NEUTRON IRRADIATION EFFECTS ON HIGH NICALON SILICON CARBIDE FIBERS - M. C. Osborne and D. Steiner (Rensselaer Polytechnic Institute), and L. L. Snead (Oak Ridge National Laboratory)

Summary

The effects of neutron irradiation on the mechanical properties and microstructure of SiC and SiC-based fibers is a current focal point for the development of radiation damage resistant SiC/SiC composites. This report discusses the radiation effects on the Nippon Carbon Hi-Nicalon^{TM} fiber system as also discusses an erratum in earlier results published by the authors on this material. The radiation matrix currently under study is also summarized.

Introduction

Due to the intrinsically low activation of silicon carbide and the relatively high toughness of SiC/SiC composites, these materials have become an attractive alternative to conventional structural materials for future fusion devices. However, it has been demonstrated that the "off-the-shelf" SiC/SiC composites undergo significant degradation at fluences as low as 1 dpa [1]. This degradation has been attributed to the densification of the Nicalon^{TM} fiber, which has been the standard fiber used in the study and development of SiC/SiC composites. The primary emphasis in the fusion community is toward a more stoichiometric SiC fiber, which should not undergo the large densification seen in regular Nicalon^{TM}, and hopefully exhibit the modest swelling typical of stoichiometric SiC. The Nippon Carbon Hi-Nicalon^{TM} fiber (Table 1) in particular has received significant attention from the fusion community owing to its very low oxygen content, typically less than a few tenths of a percent.

Table 1. Properties and Composition of SiC Fibers

	Nicalon	Hi-Nicalon	Hi-Nicalon Type S (Stoichiometric)	Dow Corning Stoichiometric	CVD SiC
Diameter (mm)	14	14	12	10	
Strength (GPa)	2.8	2.8	2.6	2.8	
Modulus (GPa)	220	270	420	400	450
Failure Strain	0.014	0.010	0.006	0.007	
Density (g/cc)	2.55	2.74	3.1	>3.1	3.21
Si (wt %)	56.6	62.4	68.9	67.7	70.0
C (wt %)	31.7	37.1	30.9	28.6	30.0
O (wt %)	11.7	0.5	0.2	0.9	
B (wt %)				2.4	
N (wt %)				0.4	

Discussion

Data from earlier work by the authors [2] shows the effects of atomic displacements on the tensile strength and tensile modulus of Hi-Nicalon™ fiber. Since this report, the fiber diameter measurement was found to be in error. In the original report, the fiber diameter was determined by optically imaging the fiber following tensile testing. The measurement problem arose from the cleaning treatment prior to imaging. Specifically, before the tensile testing the fiber was coated with silicon vacuum grease to minimize the spread of contamination and to aid in recovery of the fiber after it was broken. The fiber remnants were then collected and cleaned ultrasonically in a ethyl alcohol solution and imaged optically. These diameters were used to reduce the data shown in Fig. 1.

SEM imaging was later used on fibers which were cleaned in a similar fashion. It became apparent that not all of the silicon vacuum grease was removed during the cleaning process, thus leaving a residue and yielding inaccurate (high) diameter data. The inaccurate data as measured optically yielded a diameter of 15.3 μm, while that measured via SEM was 13.7 μm. As further confirmation some higher dose irradiated fibers (2.56×10^{25} n/m^2) have recently become available for tensile measurement and using the SEM for fiber diameter measurement. These fibers did not have vacuum grease residue and gave an average diameter of 13.4 μm.

To correct the data referred to above, it is noted that the density did not vary significantly up to 0.32 dpa and the density increased by about 4% at 2.56 dpa. Therefore, the diameters of the fibers for the previously reported data can be taken as the unirradiated average value and the strengths and moduli can be reconstructed as shown in Fig. 2. From this figure, the strength increases sharply at low damage levels and then shows a moderate increase. This is somewhat different than the behavior presented in the previous report and Fig. 1. The modulus of the fibers of Fig. 2 exhibit a decrease followed by an increase but the effect is not as drastic as previously reported (about 5% decrease rather than about a 10% decrease). Since these are only estimates of the low level irradiation conditions, the 0.1 and 0.32 dpa conditions are being repeated.

A slightly different representation of the data is given in Fig. 3. The strength, strain, and elastic strain energy density are plotted against dpa. Strain energy density is calculated as one half times the stress times the strain [3]. The strain shown in Fig. 3 is the average strain calculated using laser micrometer measurements of the gage length and displacement, no diameter measurements are involved in its calculation. The figure presents the data in a more intuitive manner by showing that as the fibers strengthen with irradiation, there is more elongation and more energy (on average) is needed to fracture the fibers. The reasons for this are still under investigation and will be discussed in a later semiannual.

