The eighth SINQ Target Irradiation Program, STIP-VIII

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A B S T R A C T

The eighth experiment of the SINQ Target Irradiation Program (STIP-VIII) included a total of 941 specimens from 25 steels, 8 zircaloys, 6 tungsten based alloys (W-alloys) and 2 silicon carbide (SiC) composites. Specimens were prepared in 2016 and 2017. Irradiation was carried out in SINQ Target-13 in two periods: June 26 to December 21, 2018 and July 30 to December 23, 2020. The total proton charge received by Target-13 is 7.8 Ah, corresponding to 1.75\(\times\)10\(^{23}\) protons. The irradiated specimens were unpacked in 2022 and 2023 and more than 98 % of the specimens were recovered. The specimens were irradiated at temperatures between 100 and 450 \(^{\circ}\)C. The maximum doses are 10.4 dpa for steels, 14.8 dpa for zircalloys, 11.5 dpa for W-alloys, and 3.5 dpa for SiC composites. The He-to-dpa ratio is in the range of 22–42 appm/dpa for steels, 13–16 appm/dpa for zircalloys, 30–36 appm/dpa for W-alloys and 90–125 appm/dpa for SiC composites.

Introduction

Since 1996 the SINQ (the Swiss Spallation Neutron Source) Target Irradiation Program (STIP) has received continuous interest from nuclear materials communities for spallation, fusion and fission applications \([1–4]\). After seven irradiation experiments and irradiating large numbers (>8000) of specimens at a wide range of doses (5–30 dpa) and temperatures (80–600 \(^{\circ}\)C), this interest is waning. Nonetheless, STIP-VIII has been implemented to meet some requirements from Europe, China and Japan.

Due to increased safety requirements following the incident occurred in SINQ Target-11 in 2016 \([5]\), the configuration of SINQ targets was changed, especially in the high proton and neutron flux region where previous STIP specimens were located. Instead of zircaloy-2 cladded lead rods or STIP specimen rods, solid zircaloy-2 rods were used in this area. STIP specimens had to be moved to the upper part of the target where the proton and neutron fluxes are lower. In this case, the maximum irradiation dose of the STIP-VIII specimens is significantly lower, around 10 dpa (in steels), instead of \(>20\) dpa in previous STIP experiments.

In STIP experiments, the production rate of spallation transmutation elements in specimens, especially helium (He) and hydrogen (H), depends strongly on the local proton and neutron spectra \([4,5]\). Compared to low-dose specimens, in high-dose specimens, the contribution of high-energy protons to displacement damage, He and H production is higher, which results in high He-to-dpa ratios typically 60–80 appm He/dpa. In STIP-VIII specimens, the He-to-dpa ratio is much lower, about 35 appm He/dpa, which is closer to the 10–15 appm He/dpa in the first wall of a fusion reactor. Therefore, STIP-VIII is attractive for fusion materials research. Similarly, fission materials research also appreciates a lower production rate of transmutation elements, since the rate is generally low in fission neutron irradiations. Consequently, STIP-VIII is dominated by materials for fusion and fission applications.

Materials and specimens

In STIP-VIII, specimens were packed in zircaloy-2 tubes with inner/outer diameters of 8.8/10.05 mm. The space between specimens, specimens and tube-wall was filled with spacers of T91 steel and the gaps were filled with pure He gas. 941 specimens from 43 kinds of materials were irradiated in 7 specimen rods. Among the specimens, 587 specimens are from 25 steels, including various ferritic-martensitic (FM) steels and ODS FM steels. The composition of the FM steels is typically: 9–12Cr, 0–1Ni, 1–2 W, 0.5–1Mn, 0.2–0.3 V, 0.1–0.2Si, 0.1C, balanced by Fe. The ODS FM steels contain about 0.3 % \(\text{Y}_2\text{O}_3\). Specimens are to be used for tensile, bend, small punch (SP) testing and transmission electron microscopy (TEM) observations. 232 specimens from 8 zircalloys were cut from cladding tubes, which were either used in SINQ targets or
Fig. 1. Dimensions of the STIP-VIII specimens.
developed for nuclear power plants. The composition of the zircaloys is typically: 0.8–1.2Sn, 0.1–0.2Cr, 0.1–0.2O, 0.1–0.2Si, balanced by Zr. The specimens are tensile specimens and $\phi 3 \times 0.25$ mm$^3$ discs for SP/TEM. 61 specimens of pure tungsten (W) and tungsten alloys were irradiated. The W-alloys contain 0.3 % Y$_2$O$_3$ or 0.3 % Re or K, which were developed specifically for fusion applications. Specimens include tensile specimens, mini-bend bars and discs of $\phi 8 \times 0.5$ mm$^3$ or $\phi 3 \times 0.25$ mm$^3$. The SiC composites were also developed for new generation nuclear plants. The composition is 69.4 % Si, 29.8 % C and 0.8 % O. 35 specimens are miniature bend bars. Also included are 26 discs of $\phi 3 \times 0.25$ mm of Zr or Pd based glass metals. They are used for TEM, SP and hardness testing. Finally, 25 dosimetry discs of $\phi 3 \times 0.1$ mm$^3$ of were irradiated with the specimens, which makes the total number of specimens to 966.

