2.1 TITAN TASK 2-3 SILICON CARBIDE BEND STRESS RELAXATION CREEP STUDY: PHASE-II EXPERIMENT

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OBJECTIVE

This report provides a brief outline of the technical planning and progress of the Phase-II irradiation experiment for the Task 2-3 on Dynamic Deformation of Fusion Materials, US/Japan TITAN collaboration on fusion materials and blanket technology.

SUMMARY

A study of irradiation creep behavior of silicon carbide ceramics and composites is part of Task 2-3 of the US/Japan TITAN collaboration on fusion materials and blanket technology. In the Phase-I experiment, low fluence irradiation creep behavior of monolithic silicon carbide ceramics was studied using the bend stress relaxation (BSR) technique. The objective of the Phase-II experiment is to gain understanding of the stress relaxation and creep behavior of silicon carbide ceramics, fibers, and composites under neutron irradiation to higher fluences at elevated temperatures. The neutron irradiation will be performed using the fixed rabbit facilities of the High Flux Isotope Reactor. The Phase-II program will irradiate 13 rabbit capsules; 8 for monolithic and composite samples and 5 for fiber samples. Target irradiation temperatures are 300, 500, 800, and 1200°C. The present schedule assumes the initiation of irradiation in early 2011.

PROGRESS AND STATUS

Introduction

Irradiation creep is an important irradiation effect phenomenon for materials for services in high radiation environments, because it is a major contributor to potential dimensional instability of materials under irradiation at such temperatures that thermal creep is not strongly anticipated.[1] Although irradiation creep often determines irradiated lifetime of metallic structural components, it may also be beneficial for inherently brittle materials like ceramics for functional applications, because creep may relax or redistribute the stresses.[2] For silicon carbide (SiC)-based nuclear components, the latter function of irradiation creep may be important, in particular when a significant temperature gradient exists and the secondary stresses developed by differential swelling can be severe. Flow channel insert in liquid metal blankets of fusion energy systems is an example of such applications.[4]

Studies on neutron irradiation creep of SiC have so far been extremely limited and insufficient. Price published the result of the irradiation creep study on chemically vapor-deposited (CVD) SiC in 1977.[6] In that work, elastically bent strip samples were irradiated in a fission reactor, and the linear-averaged creep compliance was estimated to be in the order of 10^{-38} [Pa•n/m^2 (E > 0.18 MeV)]$^{-1}$, or 10^{-7} [MPa•dpa]$^{-1}$, in a temperature range 780 - 1130°C. This result may involve very significant uncertainty because stresses in some of the samples appeared to have been completely relaxed at the end of irradiation. However, the experimental method of estimating the creep parameters based on the stress relaxation in elastically bent strip samples developed by Price was later adopted to examine the thermal creep behavior of refractory ceramic fibers and was named bend stress relaxation (BSR) method by Morscher and DiCalro.[5]

In more recent work, Katoh et al. applied it for studying the irradiation creep of high purity, stoichiometric CVD SiC, demonstrating the applicability of the BSR technique to determine the irradiation creep parameters.[3] They also revealed that the creep strains SiC were dominated by transient creep at

temperatures below ~950°C whereas steady-state creep may operate at higher temperatures with a compliance of $1.5 \pm 0.8 \times 10^{-6}$ [MPa•dpa]$^{-1}$ with the initial flexural stress magnitude ~100 MPa. However, fundamental aspects of the irradiation creep, including the effect of stress magnitude on the creep strain rate, the correlation of irradiation creep and swelling, and the responsible physical mechanism, remain to be understood.

