IRRADIATION OF FUSION MATERIALS IN THE BR2 REACTOR: THE FRISCO-F EXPERIMENT—E. Lucon (SCK•CEN) and M. A. Sokolov (Oak Ridge National Laboratory)

SUMMARY

Tensile and miniature Compact Tension specimens of eight high chromium steels of fusion relevance have been irradiated in the BR2 reactor in the framework of a collaborative project between Oak Ridge National Laboratory (ORNL) and SCK•CEN.

All samples have been irradiated at a nominal temperature of 300°C in the in-pile section 2 (IPS-2) for five cycles (02/2005 to 01/2006) up to an average fast neutron fluence of 8.02 × 10^{20} n/cm² or 1.20 dpa. The rig was rotated three times by 180° in order to reduce the radial and azimuthal neutron flux gradients.

INTRODUCTION

Material research represents a crucial issue for the assessment of fusion as a future source of energy. Structural materials, in particular, need to show a superior mechanical and chemical behavior to guarantee the safe operation of the reactor during its whole lifetime, while retaining low activation characteristics to minimize the environmental impact of the produced waste. For many aspects of the design, it is indeed material technology that will dictate the most viable concept for the commercial power plant.

In this context, specific efforts have been focused for the last twenty years, in Europe, Japan and the US, on developing suitable Reduced Activation Ferritic Martensitic (RAFM) steels as prominent structural materials. EUROFER97 has recently emerged in Europe as the reference material for the DEMO design, whereas activities in the US and Japan have been concentrating for several years on F82H. At the same time, research is in progress at Oak Ridge National Laboratory (ORNL) in the US using model alloys with chromium contents ranging from 3% to 9%.

While the final assessment of these materials under the actual reactor conditions will only occur in ITER (blanket module) and IFMIF, it is presently of primary importance to develop the scientific understanding of the mechanisms which control the physical, mechanical, and chemical behavior of such materials under radiation.

A representative selection of high chromium steels of fusion relevance has been irradiated in the Belgian Reactor 2 (BR2) located in Mol (Belgium), in the framework of a collaborative project between ORNL and SCK•CEN. The experiment, denominated FRISCO-F (Fusion and Reactor Materials Irradiation SCK•CEN/ORNL–Fusion Materials), included tensile and fracture toughness specimens of the following materials: EUROFER97, F82H, CLAM and four ORNL developmental alloys (9Cr, 5Cr, 3Cr, 3Cr+Ta); in addition, we also irradiated samples of the well-known ferritic/martensitic steel T91, which is not relevant for fusion (its chemical composition cannot be considered reduced-activation) but it's regarded as one of the reference materials for applications such as accelerated-driven systems (ADS) and future high temperature nuclear energy systems (Gen IV).

The irradiation campaign took place during five cycles of BR2 in the period June 2005/March 2006; the nominal irradiation temperature was 300°C.

On the basis of analytical calculations which have been verified by actual dosimetry measurements, the average fast neutron fluence (E > 1 MeV) for all irradiated specimens is 8.02 × 10^{20} n/cm², corresponding to 1.20 dpa.

<u>Irradiation Conditions</u>

The FRISCO-F irradiation has been conducted between April 15, 2005, and March 21, 2006, at a water temperature between 295 and 300°C in the D180 channel (IPS-2) of the CALLISTO rig in the BR2 reactor. In order to achieve uniform irradiation conditions (fluence and flux) in the radial direction, the rig has been rotated by 180° between the first and the second, the second and the third and the third and the fourth cycles.

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

- Temperature 295–300°C
- Boron (boric acid) ± 550 ppm
- Lithium (lithium hydroxide) 1.8 ppm ≤ [Li] ≤ 2.2 ppm
- pH $7.00 \leq pH_{25°C} \leq 7.08$ or $7.26 \leq pH_{300°C} \leq 7.34$
- Dissolved hydrogen 25 ccSTP/kg ≤ $[H_2]$ ≤ 35 ccSTP/kg

The specimens were in direct contact with the water.

Materials and Specimens Irradiated

The following materials have been irradiated in the FRISCO-F experiment:

- three reduced-activation ferritic/martensitic (RAFM) steels : EUROFER97 (9%Cr), F82H (8%Cr), CLAM (Chinese Low Activation Material, 9%Cr);
- four high Cr developmental alloys produced by ORNL: 9Cr-2WVTa, 5Cr-2W2.5V, 3Cr-3WV, 3Cr-3WVTa;
- one ferritic/martensitic steel denominated T91 (9Cr1MoVNb).

The chemical composition of the steels irradiated is given in Table 1 (weight %). Tensile properties at RT for the three RAFM steels and for T91 are shown in Table 2.