The dimensions of STIP-VIII specimens are shown in Fig. 1. Tensile specimens are in two sizes named as “small tensile (ST)” and “large tensile (LT)”. In addition to these specimens, some $16 \times 2 \times 0.75$ mm$^3$ tungsten mini-bend bars (MMB), $20 \times 4 \times 2$ mm$^3$ SiC bend bars and $4 \times 4 \times 1$ mm$^3$ SiC thermal conductivity specimens (TC) were also included. It should be noted that the LT specimens of zircaloys were directly cut from cladding tubes with an outer diameter of 10.05 mm and an inner diameter of 8.8 mm and were therefore curved in the width direction.

To increase comparability across the materials, all specimens were manufactured by the same company. Bars of different specimen shapes were first cut from the received materials by electron discharge machining (EDM) and then cut into pieces approximately 0.1 mm larger than the required sizes shown in Fig. 1. Finally, the 0.1 mm was removed from the pieces by milling 0.05 mm from each surface. The surfaces were ground to ensure that the surface quality meets the N6 requirement, namely an average surface roughness of 0.8 μm.

**Specimen rods**

The design of the STIP-VIII specimen rods is similar to that of previous STIP experiments. For example, Fig. 2 shows the specimens packed in Rod 1. This rod includes 12 bend bars (BB), 84 small tensile (ST) specimens, 137 TEM discs and 8 large discs (LSP). The specimens were distributed in 8 parts from part A to part H. Each type of specimen was packed into 2–3 parts to obtain different irradiation conditions. To tune the irradiation temperature, 0.1 mm deep grooves were introduced on the outer surface of spacers between the specimens and the zircaloy tube. The temperature was adjusted by the width of the grooves. Laser engraving was used to mark IDs on specimen surfaces.

One of the difficulties with such irradiation experiments is to achieve the required irradiation temperature for specimens. The previous STIP experiments have demonstrated that this is even more difficult compared to irradiation experiments in fission reactors, because both the beam current and the intensity distribution profile of the proton beam injected to a SINQ target vary greatly during the 2-year operation time [1–3]. During the design phase, the temperature of each specimen package was carefully modeled. Due to the extensive modeling work, only 2-dimensional (2D) temperature distribution over the cross-section of a specimen package was simulated using the ANSYS code. For STIP-VIII, the energy deposition values obtained from STIP-VI [4] were used to model the irradiation temperature. These modeling results have to be revised and finalized after irradiation when the neutronic simulation is done with the actual proton beam parameters of STIP-VIII. Fig. 3 shows two examples of temperature modeling results by using the neutronic simulation results described in Section 8. Fig. 3(a) depicts the temperature distribution of specimen part A of Rod 1, where 6 BB specimens were included (Fig. 2), while Fig. 3(c) is the temperature distribution of TEM specimens part B of Rod 1, where 4 groups of $\phi 3 \times 0.25$ mm discs were packed. Fig. 3(b and d) are the temperature distribution along the paths indicated in Fig. 3(a and c), respectively. The asymetric temperature distribution in Fig. 3(d) is due to different materials. The large temperature jumps at the interfaces of specimen/specimen, specimen/spacer, and spacer/outer tube are caused by helium gaps with different thicknesses from about 10 μm up to 110 μm. It is worth noting that, based on the experience from previous STIP experiments, a gap thickness of 10 μm between specimens seems to be a good approach, although the average specimen surface roughness is only 0.8 μm.

