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Light ion irradiation creep of Textron SCS-6™ silicon carbide fibers

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Abstract

Creep tests were conducted in torsion on Textron SCS-6™ fibers during an irradiation with light ions in the temperature range 500–1000 °C for doses up to 0.16 dpa. The fibers produced by chemical vapor deposition have a similar structure as a silicon carbide composite matrix produced by chemical vapor infiltration. At 600 °C, the irradiation creep curves were characterized by a continuous drop in creep rate with dose. There was approximately a square root relationship between irradiation creep strain and dose. The creep rate was a linear function of stress. On a decrease in temperature the creep rate increased. At 1000 °C, the creep rate dropped only slightly with dose and decreased if the temperature was lowered. The results are discussed in terms of concentration and mobility of point defects and the change of these quantities with temperature.

1. Introduction

Irradiation enhanced creep (IEC) is an important deformation mechanism of structural reactor components, which are exposed, simultaneously, to stresses and a high-energy particle irradiation. There is a large IEC database available for austenitic or martensitic steels. The situation is different for silicon carbide (SiC) composites, which were considered more recently for structural applications in fusion reactors [1]. Two data sets are available, which include:

(1) The bend stress relaxation (BSR) data published by Price more than 20 years ago [2]. Several monolithic β-SiC strips were bent to about half their fracture strain and held in the bent position while being irradiated at 780, 950 and 1130 °C with neutrons in the EBRII fast nuclear reactor up to a dose of 7.7×10^{25} neutron m^{-2} corresponding to 7.7 dpa. The magnitude of IEC was determined by measuring the residual curvatures of the relaxed strips. The resulting data are shown in Fig. 1: the logarithm of the creep constant κ is plotted versus $1/T$, κ defined as creep strain divided by dose and average applied stress.

(2) IEC data resulting from light ion irradiation creep tests conducted on the Textron SCS-6 fiber [3] (see Fig. 1). These tests were performed with a torsion creep machine for the temperature range 450–1100 °C. The torsion quantities, shear strain γ and stress τ, were converted into tensile equivalents with the formula $\varepsilon/\sigma = \gamma/3\tau$. The temperature dependence of both data sets is remarkably similar despite the completely different experimental methods used and despite the two orders of magnitude difference in dose accumulated in the tests. Above 900 °C, both data sets of κ-values increased with temperatures, in the range 450–800 °C, the κ-values for the SCS-fibers decreased if the temperature was increased.

Apparently, two IEC regimes existed: (a) for the range 450–800 °C, the curves were characterized by strain transients with decreasing creep rates before attaining approximately steady state values and (b) for temperatures $950 < T < 1100$ °C, the creep rate was almost constant from the beginning of the irradiation. However, the tests were limited to doses <0.06 dpa. In order to check whether or not for both regimes, a steady

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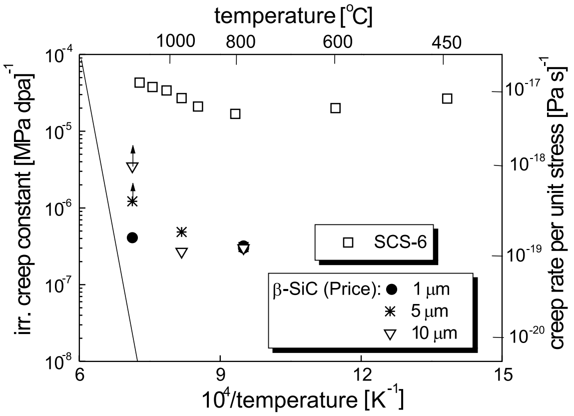

Fig. 1. Irradiation creep compliance values as a function of temperature for two different experiments: (a) BSR after high dose (~7.7 dpa) neutron irradiation of β-SiC strips of different average grain size (1, 5, 10 μm) [2]; (b) low dose (<0.07 dpa) light ion irradiation of β-SiC fibers (Textron SCS-6) in torsion [3]. The straight line represents the stress normalized thermal creep rate of monolithic β-SiC.

state situation will possibly be reached at higher doses, several specimens were irradiated to doses up to 0.15 dpa, at 600 and 1000 °C.

2. Material and experimental procedure

Single fiber specimens were made from Textron SCS-6™ fibers (Textron Specialty Material, Lowell, MA, USA) by nickel-plating both ends of a 10 cm long fiber segment to a thickness of ≈1 mm. The central fiber segment of 8 mm length, which was exposed to the irradiation, remained unplated. The Ni-plated parts can be tightly fixed into the grips of the creep machine so that shear stresses higher than 800 MPa could be applied without causing slippage or failure of the specimen at the grip position what often limits the magnitude of stress in mechanical tests of brittle materials. The experimental parameters, such as applied stress or temperature of the specimens were remotely controlled and could be changed easily during the creep tests. The specimens, before starting the irradiation, were exposed for 24–48 h to the same temperature and loading conditions imposed during the irradiation in order to exhaust strain transients of thermal origin. The specimens were irradiated with 12 MeV protons at a dpa rate up to 3.5×10^{-6} dpa/s calculated with the aid of the TRIM code [4] by assuming an overall displacement energy of 38.5 eV for the material [5]. The beam intensity, which hit the specimen, was calculated from the difference in heating power input for beam on/off conditions at a fixed specimen temperature.