Table 1. Chemical composition of the steels irradiated in FRISCO-F

Material	C	Mn	Si	S	P	Cr	V	W	Ta	Al	B	N
F82H	0.090	0.16	0.11	0.002	0.002	7.71	0.16	1.95	0.02	0.003	0.0002	0.006
EUROFER97	0.12	0.42	0.06	0.003	0.004	8.87	0.19	1.10	0.14	0.008	<0.0005	0.018
CLAM	0.10	0.45	0.25	-	-	9.0	0.20	1.5	0.07	-	-	0.02
ORNL 9Cr-2WVTa	0.11	0.44	0.21	0.008	0.015	8.90	0.23	2.01	0.06	0.017	<0.001	0.0215
ORNL 5Cr-2W2.5V	0.12	0.49	0.23	0.009	0.015	5.04	0.24	2.01	-	0.010	<0.001	0.0171
ORNL 3Cr-3WV	0.10	0.39	0.16	0.004	0.010	3.04	0.21	3.05	<0.01	0.003	0.001	0.004
ORNL 3Cr-3WVTa	0.10	0.41	0.16	0.005	0.011	3.02	0.21	3.07	0.09	0.003	0.001	0.003
T91	0.099	0.43	0.32	0.004	0.020	8.8	0.24	<0.01	-	<0.01	<0.0005	0.03

Table 2. RT tensile properties for four of the steels irradiated in FRISCO-F

Material	Yield strength	Tensile strength	Total elongation	Reduction of area
	(MPa)	(MPa)	(%)	(%)
F82H	562	664	18	81
EUROFER97	557	670	20	80
CLAM	1086	1428	15	70
T91	544	684	22	73

For each of the three RAFM steels and for the ORNL 9Cr-2WVTa material, the following samples have been irradiated:

- 3 miniature tensile specimens with cylindrical cross section and the following nominal dimensions:
  - overall length L = 24 mm;
  - length of reduced section A = 12 mm;
  - diameter of reduced section D = 2.4 mm;
  - heads M4;

- 12 miniature Compact Tension MC(T) specimens with the following nominal dimensions:
  - height H =10 mm;
  - width W = 10 mm;
  - thickness B = 4.2 mm.

For T91, one tensile and 12 MC(T) samples have been irradiated. In the case of the remaining ORNL alloys, only tensile specimens (three for 5Cr-2W2.5V, four for 3Cr-3WV and 3Cr-3WVTa) were included in the rig.

Technical drawings of the samples are provided in Annex 1 (tensile) and Annex 2 (miniature C(T)).

Activation Dosimeters

In total, 4 activation dosimeters made of pure iron (discs of 9 mm diameter and 0.5 mm thickness) have been inserted in the rig, located between the tensile specimen boxes and the MC(T) stacks in both needles.

Each dosimeter had slightly different geometrical dimensions in order to be distinguished from the others.

Pre-irradiation Operations

In the case of F82H and the four developmental alloys, samples were prepared by ORNL according to SCK•CEN specifications and shipped to Mol before irradiation. The remaining specimens were machined at SCK•CEN.

The tensile samples have been assembled in boxes, three by three at the same axial position; the MC(T)'s have been stacked in groups of 15.

Operations During and Post-irradiation

The irradiation of the FRISCO-F specimens has proceeded as planned, without any significant problem.

After the conclusion of cycle 01/2006 and a short cool-down period, the rig has been transported to the hot cells of BR2 where all the specimens have been recovered.

The irradiated samples have been transferred to the storage facility of LHMA (Laboratory for High and Medium Activities of SCK•CEN), where they are currently stored before being tested and/or shipped back to ORNL.

Evaluation of Fast Neutron Fluences and Doses

The evaluation of fast neutron fluences and doses experienced by the specimens is based upon the power of BR2, the axial position of the samples in the rig and the duration of the irradiation cycles.

The maximum fast neutron fluences (E > 1 MeV) and doses relative to the center channel of the CALLISTO rig (channel E) and the BR2 midplane (axial position Z = -72 mm) have been calculated using the code GEXBR2-TRPT3, which has been developed and validated by SCK•CEN and is based on neutron transport theory.

The calculated values for the five BR2 cycles of the FRISCO-F irradiation are: □ = 8.80×10^{20} n/cm² (E > 1 MeV) or 1.32 dpa

These values are rigorously valid only for samples located at the position of highest flux (midplane) and in the center channel of the rig (channel E); cosinusoidal axial distribution functions [1] are available and have been used to evaluate the fluence and dose associated to each individual specimen.

Fluences and fluxes measured by the 4 activation dosimeters loaded in the rig have been used to adjust the values calculated using the neutron transport code. The maximum deviation between calculated and measured fluence at the dosimeter position was 2%. Details of the dosimetry measurements are given in the Technical Note presented in Annex 3.

The values of fast fluence and dpa associated to each irradiated specimen are presented in the next section.

<u>Loading Plan/values of Fluence and dpa</u>

The two needles (G and I) containing the specimens irradiated in FRISCO-F have been loaded into the basket ST 17 of IPS 2.

All loading and unloading operations have been performed in the BR2 hot cells; other operations in the reactor pool were standard manipulations.

The loading plan for the specimens irradiated in channels G and I is given in Table 3 and Table 4 respectively,* with the values of fast fluence and dpa calculated for each individual sample.

The mean values of fast fluence and dpa associated to the entire specimen set and to each material and specimen type are presented in Table 5.

Acknowledgements

The collaboration of the personnel of the BR2 and TCH departments of SCK•CEN for all pre-, during- and post-irradiation operations is acknowledged. Thanks also to Roger Mertens for the preparation of the specimens extracted from the materials provided by SCK•CEN.

Finally, personal thanks to Marcel Wéber who followed the preparation and execution of the irradiation campaign.

Reference

[1] R. Chaouadi, A Simple Equation to Estimate the Neutron Flux Distribution in the Callisto Loop of the BR2 Reactor, SCK•CEN Open Report BLG-866 (2001).

*The axial level in Tables 3 and 4 refers to the midplane position of the specimens.

