TECHNIQUE FOR MEASURING IRRADIATION CREEP IN POLYCRYSTALLINE SiC FIBERS - G. E. Youngblood, M. L. Hamilton and R. H. Jones (Pacific Northwest National Laboratory)*

OBJECTIVE

The objective of this work is to examine irradiation enhanced creep behavior in polycrystalline SiC fibers. This data will be used in conjunction with results from irradiation creep of SiC/SiC composites to help develop a predictive creep equation for composite materials.

SUMMARY

A bend stress relaxation (BSR) test has been designed to examine irradiation enhanced creep in polycrystalline SiC fibers being considered for fiber reinforcement in SiC/SiC composite. Thermal creep results on Nicalon-CG and Hi-Nicalon were shown to be consistent with previously published data with Hi-Nicalon showing about a 100°C improvement in creep resistance. Preliminary data was also obtained on Nicalon-S that demonstrated that its creep resistance is greater than that of Hi-Nicalon.

PROGRESS AND STATUS

Introduction

A previous review examined experimental work carried out by General Atomics in the 1970's on irradiation creep in monolithic SiC.[1,2] In this work a higher rate of creep compared to thermal creep was observed, and surprisingly, the creep in this high temperature ceramic exhibited some similarity to irradiation-enhanced creep in metals. Based on this review, a systematic examination of the creep behavior of SiC/SiC composite and SiC fibers has been initiated. In particular, creep behavior of SiC/SiC composite and SiC fiber will be examined for fusion energy relevant temperatures, times and irradiation doses.

To examine creep behavior in SiC composite, a pressurized thin-walled metallic bladder will impose a constant hoop stress to a surrounding SiC/SiC tube. By a laser interference technique the circumferential strain in the SiC/SiC tube will be determined as a function of applied stress, temperature and dose. The SiC/SiC tubes will be made with different fiber types and matrix structures. Since composite creep is expected to be dominated by fiber creep, a simple bend stress relaxation (BSR) test to examine fiber creep behavior alone will be carried out in parallel to the composite creep tube test. This report presents some preliminary results for thermal control BSR tests and describes some conditions for the planned irradiated fiber BSR test.

Experimental Procedure

Theoretical and experimental details of the BSR test, developed and presented by researchers at NASA Lewis[3-7], will be closely followed. In Fig. 1, a schematic representation of a BSR test, modified to also examine the influence of irradiation, is depicted. An initial elastic bend strain (e_o) is imposed on a fiber by wrapping several coiled loops within a slightly recessed region along a SiC mandrel. The loops are captured by a split SiC jig with a known inside radius R_o. The split jig itself

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is held together within a surrounding protective SiC sleeve (not shown in the figure). The split jig and sleeve are designed to hold the fiber loop configurations during the handling necessary for the irradiation treatment within the core of a reactor. The fiber loops are then subjected to a specific time (t), temperature (T), and irradiation dose (Φ), i.e., a high temperature (HTtΦ) treatment. To separate thermal creep effects, the test also is carried out without irradiation for equivalent times and temperatures, i.e., a high temperature (HTt) treatment.