3. Results

3.1. Irradiation creep at 500–600 °C

Fig. 2 shows the strain–dose curves for two specimens irradiated at 600 °C for maximum shear stresses of 160 and 320 MPa, respectively, up to doses of 0.15 dpa. A continuous drop in creep rate is evident for both curves. A least square fit of the function $y = Ax^B$ gave the following results: for the 160 MPa curve, $A = 0.4$, $B = 0.53$, for the 320 MPa curve, $A = 0.8$ and $B = 0.55$. So, there was approximately a square root relationship between irradiation creep strain and dose, for both curves. The slopes of the straight lines drawn in Fig. 2 correspond to the mean irradiation creep rates in the dose interval 0.1–0.14 dpa. The slope of the 320 MPa curve is a factor of two larger that that of the 160 MPa test. Thus, there is a linear stress dependence of the creep rate. The magnitude of these creep rates is a factor of 2–3 smaller than those determined in the dose interval 0.03–0.06 dpa, which are reported in Fig. 1.

Previous tests, which were limited to doses <0.1 dpa, showed that the IEC rate increased on a drop in temperature. In order to check whether or not this effect appears also for higher doses, at the end of the 160 MPa tests shown in Fig. 2, the temperature was lowered from 600 to 500 °C and increased back to 600 °C after a dose of 0.03 dpa was accumulated. The maximum shear stress was kept constant at 160 MPa. The resulting strain dose relationship is plotted in Fig. 3. After the drop in temperature from 600 to 500 °C, there is a small elastic strain jump, followed by an IEC rate that was higher than that at 600 °C. The increase in temperature had the opposite effect: the IEC rate decreased. The creep rate

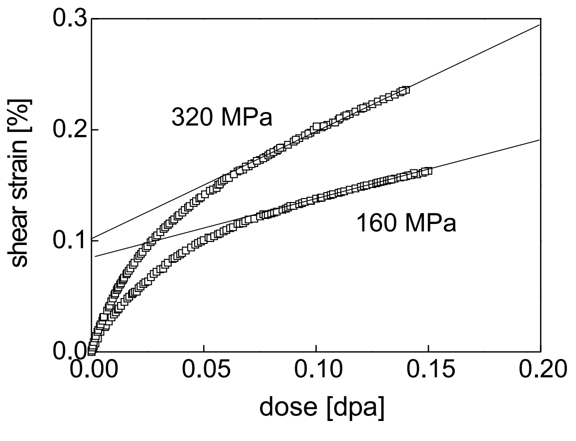

Fig. 2. Two creep curves for 600 °C and maximum shear stresses of 160 and 320 MPa. There is approximately a square root relationship between shear strain and dose. The straight lines represent the creep rates in the saturation part of the curves. The creep rates are a linear function of stress.

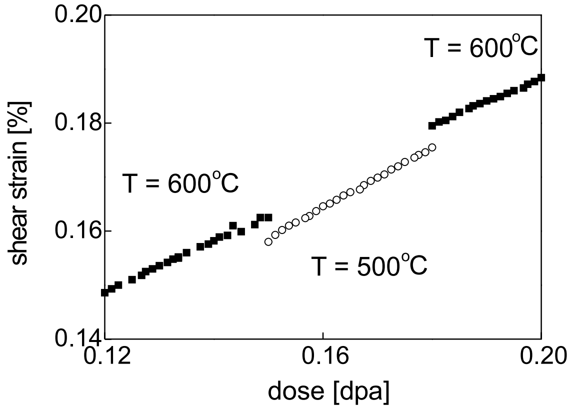

Fig. 3. Result of a temperature change test from 600 to 500 °C and back to 600 °C. The shear strain is plotted as a function of dose. The creep rate increased, when the temperature was lowered, and decreased when the temperature was increased.

dropped almost immediately and stayed at lower values in comparison to the 500 °C period.

3.2. Irradiation creep at 900–1000 °C

Fig. 4 shows the IEC curve of a test started at a temperature of 1000 °C. After a dose of 0.08 dpa the temperature was lowered to 900 °C, maintaining a constant stress of 160 MPa. During the 1000 °C period, the shear strain increased almost linearly with dose after a short transient period at the onset of the irradiation. The drop in creep rate, which amounted to about 20% if one compares the slope of the curve at 0.04 and 0.08 dpa, was much less significant than that during the 600 °C runs. After the decrease in temperature from 1000 to 900 °C, the IEC rate decreased continuously and approached, after a total dose of 0.12 dpa, a value, which was a factor of two smaller than that measured for 1000 °C.

