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The second SINQ target irradiation program, STIP-II

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Abstract

The second SINQ Target Irradiation Program (STIP-II) was performed in 2000 and 2001. More than 2000 specimens from more than 40 kinds of materials were irradiated up to 20 dpa and 1800 appm He in a temperature range of 80–450 °C. While the majority of specimens were irradiated in He gas environment, some specimens were in contact with liquid Hg and Pb-Bi. Neutron radiography was performed before and after irradiation, which indicated that Hg has leaked from one Hg-filled capsule, probably during a short temperature excursion caused by beam focusing. Specimens irradiated in He gas environment were unpacked and sorted. Except for TEM and dosimetry discs, almost all the other specimens were retrieved. About 20% of TEM and dosimetry discs were lost. Most of them are from brittle materials. Neutronics calculations were performed using the proton distribution profile obtained by γ-mapping on the beam window of the $AlMg_3$ target container. The calculated He and H concentrations will be corrected with the results of measurements on some selected samples.

1. Introduction

For the existing spallation targets of medium-power range (0.1–1 MW) as well as the high power (⩾1 MW) targets to be built in new spallation neutron sources or accelerator driven systems (ADS), the irradiation induced degradation in the mechanical properties of the target or structural materials is a key parameter limiting the lifetime of the targets. Therefore, target related materials R&D activities have been emphasized in all the target development projects worldwide [1–4].

Because of the high displacement rate and high production rates of spallation transmutation impurities, especially helium (He) and hydrogen (H), the damage induced by spallation irradiation cannot be simply simulated by fission neutron irradiation and ion–beam implantation experiments. Therefore the SINQ target irradiation program (STIP) was initiated in 1996 and conducted under a collaboration with Forschungszentrum Jülich (FZJ), Oak Ridge National Laboratory (ORNL), Commissariat a L'energie Atomique Centre D'etudes de Saclay (CEA), Japan Atomic Energy Research Institute (JAERI) and Los Alamos National

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Table 1
Irradiation matrix of STIP-II

Irradiation experiment									
ID	Materials	Tensile S	Tensile L	B-fatigue	Bend bar	Charpy	SP/TEM	CT	Supplier
A	EC 316LN	24	18	12			21	12	ORNL
		16	16		10		41		PSI
B	316LN CW	24	12	12			21	6	ORNL
C	316N EBW	20	5	6			11		PSI
D	JPCA	28	9	12			13		JAERI
E	Alloy 800H	20	9	12			15		JAERI
F	T91(9Cr–	20				8	24		CEA
	1MoVNb)		2	18			4		PSI
G	EM10	24	12			16	26		CEA
			7	18	7		20		PSI
H	EM10 EBW	16					8		PSI
I	Optifer-IX	20	9	18	5	16	21		PSI
J	Optifer-IX EBW	16					10		PSI
K	F82H	20	17	18	8	16	44		PSI
L	F82H EBW	16					10		PSI
M	EP823		10	12	5		21		LANL
N	HT-9		12		5		15		LANL
O	MANET-II	20		6			6		PSI
P	MANET-II EBW	16					8		PSI
Q	MA957(ODS)	16					13		CEA
R	MA956(ODS)	16					19		FZJ/UAI
IJ	EuroFer-97	16	6				19		PSI
IK	PCr01WVTa	16					18		CEA
S	Zircalov-2	20	13	12	7		28		PSI
T	Inc. 600		6	6			15		LANL
U	Inc. 718 SPF		6	6			13		LANL
V	Inc. 718 Ann	20		12			13	6	ORNL
W	Inc. 718 CW	20		3			11	6	ORNL
X	Inc. 718 EBW	20		3			9	6	ORNL
IN	Ni sup. alloy						10		LANL
IO	Ni sup. alloy	4							PSI
Y	Au	8		3			4		JAERI
Z	Pt	8		3			6		JAERI
IA	Nb	8		3			5		JAERI
IB	Ta	8		3			10		JAERI
		20	17	15	8	8	51		PSI
IC	Cr	8		3			6		JAERI

ID	W/Nb(AZr)			6			4		JAERI
IE	W/Ta			5			4		JAERI
IF	W/Cr			6			4		JAERI
IG	W	20					24		JAERI
							6		LANL
IH	SiCf/SiC				3				JAERI
IP	ZrO_2				2				PSI
									Sub-total
Sum		528	186	233	60	64	631	36	1738

Irradiation + corresion in Hg (ORNL)

ID	Materials	Tensile S	Tensile L	B-fatigue	Capsule	TEM
A	EC316LN		6		3	12
IM	9Cr–2WVTa		3		3	12
				Sub-total		39

Irradiation + corrsion in Pb-Bi (PSI)

ID	Materials	Tensile S	Tensile L	B-fatigue	Capsule	TEM
A	EC316LN	4	2	2	2	4
B	316LN CW					4
C	316LN EBW	2		1		2
D	JPCA		3			4
F	T91		3			4
G	EM10	2	3	2		4
H	EM10 EBW					4
I	Optifer	2		2		2
K	F82H	4	2	2		2
L	F2HEBW	2				1
O	MANET-III					4
IJ	Eurofer		3			4
IM	PCr–2WVTa		2		2	1
V	Inc.718 Ann			2		4
IB	Ta	4	2	2		4
	FZK coated discs					24
	Sum	20	23	13	7	123
				Sub-total		147

It includes additionally:

(a) 10 SANS and 5 TAP samples of EM10 and T91 from CEA.
(b) 25 carbon/carbon composites samples from CEA to study the thermal properties of the composites.
(c) 140 dosimetry disks: 28 Al, 18 Au, 12 Co, 28 Cu, 14 Fe, 12 Nb, 14 Ni and 14 Ti.
(d) 2 Ta-clad-W cylinders from ISIS, UK.
(e) 1 SiC/SiC SANS sample from ATI, Austria.