HFIR Experiments In Progress

The fibers currently under evaluation (under irradiation or irradiated) are listed in Table 1. This table gives unirradiated fibers mechanical properties and nominal composition, as provided by the manufacturers [4, 5].

Table 2 summarizes the fiber irradiation program in the HFIR hydraulic rabbits positions HT-2, HT-7, or HT-3. The times in each position are varied to get the appropriate fluence for E>0.1 MeV. The fiber temperature is achieved through defining a gas gap between the fiber holder and the rabbit internal wall. Each rabbit includes a silicon carbide temperature monitor. The conditions for the irradiations are shown in Table 2. It is expected that the irradiations will be complete by November 1996, with the evaluation of tensile properties completed by years end.

Table 2. Irradiation Conditions of SiC Fibers

	0.1 dpa	**0.5 dpa**	**2.0 dpa**	**5.0 dpa**
100°C	completed	completed	completed	completed
500°C	completed	completed	completed	in progress
800°C	in progress	in progress	in progress	in progress

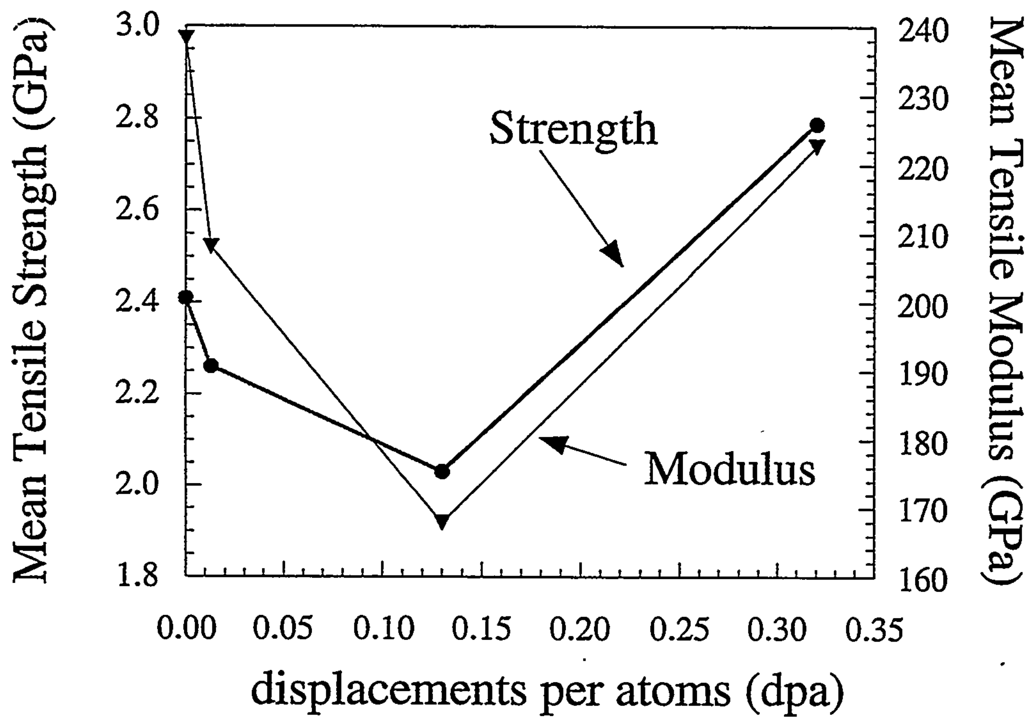

Fig. 1. Mean strength and modulus vs damage level as originally reported in [1].

