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Abstract:	<p>The IRFUMA-IV irradiation campaign was the follow-up of the IRFUMA -I, IRFUMA-II and IRFUMA-III experiments; the acronym stands for IRradiation of FUSion MAterials. The IRFUMA-IV experiment consisted of a total of seven irradiation cycles in the Belgian Reactor 2 (BR2) of Mol.</p> <p>All the IRFUMA experiments are devoted to materials for Fusion applications, mainly RAFM (Reduced Activation Ferritic-Martensitic) steels such as the European reference structural steel EUROFER97.</p> <p>The IRFUMA-IV irradiation, spanning from the third BR2 cycle of 2004 (03/2004) to the fourth cycle of 2005 (04/2005), started on July 21, 2004 and finished on November 2, 2005.</p>		
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**Final Report - IRFUMA IV:
Irradiation of EUROFER97 joints
and EUROFER97 ODS at 300 °C up
to 2 dpa in the BR2 reactor**

EFDA Technology Workprogramme
Tritium Breeding and Materials - Materials
Development
TW2-TTMS-001b (Del. 14) and TW2-TTMS-006
(Del. 6)

E. Lucon and M. Wéber

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1. Introduction

The IRFUMA-IV irradiation campaign was the follow-up of various IRFUMA experiments (IRFUMA-I [1], IRFUMA-II [2] and IRFUMA-III [3]); the acronym stands for Irradiation of Fusion Materials. The IRFUMA-IV experiment consisted of a total of seven irradiation cycles in the Belgian Reactor 2 (BR2) of SCK•CEN in Mol.

All the IRFUMA experiments have been devoted to materials for Fusion applications, mainly RAFM (Reduced Activation Ferritic-Martensitic) steels, also in ODS (Oxide Dispersion Strengthened) form.

The IRFUMA-IV irradiation, spanning from the third cycle of 2004 (03/2004) to the fourth cycle of 2005 (04/2005), started on July 21, 2004 and finished on November 2, 2005.

The irradiation report for the first part of the experiment (BR2 cycles 03-05/2004) has been issued as a progress report at the end of 2004 [4]; the present document focuses on the second part of the irradiation campaign (BR2 cycles 01-04/2005).

The publication and distribution of this report officially closes the sub-tasks TW2-TTMS-001b Del. 14 and TW2-TTMS-006 Del. 6.

2. Irradiation conditions

The irradiation has been conducted at a temperature between 295 °C and 300 °C in the D180 channel (IPS 2) of the CALLISTO rig of the BR2 reactor in Mol. Throughout the IRFUMA-IV experiments, the channel has been shared with specimens irradiated within other campaign, which have been loaded and unloaded at different stages.

The specimens of IRFUMA-IV have been irradiated during three reactor cycles in 2004 and four reactor cycles in 2005. All the samples have been unloaded at the end of 2005 (November/December) and will be submitted to post-irradiation examinations (PIE) during the course of 2006.

The foreseen neutron dose, estimated for 161 days (7 cycles), is approximately 2 dpa at the midplane. Detailed dosimetry measurements are currently being carried out and will be reported in parallel with the mechanical characterization (PIE); theoretical predictions are however available, allowing to estimate the maximum fluence and dose accumulated by the samples at the midplane level.

In order to compensate for the flux gradient among the needle positions and to homogenize the accumulated dose in the radial direction, the basket has been twice rotated by 180 °C (after cycle 03/2004 and after cycle 02/2005).

The parameters relative to the coolant had been chosen in conformity with the technical specifications of PWR primary water chemistry:

- Temperature : 290–300°C
- Boron (boric acid) ± 400 ppm
- Lithium (lithium hydroxide) $1.8 \text{ ppm} \leq [\text{Li}] \leq 2.2 \text{ ppm}$
- pH: $7.00 \leq \text{pH}_{25^\circ\text{C}} \leq 7.08$ or $7.26 \leq \text{pH}_{300^\circ\text{C}} \leq 7.34$
- Dissolved hydrogen: $25 \text{ ccSTP/kg} \leq [\text{H}_2] \leq 35 \text{ ccSTP/kg}$.

The specimens were in direct contact with the cooling water.