Total 2093 specimens.

Laboratory (LANL) in order to meet the needs from different spallation source development projects. The first irradiation experiment (STIP-I) was performed through 1998 and 1999, where more than 1500 samples from nearly 40 kinds materials were irradiated to doses up to 12 dpa and 1100 appm He (in steels) in a temperature range of about 80–400 °C [5]. With and, in response to strong interests from the STIP partners in higher irradiation doses and temperatures along with some new materials selections, the second experiment (STIP-II) was conducted in 2000 and 2001, taking advantage of the increased proton beam current at the SINQ target. More than 2000 samples of more than 40 kinds of materials were irradiated. The present paper will present information about the irradiation.

2. Materials selection and specimens

The majority of the materials selected for STIP-II is similar to that of STIP-I, namely austenitic and martensitic/ferritic (MF) steels, as can be seen from Table 1. Special emphases were given to MF steels because of their prospective applications in the ADS devices. The selection included not only well studied steels such as T91(9Cr–1MoVNb), EM10(9Cr–1Mo), F82H(8Cr–2WTa), HT-9(12Cr–1MoVNb) and EP823(12Cr–1Si), but also the ones newly developed in the fusion program such as Optifer-IX(9Cr–1W) and Eurofer-97 (9Cr–1WTa), and ODS MF steels such as MA956 and MA957. Apart from austenitic and martensitic steels, Ni-based Inconel alloys were widely included for their possible applications as the beam window materials in spallation sources as suggested by the relatively good experience from LANL [6]. Another highly interesting material was pure tantalum which showed excellent ductility after irradiation in the ISIS target to about 12 dpa [7]. Tentative coating materials and some high-Z pure metals were supplied by JAERI for developing high-power solid targets.

Although most of the specimens of STIP-II are miniature type similar to those of STIP-I, STIP-II includes a much larger variety of specimen types, shown in Fig. 1. The specimens were irradiated for different purposes: (1) mechanical testing to obtain tensile, fatigue, fracture and impact properties, (2) microstructure studies with transmission electron microscopy (TEM), small angle neutron scattering (SANS) and tomographic atom probe (TAP) analyses, and (3) investigations on basic physical properties such as thermal capacity and conductivity.

The structure of the specimen package was similar to what was used in STIP-I [6], namely specimens were packed in specimen holders layer by layer and then enclosed in SS 316L tubes. The specimen holders were modified for easier loading and better heat transfer from the specimens to the cooling water. Fig. 2 illustrates the structure of a package which contains ten layers of specimens and one thermocouple.

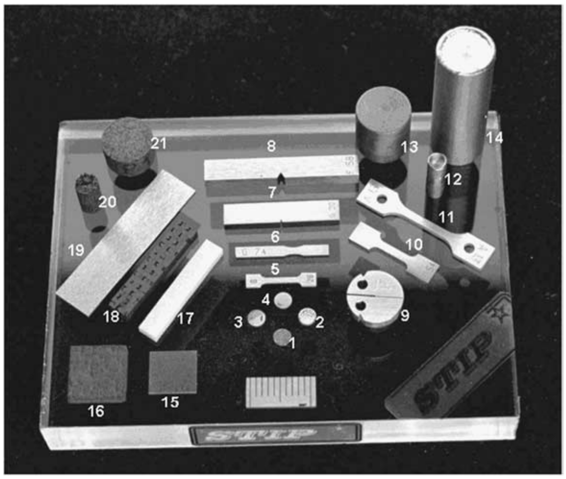

Fig. 1. A photograph shows the samples of different types and some kinds of materials. 1: dosimetry disc, 2: surface-treated disc for corrosion test, 3: W-Cr joining sample for coating materials study, 4: TEM/SP sample, 5: miniature tensile sample, 6: bending fatigue sample, 7: precracked bend bar sample, 8: 1/3 size charpy sample, 9: small CT test disk, 10: subsize tensile sample, 11: SS-3 size tensile sample, 12: stressed steel capsule for corrosion test, 13: Ta-clad-W sample, 14: Hg filled capsule containing 2 stressed capsules, 3 SS-3 tensile samples and 8 TEM disks, 15: steel plate for SANS study, 16: SiC/SiC sample for SANS study, 17: ZrO_2 bending sample for coating materials study, 18: SiC/SiC bending sample, 19: steel plate for TAP study, 20: Carbon fibre composite cylinder for heat capacity study, 21: Carbon fibre composite cylinder for thermal conductivity study.