After specimens inserted into zircaloys-2 tubes, two end-plugs were welded using electron beam welding at one end, the same as normal target rods, and laser welding at the other end. For the laser welding, a specimen rod was loaded into a specially prepared chamber, which was first evacuated to low vacuum and then filled with 2-bar helium. Laser beam penetrates through a glass window onto the specimen rod. In order to check the quality of the laser welding, metallographic inspection (Fig. 4(a)) and a burst test (Fig. 4(b)) were performed. The results demonstrate the good quality of the laser welding.

The 7 specimen rods were inserted into the target in the middle part of rows 20 to 24, as shown in Fig. 5. Among the 7 rods, two of them are equipped with thermocouples, namely Rod 3 in row 20 and Rod 6 in row 24, for monitoring the temperature during irradiation.

It can be seen in this figure that, a large number of full zircaloy rods were used in the high proton and neutron flux area. This was a test conducted to improve the safe operation of the SINQ target after the incident happened in 2016 [5]. The two 75 % Pb-filled rods were used for the same reason.

**Irradiation at SINQ**

The irradiation took place in 2018 and 2020, with an interruption in 2019 due to an upgrade of some neutron beam lines. The overall
The situation is similar in both years. Fig. 6 shows the maximum proton beam current and the average beam current for each irradiation week in 2018 and 2020. In the first 1–3 weeks, there were many beam tuning tests. During this period, the beam current gradually increased. Afterwards, the beam current was maintained at the desired level. It is clear that, a smaller difference between the maximum and the average values means that the proton beam is more stable during this week. For example, Fig. 7 presents the beam status in two weeks, week 37 and week 38 in 2020. Week 37 was normal, while week 38 had more than 3 days dedicated to accelerator service. In a service week, the proton beam is less stable due to beam tests, resulting in a larger difference between the maximum and the average beam current value, as shown in Fig. 6. It can also be seen from Fig. 9 that this kind of service (indicated on the X-axis) was performed every month in 2018 and 2020.

In Fig. 7, the temperature measured at the center of the central target rod in row 12 (CT009) is also presented. As usual, the temperature changed with the proton beam current [1]. The temperature excursions were introduced by proton beam excitations. The majority of the beam excitations were introduced by the operation of the UCN (Ultra Cold Neutron source) facility [7]. UCN is operated in pulsed mode using the full proton beam from the accelerator at a repetition rate of one pulse every 5 min, as shown in Fig. 8. The duration of each beam excursion is about 30 s. Such a beam excursion consists of a sudden beam interruption of 8 s and followed by a slow rise of beam current in the rest 22 s. Accompanied by the absence of the proton beam at the SINQ target, the temperature drops to ~50 °C during the 8 s of the beam interruption. There were 38,362 beam excitations for Target-13, 22,299 in 2018 and
The irradiation temperature of the STIP specimens was monitored by the two thermocouples in row 20 (Rod 3) and row 24 (Rod 6) (Fig. 5). The measurement results are presented in Fig. 9, where each data point is averaged over one hour. Therefore, the short temperature excursions (Fig. 8) cannot be seen here. During a long beam excursion, the temperature drops to 30 °C, the temperature of the cooling water.

In 2018, the temperature was relatively stable, except for the first week, as shown in Fig. 9. The average temperature for the full-beam-on period was 229 °C in row 20 and 179 °C in row 24. It should be noted that the thermocouple in row 20 was placed at 8 mm away from the central line and that in row 24 is 28 mm away from the central line of the target. Therefore, the temperatures were not the highest ones in the corresponding rods.

The temperature measurement results of 2020 are similar to those in 2018. However, the overall temperature values are slightly higher. The reason for this small increase is not clear. It could be due to a slight change in the proton beam intensity profile, which was adjusted at some point by the SINQ operation group. The data was missing in the period from September 9 to October 10 due to a technical problem. However, the irradiation during this period was normal, as shown by the beam current measurement (Fig. 6).

Although some temperature data are missing, it should be mentioned that the online temperature measurement in four target rods indicated that no overfocused beam experienced during the entire irradiation period of STIP-VIII.

Extraction of specimen rods and target beam window

In May 2022 Target-13 was loaded into the hotcell (ATEC) near the nuclear fuel storage. Fig. 5. A sketch showing SINQ Target-13. The insert on the right shows the position of the 7 STIP rods.