Table 3. Loading plan with fluence and dpa values for needle G

Axial level (mm)	Material	Specimen		Fast fluence (10^{20} n/cm², E > 1 MeV)	Dose (dpa)
		Type	Id.		
	ORNL 3Cr	Tensile	K1	7.56	1.13
-209	ORNL 3Cr	Tensile	K2	7.56	1.13
	ORNL 3Cr	Tensile	K3	7.56	1.13
	F82H	Tensile	F1	7.86	1.18
-185	EUROFER97	Tensile	E97-140	7.86	1.18
	CLAM	Tensile	CL28	7.86	1.18
-172.5	Fe	Dosimeter	NFE1	8.00	1.20
-147.5	T91	Mini-CT	T91-93	8.23	1.23
-143.3	T91	Mini-CT	T91-94	8.26	1.24
-139.1	T91	Mini-CT	T91-95	8.29	1.24
-134.9	F82H	Mini-CT	F4	8.32	1.25
-130.7	EUROFER97	Mini-CT	E97-128	8.35	1.25
-126.5	CLAM	Mini-CT	CL13	8.37	1.26
-122.3	ORNL 9Cr	Mini-CT	I4	8.40	1.26
-118.1	F82H	Mini-CT	F5	8.42	1.26
-113.9	EUROFER97	Mini-CT	E97-129	8.44	1.27
-109.7	CLAM	Mini-CT	CL14	8.46	1.27
-105.5	ORNL 9Cr	Mini-CT	I5	8.47	1.27
-101.3	F82H	Mini-CT	F6	8.49	1.27
-97.1	EUROFER97	Mini-CT	E97-130	8.50	1.27
-92.9	CLAM	Mini-CT	CL15	8.51	1.28
-88.7	ORNL 9Cr	Mini-CT	I6	8.52	1.28
-61.5	F82H	Mini-CT	F7	8.53	1.28
-57.3	EUROFER97	Mini-CT	E97-131	8.52	1.28
-53.1	CLAM	Mini-CT	CL16	8.51	1.28
-48.9	ORNL 9Cr	Mini-CT	I7	8.50	1.27
-44.7	F82H	Mini-CT	F8	8.49	1.27
-40.5	EUROFER97	Mini-CT	E97-132	8.48	1.27
-36.3	CLAM	Mini-CT	CL17	8.46	1.27
-32.1	ORNL 9Cr	Mini-CT	I8	8.45	1.27
-27.9	F82H	Mini-CT	F9	8.43	1.26
-23.7	EUROFER97	Mini-CT	E97-133	8.41	1.26
-19.5	CLAM	Mini-CT	CL18	8.39	1.26
-15.3	ORNL 9Cr	Mini-CT	I9	8.36	1.25
-11.1	T91	Mini-CT	T91-96	8.33	1.25
-6.9	T91	Mini-CT	T91-97	8.31	1.25
-2.7	T91	Mini-CT	T91-98	8.28	1.24
21	Fe	Dosimeter	NFE2	8.08	1.21
	F82H	Tensile	F2	7.95	1.19
33.5	EUROFER97	Tensile	E97-141	7.95	1.19
	CLAM	Tensile	CL29	7.95	1.19
	ORNL 3Cr+Ta	Tensile	L1	7.90	1.18
37.5	ORNL 3Cr+Ta	Tensile	L2	7.90	1.18
	ORNL 3Cr+Ta	Tensile	L3	7.90	1.18