To study possible irradiation assisted liquid metal corrosion and embrittlement effects, three target rods were prepared which contained Hg- or Pb-Bi-filled capsules. Fig. 3 shows neutron radiographs taken from these rods. Rod A was composed of three Hg-filled capsules (see Fig. 1, sample 14) and four Chapy samples. The Hg-filled capsules were fabricated by ORNL. Each Hg-filled capsule contained two small Al-stressed steel capsules, three SS-3 size tensile samples and eight TEM disks. Fig. 4 shows schematically the structure of the Hg-filled capsules. To increase the protection against Hg flowing out of the rod, a protective capsule was added between the containment capsule and the target tube. Furthermore, the space between the containment capsule and the protective capsule was filled with silver (Ag) coils, so that amalgam would be formed by Hg leaking out of the containment capsule. Rods B and C are made up of Pb-Bi-filled capsules which contain four stressed capsules and a number of tensile, bending-fatigue and TEM specimens. A thermocouple was installed

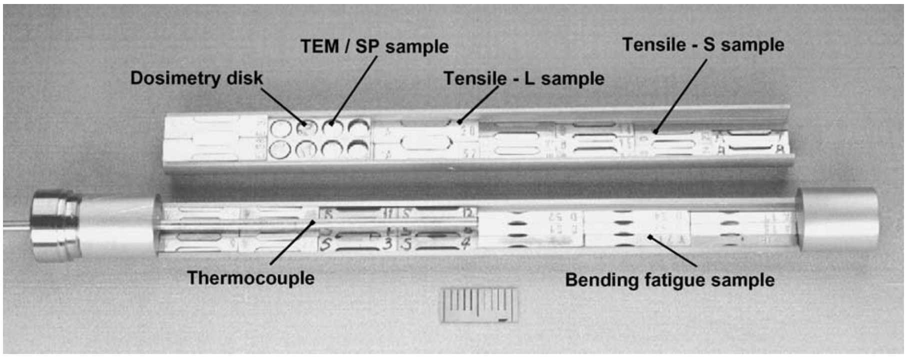

Fig. 2. A photograph shows the structure of specimen package. The scale in the picture represents 1 cm.

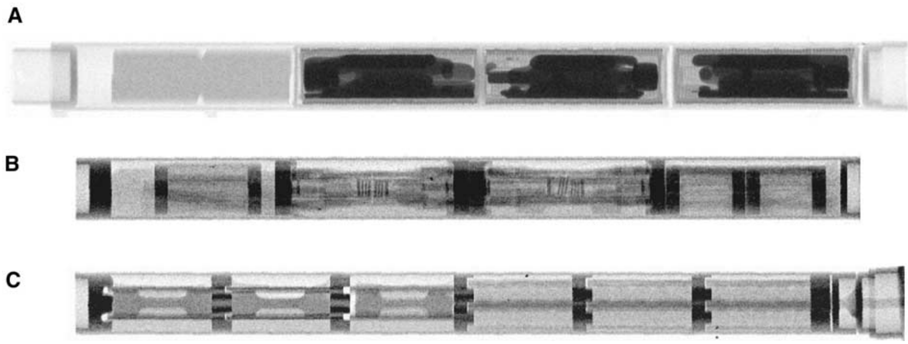

Fig. 3. Neutron radiography pictures showing Target Rod-A to -C. A is Rod-A which contains three Hg (about 19 g in total) filled capsules and one steel sample package. There is about 25% free space in each Hg filled capsule. B and C are the Pb-Bi filled capsules put into Target Rod-B and -C, where test samples are embedded in Pb-Bi (about 38 g for each capsule). There is about 10% free space filled with 2 bars He in each of them.

in Rod C, which is clearly visible in the right hand half of the radiograph.

Finally, to support solid target development, two Ta-clad-W cylinders of 9.7 mm diameter and 10 mm length were prepared by ISIS and included for studying the behaviour of the bonding between the W-cylinder and the Ta cladding after extensive irradiation.

Detailed information about the specimens for the irradiation and corrosion under irradiation in Hg and PbBi experiments is presented in Table 1. Fig. 5 shows the arrangement of the 17 specimen rods in the target with the information about the samples in each specimen rod given in the right-lower corner of the sketch. Instead of zircaloy rods as used in the previous targets [5], the normal target rods of this target (Target-4) are lead (Pb) rods clad with SS 316L tubes. This increased the neutron production by more than 30% [8]. Due to the more dense target rods, the length of the target block was reduced from 57 cm of the previous zircaloy targets to 44 cm of the present target. To ease the manufacturing process the cross-section of the target is now of square shape rather than the hexagonal shape of the earlier versions. The target enclosure, the double walled $AlMg_3$ container remains the same as before.

3. Proton and neutron spectra, irradiation damage and gas production

The irradiation was started on the 20th of March, 2000 and interrupted on the 23rd of December. After a long break it was started again on the 15th of May and finally ended on the 23rd of December, 2001. The total irradiation period was about 16 months. In the 9 months of 2000, a total proton charge of 5.56 Ah was received, and in the 7 months of 2001, another 4.47 Ah was reached, which gave a total proton charge of 10.03 Ah.

