ELSEVIER

Journal of Nuclear Materials 283–287 (2000) 372–375

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

Irradiation creep of advanced silicon carbide fibers

R. Scholz [a,*], G.E. Youngblood [b]

[a] *Commission of the European Community, JRC. IHCP, TP 202, Via E. Fermi 1, 21020 Ispra (VA), Italy*
[b] *Pacific Northwest National Laboratory, Richland, WA 99352, USA*

Abstract

The bend stress relaxation (BSR) method was applied to study irradiation enhanced creep (IEC) of small diameter silicon carbide (SiC) fibers after 10 MeV proton irradiation. A first series of tests was conducted on Sylramic™ fibers irradiated at 600°C with average bending stresses of 400 and 667 MPa and for irradiation doses smaller than 0.04 dpa. The BSR results are compared to previously obtained torsional creep test results for the Textron SCS-6™ type SiC fibers by calculating the tensile equivalents for both testing methods. For the Sylramic fibers, the creep constant $\kappa = 4.7 \times 10^{-6}$ Mpa^{-1} dpa^{-1}, was a factor of 6 smaller than the κ-value determined for SCS-6 fibers at 600°C. In contrast, for $T < 900$°C the κ-value determined by R.J. Price [Nucl. Technol. 35 (1977) 320] for high purity monolithic β-Si after 7.7 dpa neutron irradiation was only 0.4×10^{-6} MPa^{-1} dpa^{-1}. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Several fusion reactor concepts have been proposed that use silicon carbide (SiC) composites as the primary structural material for the first wall and blanket [1]. Continuous SiC fiber-reinforced SiC composites consisting of a SiC matrix produced by chemical vapor infiltration (CVI) and reinforced by small diameter fibers have received the most attention [2]. In such composites operating at high temperatures, creep is an important deformation mechanism. Usually the fibers are less creep resistant than the matrix [3]. As the fibers creep under load, they tend to transfer part of their load to the matrix. With time, the stresses that build up in the matrix may become sufficiently large to cause matrix cracking by a mechanism called slow or sub-critical crack growth, whereby matrix cracking can proceed at stress levels below the fast-fracture matrix cracking stress. Slow crack growth in SiC–SiC is directly related to the creep rate of the reinforcing fibers [4]. Irradiation may enhance the creep rate, as has been observed for irradiated metals [5]. In particular, an enhancement of the fiber creep rate could lead to a life-limiting condition for using SiC–SiC composites as structural reactor components. Therefore, a concerted effort to examine irradiation creep of advanced SiC fibers has been initiated in Europe and in the US.

Initially, torsion creep tests were conducted on Textron SCS-6 monofibers under light ion irradiation for the temperature range 450–1100°C [6]. The SCS-6 fibers (140 μm diameter) were produced by vapor depositing SiC on a carbon core. The resulting β-SiC grain structure and orientation were considered representative of a CVI-SiC matrix deposited onto small diameter fibers in a SiC–SiC composite [7]. These tests showed that:

1. significant irradiation enhanced creep (IEC) occurred over the entire temperature range examined;
2. for the temperature range $450 < T < 800$°C, the IEC curves were characterized by strain transients with decreasing creep rates before attaining approximately steady-state values;
3. at 600°C, the apparent steady-state creep was proportional to stress and flux;
4. for the temperature range $900 < T < 1100$°C, the creep rate was constant from the beginning of the irradiation.

Over the temperature range 900–1100°C, an activation energy $E = 48 \pm 15$ kJ mol^{-1} was determined. This value is much smaller than the activation energy of 590 kJ

---

[*] Corresponding author. Tel.: +39-0332 789 260; fax: +39-0332 789 355.
E-mail address: reinhard.scholz@jrc.it (R. Scholz).

0022-3115/00/$ - see front matter © 2000 Elsevier Science B.V. All rights reserved.
PII: S0022-3115(00)00264-6

mol^{-1} determined by Morscher et al. [8] for thermal creep of SCS-6 fibers.

More than 20 years ago, Price examined IEC in monolithic β-SiC by using a bend stress relaxation (BSR) technique [9]. Several CVD-deposited strips were bent to about half their fracture strain and held in the bent position while being irradiated at 780°C, 950°C and 1130°C with neutrons in the EBR II fast nuclear reactor. After irradiation to a dose of about 7.7 dpa (assuming 1 dpa $= 10^{25}$ n m^{-2}), the residual curvatures of the relaxed strips were measured to estimate the magnitude of IEC.