Table 4. Loading plan with fluence and dpa values for needle I

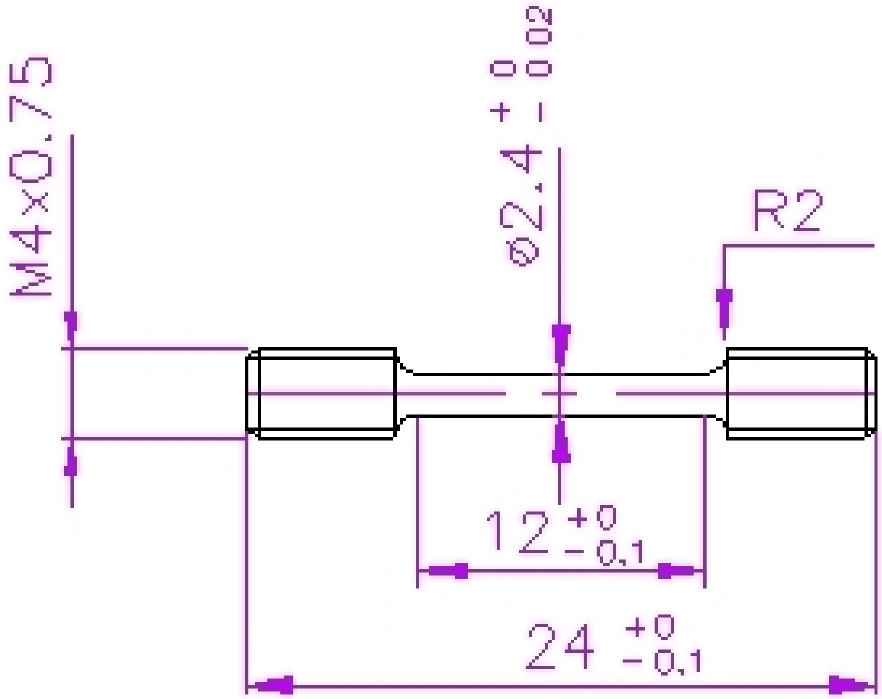
Axial level (mm)	Material	Specimen		Fast fluence (10^{20} n/cm², E > 1 MeV)	Dose (dpa)
		Type	Id.		
	ORNL 5Cr	Tensile	J1	7.59	1.14
-209	ORNL 5Cr	Tensile	J2	7.59	1.14
	ORNL 5Cr	Tensile	J3	7.59	1.14
	F82H	Tensile	F3	7.89	1.18
-185	EUROFER97	Tensile	E97-142	7.89	1.18
	CLAM	Tensile	CL30	7.89	1.18
-172.5	Fe	Dosimeter	NFE3	8.03	1.20
-147.5	T91	Mini-CT	T91-99	8.26	1.24
-143.3	T91	Mini-CT	T91-100	8.29	1.24
-139.1	T91	Mini-CT	T91-101	8.32	1.25
-134.9	F82H	Mini-CT	F10	8.35	1.25
-130.7	EUROFER97	Mini-CT	E97-134	8.38	1.26
-126.5	CLAM	Mini-CT	CL19	8.40	1.26
-122.3	ORNL 9Cr	Mini-CT	I10	8.42	1.26
-118.1	F82H	Mini-CT	F11	8.45	1.27
-113.9	EUROFER97	Mini-CT	E97-135	8.47	1.27
-109.7	CLAM	Mini-CT	CL20	8.48	1.27
-105.5	ORNL 9Cr	Mini-CT	I11	8.50	1.27
-101.3	F82H	Mini-CT	F12	8.51	1.28
-97.1	EUROFER97	Mini-CT	E97-136	8.53	1.28
-92.9	CLAM	Mini-CT	CL21	8.54	1.28
-88.7	ORNL 9Cr	Mini-CT	I12	8.55	1.28
-61.5	F82H	Mini-CT	F13	8.55	1.28
-57.3	EUROFER97	Mini-CT	E97-137	8.55	1.28
-53.1	CLAM	Mini-CT	CL22	8.54	1.28
-48.9	ORNL 9Cr	Mini-CT	I13	8.53	1.28
-44.7	F82H	Mini-CT	F14	8.52	1.28
-40.5	EUROFER97	Mini-CT	E97-138	8.51	1.28
-36.3	CLAM	Mini-CT	CL237	8.49	1.27
-32.1	ORNL 9Cr	Mini-CT	I14	8.48	1.27
-27.9	F82H	Mini-CT	F15	8.46	1.27
-23.7	EUROFER97	Mini-CT	E97-139	8.44	1.26
-19.5	CLAM	Mini-CT	CL24	8.41	1.26
-15.3	ORNL 9Cr	Mini-CT	I15	8.39	1.26
-11.1	T91	Mini-CT	T91-102	8.36	1.25
-6.9	T91	Mini-CT	T91-103	8.33	1.25
-2.7	T91	Mini-CT	T91-104	8.30	1.24
21	Fe	Dosimeter	NFE4	8.10	1.21
	ORNL 9Cr	Tensile	I1	7.97	1.20
33.5	ORNL 9Cr	Tensile	I2	7.97	1.20
	ORNL 9Cr	Tensile	I3	7.97	1.20
	ORNL 3Cr	Tensile	K4	7.93	1.19
37.5	ORNL 3Cr+Ta	Tensile	L4	7.93	1.19
	T91	Tensile	T91-105	7.93	1.19

Table 5. Mean values of fast fluence and dpa calculated for the materials irradiated in the FRISCO-F experiment

Material	Specimen type	Fast fluence (10^{20} n/cm², E > 1MeV)	Dose (dpa)
ORNL 3Cr	Tensile	7.65	1.15
ORNL 3Cr+Ta	Tensile	7.91	1.19
ORNL 5Cr	Tensile	7.59	1.14
ORNL 9Cr	Tensile	7.97	1.20
	MC(T)	8.46	1.27
	All	8.37	1.25
CLAM	Tensile	7.90	1.18
	MC(T)	8.46	1.27
	All	8.35	1.25
EUROFER97	Tensile	7.90	1.18
	MC(T)	8.46	1.27
	All	8.35	1.25
T91	Tensile	7.93	1.19
	MC(T)	8.30	1.24
	All	8.27	1.24
F82H	Tensile	7.90	1.18
	MC(T)	8.46	1.27
	All	8.35	1.25
All	Tensile	7.83	1.17
	MC(T)	8.43	1.26
	All	8.26	1.24

