TITAN TASK 2-3 SILICON CARBIDE BEND STRESS RELAXATION CREEP EXPERIMENT —

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OBJECTIVE

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

SUMMARY

A study on transient irradiation creep deformation of silicon carbide is being undertaken as a part of Task 2-3 of the US/Japan TITAN collaboration on fusion materials and blanket technology. The Phase-IA program is the first of the two irradiation campaigns planned for studying the bend stress relaxation (BSR) creep of ceramics and composites under neutron irradiation. The objective of experiment is to gain understanding of the stress relaxation and transient creep behavior of SiC composites and constituents under neutron irradiation at elevated temperatures. The neutron irradiation will be performed using hydraulic and fixed rabbit facilities of the High Flux Isotope Reactor. The Phase-IA program irradiates 8 rabbit capsules; 5 in hydraulic and 3 in fixed facility. Target irradiation temperatures in the Phase-I are 300, 500, 800, and 1200ºC. The minimum number of rabbit capsules to be irradiated in entire Phase-I campaign will be 15. The present schedule assumes the initiation of irradiation in early 2009.

PROGRESS AND STATUS

Introduction

Irradiation creep is an important irradiation effect phenomenon for materials for radiation services, 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 is not necessarily undesirable for brittle materials like ceramics for functional applications, because it may help relaxing or redistributing 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 - 1130ºC. 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 a recent work, Katoh et al. applied it for studying the irradiation creep of high purity, stoichiometric CVD SiC, demonstrating that the BSR technique is effective to determine the irradiation creep parameters. [3] They also revealed that the creep strains SiC were dominated by transient creep at temperatures below ~950ºC whereas steady-state creep is likely to dominate 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 Phase-I program of Task 2-3 on dynamic deformation of fusion materials in the US Department of Energy /Japan MEXT (Ministry of of Education, Culture, Sports, Science and Technology) TITAN Collaboration. The objective of the Phase-I study is to gain understanding of the transient creep and stress relaxation behavior of SiC ceramics, model composites, and composite constituents during neutron irradiation. More specifically, the study aims at determining the dose, temperature, and stress dependent BSR behavior of those materials and the correlation between the point defect swelling and the transient creep deformation.

Technical Plan Details

Irradiation Matrix

The Phase-IA irradiation program consists of irradiation of 8 rabbit capsules including 5 hydraulic rabbits and 3 fixed rabbits. The planned irradiation matrix is shown in Table 1. The irradiation conditions were chosen so that 1) the temperature range of technological interest is covered, 2) the irradiation creep – swelling coupling could be studied in a broad range of swelling magnitude, and 3) the effect of irradiation temperature may be compared both at the same dose and at the similar swelling magnitude.

Table 1 – Irradiation conditions planned for Phase-IA.

Capsule ID in Titan	Capsule Type	Target Temperature (ºC)	Target Dose ($x10^{25}$ n/m^2 fast)	Anticipated Swelling	Facility
T08-01J	Titan-BSR1	300	0.01	~0.2%	Hydraulic
T08-02J	Titan-BSR1	300	0.1	~1.0%	Hydraulic
T08-03J	Titan-BSR1	500	0.01	~0.1%	Hydraulic
T08-04J	Titan-BSR1	500	0.1	~0.6%	Hydraulic
T08-05J	Titan-BSR1	500	1	~1.2%	PTP or TH
T08-06J	Titan-BSR1	800	0.1	~0.1%	Hydraulic
T08-07J	Titan-BSR1	800	1	~0.6%	PTP or TH
T08-08J	Titan-BSR1	1200	1	~0.1%	PTP or TH

Irradiation Capsules

The standard “SiC Bend Bar” type capsule housing configuration 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. Both the sleeves and the coffins were made of molybdenum.

Each coffin 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. The end pillars have to withstand the transient mechanical loading during the operation of the hydraulic rabbit facility.

Four BSR assembly units are accommodated in each coffin. Each BSR assembly 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. 2. 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.

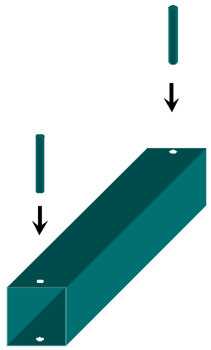

Fig.1 – A schematic illustration of the “coffin” container with retaining pillars.