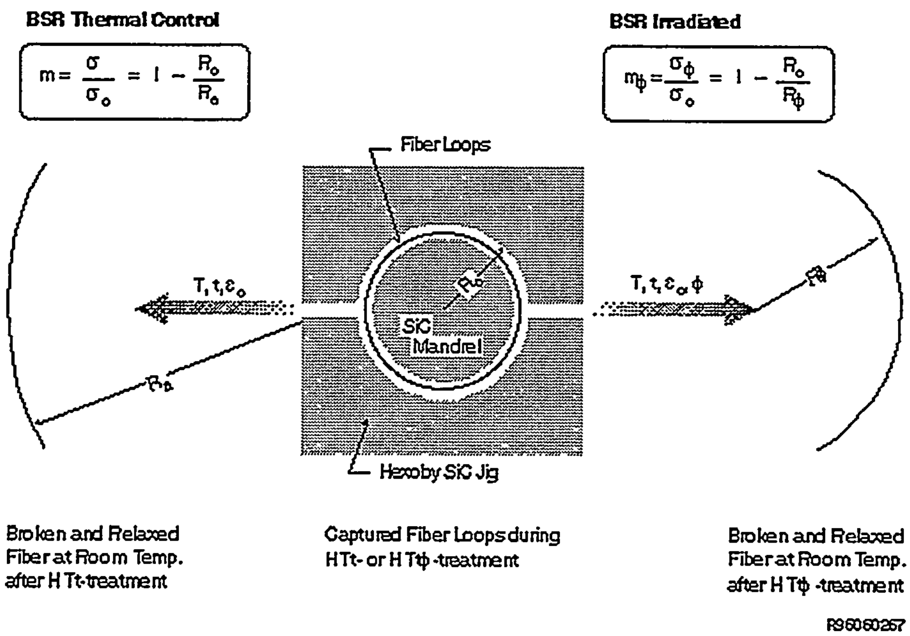


Figure 1. Schematic representation of a BSR fiber test to examine irradiation creep.

A BSR parameter "m", defined by Morscher and DiCarlo as the ratio of final to initial stress[3], quantifies the stress relaxation that occurs during either treatment:

$$m = \sigma(T,t,\Phi)/\sigma(T,0,0) = 1/[1 + (e_c/e_o)] \quad (1)$$

In Eq. (1), e_c and e_o are the total treatment-induced creep strain and the local initial elastic strain, respectively. Subject to several assumptions, m can be experimentally determined by the relations:

$$m = 1 - R_o/R_a \quad \text{(BSR thermal control)} \quad (2)$$

$$m = 1 - R_o/R_\Phi \quad \text{(BSR Irradiated)} \quad (3)$$

where R_a and R_Φ are the arc radii of the broken and relaxed fiber segments at room temperature after the HTtΦ or the HTt treatments, respectively. Values of m range from 1 to 0. If m = 1, the fiber behaved elastically or did not relax (creep) for the given time/temperature treatment. If m = 0, the fiber completely relaxed and retained the curvature subjected by the BSR jig. If 1 >m > 0, the fiber partially relaxed during the treatment.

The assumptions necessary to arrive at the simple geometric relations given by Eqs. (2) and (3) have been critically examined by Morscher and DiCarlo.[3,4,6] They found that e_c generally was linearly dependent on stress, as expected for polycrystalline materials with stress relaxation (creep) due to grain boundary sliding mechanisms. This means that creep at each local position within the bent fiber follows the same time-temperature dependence. In pure bending, the local applied strains (held constant during treatment) increase linearly from the fiber neutral central plane to reach a maximum ($e_o = r_f/R_o$ where r_f is the fiber radius) at the fiber outer surface. Morscher and DiCarlo also observed that m generally was independent of most initial values of e_o, i.e., initial values of R_o. Finally, in an analysis of creep in polycrystalline -SiC fibers, Morscher et al[6] determined that creep determined by the BSR method was a consistent factor of two lower than that determined by a conventional tensile method. However, creep determined by either method exhibited the same time and temperature dependence. Hence fiber tensile creep, if desired, can be estimated from results for the much easier to perform BSR test.

Thermal BSR tests were performed initially on four different polymer-derived SiC fiber types. Relevant properties are given in Table 1 for the tested fiber types, which are listed in order of increasing creep resistance.

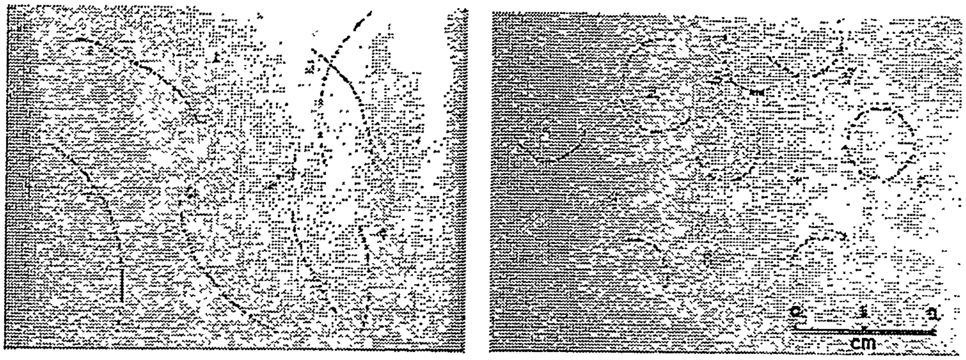
Table 1. Fiber Specimens and Properties

Type	Diameter (um)	Primary Phases	Elastic Modulus (GPa)
Tyranno S#	6.9±0.5	β-SiC, Si-O-C-Ti + C	190
Nicalon-CG^	11.4±2.5	β-SiC, Si-O-C + C	190
Hi-Nicalon^	12.0±1.6	β-SiC + C	270
Nicalon-S^	11.3±1.1	β-SiC	420

#UBE Industries, Tokyo, Japan; ^Nippon Carbon Co., Tokyo, Japan.