As described in the previous paper [5] on STIP-I, the intensity and profile of the incident proton beam on the

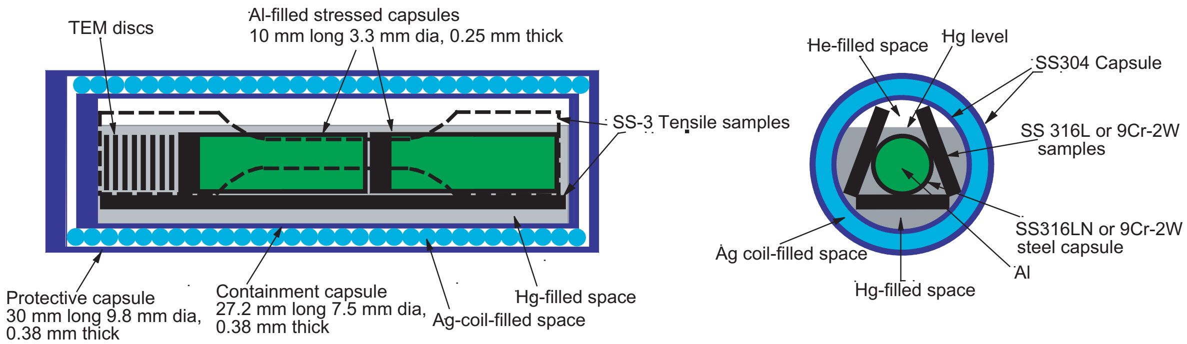

Fig. 4. The sketches illustrate schematically the structure of the Hg capsules.

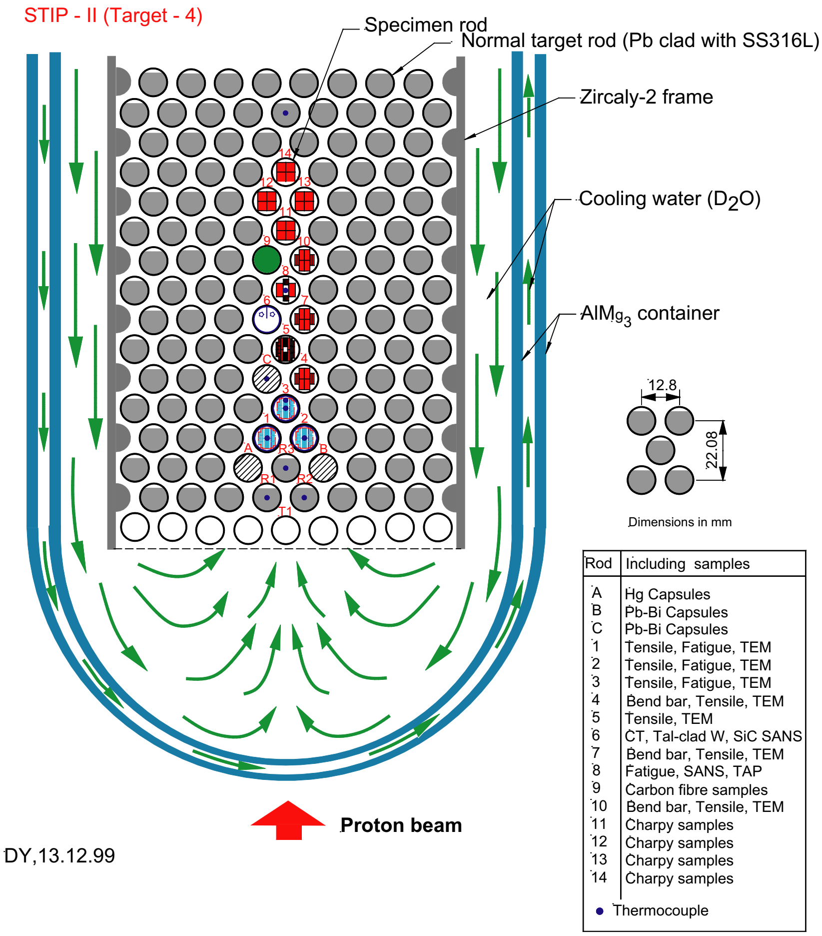

Fig. 5. A sketch shows the positions of the specimen rods in the lower part of the target. The information about the samples in each specimen rod is briefly described at the right-lower corner.

SINQ target depends on the thickness of Target-E which is a graphite target of 6 cm or 4 cm thickness installed in the beam line upstream of the SINQ target. The beam profile on the SINQ target can be roughly represented as a truncated two-dimensional Gaussian distribution. For precise determination of the irradiation dose, a γ-mapping has been performed on the beam entrance area of the $AlMg_3$ container. Fig. 6(a) shows the view of the

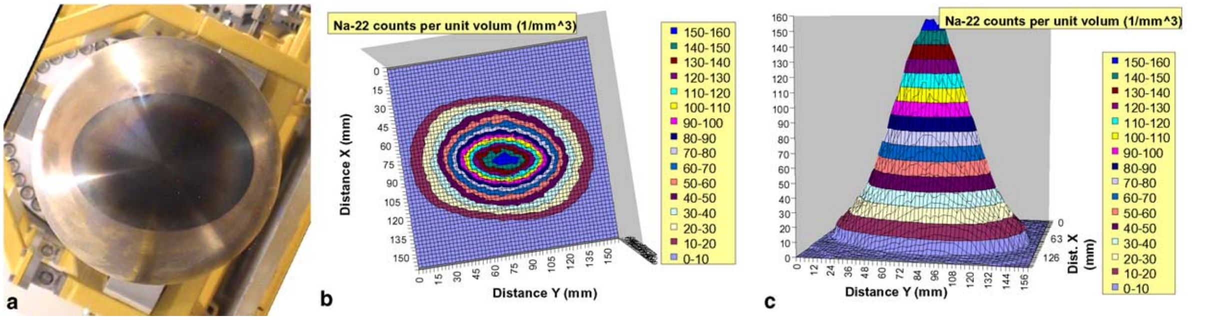

Fig. 6. (a) A photo shows the beam window of the $AlMg_3$ container of Target-4. (b) and (c) present the distribution of ^{22}Na counts per cubic millimetre (mm^3) measured with γ-mapping, which indicates the integrated proton beam intensity of Target-4 after 2-year irradiation.

bottom end (beam window) of the $AlMg_3$ container after irradiation. Fig. 6(b) and (c) present the results of the γ-mapping. Since the neutron flux is much less than the proton flux at the beam window [9], the ^{22}Na nuclei were essentially produced by protons. This can be verified from Fig. 6(b): The area of the ellipse reflects more or less the actual size of the black proton beam footprint on the outer surface, with virtually no ^{22}Na activity beyond it. Like the beam profile given by Rohrer before [10], the γ-mapping data shown in Fig. 6(c) cannot be simply fit with a two-dimensional Gaussian distribution.