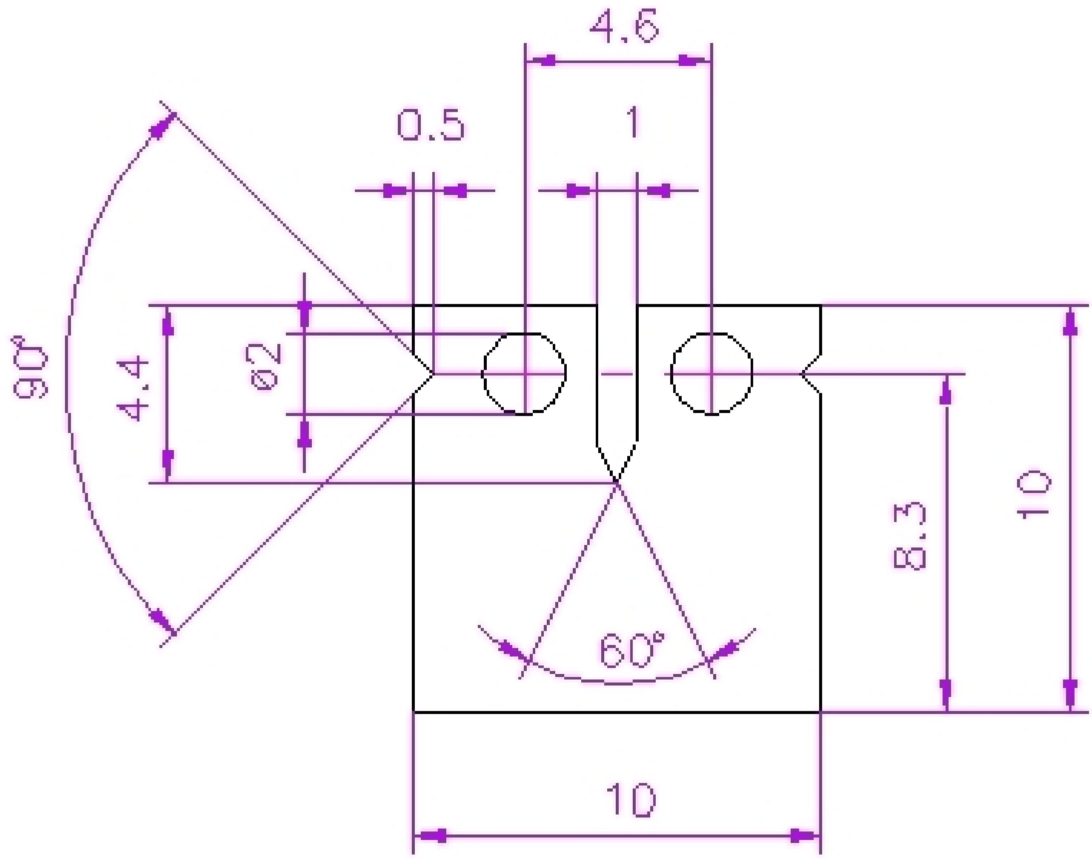
ANNEX 1

Technical drawing of the sub-size tensile specimen


M4×0.75
ø2.4 +0 -0.02
R2
12 +0 -0.1
24 +0 -0.1

ANNEX 2

Technical drawing of the miniature C(T) specimen



Thickness 4.2

ANNEX 3

Dosimetry measurements

**(Technical Note
RF&M/Vwi/vwi RF&M/Vwi/vwi
32.D049011-205/06 09)**

Introduction

FRISCO stands for Fusion and Reactor material Irradiation SCK•CEN–ORNL. The specimens from the FRISCO-F experiment are made of different fusion materials.

The specimens were inserted in capsules which were assembled to a needle. These needles were loaded in the CALLISTO loop IPS2. Two needles were loaded with Fe dosimeters. The Fe dosimeters were inserted at the end of the specimens set in each capsule (see annex Fig. 1). Both dosimeters are small discs made of pure iron. Five irradiation cycles are foreseen for needles G and I in IPS2. This report describes how the fission flux and fluence is determined from the activity measurements of the dosimeters that have been irradiated during cycles 02A & 03A & 04B & 04C & 05A/2005 and 01A/2006.

Irradiation

The irradiation was performed in the BR2 reactor in channel D180 (IPS2) of the CALLISTO loop at a nominal temperature of 295°C. The basket containing the FRISCO-F samples was rotated 180° between cycles 02A and 03A, 03A and 04B, 04C and 05A/2005 in order to get the most uniform fluence at the different specimen positions (see annex Fig. 2).

Fe activation dosimeters are used for the determination of the fast neutron flux and fluence. The dosimeters are discs with a diameter of 9 mm and a thickness of 0.5 mm.

Table 1 summarizes the identification, dosimeter name, needle number, axial position in the reactor, irradiation cycle, and rig (IPS).

Table 1. Overview of the dosimeters

Identification name	Dosimeter number	Needle	Reactor position	Irradiation cycle	Irradiation rig
FRIS-1	NFE1	G	-170	02A&03A&04BC&05A/2005 and 01A/2006	IPS 2
FRIS-2	NFE2	G	+20	02A&03A&04BC&05A/2005 and 01A/2006	IPS 2
FRIS-3	NFE3	I	-170	02A&03A&04BC&05A/2005 and 01A/2006	IPS 2
FRIS-4	NFE4	I	+20	02A&03A&04BC&05A/2005 and 01A/2006	IPS 2

The irradiation history is given in Table 2.

Table 2. Irradiation history

BR2 cycle	Start date of the irradiation	End date of the irradiation	Nominal Power (MW)	Duration (days)
02A/2005	2005-07-29 07:05	2005-08-23 21:15	57	25.48
03A/2005	2005-07-29 07:05	2005-08-23 21:15	57	25.48
04B/2005	2005-10-12 07:45	2005-10-20 10:11	60	8.02
04C/2005	2005-10-22 11:10	2005-11-02 18:42	60	11.31
05A/2005	2005-11-29 07:45	2005-12-19 00:48	60	19.39
01A/2006	2006-02-28 07:52	2006-03-21 23:19	57	19.73

A plot of the irradiation history is given in the annex (Fig. 3).

Dismantling of the Dosimeters

The capsule with the dosimeters was unloaded in the hot cells of BR2. All the dosimeters were recovered.

Results

The equivalent fission flux was calculated from the ^{54}Mn activity formed by the following reaction:

$$^{54}Fe(n,p)^{54}Mn$$

The neutron flux was calculated using the ^{235}U fission spectrum averaged $^{54}Fe(n,p)^{54}Mn$ cross-section $<\sigma>= 81.7$ mb adopted from [1] (the $^{54}Fe(n,p)$ reaction having an effective threshold energy of 2.80 MeV [1]).

