cracked Charpy



Detail "Y"



* Identification: 5.01.1 — Layer- or remaining material number
 Blocknumber Slicenumber

CV: 60X
 PCCV: 60X
 Tensile: 24X

Tek. Des. Draw.	Mertens R.				
Verificat.					
Approbat.	Ind.	Dat.	Wijziging - Modification - Up dating	Par.	App.
L.H.M.A.		Wijmaat tol. Tol. libre TOL Free tol.	Afwerking Et. de surface AFW Finishing grade	Schaal Echelle Scale	
File EvW08		Datum-Date-Date 10.12.97		Nr. RMO-87	
RR VVER 440					

