## 8.2 Reseach Plan and Status of High-Dose Rabbit irradiation Experiment for Silicon Carbide Composites

— T. Nozawa, K. Ozawa, H. Tanigawa (Japan Atomic Energy Agency), Y. Katoh, L. L. Snead, and R. E. Stoller (Oak Ridge National Laboratory)

### OBJECTIVE

This report provides a brief summary of research plan and present status of the high-dose rabbit irradiation campaign for silicon carbide composites under the JAEA-DOE collaboration on fusion reactor materials.

### SUMMARY

In order to examine the high-dose and high-temperature neutron irradiation effects on (1) mechanical properties of SiC, SiC/SiC composites and the fiber/matrix (F/M) interface, and (2) microstructural change of pyrolytic carbon (PyC) as the F/M interphase, new HFIR irradiation experiments were planned. Two types of rabbit capsules were designed: (1) composites' compact flexure rabbits for macro-strength evaluation and (2) mini-composites' rabbits for the F/M interfacial evaluation.

### PROGRESS AND STATUS

*Introduction*

SiC/SiC composites are candidates for the advanced blanket system of a fusion DEMO reactor due to the excellent thermo-mechanical and physical/chemical properties [1-3]. Since these composite's functionalities are readily affected by the F/M interfacial properties, the irradiation stability of the interface needs to be proven for the practical design of the DEMO components. Specifically, understanding the correlation between the strength and microstructure is useful. The key issue is, therefore, to identify the dose-dependent strength of the F/M interface coupled with summarizing the irradiation-induced microstructural changes of SiC and PyC as a combined form of the F/M interface. High-dose irradiation effects data are lacking and specifically needed. Identifying the micro- and macro-strength interaction is another issue to be solved.

The effect of neutron irradiation on the F/M interfacial strength was evaluated by adopting the modified shear-lag model, which gives an interfacial debond shear strength and an interfacial friction stress [4-7]. Up to 10 dpa, they were revealed to slightly decrease by irradiation [4, 5]. The probable explanation for such changes is a decreasing surface roughness produced by the irradiation-enhanced change of the cracking path at the F/M interface from inside the PyC interphase to the fiber surface, although this is still uncertain because of the scarce of data of the irradiation-induced morphology change of the PyC interface. In contrast, at higher doses up to 40 dpa, there was no significant degradation of the bend strength, implying no potential degradation of the F/M interfacial strength [6]. Recent work by the authors has shown good irradiation tolerance of the interface up to 40 dpa for certain conditions [7]. This is somewhat surprising since it has been believed that carbon materials are generally unstable at higher dose irradiation. Therefore, the key mechanism of the good irradiation stability is still unsolved and needs to be identified by further experiments.

## DETAILS OF THE TECHNICAL PLAN

*Irradiation Matrix*

The primary scope of this irradiation campaign is to examine the effects of neutron irradiation at high temperatures (~1000 ºC) and high-doses (~100 dpa). Specifically, the following two domains are to be evaluated: (1) mechanical properties of both monolithic chemical-vapor-deposited (CVD) SiC and "nuclear-grade" SiC/SiC composites and micromechanical properties of the F/M interface and (2) nano-structural change of the F/M interface, for materials such as PyC. This program includes 8 rabbits containing compact composite flexure specimens (hereafter JCR-CF) and 4 rabbits containing mini-composite specimens (hereafter JCR-MC). Table 1 shows the irradiation matrix including the specimen types to be irradiated.

Table 1 – Summary of irradiation conditions planned in high-dose rabbit experiments.

| Rabbit ID | Configuration Type | Target Temperature [ºC] | Target Dose [x$10^{25}$ n/m$^2$ fast] | Materials |
|---|---|---|---|---|
| JCR11-01 | JCR-CF | 950±50 | 10 | · Composites for flexure<br>· SiC for thermometry |
| -02 | JCR-CF | 950±50 | 10 | |
| -03 | JCR-CF | 950±50 | 30 | |
| -04 | JCR-CF | 950±50 | 30 | |
| -05 | JCR-CF | 950±50 | 50 | |
| -06 | JCR-CF | 950±50 | 50 | |
| -07 | JCR-CF | 950±50 | 100 | |
| -08 | JCR-CF | 950±50 | 100 | |
| -09 | JCR-MC | 950±50 | 4 | · Mini-composites for the F/M interfacial tests<br>· SiC for equi-biaxial flexure and thermometry |
| -10 | JCR-MC | 950±50 | 10 | |
| -11 | JCR-MC | 950±50 | 30 | |
| -12 | JCR-MC | 950±50 | 100 | |