Based on the previous demonstration of the experimental technique and the recognized importance of the irradiation creep, a more detailed study on the BSR irradiation creep of SiC ceramics and composites was planned as the main focus of Task 2-3 on dynamic deformation of fusion materials in the US Department of Energy /Japan MEXT (Ministry of Education, Culture, Sports, Science and Technology) TITAN Collaboration. In the Phase-I study, understanding of the transient creep and stress relaxation behavior of various SiC ceramics, including high purity, polycrystalline beta-phase SiC, monocrystalline alpha SiC, and liquid phase-sintered SiC, during neutron irradiation was obtained. The Phase-I study also demonstrated that the rapid stress relaxation in SiC at the onset of irradiation is very substantially suppressed when the as-grown defects are annealed by pre-irradiation heat treatment, with large fractions of the initial stress remaining after low fluence irradiation. This result indicates that the crystalline SiC with sufficiently low as-grown defect density makes an exceptionally radiation-resistant spring material. Moreover, it indicates that the stress relaxation technique may be used for experiments to higher fluences. The Phase-II study is intended to explore the irradiation creep or stress relaxation behavior of monolithic SiC, SiC fibers, and SiC composites.

Technical Plan Details

Irradiation Matrix

The Phase-II irradiation program consists of irradiation of 13 rabbit capsules including 8 rabbits for monolithic and composite materials and 5 rabbits for fiber specimens. The irradiation matrix is shown in Table 1.

Table 1 – Irradiation conditions planned for Phase-II SiC irradiation creep.

Capsule ID in Titan	Capsule Type	Target Temperature (ºC)	Target Dose ($x10^{25}$ n/m^2 fast)	Materials	Facility
T10-01J	Titan-BSR1	300	1	Monolithic and Composites	PTP or TH
T10-02J	Titan-BSR1	300	10		
T10-03J	Titan-BSR1	300	20		
T10-04J	Titan-BSR1	500	10		
T10-05J	Titan-BSR1	500	20		
T10-06J	Titan-BSR1	800	10		
T10-07J	Titan-BSR1	800	20		
T10-08J	Titan-BSR1	1200	10		
T10-09J	Titan-BSR2	500	1	Fibers	PTP or TH
T10-10J	Titan-BSR2	500	10		
T10-11J	Titan-BSR2	500	20		
T10-12J	Titan-BSR2	1200	1		
T10-13J	Titan-BSR2	1200	10		

Irradiation Capsules

The standard "SiC Bend Bar" type capsule housing configurations are employed, using the existing HFIR capsule design X3E020977A325. Inside the standard sleeve, which holds two ceramic bend bar samples in the standard configuration, a rectangular casing (denoted "coffin" hereafter) is accommodated for the Titan-BSR1 configuration. Both the sleeves and the coffins were made of molybdenum. In the Titan-BSR2 configuration, ceramic fiber specimens are accommodated in fixture made of nuclear grade graphite.

Each coffin for the Titan-BSR1 configuration consists of a straight rectangular tube with end pillars on both ends, Fig. 1. The end pillars are for 1) fixing the internal parts and specimens in appropriate positions, and 2) retaining the internal parts and specimens during the capsule disassembly process until right before the specimen examination in the post-irradiation examination facility. The end pillars are made of the same material with the tube.