The proton and neutron fluences, irradiation dose, helium and hydrogen production for STIP-II samples have been calculated with the MCNPX code using the γ-mapping data. Fig. 7(a)–(d) illustrate the distributions

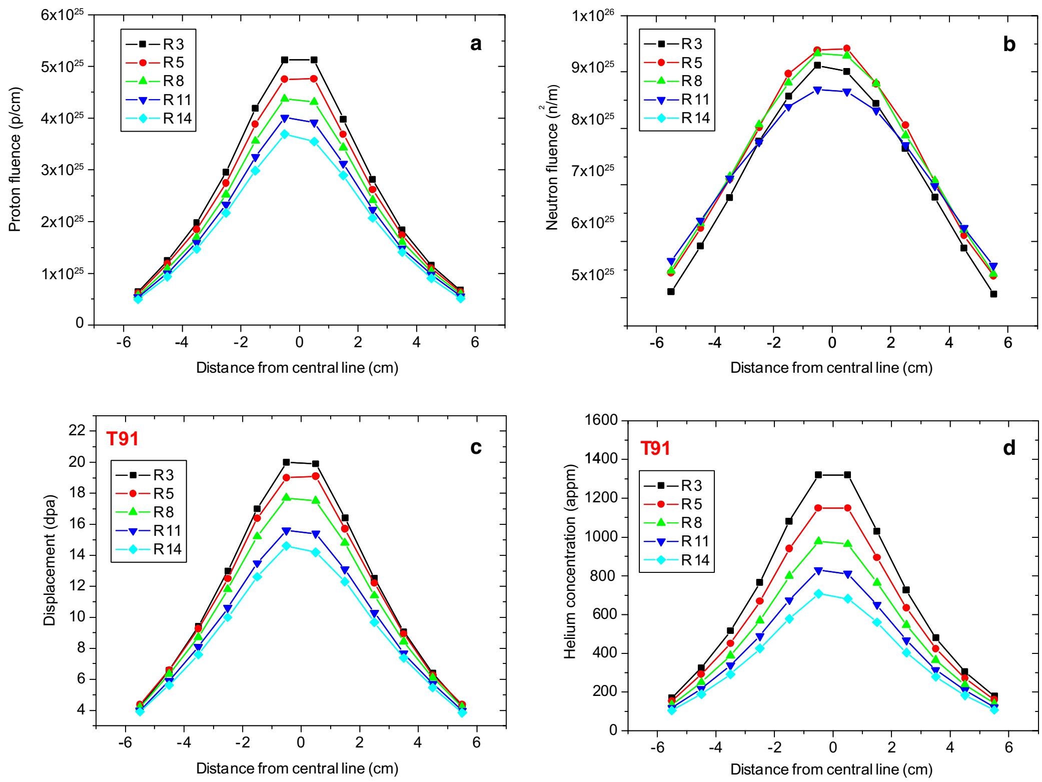

Fig. 7. Plots (a)–(d) show the distributions of the proton and neutron fluences, the irradiation dose and He concentration (for T91 steel) along specimen rods in the central column, respectively.

of the proton and neutron fluences, irradiation dose and He concentration along the five specimen rods (Rods-3, -5, -8, -11 and -14, see Fig. 5) in the central column. It shows that the maximum dose of the STIP-II samples is about 20 dpa (in steel).

To confirm the results of the neutronics calculations, γ-spectrometry measurements have been performed on dosimetry discs. Preliminary results suggest that the proton and neutron fluences deduced from the measured results are slightly (∼10%) lower than the calculated values. The detailed analysis is going on and the results will be reported elsewhere [11].

As in STIP-I, a number of TEM discs from different materials were selected for He and H measurements. The results of He measurements from four F82H discs indicate that the measured concentrations are systematically greater than the calculated ones. The ratio between the measured and calculated values is about 1.3 for the