*Materials and Specimens*

Materials to be irradiated are summarized in Table 2. Four types of SiC/SiC composites are included for compact flexural tests: (1) P/W Hi-Nicalon Type-S/ PyC/CVI-SiC, (2) S/W Hi-Nicalon Type-S/ML/CVI-SiC, (3) P/W Tyranno-SA3/PyC/CVI-SiC and (4) P/W Tyranno-SA3/ML/CVI-SiC (Hyper-Therm High- Temperature Composites, Inc.); where P/W = plain-weave, S/W = satin-weave, and ML = multilayer. The multilayer interface consists of 150 nm PyC as the inner layer surrounded by 4 sequences of 100 nm SiC and 20 nm PyC. The size of the compact flexural specimen is 25 mm (L) x 2.8 mm (W) x 1 mm (T). At least two representative repeating fabric units are included in width of this rectangular composite specimen. The number of plies in thickness is in most cases five or more. The compact flexural tests after irradiation will use the four-point method with a support span of 20 mm and a loading span of 10 mm at room-temperature.

Table 2 – List of materials for irradiation.

| ID | Fiber | Architecture | Interface | | # of spec. per capsule | Capsule Type |
|---|---|---|---|---|---|---|
| | | | Type | $e_{PyC}$ [nm]* | | |
| PW-HS-150 | Hi-Nicalon Type-S | P/W | Monolayer | 150 | 5 | JCR-CF |
| SW-HS-ML | Hi-Nicalon Type-S | 5H S/W | Multilayer | 150 | 5 | |
| PW-SA-150 | Tyranno-SA3 | P/W | Monolayer | 150 | 5 | |
| PW-SA-ML | Tyranno-SA3 | P/W | Multilayer | 150 | 5 | |
| UD-HS-70 | Hi-Nicalon Type-S | UD | Multilayer | 70 | 1 | JCR-MC |
| UD-HS-240 | Hi-Nicalon Type-S | UD | Multilayer | 240 | 1 | |
| UD-HS-1150 | Hi-Nicalon Type-S | UD | Multilayer | 1150 | 1 | |
| UD-SA-70 | Tyranno-SA3 | UD | Multilayer | 70 | 1 | |
| UD-SA-230 | Tyranno-SA3 | UD | Multilayer | 230 | 1 | |
| UD-SA-1000 | Tyranno-SA3 | UD | Multilayer | 1000 | 1 | |
| UD-Syl-150 | Sylramic | UD | Monolayer | 150 | 1 | |
| UD-iBN-0 | Sylramic-iBN | UD | None | 0 | 1 | |
| R&H-CVD | - | - | - | - | 4 | |

*$e_{PyC}$: thickness of the inner PyC interphase adjacent to the fiber

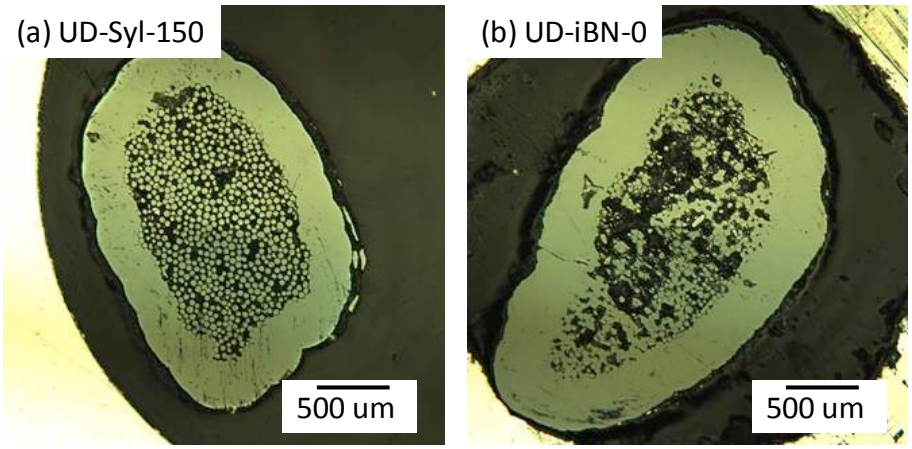


Fig. 1. Typical cross-sectional images of mini-composites: (a) Sylramic/PyC/CVI-SiC and (b) Sylramic- iBN/CVI-SiC.