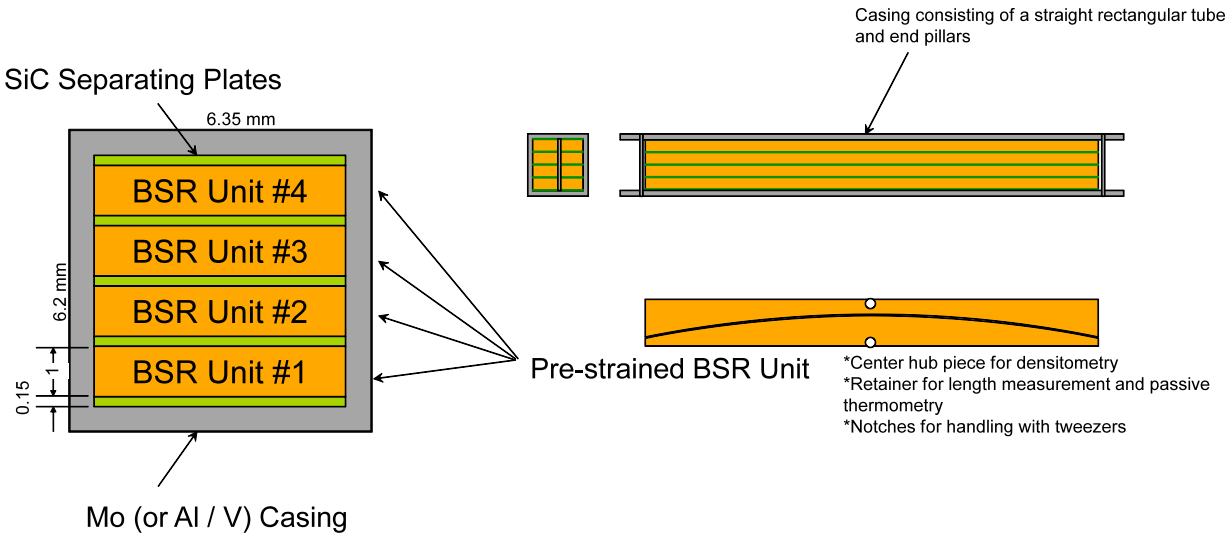

Fig. 1 – Schematic illustration of monolithic/composite SiC ceramics BSR fixture and loading into coffin.

Thin strip specimens of monolithic ceramics and ceramic composites are loaded into fixture, making an individual BSR unit. The fixture, once held in the coffin, keeps the specimens uniformly bent to the pre-determined curvatures, which are typically 100 mm radius for the monolithic specimens and 200 mm radius for the composite specimens. Fig. 2 shows the specimens in fixture, held in an assembly tool, along with the specimens and nickel foils used for in-situ helium injection (as described in the next section).

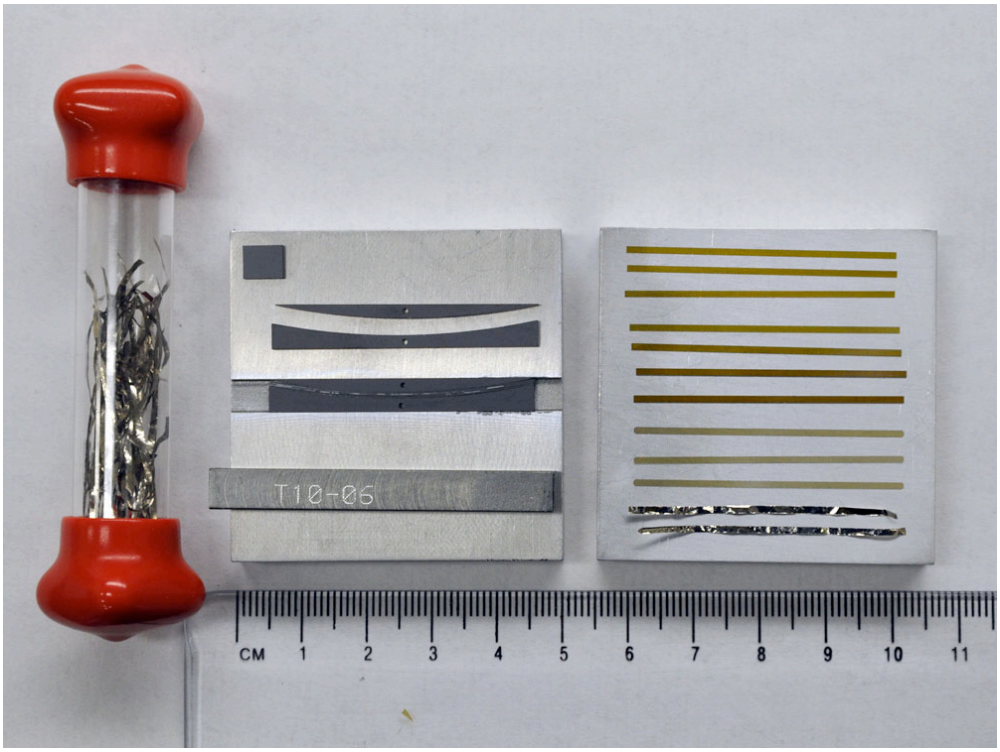

Fig. 2 – CVD SiC BSR fixture with specimens loaded in assembly tool (middle), monolithic ceramic specimens before loading (right), and nickel foil strips (left) for in-situ helium injection.