In Fig. 1, the temperature dependence of the irradiation creep constant κ (defined here as creep strain divided by dose and average applied stress) is plotted for monolithic β-SiC (data taken from [9]). For comparison, the κ-values of SCS-6 fibers, determined for light ion irradiation with a torsional creep device, are superimposed onto Fig. 1. These κ-values are calculated using the relationship $\kappa =$ (steady state creep rate)/(dpa – rate x applied maximum shear stress) and converted to tensile equivalents with the formula $\varepsilon/\sigma = \gamma/3\tau$, where ε/σ is the tensile strain to stress ratio, τ and γ are the maximum shear stress and strain, respectively [7]. The temperature dependence exhibited by both data sets are remarkably similar despite the completely different dose ranges and experimental methods used. Below 900°C, the κ-values were relatively temperature independent for both the β-SiC strips and the SCS-6 fibers even though the equivalent κ-values of the two data sets differed by one-to-two orders of magnitude. Above 900°C, both sets of κ-values increased with increasing temperatures, though probably at different rates.

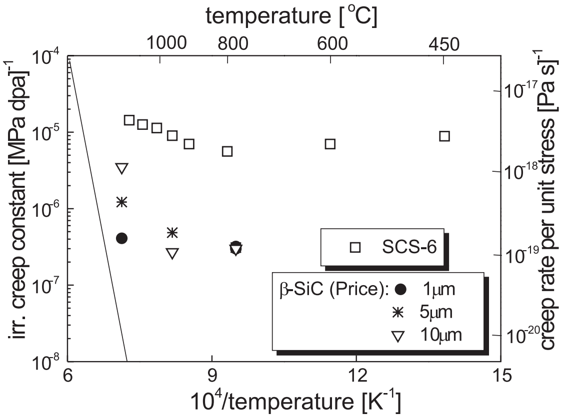

Fig. 1. The logarithm of the irradiation creep compliance is plotted versus $1/T$ for two data sets: low dose (<0.07 dpa) light ion creep of SCS-6 fibers; high dose (≈7.7 dpa) BSR test using β-SiC strips of different average grain size (1, 5 and 10 μm) [9]. The straight line represents the stress normalized thermal creep rate of monolithic β-SiC.

The torsion method cannot be adopted to examine creep of small diameter (≈15 μm) fibers used as reinforcement in SiC–SiC composites, and which are of keen interest for fusion reactor application. However, a BSR technique was used to examine these fibers, apparently without major difficulties. The BSR method is most suitable for in-pile tests where space restrictions dictate a small, robust test configuration. Two BSR fiber creep experiments are currently underway in the ATR and the HFIR reactors [10]. Since it will require about a year to obtain any results from the in-reactor BSR tests, the BSR method also has been adopted to obtain some initial IEC data for light ion irradiation tests. These tests can be carried out in a few days. Initial BSR fiber creep results for the near stoichiometric Dow Sylramic™ SiC fiber obtained after light ion irradiation are reported here. Continuing light ion BSR creep tests of other near stoichiometric SiC fibers, namely, Hi-Nicalon, Type S Nicalon and Tyranno SA fibers are underway.

2. Experimental details

2.1. The BSR test

BSR creep testing of small diameter fibers was first developed by Morscher and DiCarlo and is described in detail in [11]. For carrying out BSR creep tests of fibers irradiated with light ions, the fibers were wrapped around a cylindrical graphite rod of radius R_o and kept in the bent position while being irradiated from one direction. After the irradiation, the residual curvatures of the irradiated semi-arcs were determined by measuring the arc radius, R_c, of each relaxed fiber segment. From these curvatures, a creep deformation parameter $m = 1 - (R_o/R_c)$, was calculated. The parameter m varies between 0 for complete relaxation and 1 for no relaxation. The IEC strain may be approximated $\Delta\varepsilon_c = \varepsilon_o(1/m_{irr} - 1/m_{th})$, where ε_o stands for the average applied bending strain imposed during the irradiation [10]. At 600°C under thermal conditions creep relaxation can be neglected ($m_{th} = 1$) and the irradiation creep strain $\Delta\varepsilon_c = \varepsilon_o(1/m_{irr} - 1)$. In principle, by plotting $\Delta\varepsilon$ as a function of dose, an irradiation creep curve can be constructed, except that singular data points are obtained for each dose. In contrast, a continuous set of data points are obtained when the test is carried out at constant load, which was the case when fiber creep was determined by the torsional method.