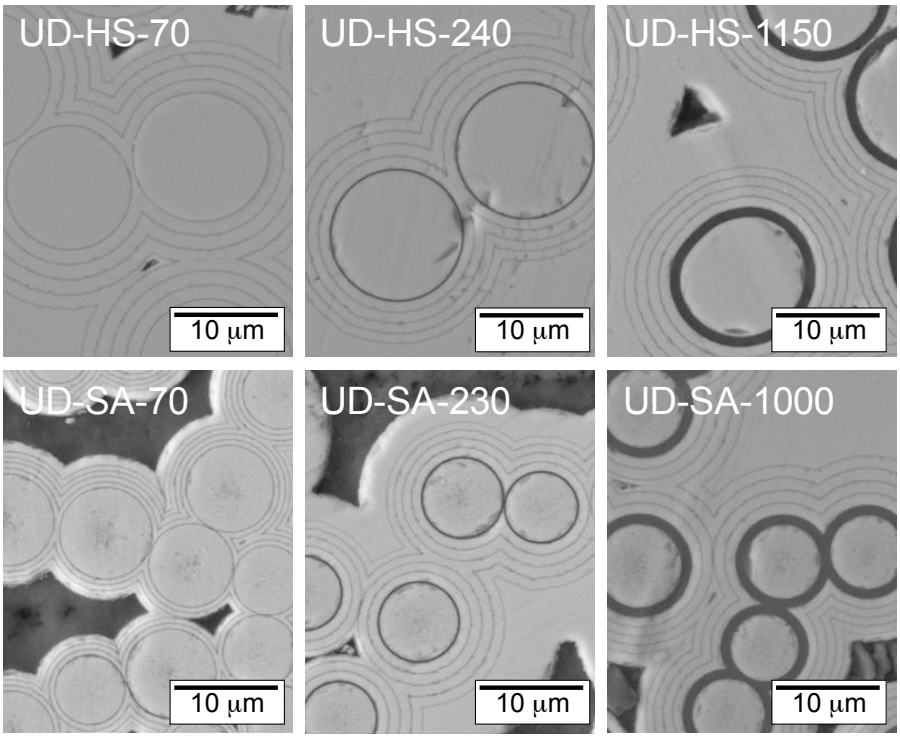


Fig. 2. Typical microstructural images of the F/M interface of mini-composites (UD-HS-70, UD-HS-240, UD-HS-1150, UD-SA-70, UD-SA-230 and UD-SA-1000). No image is presently available for UD-Syl-150 and UD-iBN-0.

Also, eight variations of mini-composites are included to evaluate irradiation tolerance of the F/M interface at high doses and high temperatures (Table 2). The F/M interfacial shear properties and microstructures of PyC will be evaluated by the single fiber push-out test and TEM observation, respectively. Typical cross-sectional images of mini-composites are as shown in Figs. 1 and 2. Thickness of the inner PyC was varied as shown in the table. The subsequent layers are fixed for all types of mini-composites: (1000 nm SiC + 20 nm PyC) x 4 sequences. Fiber volume fractions were approximately 42~50% for UD-Syl-150 and UD-iBN-0 and 5~10 % for the other types. Additionally, a high-resistivity CVD-SiC (Rohm and Haas Co., Ltd., presently The Dow Chemical Company) is used as mechanical test coupons (equi-biaxial flexure) and temperature monitors. A schematic drawing of the equi-biaxial flexural bar is as shown in Fig. 3. This bar has 7 V-shaped grooves on each surface and this can easily be broken into 6 mm x 6 mm pieces. This enables 8 tests from one coupon. A ring-on-ring test with 5 mm support diameter will be used to test these specimens. The experimental details are described elsewhere [8].

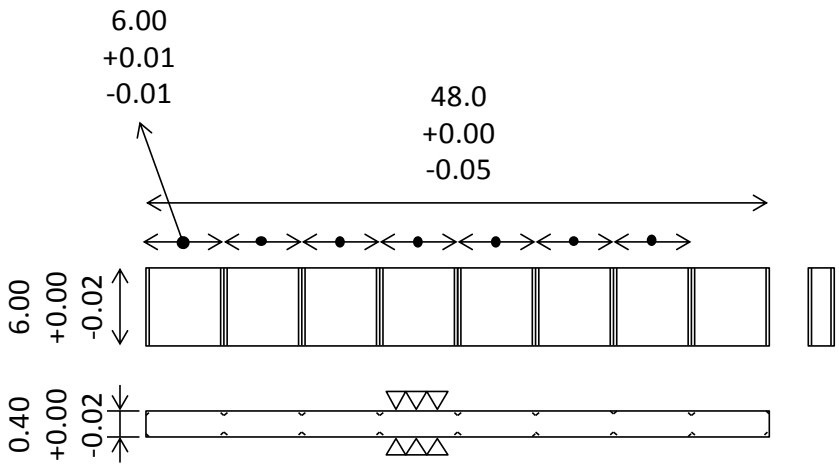


Fig. 3. Schematic drawing of the equi-biaxial flexural specimen (unit in mm).