Four pre-strained BSR units are accommodated in each coffin. Each unit is of size approximately 48 mm x 5.1 mm x 1 mm. The BSR assembly units are stacked together with CVD SiC liners/separator plates at the top, bottom, and between the units, Fig. 3. The CVD SiC liners/separators prevent potential undesirable reaction between the samples and the metallic parts or fixture, as well as help aligning the BSR assembly units in proper positions.

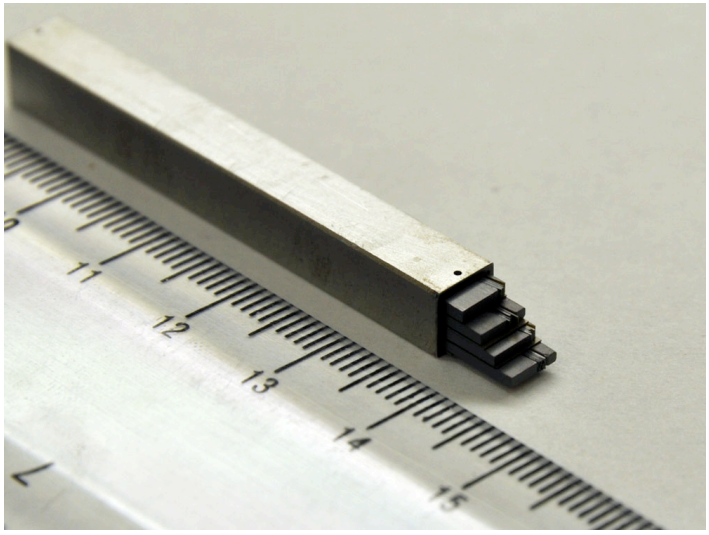

Fig. 3 – Photograph showing a set of four monolithic/composite SiC ceramic BSR units being loaded, separated by SiC sheets, into a molybdenum coffin.

In the Titan-BSR2 configuration, instead of the coffin, a pair of fiber BSR units are accommodated in the sleeve. Each assembled unit measures 25 mm x 6.35 mm x 6.22 mm and holds fibers in two separate bundles. The fiber bundles are elastically bent to three different curvatures as shown in Fig. 4. The unit is held together by a graphite pin penetrating throughout the fixture length with help by the spring force of the fiber samples. Appearance of the assembled fiber BSR unit is shown in Fig. 5.

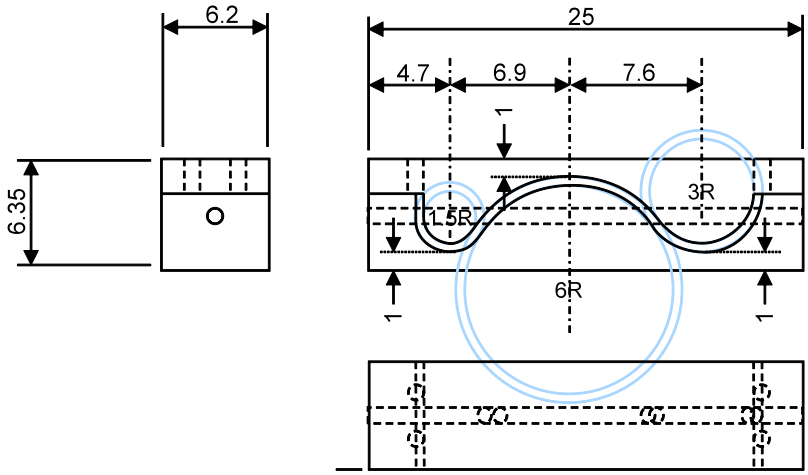

Fig. 4 – Graphite fixture for fiber BSR irradiation creep.