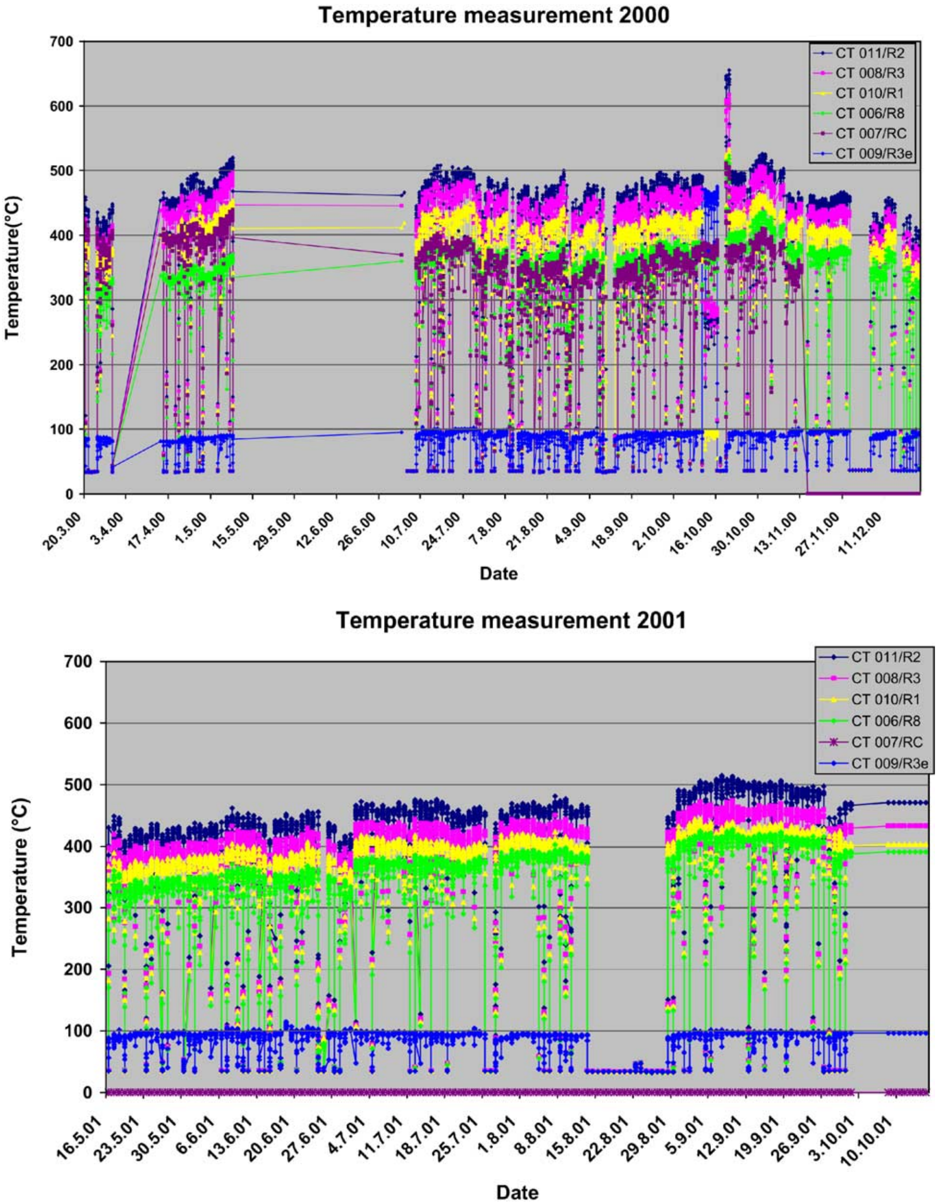

Fig. 8. The temperatures measured at the centre positions of Rod-1, -2, -3, -8 and -C, and the edge position of Rod-3. Some data were lost during periods of May to July, 2000, and October to December, 2001. There was a shutdown of about 2 weeks in August, 2001.

two discs from the centre areas of Rod-3 and Rod-10, and about 1.5 for the two discs from the edge areas of Rod-3 and Rod-10. The evaluation is still ongoing. The calculated He and H concentrations will be corrected using the measured results.

4. Irradiation temperature

As illustrated in the previous paper [5], the irradiation temperature is influenced greatly by the proton beam current and frequent beam trips. The proton beam current depends strongly on the thickness of Target-E. During this irradiation, the thickness of Target-E was 4 cm for the first 14 months and 6 cm for the last 2 months. The change of Target-E induced a temperature drop of about 20%. Furthermore the beam current also varied during irradiation with the same Target-E. It fluctuated between 1.0 mA and 1.25 mA in the first 14 months and between 0.92 mA and 1.02 mA in the last 2 months. Therefore, one would expect that the irradiation temperature should show a similar large variation. Fig. 8 illustrates the temperatures measured at the centre positions of Rod 1, 2, 3, 8 and C and the edge position of Rod 3 during 2000 and 2001. The results are summarized in Table 2.

Table 2
The main range, average value and variation of the temperature measured at each position

	Rod-1 centre	Rod-2 centre	Rod-3 centre	Rod-8 centre	Rod-C centre	Rod-3 edge
Main range (°C)	345–450	390–510	365–485	310–410	320–400	80–106
Average (°C)	395	450	425	360	360	93
Variation (°C)	±55	±60	±60	±50	±40	±13

Note: Temperatures during short beam trips are excluded from the main temperature range and also for evaluating the average temperature values.

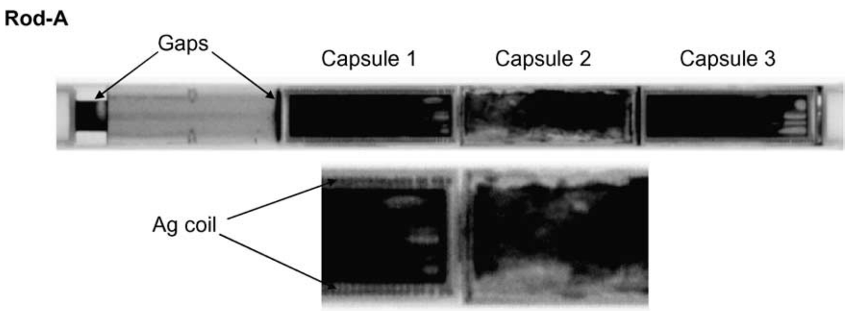

Fig. 9. Neutron radiography picture showing Rod-A after irradiation.