2.2. Material and experimental procedure

Sylramic fibers, with near stoichiometric SiC composition and a radiation resistance expected to be similar to pure monolithic SiC, were selected for the initial BSR creep tests under light ion irradiation. Single Sylramic

Table 1
BSR data for irradiated Sylramic fibers

Dose	$\bar{\varepsilon}_o$ (%)	$\bar{\sigma}$ (MPa)	R_c (mm)	$m = 1 - (R_o/R_c)$	$\varepsilon_c = \bar{\varepsilon}_o(1/m - 1)$ (%)
0.025	0.165	665	32	0.953	0.0081
0.04	0.165	665	19	0.92	0.014
0.04	0.100	400	34	0.926	0.008

fibers (diameter ≈10 μm) were carefully wrapped around two graphite cylinders (radius $R_o = 1.5$ or 2.5 mm) to examine stress dependence of IEC. The fibers were first glued at one end of the cylinder, wrapped around, and then glued at the other end of the cylinder. The fiber-wrapped cylinder was fastened onto an aluminum sample holder covered with alumina cement, which retained the fibers in the bent position during irradiation. Related tests under thermal conditions showed that the alumina fixing was sufficient to hold the fibers. The samples were irradiated with 10 MeV protons in a helium atmosphere at a temperature of 600°C for two doses 0.025 and 0.04 dpa calculated by using the TRIM code and assuming an average displacement energy of 38.5 eV [12]. A specimen temperature of 600°C was maintained by adjusting the helium pressure in the irradiation chamber to achieve an equilibrium between beam heating and heat losses by convection and conduction. The temperature was measured with an infrared pyrometer and a thermocouple shielded from the beam inside the graphite cylinder. After irradiation, photographs were taken under a microscope of each irradiated fiber semi-arc to determine their bend radii. An average bend radius R_c was determined from measurements of at least four semi-arcs for each condition.

3. Results and discussion

The BSR data for Sylramic fibers ion-irradiated at 600°C for doses of 0.025 and 0.04 dpa are given in Table 1. The applied average strain and stress values, $\bar{\varepsilon}_o$ and $\bar{\sigma}$, are listed, together with the mean bend radii R_c and calculated m- and ε_c-values.

In Fig. 2, the ε_c-values of Table 1 are plotted versus dose together with a creep curve measured on a Textron SCS-6 fiber under torsional loading irradiated with light ions at 600°C for a maximum shear stress of 320 MPa (tensile equivalent $\sigma = 554$ MPa). For temperatures $T < 800$°C, the IEC curves of the SCS-6 fibers were characterized by strain transients during which the creep rate decreased before reaching approximately constant values, e.g., the creep rate after 0.05 dpa for the SCS-6 fiber shown in Fig. 2 is almost an order of magnitude smaller than at the beginning of the irradiation. In contrast, for $T > 900$°C the IEC rates appeared to attain steady state almost immediately.

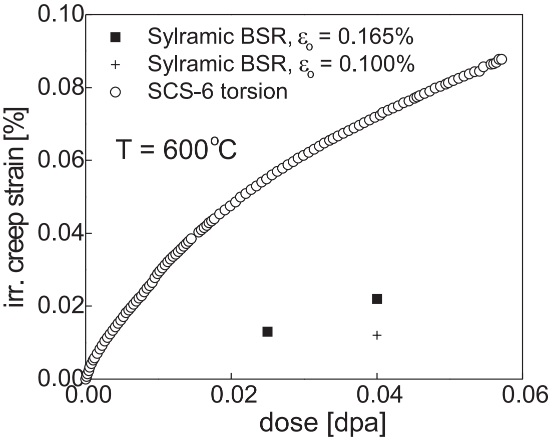

Fig. 2. SCS-6 torsional irradiation creep curve (tensile equivalent stress $\sigma = 554$ MPa) in comparison to irradiation creep strain of Sylramic fibers calculated from BSR tests, ε_o – average bending strain.