Fig. 6. The maximum and average proton beam currents of Target-13 in each irradiation week in 2018 and 2020.
SINQ target station. The 7 specimen rods were removed from the target. As an example, Fig. 10 shows Rod 4 when it was pulled out of the target. All 7 rods were pulled out smoothly. Video inspections did not reveal any damages such as pits and cracks. The proton beam trace can be seen from the color variation on the surface of the rod.

The target beam window, namely the calotte of the target aluminum (Al) container, was cut with a band saw, as shown in Fig. 11. The appearance of the beam window looks different from the previous ones with a dark oval proton beam footprint on the outer surface [1,2]. For this target, the central area of the calotte only looks slightly brown. This indicates the better vacuum in the proton beam line.

Gamma mapping of the Al calotte

In order to accurately evaluate the irradiation dose of the irradiated specimens, it is essential to obtain the distribution profile of the accumulated proton fluence. For STIP irradiation experiments, except for STIP-I, this is done by conducting gamma mapping on the calottes of the Al containers in PSI Hot Laboratory (Hotlab). An area of $160 \times 160 \text{ mm}^2$ was scanned in 4 mm steps [2–4]. 1681 points were measured and each measurement lasted 10 minutes. The activity distribution of $^{22}\text{Na}$ was determined. Since $^{22}\text{Na}$ is mainly produced by protons with energies above about 30 MeV [8], the distribution of $^{22}\text{Na}$ reflects the distribution of the accumulated proton fluence. The derived proton fluence distribution is shown in Fig. 12. The maximum proton fluence is $4.1 \times 10^{25} \text{p/cm}^2$. Comparison with the 2D plots in Ref. [2–4] shows that the distribution of accumulated proton fluence of STIP-VIII is different from all previous ones. This indicates the necessity of the gamma mapping for determining the irradiation dose of STIP specimens. However, it should be noted that the gamma mapping provides only a best estimation of the proton beam distribution accumulated throughout the irradiation period. The intensity profile of the proton beam at SINQ is not stable at all. Unfortunately, during operation there is no precise measurement of the actual beam intensity profile, although the beam position and beam over-focus are monitored.

Unpacking irradiated specimens

The irradiated specimens were unpacked in a hotcell of Hotlab, which was very time-consuming. First, the endplugs of the specimen rods had to be cut off (Fig. 13(a)). Then, cutting along the rod is needed (Fig. 13(b)). A diamond disc saw was used for the cutting.

More than 98 % of specimens were recovered. Only a few small SiC bend bars were broken during unpacking. Fig. 14 presents two photos of some unpacked tensile specimens of zircaloy cladding tubes and some...
TEM/SP specimens. These specimens were contaminated during the above cutting process. They were further cleaned with ethanol in an ultrasonic bath. After cleaning, most of them were identified and stored in a lead-shielded cupboard, and some specimens taken directly for testing.

Neutronic calculation of irradiation parameters

Using the proton distribution profile (Fig. 12) as input data, the irradiation parameters such as displacement damage dose (dpa), He and H concentrations and energy deposition in specimens were calculated using the MCNPX code. Representative results are shown in Fig. 15-19 for three rods in the central column, namely Rod 3, 1 and 6 in rows 20, 22 and 24 (Fig. 5).

The proton and neutron spectra at the center of Rod 1, 3 and 6 are presented in Fig. 15. As the penetration depth increases from row 20 (Rod 3) to row 24 (Rod 6), both the energy and intensity of proton beam decrease. The maximum proton fluxes are $9.1 \times 10^{13}$, $8.3 \times 10^{13}$ and $7.8 \times 10^{13}$ p/cm$^2$/s for the three rods, decreased about 15% from Row 20 (Rod 3) to Row 24 (Rod 6).

The overall distribution of the spallation neutron flux in the target is different from that of the proton flux [2–4]. The energy of spallation neutrons varies essentially from 0 to 570 MeV. The total neutron fluxes are $2.8 \times 10^{14}$, $2.5 \times 10^{14}$ and $2.3 \times 10^{14}$ n/cm$^2$/s for Rod 3, 1 and 6, respectively. The corresponding fast components with energy above 0.1 MeV are $1.9 \times 10^{14}$, $1.6 \times 10^{14}$ and $1.5 \times 10^{14}$ n/cm$^2$/s. Fig. 13(b) shows the fast neutron spectra of the three rods.