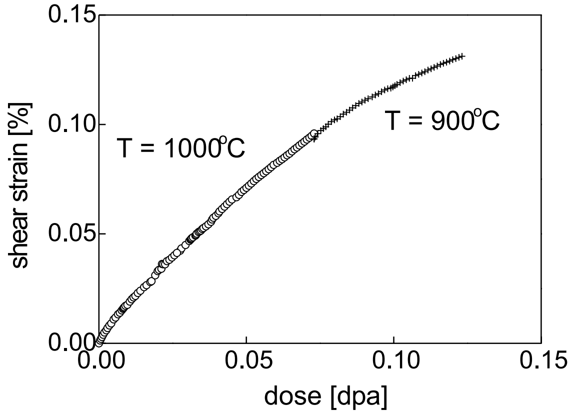

Fig. 4. The effect of a change in temperature from 1000 to 900 °C. At 900 °C, the creep rate approaches a steady state value smaller than that at 1000 °C.

4. Discussion

IEC is primarily due to the concentration and mobility of the irradiation-induced point defects, which, in the case of SiC, are vacancies and interstitial atoms of silicon and carbon. For the experimental conditions imposed, point defects were present at high supersaturating levels in comparison to thermal equilibrium concentration.

For temperatures between 500 and 600 °C, only the carbon interstitial is presumably mobile [6] and the difference in irradiation creep observed in this temperature range may be attributed to the difference in mobility and concentration of this defect. After being generated, the point defects may be accumulated in the lattice or, if mobile, undergo several reactions: they can recombine, be captured by pre-existing sinks as dislocations and grain boundaries or form agglomerations of equal defect type, such as dislocation loops. Which of these different reactions prevails, depends on the defect mobility and concentration, thus, on temperature if the other parameters, such as beam intensity and sink concentration are held constant. For the low temperature tests ($T \leqq 600$ °C), the concentration of immobile carbon vacancies increased with irradiation dose. The amount of carbon interstitial atoms, which escape recombination drops accordingly. The same dependency holds for the irradiation creep rate that is high at the beginning of the irradiation and decreases with dose (see Fig. 2). In this picture, the high creep rates measured for temperatures <800 °C are a low dose effect.

The connection between IEC and swelling is evident. For temperatures below 1000 °C, swelling occurred without an incubation dose and saturated rapidly within a total dose of about 0.3 dpa. The magnitude of swelling observed after saturation decreased with increasing irradiation temperature from about 1% at room temperature to 0.05% at 1000 °C [6]. It has been attributed to the accumulation of single defects and small defect clusters [7]. A cessation of swelling means that the concentration of immobile vacancies had reached its maximum value. At this point, the interstitial atoms generated by the irradiation are predominantly or completely annihilated by vacancies and, thus, are not available for the IEC process. The irradiation creep rate will reach a minimum, which might be close or equal to zero. Since a drop in temperature caused the IEC rate to grow (see Fig. 3) one may further conclude that the total amount of creep strain, accumulated before the saturation of swelling is reached, will decrease with temperature such as the magnitude of swelling itself for temperatures below 1000 °C.

At temperatures >1000 °C, the irradiation creep curves had an almost constant slope from the beginning of the irradiation [3], i.e. steady state conditions were established almost immediately. If it is assumed, in a first approximation, that the point defects in the carbon sublattice are rate determining for the IEC process, one may conclude that both defects, the carbon interstitial and vacancy, are now mobile. For this case, the time to reach steady state of the point defect concentrations is inversely proportional to the mobility of the slower defect if annihilation at sinks is the dominant annihilation mechanism [8]. Computer simulations gave about 2.7 eV for the migration energy of both the Si and C vacancy [9] indicating that these vacancies have a significant mobility at 1000 °C. If the temperature of the specimen is lowered from 1000 to 900 °C, the mobility of the vacancies is reduced drastically and their concentration will grow with irradiation time [8], thus, increasing the magnitude of pair recombination. So, the amount of interstitial atoms available for the creep process decreased, the IEC rate declined after the temperature change from 1000 to 900 °C (see Fig. 4).

5. Conclusions

Proton irradiation creep tests have been conducted in torsion on Textron SCS-6™ silicon carbide fibers in the temperature range 500–1000 °C for doses up to 0.15 dpa. Two irradiation creep regimes could be distinguished:

(a) For a specimen temperature of 600 °C, there was approximately a square root relationship between creep strain and dose. The creep rate increased if the temperature was decreased from 600 to 500 °C.
(b) For a specimen temperature of 1000 °C, the slope of the creep curves decreased only slightly with dose. The creep rate decreased after a drop in temperature from 1000 to 900 °C.

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