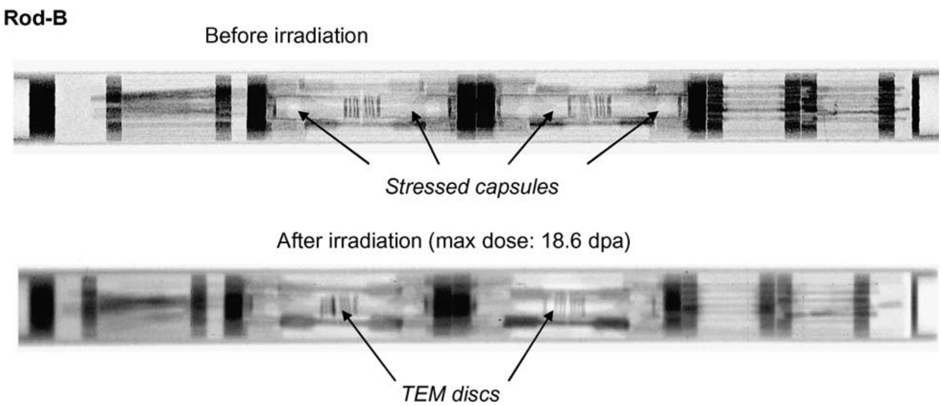

Fig. 10. Neutron radiography pictures showing Rod-B before and after irradiation.

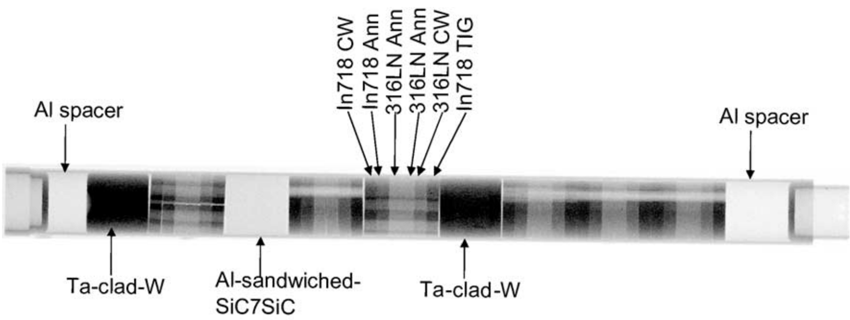

Fig. 11. Neutron radiography picture of Rod-6 which consists of two Ta-clad-W cylinders, one Al-sandwiched-SiC/SiC samples, and 36 CT specimens from cold worked Inconel 718 (In718 CW), annealed Inconel 718 (In718 Ann), TIG weld of Inconel 718 (In718 TIG), cold worked SS 316LN (316LN CW) and annealed SS 316LN (316LN Ann).

During irradiation there were many proton beam trips with durations of a few seconds to a few minutes. Each beam trip resulted in a temperature variation, rapidly (in few seconds) dropping from the irradiation temperature to the cooling water temperature, ~35 °C, and then relatively slowly (in few minutes) recovering with the gradual controlled increase of the proton beam intensity. Normally, there are about 50 proton beam trips per day.

In the evaluation of the average temperature value, the data from short beam trips are not taken into account, only those data from mainly stable beam periods are used. The result of the evaluation is presented in Table 2, which lists the range, the average value and the variation of the temperature measured at each position. It can be noted that the variation is about ±15% of the average value for any position.

Besides the above factors, which more or less represent the routine of SINQ operation, some mistakes in beam tuning resulted in unfortunate incidents like the over focused beam in STIP-I [6], which killed about 300 valuable specimens. In STIP-II, such a mistake happened again. In Fig. 8 one may note that there is an unusual period in October 2000, where the temperature at the edge position of Rod-3 is much higher than that at the centre positions. The period was followed by a high temperature period of about 22 h. Fortunately, the maximum temperature was not very high and only small damage was detected during retrieving the samples. Nevertheless, a high temperature period of 22 h might have introduced some annealing effects in some samples, which should be considered when interpreting the post-irradiation-examination results.[2]

[2] In the process of preparing for an experiment with a liquid metal target (MEGAPIE), beam profile and beam intensity monitoring devices have been installed, which should help to eliminate such incidents in the future.

The assessment of the temperature of an individual specimen has to rely on calculations using the ANSYS code. Such calculation have been extensively performed for STIP-I. For STIP-II, similar calculations were done for some specimen rods [12].