The distribution of proton and fast neutron fluences of the three rods is presented in Fig. 16. The difference between the values of the three rods becomes smaller at the edge of the rods. The same trend can also be expected for the other irradiation parameters.

The energy deposition is determined directly from the MCNP simulation. In order to avoid the differences caused by the different densities of the materials in the specimen rods, the energy deposition in the zircaloy cladding tubes was calculated. The results of the three rods are shown in Fig. 17, which are for the case of the average proton beam flux.
current of 1.25 mA. It can be seen that the trend looks similar to that of the proton fluences in Fig. 16. This is because the energy deposition is primarily generated by protons.

In the same way as the previous STIP experiments, the values of dpa, He and H concentrations were calculated from the above proton and neutron spectra and the corresponding cross-section data [2–4,9,10]. Fig. 18 presents the distribution of irradiation dose (dpa) as well as He and H concentrations for steels, zircaloys and tungsten specimens in the three rods. From this data, the He-to-dpa and H-to-dpa ratios can be easily calculated. Since the results are almost the same for the three rods, the results of Rod 1 are plotted as an example in Fig. 19. As expected, the He-to-dpa ratio of steel specimens varies between 25 and 40 appm He/dpa.

As gas release measurements show [11,12], the H content of a specimen depends strongly on its material, irradiation temperature and other specimens/materials in the same rod. The difference between the calculated and measured H concentrations can be very large. Therefore, the calculated H concentrations are for reference only.

Table 1 presents a brief summary of irradiation parameters of the specimens in different rods. From the irradiation parameters one can see the importance and limitations of the irradiated specimens for fusion, fission and spallation applications. For fusion applications, the helium and hydrogen content of the specimens is about 2–3 times that of structural materials in fusion reactors. Nevertheless, these specimens are still among the best specimens that have relatively high doses and...
Fig. 14. (a) Unpacked tensile specimens of zircaloy cladding tubes, (b) unpacked TEM/SP specimens.
reasonable good He-to-dpa ratios and can provide bulk mechanical and thermal properties. In nuclear fission applications, the helium concentrations of STIP-VIII specimens are very high as compared to materials irradiated in fission reactors. However, at least the results of ferritic/martensitic steels irradiated in STIP show that the helium effects are not pronounced when helium concentration is below about 500 appm [13,14]. In this case, the mechanical properties of STIP specimens are consistent with those of fission neutron irradiated specimens. Since the helium concentrations of current specimens are below 450 appm. Therefore, it is believed that the results of these specimens will not differ significantly from those after fission neutron irradiation at similar irradiation doses and temperatures. In any case, the results of STIP specimens, even if not realistic, are conservative for nuclear fission and fusion application due to the higher helium and hydrogen content. For spallation applications, STIP-VIII covers the irradiation conditions of most spallation targets worldwide today. Its limitation would be the relatively low irradiation dose for a spallation source such as SINQ, which can reach a higher irradiation dose.

Summary and outlook

In STIP-VIII a total of 941 specimens of steels, zircaloys, W-alloys, and SiC composites were irradiated in 2018 and 2020 in SINQ Target 13. With the exception of a few small SiC bend bars, more than 98% of specimens were successfully recovered. The maximum doses obtained were 10.4 dpa for steels, 14.8 dpa for zircaloys, 11.5 dpa for W-alloys, and 3.5 dpa for SiC composites. The He-to-dpa ratio is in the range of 22–42 appm/dpa for steels, 13–16 appm/dpa for zircaloys, 30–36 appm/dpa for W-alloys and 90–125 appm/dpa for SiC composites. The specimens were irradiated at temperatures between 100 and 450 °C.

The post-irradiation examination (PIE), particularly the mechanical testing of the steel and zircaloy specimens, is being performed. Many PIE results will be obtained in 2024 and 2025. Some results will be published elsewhere soon.

CRediT authorship contribution statement

Fig. 18. The distribution of the dpa, He and H concentrations of steels, zircalloys and tungsten for Rod 1, 3 and 6.
Fig. 19. The distribution of He-to-dpa and H-to-dpa ratios for steels, zircalloys and tungsten in Rod 1.

Table 1
A summary of irradiation parameters of materials irradiated in different rods of STIP-VIII.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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