For the Sylramic fibers, the irradiation creep strain ε_c appears to be approximately proportional to dose and to the average stress. Furthermore, at a dose of 0.04 dpa the magnitude of ε_c was less than that determined for the SCS-6 fiber at a similar stress.

In Table 2, the κ-values determined for the SCS-6 and Sylramic fibers after light ion irradiation (low dose) are compared to the κ-values determined by Price for monolithic β-SiC after high dose neutron irradiation [9]. Both sets of κ-values were obtained for $T < 900$°C, where IEC appears to be temperature independent.

Table 2
Comparison of irradiation creep constant κ determined for SCS-6, Sylramic SiC fibers and β-SiC strips [9]

Fiber	T (°C)	Method	Dose (dpa)	Particles	κ (MPa^{-1} dpa^{-1})
SCS-6	600	Torsion	0.04	Protons	30×10^{-6}
Sylramic	600	BSR	0.04	Protons	4.7×10^{-6}
β-SiC	800	BSR	7.7	Neutrons	0.4×10^{-6}

Table 2 shows that the κ-values observed for the SiC-fibers were much larger than Price observed for monolithic β-SiC.

4. Conclusions

The BSR-method was applied to study irradiation creep of small (<15 μm) diameter fibers under proton irradiation (total dose < 0.05 dpa). The first tests were successfully conducted on the near stoichiometric Sylramic™ fiber at 600°C. The test results, together with results from light ion-irradiated Textron™ SCS-6 fibers and in-pile BSR tests on β-SiC, show that:

- The irradiation creep compliance κ is smaller for Sylramic™ than for SCS-6 fibers at 600°C.
- The low dose (0.05 dpa) κ-value of Sylramic fibers is about an order of magnitude higher than the high dose (7.7 dpa) creep compliance of β-SiC [9].
- Whereas a κ-value of 0.4×10^{-6} Mpa^{-1} dpa^{-1} observed for monolithic β-SiC might be considered manageable for a fusion structural application, the high κ-values observed so far for two types of SiC fibers would not. Such high κ-values suggest that subcritical crack growth in SiC–SiC composites, brought about by fibers that creep excessively, would represent a life-limiting condition when using SiC–SiC in a fusion structural application.

References

[1] R.H. Jones, D. Steiner, H.I. Heinisch, G.A. Newsome, H.M. Kerch, J. Nucl. Mater. 245 (1997) 87.
[2] L.L. Snead, R.H. Jones, A. Kohyama, P. Fenici, J. Nucl. Mater. 233–237 (1996) 26.
[3] A.G. Evans, C. Weber, Mater. Sci. Eng. A 208 (1) (1996) 1.
[4] C.A. Lewinsohn, R.H. Jones, G.E. Youngblood, C.H. Henager Jr., in: R.H. Jones, A. Kohyama (Eds.), The Third IEA Workshop on SiC–SiC Ceramic Composites for Fusion Structural Applications, 29&30 January 1999, Cocoa Beach, FL, p. 76.
[5] M.B. Toloczko, F.A. Garner, C.R. Eiholzer, J. Nucl. Mater. 258–263B (1998) 1163.
[6] R. Scholz, J. Nucl. Mater. 258–263 (1998) 1533.
[7] R. Scholz, in: R.H. Jones, A. Kohyama (Eds.), The Third IEA Workshop on SiC–SiC Ceramic Composites for Fusion Structural Applications, 29&30 January 1999, Cocoa Beach, FL, p. 162.
[8] G.N. Morscher, J.A. DiCarlo, X.J. Ning, P. Pirouz, Adv. Ceram. Matrix Compos. (1995) 679.
[9] R.J. Price, Nucl. Technol. 35 (1977) 320.
[10] G.E. Youngblood, R.H. Jones, G.N. Morscher, R. Scholz, A. Kohyama, Eng. Sci. Proc. 19 (4) (1998) 341.
[11] G.N. Morscher, J. DiCarlo, J. Am. Ceram. Soc. 75 (1) (1992) 136.
[12] P. Jung, Z. Zhu, J. Chen, J. Nucl. Mater. 251 (1997) 276.