5. Neutron radiography inspections on irradiated specimens

Neutron radiography was applied to inspect specimen rods before and after irradiation. While Fig. 3 presents the graphs of the Hg and Pb-Bi rods before irradiation, Figs. 9 and 10 show the graphs of Rod A (Hg) and Rod B (PbBi), respectively after irradiation. It is known that Hg does not wet steels at room temperature. This is also obvious in Fig. 3(a). The situation was changed significantly after irradiation. In Fig. 9, one can see that the Hg quite homogenously spread in Capsule 1 and Capsule 3, which indicates that the specimens are wetted by Hg. The wetting is believed to have been improved by irradiation, heating to high temperatures and, perhaps, by the spallation products in Hg as well. Furthermore, it is clear that Capsule-2 was damaged during irradiation and the Hg leaked out of the capsule and flowed into the gaps. Fortunately, the last barrier, the SS 316L tube was not penetrated. However, it is striking to see that the Hg penetrated the two SS 304 capsules of 0.38 mm thickness (the containment and protective capsules, see Fig. 4). The detailed temperature calculation for this rod has not yet been done. A rough estimation indicates that the temperature of the Hg should be above 400 °C before the containment capsule was penetrated. After the penetration of the containment capsule, some Hg obviously reacted with the Ag coils to form amalgam. This is confirmed by the neutron radiography picture. It can be seen from the enlarged view in Fig. 9 that the Ag coils disappeared in Capsule 2. However, Ag–Hg amalgam can be decomposed at above 300 °C [13]. This means that, in all likelihood

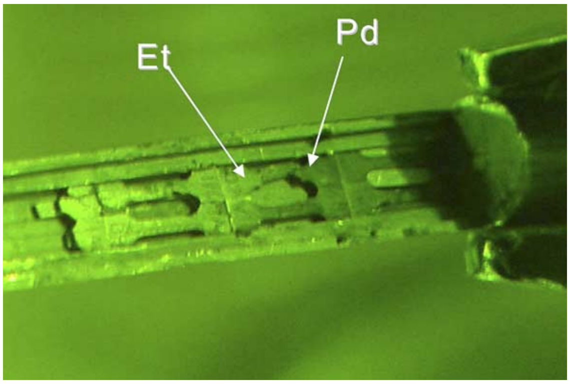

Fig. 12. A picture showing the broken Cr and Nb samples. The green colour is due to that the photo was taken from the outside of the hot cell through a very thick glass window.

the protective capsule got in contact with Hg after the first penetration. The temperature at the inner surface of the protective capsule is expected to be between 300 and 400 °C. At such temperature, Hg corrosion on stainless steels is not so significant without irradiation [14,15]. On the other hand, the temperature at the inner surface of the containment capsule of Capsule-1 should be above 300 °C, too. However, this capsule has not been penetrated. Therefore, a conclusive explanation cannot be given at this point. Extensive examinations are needed, involving the opening of the capsules.

Fig. 10 presents the view of Rod B, one of the two Pb-Bi rods. Due to the high activity of the rod, an imaging technique had to be applied after irradiation which does not yield as good pictures as the one used before irradiation. The image looks more blurred. Nevertheless, the specimens, even the small TEM discs, 0.25 mm thick, can still be clearly recognized. The stressed capsules can also been seen. This may be taken as an indication that these specimens were not significantly corroded. The maximum temperature at the surface of the stressed capsules was about 345 ± 45 °C during irradiation. One can see that the positions of the specimens changed slightly. This indicates that the PbBi had completely melted during irradiation, as would be expected (melting point 125 °C). Some PIE will be performed on this rod soon. The results will be available in 2005.

Another radiograph, from Rod6, is shown in Fig. 11. Compact tension (CT) specimens of SS 316LN, Inconel 718 in annealed and cold worked conditions, two Ta-clad-W cylinders and one Al sandwiched-SiC/SiC specimen were included in this rod. It is interesting to note the different contrast of materials and in different conditions. While one would expect the neutron beam attenuation to be highest for Ta-clad-W and weakest for the Al sandwiched SiC/SiC, it is interesting to note that cold worked Inconel 718, and cold worked SS 316LN show stronger attenuation than the annealed varieties of the same materials. Again, the stronger attenuation by Inconel 718 relative to SS 316LN is as expected.

6. Retrieving the specimens

The specimen rods were dismounted from the target in hot cell near the SINQ target station, and then transferred to hot cells in PSI Hotlabor. There, except for those irradiated in Hg and PbBi, all specimens were

Table 3
The list of specimens irradiated and retrieved for different institutes

Institute		Tensile S	Tensile L	B-fatigue	Bend bar	Charpy	SP/TEM	CT	Sum
CEA	Irradiated	76	12			24	81		193
	Retrieved	75	12			24	51		162
JAERI	Irradiated	108	18	56	3		95		280
	Retrieved	99	18	52	3		80		252
LANL	Irradiated		34	24	10		80		148
	Retrieved		34	24	10		61		129
ORNL	Irradiated	108	30	42			75	36	291
	Retrieved	108	30	42			65	36	281
UAI	Irradiated	16					19		35
	Retrieved	16					18		34
PSI	Irradiated	210	79	111	47	40	234		721
PSI	Retrieved	208	79	111	47	40	209		694
Total	Irradiated	518	173	231	60	64	580	36	1738
	Retrieved	506	173	231	60	64	484	36	1520

Note: UAI is for University of Ancona, Italy.

unpacked and sorted. Although there was no serious incident during irradiation, some specimens were still broken after unpacking, particularly TEM discs. Fig. 12 shows the pure Cr (the lower layer) and Nb (the upper layer) tensile specimens which were broken during retrieving even with careful handling. It was also noticed that some particles or powders were produced which is believed to stem from the TEM discs of some brittle materials such as Cr, Nb, W and W-alloys, because a several of them were missing. Due to the short period of high temperature, the Al dosimetry discs at the centre areas had melted and fused together some other TEM discs or dosimetry discs. Also, a several TEM discs were heavily polluted and could not be identified. Alltogether, about 120 TEM and dosimetry discs were lost. Table 3 presents the information of specimens irradiated and retrieved for different institutes.

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