3. Description of the irradiation load

3.1 Materials

The materials irradiated in the IRFUMA-IV experimental were the following:

- Three different types of EUROFER97 joints:
 - (a) Laboratory Joint (first wall mock-up, code TW0-TTBB2.3 – section n°6), obtained by diffusion welding of two plates of EUROFER97 with original thickness 25 mm [5,6].
 - (b) Laboratory Joint (weld-diffusion joint, code TTMS4.6 series 3 – upper joint), corresponding to one of the joints in a piece constituted by four different slices welded by diffusion [5,6].
 - (c) TIG Fusion Welded Joint (code F2002.4), also obtained from EUROFER97 25 mm-thick plates.

The first two joints were produced by CEA Grenoble and the third by CEA Saclay.
- EUROFER97 ODS (alloyed with 0.3% Y_2O_3 using the Oxide Dispersion Strengthening technique), originally produced by Plansee [7] and later improved at FZK by means of optimized thermo-mechanical heat treatments, including hot rolling [8].

The chemical composition of EUROFER97 (as given by the manufacturer) is given in Table 1. The main mechanical properties are reported in Table 2.

Table 1 - Chemical composition of the EUROFER97 steel (% weight).

C	Si	Mn	P	S	Cr	Mo	Ni	V
0.12	0.06	0.42	0.004	0.003	8.87	<0.001	0.0075	0.19
W	Cu	Co	Ti	Al	Nb	B	N	Ta
1.10	0.021	0.005	0.008	0.008	<0.001	<0.0005	0.018	0.14
O	As	Sn	Sb	Zr				
0.001	<0.005	<0.005	<0.005	<0.005				

Table 2 - Mechanical properties of the EUROFER97 steel.

<i>Tensile properties</i>				
Temperature (°C)	R _{p0.2} (MPa)	R _m (MPa)	A (%)	Z (%)
R.T.	548	690	21	74
550	367	418	18	86
<i>Impact tests</i>				
Temperature (°C)	Charpy-V impact energy (J)			
+ 20	203	212	205	
0	174	196	200	
- 20	158	155	167	
Grain size according to ASTM E 112: 10				
Ferrite Content according to AMS 2315: 0%				
Vickers hardness: 221 ± 4 HV				

The original heat treatment of the steel consisted in the following:

- hardening at 980 °C during 110 min, followed by air cooling;
- tempering at 720 °C during 3 hr 40, followed by air cooling.

3.2 Test specimens

Three types of test specimens were loaded in IRFUMA-IV:

- Sub-size impact specimens of the KLST Charpy V-notch type (Cv), having the nominal dimensions specified in Table 3. Part of these samples will be fatigue precracked after irradiation and tested for the characterization of fracture toughness (MPCCv); the rest will be tested under impact conditions using a pendulum machine.

Table 3 – Nominal dimensions of the KLST Charpy V-notch specimens (mm).

Length	Width	Thickness	Notch depth	Notch root radius
27	4	3	1	0.1

- Sub-size tensile specimens with cylindrical cross section, having the nominal dimensions specified in Table 4.

Table 4 – Nominal dimensions of the tensile specimens (mm).

Overall Length	Reduced section Length	Reduced section Diameter	Heads
24	12	2.4	M4

Overall, 27 tensile (9 per material), 40 KLST Charpy (10 per material) and 56 MPCCv (14 per material) specimens have been irradiated in IRFUMA-IV.

The detailed loading scheme is given in Table 5 (needle A – Laboratory Joint, first wall mock-up), Table 6 (needle B – Laboratory Joint, weld diffusion joint), Table 7 (needle C – TIG Fusion Welded Joint) and

Table 8 (needle E – EUROFER97 ODS). Specimens from the three joints are designated with “MJ”, “LJ” and “WJ” respectively; the code for ODS samples begins with “O”. Each material was loaded in a different needle.

Table 5 - IRFUMA-IV loading scheme (needle A, Mock-up joint); ∇ indicates the midplane level.