The determined equivalent fission fluxes and fluences are given in Table 3 and Fig. 4. All neutron fluxes are calculated at reference power 57.0 MW.

Table 3. Dosimetry results obtained from the ^{54}Mn activity measurements

Identification number	Needle	Position (mm)	Spec. activity (Bq/g)	Reaction rate (s^{-1})	Eq. fis. flux (n/cm²/s)	Eq. fis. fluence (n/cm²)
NFE1	G	-170	7.18E+08	6.66E-12	8.15E+13	7.82E+20
NFE2		20	7.15E+08	6.62E-12	8.11E+13	7.79E+20
NFE3	I	-170	7.19E+08	6.66E-12	8.16E+13	7.83E+20
NFE4		20	7.19E+08	6.66E-12	8.15E+13	7.83E+20

The uncertainty (1σ) on the specific activity < 3%.

Due to the rotation of the basket between the cycles an error is introduced in the neutron flux (fluence) determination for all needles. In view of the small flux level differences expected between axial positions -170 mm and 20 mm this error is estimated to be ≤ 5%. This is confirmed by the converging results for the neutron fluence (see Table 3).

Table 4 summarizes how the equivalent fission flux (EFF) can be converted to flux > 0.1 MeV, flux > 0.5 MeV, and flux > 1 MeV[2, 3].

Table 4. Fast neutron flux conversion factors

Irradiation rig	(flux > 0.1 MeV)/EFF	(flux > 0.5 MeV)/EFF	(flux > 1 MeV)/EFF
IPS 2	2.54	1.61	1.03

In order to obtain dpa rates, multiply the neutron flux > 1 MeV by 1440 barn [2, 3].

References

[1] J. H. Baard et al., Nuclear Date Guide for Reactor Neutron Metrology, Kluwer (1989).
[2] V. Kuzminov, SCK•CEN Technical Note BR2-SCU/VK/F04302/fluxes-dpa/23.06.04.
[3] V. Kuzminov, private communication.

ANNEX

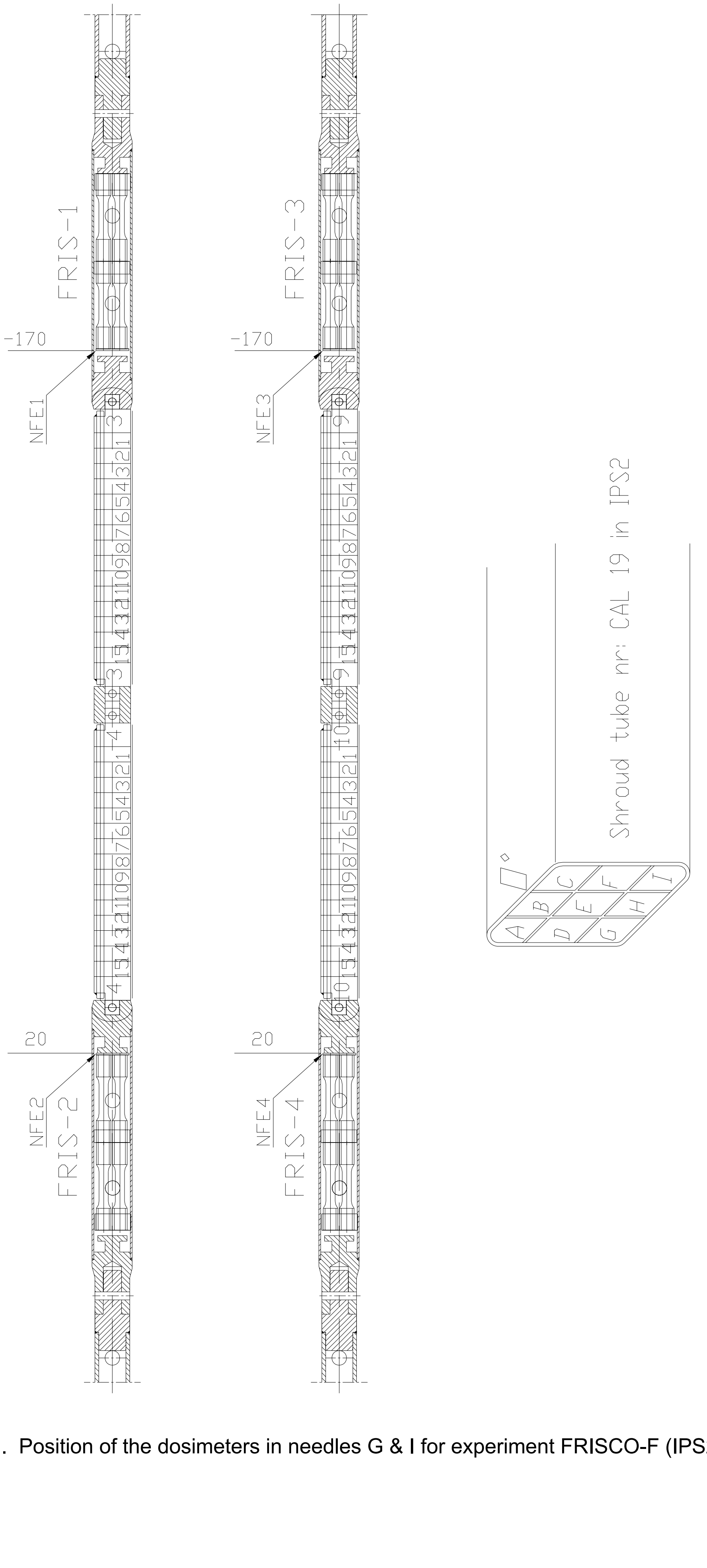


Fig. 1. Position of the dosimeters in needles G & I for experiment FRISCO-F (IPS2).