Except for the developmental fiber Nicalon-S, NASA Lewis has published background thermal BSR creep data for these fibers. Because the irradiation performance of monolithic SiC is quite attractive for fusion energy applications and because the Nicalon-S composition is near stoichiometric SiC, the irradiation performance of Nicalon-S fiber also is expected to be attractive. Thus, the detailed examination of the irradiation performance of Nicalon-S fiber in comparison to other SiC fibers is of current high interest. The primary goal of these initial thermal BSR tests was to assess the reliability of the modified jig and experimental procedure for use later in reactor tests. In these initial tests, several loops of a single fiber type were coiled at constant tension (≈ 7 MPa) around an alumina mandrel. The ends of the coil were temporarily held in place with a drop of Duco cement. An alumina sleeve with a 4.89 mm inside diameter, which set the applied strain at 0.24% for each case, was slipped over the fiber-wound mandrel to permanently hold the fiber loops in place during their thermal treatments. The initial maximum bend stresses, which depend on the fiber elastic modulus, ranged from 446 to 970 MPa. The alumina jigs containing the fibers to be treated were rapidly heated (5°C/min) inside a temperature controlled tube furnace and held for either one or twenty hours at 1000 or 1100°C. The BSR testing all took place in flowing argon (≈0.5 scfh) at approximately atmospheric pressure.

At the conclusion of the BSR test, the fiber coils either naturally broke up or were cut into several arc segments. Two sets of typical relaxed segments are shown in Fig. 2. The radii of the individual arc segments were determined with ruler and compass from x13 images.



a) Nicalon-S b) Hi-Nicalon

Figure 2. Fiber segments of Nicalon-S and Hi-Nicalon after thermal BSR testing for 20 h at 1100°C in argon. Segment radius of curvature is related to creep resistance and m-value through Eqs. 1-3.

Results and Discussion

Qualitatively from Fig. 2, it is easy to ascertain that Hi-Nicalon fibers exhibit significantly more relaxation (creep) than Nicalon-S fibers for the BSR treatment conditions: 1100°C, 20 hours, argon and $e_o = 0.24\%$.

In Fig. 3, m values determined for our BSR tests on Nicalon-S, Hi-Nicalon and Nicalon-CG fibers are compared to NASA Lewis BSR data for Hi-Nicalon and Nicalon-CG. In our initial test, only two Nicalon-CG segments were recovered for measurements, thus the error bars were quite large (±0.09). To reduce chances for accidental loss of experimental information, an important consideration for the planned reactor experiments, in later tests two separate coils of each fiber type were wrapped around the same mandrel. By determining the average relaxed radii of curvature for 8-10 segments, the typical error bars were reduced to less than ±0.04 standard deviation. For the Nicalon-S fiber test, one of the coils inadvertently unraveled. The error bars for the average m value determined for the remaining three segments were somewhat larger (±0.05). In this figure, the m values for the three 20 hour PNNL BSR tests fall between the data for the 1 and 100 hour NASA Lewis BSR tests, as expected. The m value shown for the PNNL 1 hour Nicalon-CG test coincides with the NASA Lewis data for that fiber. One hour BSR test results for the Tyranno S fiber at 1000 and 1100°C (data not shown) also agreed with NASA Lewis BSR results. It is obvious that the BSR test provides a convenient and reproducible method for evaluating fiber creep resistance.

The transition temperature range, where the m values appear to change from 1 to 0 over a rather narrow ≈ 200°C range, is an important concept. DiCarlo has shown that tensile strength data for most polycrystalline fibers exhibit a decrease at test temperatures where the rapid stress relaxation transition occurs.[3] The treatment conditions that lead to an m value of ≈0.5 might then be used as a practical indication of a fiber use temperature limit. Thus, for a 100 hour exposure, Nicalon-CG and Hi-Nicalon fibers would have an upper use temperature of about 1000 and 1120°C, respectively. Unfortunately, as yet BSR tests have not been completed to high enough temperature to define this transition for the Nicalon-S fiber.