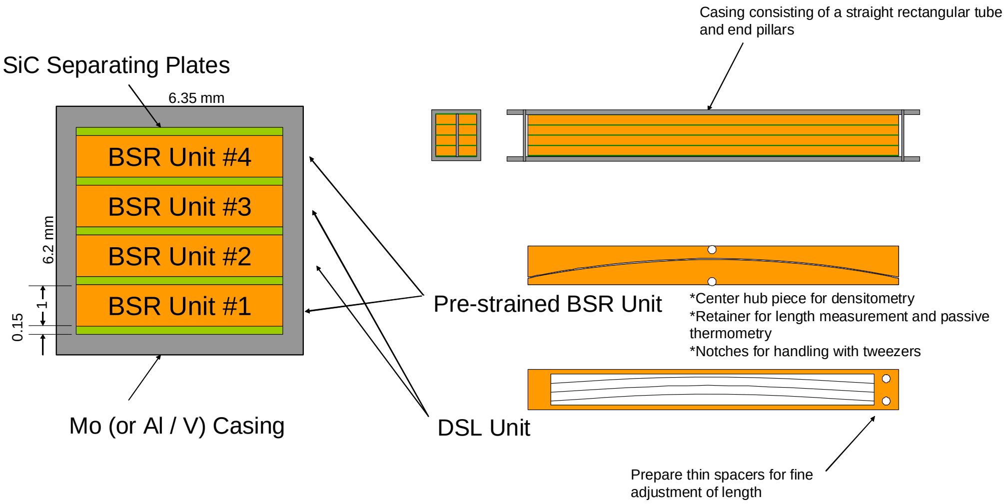

Fig. 2 – An illustration of BSR assembly units and their stacking in a metallic casing.

BSR Assembly

Two types of BSR assembly units are used: 1) conventional pre-strained (PS) BSR and 2) dynamic self-loading (DSL) BSR (Fig. 2). For each rabbit, two PS units and two DSL units are irradiated. Each unit contains several BSR strip specimens. The PS units are consisted of a pair of fixture which is made of CVD SiC. The frame for the DSL unit is made of same material with the coffin. Discussion about the DSL creep experiment is provided in a separate section.

The components of the BSR assembly and the capsule internal are shown in Fig. 3. The BSR assembly modules (fixtures of both types loaded with the specimens) are shown in Fig. 4. The assembled modules being loaded into the coffin are shown in Fig. 5.

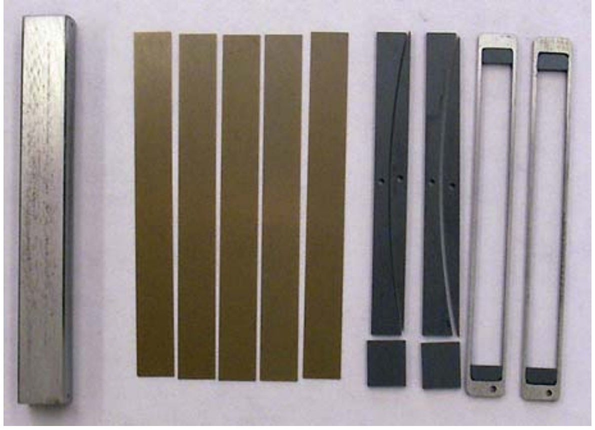

Fig.3. Components for the BSR assembly and the capsule internals. From left to right, coffin, CVD SiC lining/separating sheets, pre-strained BSR fixtures (and spacers), and dynamic self-loading fixtures.

Fig.4. Dynamic self-loading BSR fixture (middle) and pre-strained BSR fixture (bottom) both loaded with specimens. Molybdenum coffin is shown on the top.

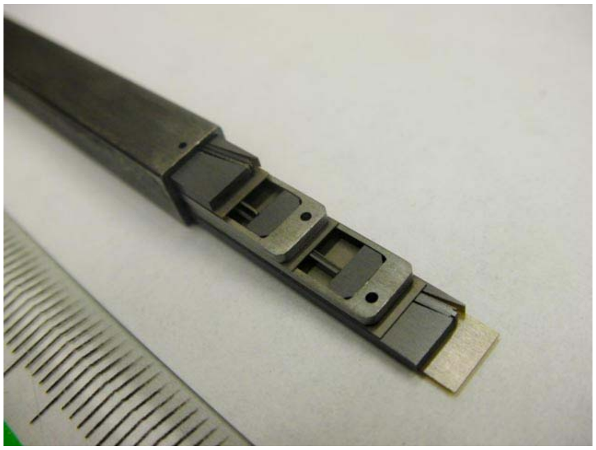

Fig.5. Loading of BSR assembly modules into the coffin. Two dynamic self-loading BSR modules are loaded in the middle and two pre-strained BSR modules are loaded on the top and the bottom. Each module and the internal wall of the coffin are separated with CVD SiC sheets.