Level	Type	Code
∇ +58	Tensile	MJT-9
∇ +58	Tensile	MJT-8
∇ +58	Tensile	MJT-7
∇ +32.5	Mini-CV	MJ24
∇ +32.5	Mini-CV	MJ23
∇ +32.5	Mini-CV	MJ22
∇ +5.5	Mini-CV	MJ21
∇ +5.5	Mini-CV	MJ20
∇ +5.5	Mini-CV	MJ19
∇ -21.5	Mini-CV	MJ18
∇ -21.5	Mini-CV	MJ17
∇ -21.5	Mini-CV	MJ16
∇ -48.5	Mini-CV	MJ15
∇ -48.5	Mini-CV	MJ14
∇ -48.5	Mini-CV	MJ13
∇ -74	Tensile	MJT-6
∇ -74	Tensile	MJT-5
∇ -74	Tensile	MJT-4
∇ -88	Fe-Dosimeter	NFE 1
∇ -101.5	Mini-CV	MJ12
∇ -101.5	Mini-CV	MJ11
∇ -101.5	Mini-CV	MJ10
∇ -128.5	Mini-CV	MJ09
∇ -128.5	Mini-CV	MJ08
∇ -128.5	Mini-CV	MJ07
∇ -155.5	Mini-CV	MJ06
∇ -155.5	Mini-CV	MJ05
∇ -155.5	Mini-CV	MJ04
∇ -182.5	Mini-CV	MJ03
∇ -182.5	Mini-CV	MJ02
∇ -182.5	Mini-CV	MJ01
∇ -208	Tensile	MJT-3
∇ -208	Tensile	MJT-2
∇ -208	Tensile	MJT-1

Table 6 - IRFUMA-IV loading scheme (needle B, Laboratory Joint); ∇ indicates the midplane level.

Level	Type	Code
∇ +58	Tensile	LJT-9
∇ +58	Tensile	LJT-8
∇ +58	Tensile	LJT-7

Level	Type	Code
▽ +32.5	Mini-CV	LJ24
▽ +32.5	Mini-CV	LJ23
▽ +32.5	Mini-CV	LJ22
▽ +5.5	Mini-CV	LJ21
▽ +5.5	Mini-CV	LJ20
▽ +5.5	Mini-CV	LJ19
▽ -21.5	Mini-CV	LJ18
▽ -21.5	Mini-CV	LJ17
▽ -21.5	Mini-CV	LJ16
▽ -48.5	Mini-CV	LJ15
▽ -48.5	Mini-CV	LJ14
▽ -48.5	Mini-CV	LJ13
▽ -74	Tensile	LJT-6
▽ -74	Tensile	LJT-5
▽ -74	Tensile	LJT-4
▽ -88	Fe-Dosimeter	NFE 2
▽ -101.5	Mini-CV	LJ12
▽ -101.5	Mini-CV	LJ11
▽ -101.5	Mini-CV	LJ10
▽ -128.5	Mini-CV	LJ09
▽ -128.5	Mini-CV	LJ08
▽ -128.5	Mini-CV	LJ07
▽ -155.5	Mini-CV	LJ06
▽ -155.5	Mini-CV	LJ05
▽ -155.5	Mini-CV	LJ04
▽ -182.5	Mini-CV	LJ03
▽ -182.5	Mini-CV	LJ02
▽ -182.5	Mini-CV	LJ01
▽ -208	Tensile	LJT-3
▽ -208	Tensile	LJT-2
▽ -208	Tensile	LJT-1

Table 7 - IRFUMA-IV loading scheme (needle C, Fusion Welded Joint); ∇ indicates the midplane level.

Level	Type	Code
∇ +58	Tensile	WJT-9
∇ +58	Tensile	WJT-8
∇ +58	Tensile	WJT-7
∇ +32.5	Mini-CV	WJ24
∇ +32.5	Mini-CV	WJ23
∇ +32.5	Mini-CV	WJ22
∇ +5.5	Mini-CV	WJ21
∇ +5.5	Mini-CV	WJ20
∇ +5.5	Mini-CV	WJ19
∇ -21.5	Mini-CV	WJ18
∇ -21.5	Mini-CV	WJ17
∇ -21.5	Mini-CV	WJ16
∇ -48.5	Mini-CV	WJ15
∇ -48.5	Mini-CV	WJ14
∇ -48.5	Mini-CV	WJ13
∇ -74	Tensile	WJT-6
∇ -74	Tensile	WJT-5
∇ -74	Tensile	WJT-4
∇ -88	Fe-Dosimeter	NFE 3
∇ -101.5	Mini-CV	WJ12
∇ -101.5	Mini-CV	WJ11
∇ -101.5	Mini-CV	WJ10
∇ -128.5	Mini-CV	WJ09
∇ -128.5	Mini-CV	WJ08
∇ -128.5	Mini-CV	WJ07
∇ -155.5	Mini-CV	WJ06
∇ -155.5	Mini-CV	WJ05
∇ -155.5	Mini-CV	WJ04
∇ -182.5	Mini-CV	WJ03
∇ -182.5	Mini-CV	WJ02
∇ -182.5	Mini-CV	WJ01
∇ -208	Tensile	WJT-3
∇ -208	Tensile	WJT-2
∇ -208	Tensile	WJT-1