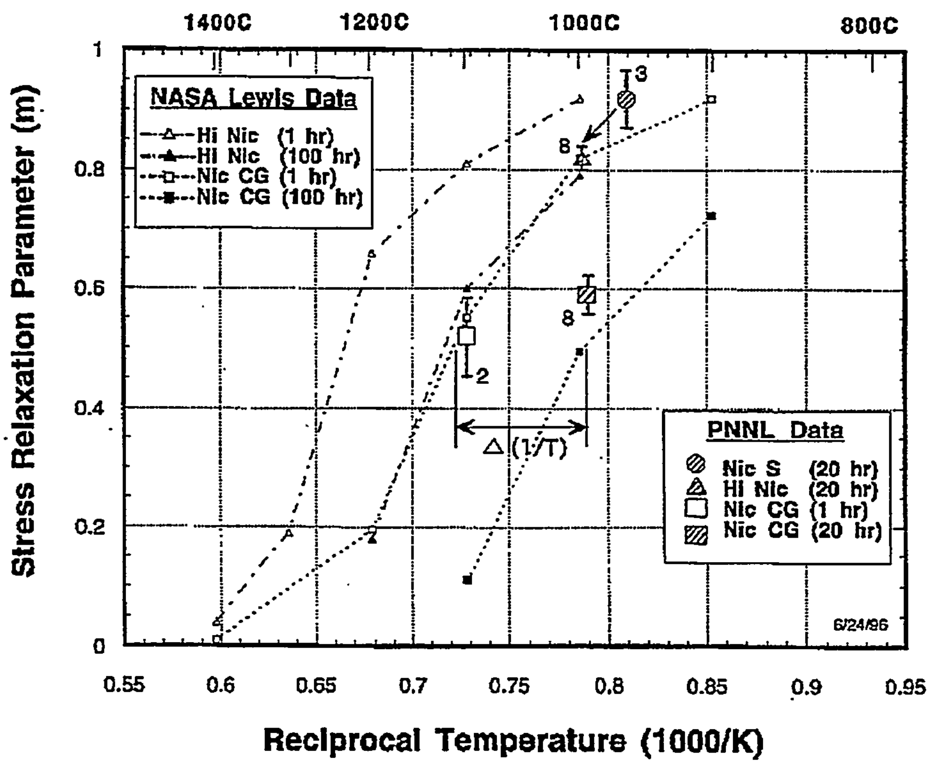


Figure 3. Comparison of PNNL BSR data with NASA Lewis BSR data for SiC type fibers.

From Fig. 3 it is apparent that the 100 hour BSR curves uniformly shift to a lower temperature range when compared to the 1 hour BSR curves. Morscher et al.[6] used the cross-cut method to provide an effective activation energy from such BSR data. They demonstrated reasonable agreement with an activation energy obtained from conventional fiber tensile tests for Nicalon-CG fiber. In this method, for one order of treatment time change the effective activation energy Q is given by:

$$Q = 2.303R/\Delta(1/T) \tag{4}$$

where R is the gas constant (8.314 J/mole). In Fig. 3, $\Delta(1/T)$ is indicated for two orders of time change, so the numerical factor in Eq. (4) must be changed to 4.606. For example, for Nicalon-CG and Hi-Nicalon fibers, Q = 555 or 722 J/mole, respectively.

CONCLUSIONS

1. The BSR test provides a convenient and reproducible method for evaluating fiber creep resistance and should be adaptable for assessing the influence of irradiation enhanced creep in SiC fibers.

2. Preliminary stress-relaxation data suggests that Nicalon-S is more creep resistant than Hi-Nicalon. This is the first creep data for this fiber.

FUTURE WORK

1. Continue thermal control BSR tests of SiC fibers at temperatures in the 1100 to 1300°C range. These tests will be carried out in a prototype SiC irradiation jig to avoid interactions between the fibers and the jig.

2. Initiate irradiation BSR tests in either the ATR reactor (Idaho Falls) or the HFBR reactor (Brookhaven).

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