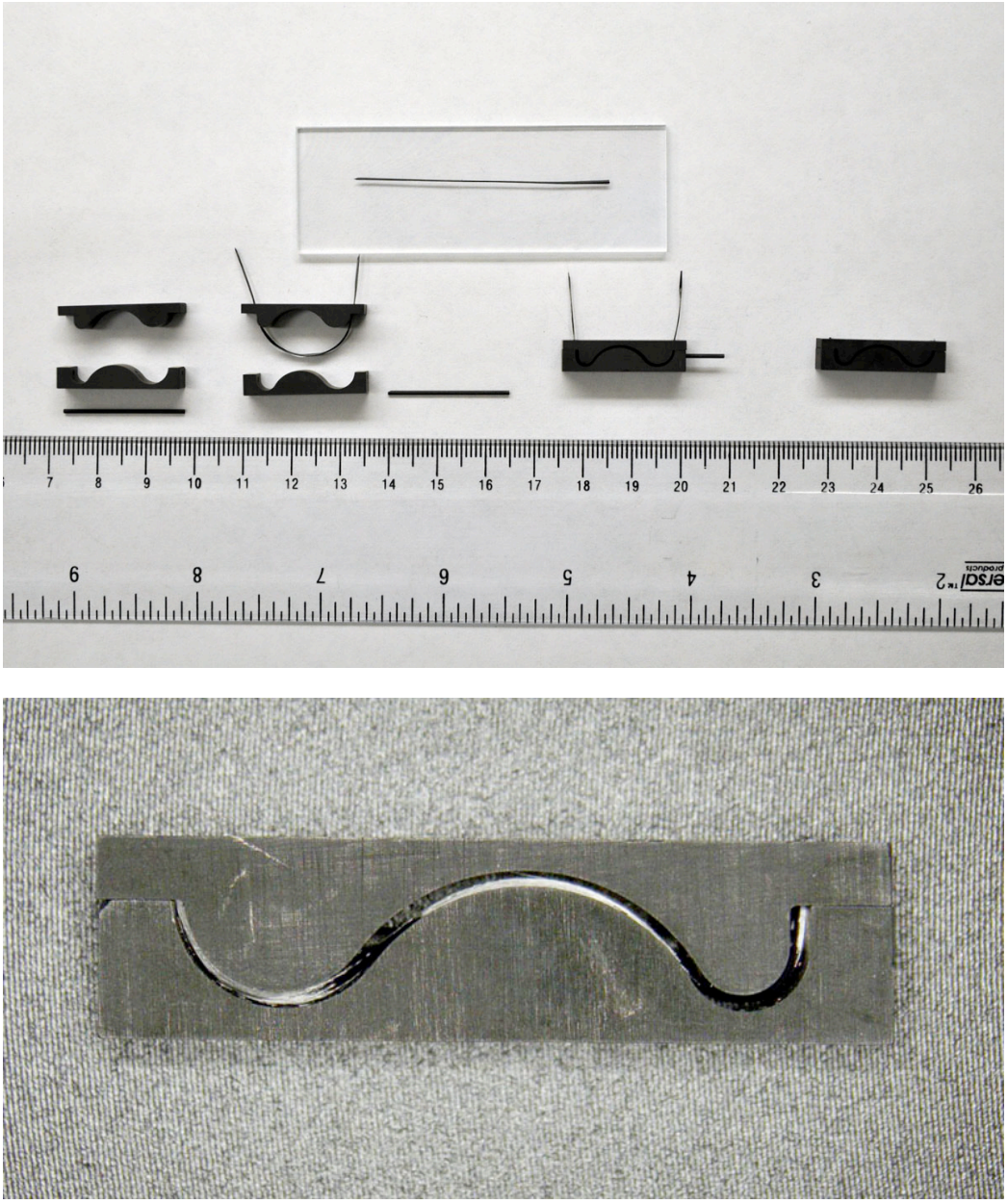

Fig. 5 – Photograph showing assembly process for fiber BSR unit (top) and magnified view of assembled unit (bottom).

Materials and Specimens

Monolithic and composite materials to be studied and the specimen loading to each rabbit are summarized in Table 2. The Cree 4H single crystal samples were machined for the surface parallel with {0001} and the length parallel with <1120>.