Table 8 - IRFUMA-IV loading scheme (needle E, ODS material); ▽ indicates the midplane level.

Level	Type	Code
▽ +58	Tensile	ODS-T9-9
▽ +58	Tensile	ODS-T8-8
▽ +58	Tensile	ODS-T7-7
▽ +32.5	Mini-CV	OC24
▽ +32.5	Mini-CV	OC23
▽ +32.5	Mini-CV	OC22
▽ +5.5	Mini-CV	OC21
▽ +5.5	Mini-CV	OC20
▽ +5.5	Mini-CV	OC19
▽ -21.5	Mini-CV	OC18
▽ -21.5	Mini-CV	OC17
▽ -21.5	Mini-CV	OC16
▽ -48.5	Mini-CV	OC15
▽ -48.5	Mini-CV	OC14
▽ -48.5	Mini-CV	OC13
▽ -74	Tensile	ODS-T6-6
▽ -74	Tensile	ODS-T5-5
▽ -74	Tensile	ODS-T4-4
▽ -88	Fe-Dosimeter	NFE 4
▽ -101.5	Mini-CV	OC12
▽ -101.5	Mini-CV	OC11
▽ -101.5	Mini-CV	OC10
▽ -128.5	Mini-CV	OC09
▽ -128.5	Mini-CV	OC08
▽ -128.5	Mini-CV	OC07
▽ -155.5	Mini-CV	OC06
▽ -155.5	Mini-CV	OC05
▽ -155.5	Mini-CV	OC04
▽ -182.5	Mini-CV	OC03
▽ -182.5	Mini-CV	OC02
▽ -182.5	Mini-CV	OC01
▽ -208	Tensile	ODS-T3-3
▽ -208	Tensile	ODS-T2-2
▽ -208	Tensile	ODS-T1-1

3.3 Pre-irradiation dimensional measurements

Before loading the samples in the irradiation rig, all specimens were dimensionally checked for compliance with machining tolerances; the results of such dimensional controls can be found in Annex 1 (joints) and Annex 2 (ODS).

4. Loading of the specimens

The specimens have been assembled to form "rods" as described above and then loaded into a CALLISTO shroud tube (CAL17); modifications of the load have been performed in the hot cells of BR2, in order to accommodate specimens from other experiments. All remaining operations in the reactor pool were standard manipulations.

It should be mentioned that the last cycle of IRFUMA-IV (04/2005) was actually split into three parts (A,B,C); part A was devoted to a different experiment, which needed a lower irradiation temperature. IRFUMA-IV samples were reloaded into the reactor for parts B and C.

5. Neutron fluences and doses

Neutron fluxes and fluences experienced by the specimens are determined by the power of BR2 and by the duration of the irradiation cycles.

The maximum fast neutron fluences (on the midplane, $E > 1$ MeV) and the corresponding doses accumulated by the IRFUMA-IV specimens, calculated using the code GEXBR2-TRPT3¹ for the seven irradiation cycles (03/2004 to 04/2005), are as follows [9]:

- Cycle 03/2004 ($E = 1511.8$ MWd): $\Phi = 2.16 \times 10^{20}$ n/cm², corresponding to **0.32 dpa**;
- Cycle 04/2004 ($E = 1119.2$ MWd): $\Phi = 1.63 \times 10^{20}$ n/cm², corresponding to **0.24 dpa**;
- Cycle 05/2004 ($E = 1142.4$ MWd): $\Phi = 1.58 \times 10^{20}$ n/cm², corresponding to **0.24 dpa**;
- Cycle 01/2005 ($E = 1237.0$ MWd): $\Phi = 1.78 \times 10^{20}$ n/cm², corresponding to **0.27 dpa**;
- Cycle 02/2005 ($E = 1463.9$ MWd): $\Phi = 2.13 \times 10^{20}$ n/cm², corresponding to **0.32 dpa**;
- Cycle 03/2005 ($E = 1442.0$ MWd): $\Phi = 1.93 \times 10^{20}$ n/cm², corresponding to **0.29 dpa**;
- Cycle 04B/2005 ($E = 471.1$ MWd): $\Phi = 0.66 \times 10^{20}$ n/cm², corresponding to **0.10 dpa**;
- Cycle 04C/2005 ($E = 674.3$ MWd): $\Phi = 0.96 \times 10^{20}$ n/cm², corresponding to **0.14 dpa**.

The total accumulated fast neutron fluence ($E > 1$ MeV) during the IRFUMA-IV experiment, based on these calculations, is therefore 1.28×10^{21} n/cm², corresponding to a total dose of **1.92 dpa**. Such values are rigorously valid only for samples located at the position of highest flux (midplane); axial distribution functions [10] will be used in order to evaluate the fluence associated to each individual specimen.

Detailed dosimetry measurements are currently being performed and will be provided together with the results of the PIE.

6. Post-Irradiation Examinations (PIE)

After the irradiation was concluded, the shroud tube was transported to the BR2 hot cell. The assembled rods were dismantled and the specimens retrieved.

Post-Irradiation Examination (PIE) of the mechanical specimens irradiated in IRFUMA-IV will take place in the course of 2006; the planned testing activity is as follows:

- tensile tests at different temperatures;
- instrumented impact tests over a temperature range covering the whole transition curve;
- quasi-static fracture toughness tests in the transition regime.

Detailed results of the PIE will be available in the second half of 2006.

¹GEXBR2-TRPT3 is a validated SCK•CEN home-made neutron code, based on neutron transport theory.

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ANNEX 1

Dimensional measurements on mechanical specimens of EUROFER97 joints

Laboratory Joint					
ID	L	Lo	D1	D2	D3
LJ T-1	24.101	12.049	2.406	2.374	2.412
LJ T-2	24.066	11.740	2.399	2.380	2.408
LJ T-3	24.089	12.065	2.408	2.401	2.416
LJ T-4	24.043	12.021	2.405	2.376	2.413
LJ T-5	24.083	12.019	2.400	2.384	2.401
LJ T-6	24.055	11.973	2.400	2.361	2.407
LJ T-7	24.090	12.038	2.409	2.391	2.415
LJ T-8	24.059	11.962	2.398	2.370	2.406
LJ T-9	24.050	11.941	2.394	2.387	2.404
LJ T-10	24.114	12.072	2.407	2.395	2.405
Lab. Mock-up Joint					
MJ T-1	24.049	12.086	2.405	2.386	2.418
MJ T-2	24.075	11.931	2.401	2.386	2.408
MJ T-3	24.082	11.881	2.409	2.392	2.409
MJ T-4	24.056	12.038	2.397	2.387	2.398
MJ T-5	24.050	12.082	2.402	2.392	2.411
MJ T-6	24.122	12.050	2.403	2.383	2.406
MJ T-7	24.121	12.063	2.407	2.385	2.411
MJ T-8	24.029	12.098	2.408	2.396	2.412
M T-09	24.110	12.112	2.403	2.399	2.410
MJ T-10	24.058	12.083	2.396	2.385	2.404
Fusion Welded Joint					
WJ T-01	24.052	12.034	2.399	2.374	2.396
WJ T-2	24.075	11.961	2.403	2.387	2.412
WJ T-3	24.050	12.034	2.389	2.375	2.397
WJ T-4	24.058	12.035	2.404	2.384	2.410
WJ T-5	24.049	11.928	2.405	2.387	2.417
WJ T-6	24.041	12.081	2.393	2.385	2.407
WJ T-7	24.031	12.019	2.407	2.390	2.408
WJ T-8	24.035	12.114	2.408	2.396	2.411
WJ T-9	24.092	12.024	2.405	2.380	2.401
WJ T-10	24.030	12.130	2.401	2.390	2.407

Legend – L = overall specimen length; L_o = length of the reduced section; D1, D2, D3 = multiple measurements of diameter of the reduced section, taken at different axial positions.