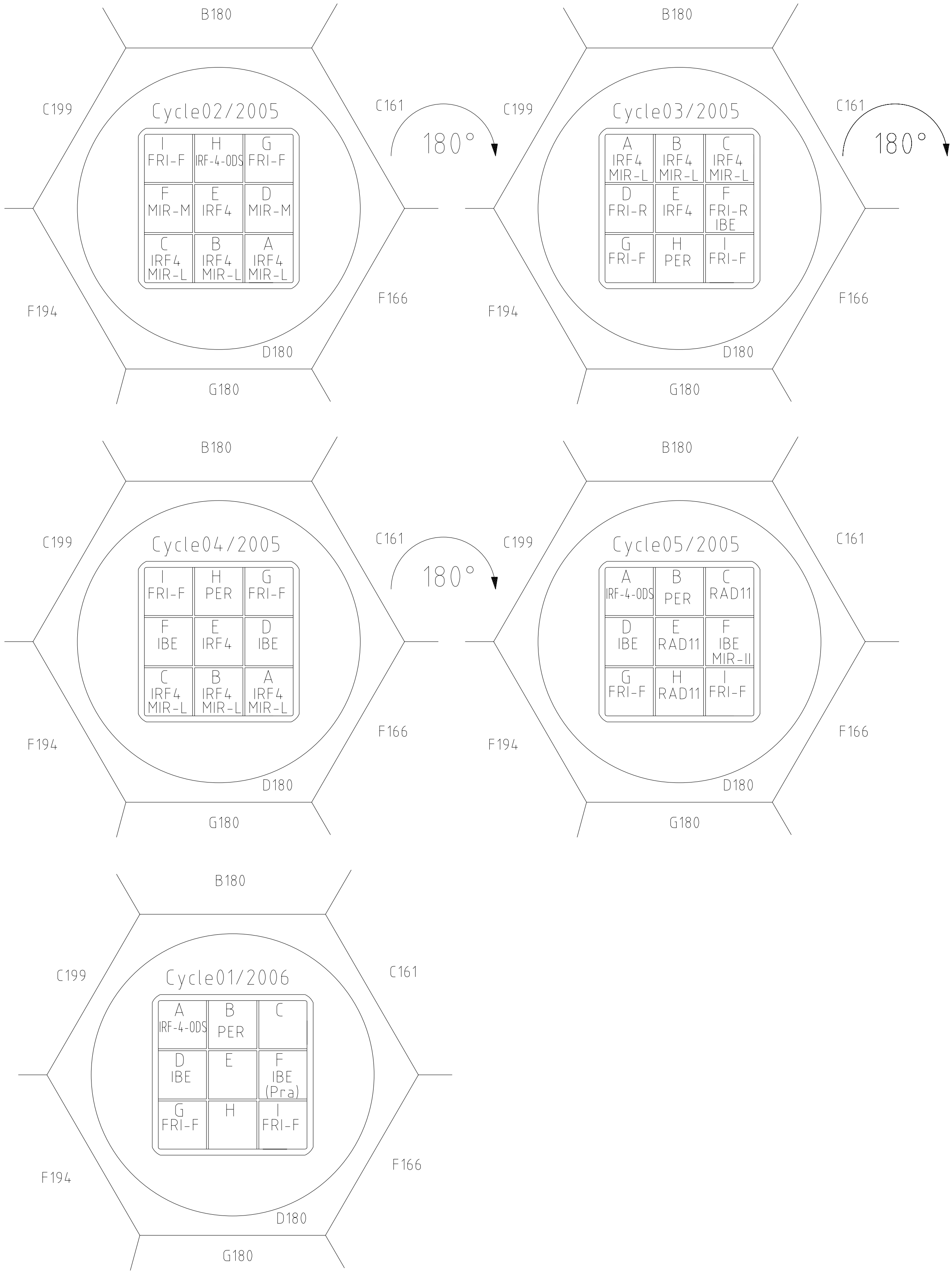


Fig. 2. Position of the needles in the Callisto rig for experiment FRISCO-F.