The dimensions of the BSR creep specimens are 40 mm (l) x 1 mm (w) x varied thickness. Some of the materials were machined into slightly reduced width for materials identification. The thickness was varied to allow determination of the effect of stress magnitude. Typical initial flexural stresses for the specimen thicknesses 0.05, 0.1, and 0.15 mm are ~100, ~200, and ~300 MPa, respectively, for monolithic samples. Typical initial flexural stresses for the composite specimens are ~100 and ~200 MPa for thicknesses 0.1 nad 0.2 mm, respectively. Some of the sample strips are loaded with thin nickel foils on both faces or one face for the purpose of in-situ helium injection. The inclusion of nickel foils was limited to capsules for irradiation at below 500°C to avoid reaction with the SiC samples.

Table 2 – SiC materials and specimens for the BSR creep experiment.

Material ID	Material	Specimen Thickness (mm)	Typical # of specimens in a rabbit
RH	R&H CVD-SiC, polycrystalline beta SiC	0.05 0.10 0.15	2 4 4
CT	Coorstek CVD SiC polycrystalline beta SiC	0.05 0.10 0.15	1 1 1
SX	Cree 4H SiC, W4NRF0X-0D00, Lot# FX0778-28/161430	0.05 0.10 0.15	1 1 1
NT1	NITE matrix material, standard	0.075 0.10 0.15	1 2 1
NT2	NITE matrix material, reduced additives	0.05	2
NITE	NITE SiC/SiC composite, uni-directional	0.10 0.20	2 2
RH with Ni	R&H CVD-SiC, in contact with nickel 2 micron-thick foil(s)	0.05	3
CT with Ni	Coorstek CVD-SiC, in contact with nickel 2 micron-thick foil(s)	0.05	3

The ceramic fiber materials to be studied and the specimen loading to each rabbit are given in Table 3. The fibers are bent to three different nominal curvature radii, namely 6, 3, and 1.5 mm. The anticipated flexural stresses for typical SiC fibers with 0.01 mm-diameter and Young's modulus 400 GPa are 333, 667, and 1333 MPa, respectively, ignoring Poisson effect. An example of the actually measured curvature profile is presented in Fig. 6.

Table 3 – Materials for the fiber BSR creep experiment.

Material ID	Material	Fiber Diameter (micron)
HNLS	Hi-Nicalon™ Type-S	~11
SA3	Tyranno™-SA3	~7.5
Syl-B11	Experimental Sylramic	~10
Syl-iB11N	Experimental Sylramic-iBN	~10

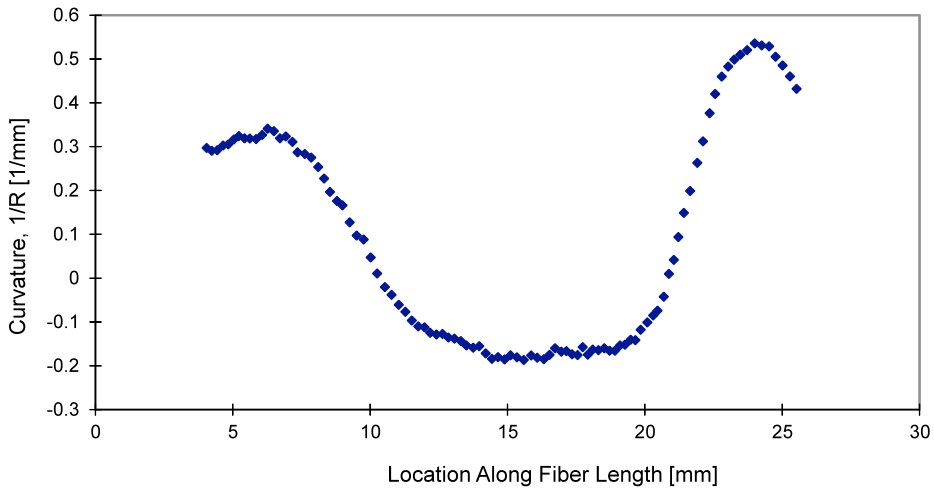

Fig. 6 – An example curvature profile of a SiC fiber specimen loaded in fiber BSR fixture.

References

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