Laboratory Joint			Lab. Mock-up Joint			Fusion Welded Joint		
ID	W	B	ID	W	B	ID	W	B
LJ01	4.000	2.998	MJ01	4.000	2.998	WJ01	4.002	3.005
LJ02	3.998	3.003	MJ02	4.000	3.004	WJ02	3.999	3.006
LJ03	4.003	3.002	MJ03	4.000	3.000	WJ03	4.000	3.000
LJ04	3.998	2.998	MJ04	3.994	3.000	WJ04	4.000	3.008
LJ05	3.993	2.995	MJ05	4.002	3.010	WJ05	3.998	3.003
LJ06	3.994	2.980	MJ06	4.000	3.002	WJ06	4.002	3.002
LJ07	3.991	2.995	MJ07	4.003	2.999	WJ07	4.000	2.994
LJ08	3.999	3.002	MJ08	4.000	3.000	WJ08	4.000	3.003
LJ09	3.993	3.001	MJ09	4.000	3.001	WJ09	4.008	2.998
LJ10	3.990	2.991	MJ10	3.998	3.000	WJ10	4.005	2.999
LJ11	3.998	2.998	MJ11	4.002	3.002	WJ11	3.998	3.000
LJ12	3.993	3.000	MJ12	4.002	3.002	WJ12	3.996	3.005
LJ13	4.002	3.000	MJ13	3.995	2.995	WJ13	3.999	3.004
LJ14	4.000	2.995	MJ14	4.008	3.000	WJ14	3.998	3.001
LJ15	3.995	3.008	MJ15	4.000	2.998	WJ15	3.995	3.010
LJ16	3.998	2.999	MJ16	4.002	3.005	WJ16	3.998	3.000
LJ17	3.999	2.998	MJ17	4.000	2.994	WJ17	3.999	3.008
LJ18	3.998	3.001	MJ18	4.000	3.001	WJ18	3.998	3.002
LJ19	3.998	3.008	MJ19	4.000	3.000	WJ19	4.002	3.003
LJ20	4.002	3.003	MJ20	3.999	2.997	WJ20	4.000	3.003
LJ21	4.000	3.003	MJ21	4.000	2.999	WJ21	3.999	3.002
LJ22	4.000	3.010	MJ22	3.995	3.001	WJ22	3.997	3.005
LJ23	3.993	3.008	MJ23	4.002	2.999	WJ23	4.002	3.005
LJ24	3.993	2.999	MJ24	4.000	3.001	WJ24	4.000	3.002

Legend – W = specimen width; B = specimen thickness.

ANNEX 2

Dimensional measurements on mechanical specimens of EUROFER97 ODS

ID	L	L _o	D1	D2	D3
ODS-T1	24.018	12.02	2.395	2.399	2.408
ODS-T2	23.94	11.945	2.413	2.394	2.406
ODS-T3	23.968	11.941	2.41	2.391	2.402
ODS-T4	23.949	11.982	2.41	2.384	2.412
ODS-T5	23.965	11.898	2.41	2.391	2.41
ODS-T6	23.997	11.98	2.424	2.404	2.419
ODS-T7	23.975	11.968	2.399	2.393	2.427
ODS-T8	24.029	12.019	2.415	2.401	2.421
ODS-T9	23.98	12.079	2.425	2.414	2.427

Legend – L = overall specimen length; L_o = length of the reduced section; D1, D2, D3 = multiple measurements of diameter of the reduced section, taken at different axial positions.

ID	W	B
0C01	4.000	2.998
0C02	4.003	3.007
0C03	4.004	2.999
0C04	4.003	3.001
0C05	3.999	2.999
0C06	4.007	3.002
0C07	4.000	3.003
0C08	4.001	3.003
0C09	4.007	2.998
0C10	4.003	2.997
0C11	4.005	2.999
0C12	4.005	2.996
0C13	4.002	3.005
0C14	4.003	2.999
0C15	4.000	3.000
0C16	4.001	2.996
0C17	4.003	3.002
0C18	3.998	3.003
0C19	4.003	3.000
0C20	4.001	3.001
0C21	3.999	3.003
0C22	4.001	3.001
0C23	4.007	3.004
0C24	4.009	3.001

Legend – W = specimen width; B = specimen thickness.