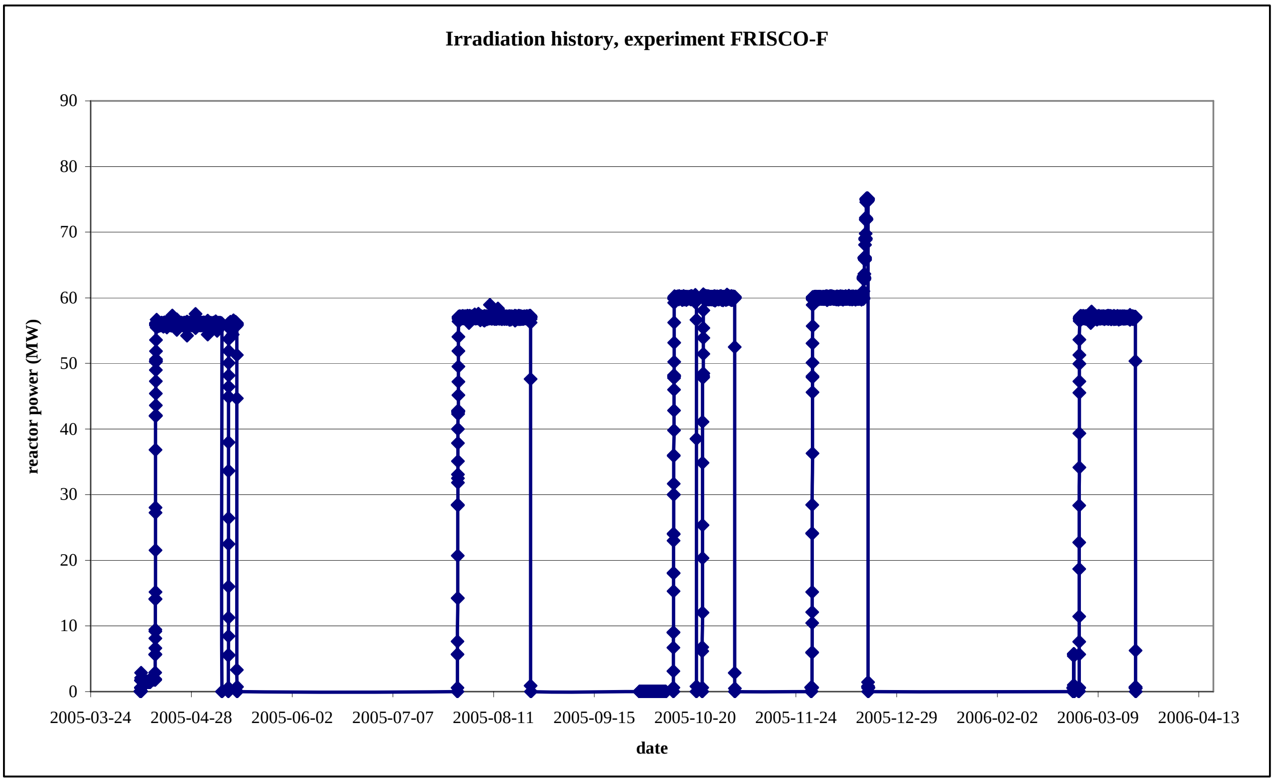


Fig. 3. Irradiation history for experiment FRISCO-F in the BR2 reactor.

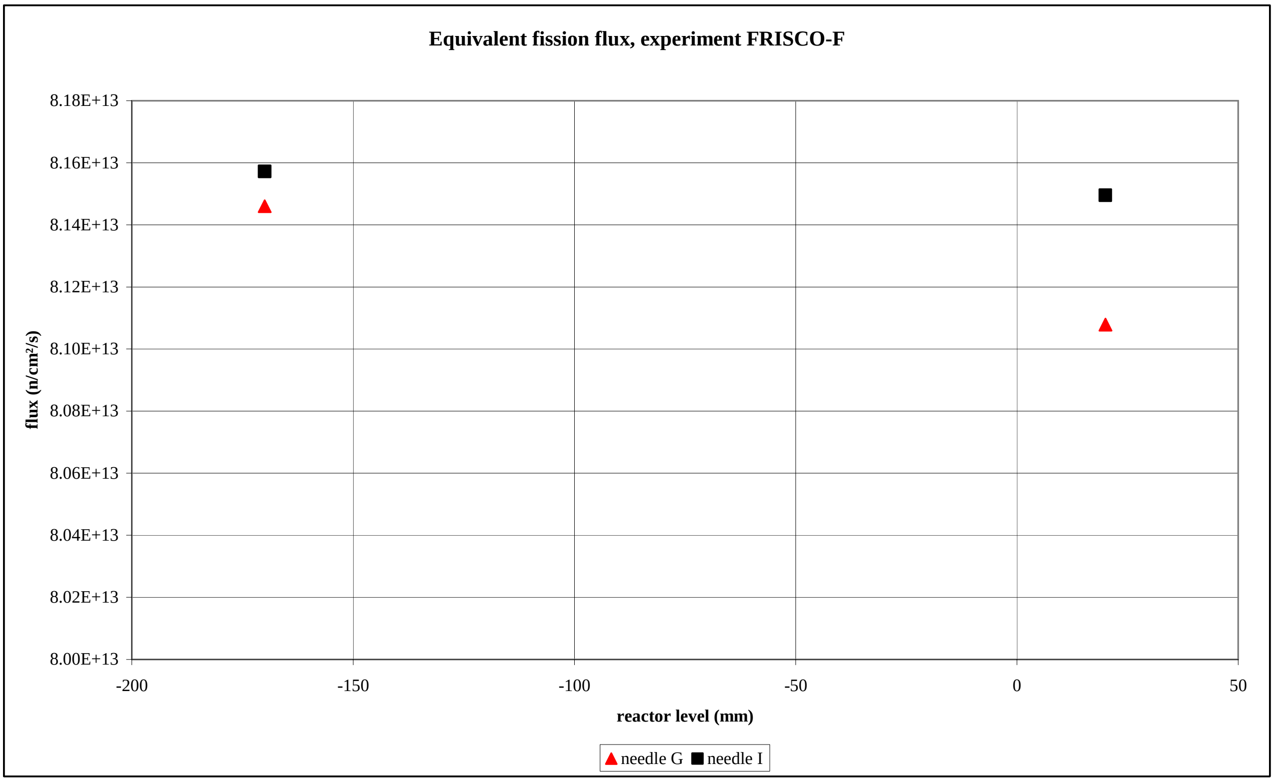


Fig. 4. The equivalent fission flux determined from the activity measurements.