Materials and Specimens

Materials to be studied and specimen loading to each rabbit are summarized in Table 3. The Cree 6H 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 thicknesses 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.

Table 3 – Materials and specimens for the BSR creep experiment. Number of specimens per rabbit capsule. Parenthesis indicates specimens are backup.

Material ID	Material	Specimen Thickness (mm)	# of specimens in PS units	# of specimens in DSL units
RH	R&H CVD-SiC, polycrystalline beta SiC	0.05	2	
		0.10	1	1
		0.15	1	1
		0.20		1
CT	Coorstek CVD SiC polycrystalline beta SiC	0.05	1	
		0.10	1	
		0.15	1	
SX	Cree 4H SiC, W4NRF0X-0D00, Lot# FX0778-28/161430	0.05	1	
		0.10	1	
NT1	NITE matrix material, standard	0.075	1	
		0.10	1	1
		0.15		1
		0.20		1
NT2	NITE matrix material, reduced additives	0.05	2	
		0.10	1	
		0.15	1	

Dynamic Self-Loading Technique

The dynamic self-loading (DSL) is an experimental technique that makes use of the isotropic swelling of SiC strip sample to apply a flexural strain to the sample itself. When a flat strip sample mechanically constrained at both ends undergoes longitudinal expansion, the sample elastically bows out developing flexural loading. The extent of bow-out and the flexural strain are calculated and shown in Fig. 6 as a function of linear swelling. Advantages of the DSL technique include 1) flexural strain in excess of the fast fracture strain (typically <0.1% for SiC) can be applied, and 2) early stress relaxation due to the transient irradiation creep may be compensated by the additional strain application with the progress of irradiation. The DSL experiment, if successfully performed, helps determining adequacy of theory and models of irradiation creep. With a known stress exponent for the irradiation creep rate and an adequate model, irradiation creep parameter such as a swelling-creep coupling coefficient may be determined by the DSL experiment.

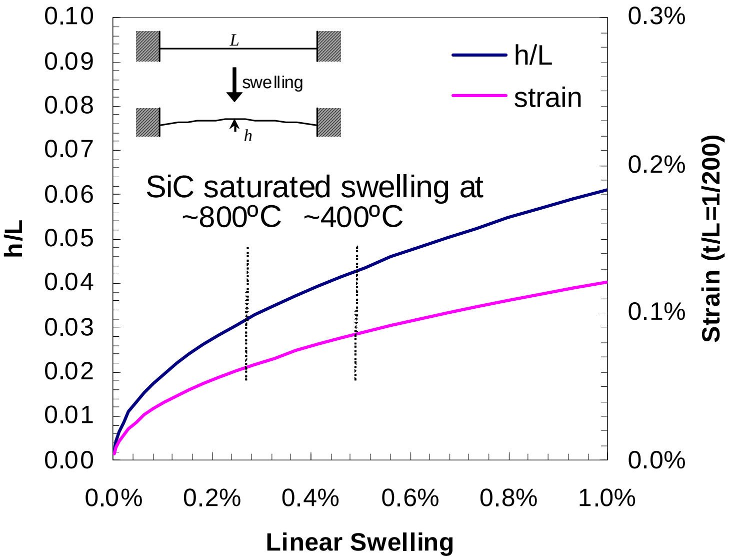

Fig.6. Calculation of bow-out magnitude (h/L) and flexural strain as a function of linear swelling. L = specimen length, h = bow-out height, and t = specimen thickness. Initial h was set to zero in this calculation.

References

[1] K. Ehrlich, Journal of Nuclear Materials 100 (1981) 149-166.
[2] Y. Katoh, S. Kondo, and L.L. Snead, Journal of Nuclear Materials 382 (2008) 170-175.
[3] Y. Katoh, and L.L. Snead, Ceramic Engineering and Science Proceedings 26 (2005) 265-272.
[4] N.B. Morley, Y. Katoh, S. Malang, B.A. Pint, A.R. Raffray, S. Sharafat, S. Smolentsev, and G.E. Youngblood, Fusion Engineering and Design 83 (2008) 920-927.
[5] G.N. Morscher, and J.A. DiCarlo, Journal of the American Ceramic Society 75 (1992) 136-40.
[6] R.J. Price, Nuclear Technology 35 (1977) 320-336.