*Irradiation Capsules*

The standard rabbit capsule design which has a volume of 6.2 x 6.2 x 50 mm and two bend bars can be stored in the standard employment was employed in this study, with the inner configuration modified to meet our requirements. The capsule was made of molybdenum.

A JCR-CF type capsule contains 20 compact flexural (CF) specimens (= 4 composites' type x 5 each) and 4 CVD-SiC temperature monitor (TM) specimens. The loading configuration is shown in Fig. 4. In this configuration, SiC liners should be used to prevent the specimen surfaces from touching the inner capsule surfaces.

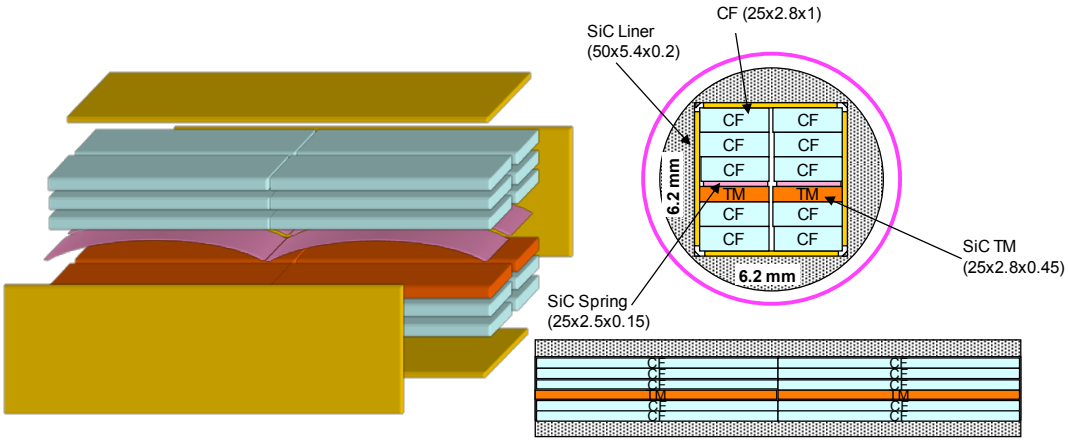


Fig. 4. Schematic drawing of the compact flexure rabbit capsule (unit in mm).

A JCR-MC type capsule contains 8 mini-composite (MC) specimens (= 8 mini-composites' type x 1 each), 4 equi-biaxial flexural (BF) specimens, and 2 CVD-SiC thermometry (TM) specimens.

The loading configuration is shown in Fig. 5. In this configuration, SiC springs (40 x 1.5 x 0.2 mm) will be used to hold mini-composites in place and in good thermal contact. The total number of the MC specimens will be adjusted to fit the available space.

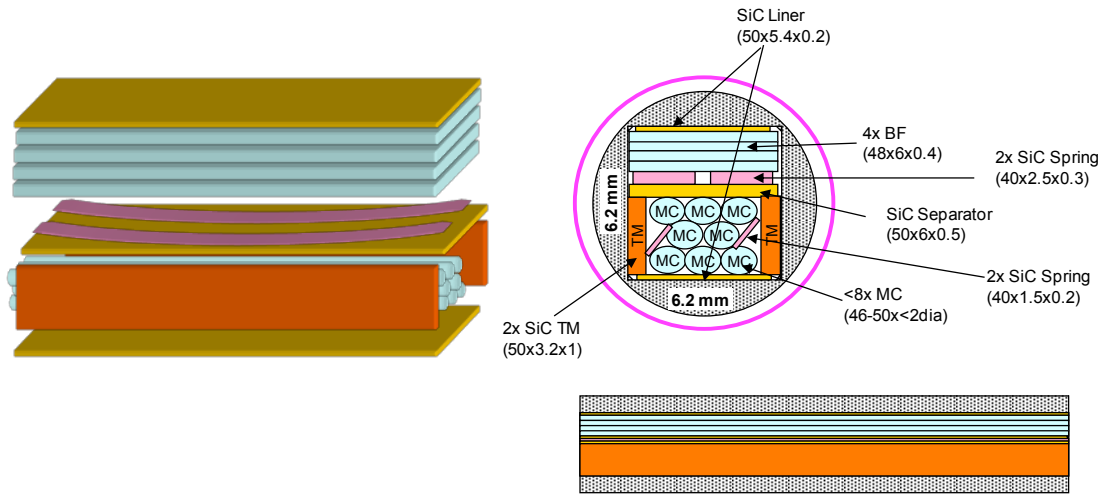


Fig. 5. Schematic drawing of the mini-composite rabbit capsule (unit in mm).

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