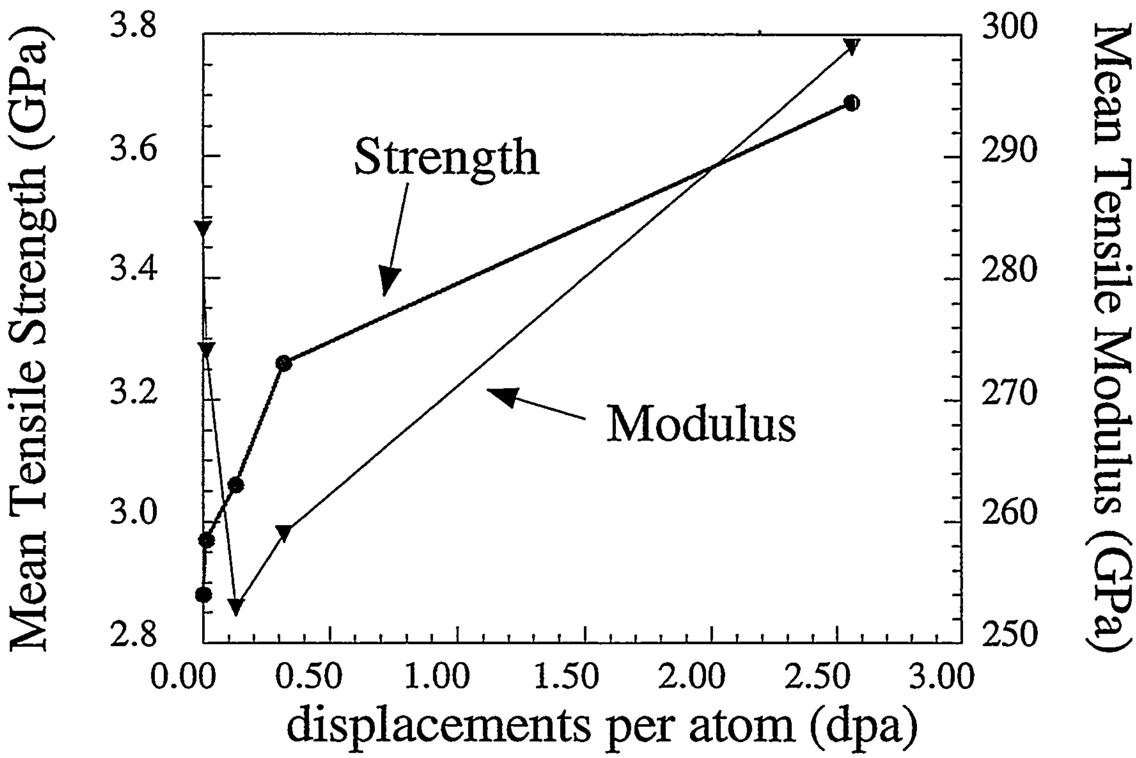

Fig. 2. Corrected strength and failure strain vs damage.

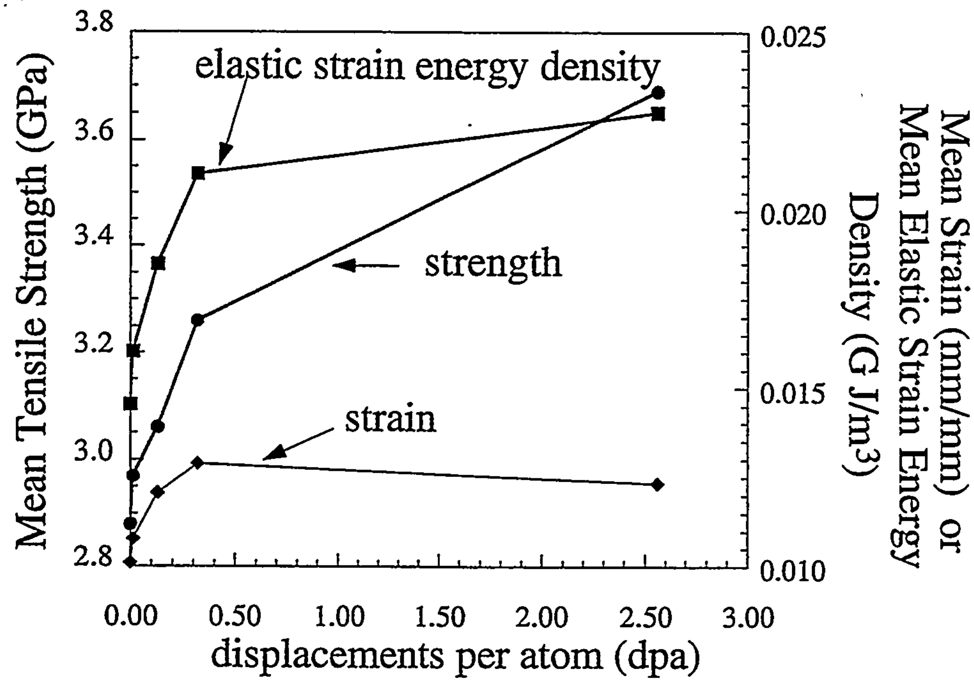

Fig. 3. Mean strength, mean strain, and mean elastic strain energy density vs damage.

References:

[1] L. L. Snead, S. J. Zinkle and D. Steiner, "Measurement of the Effect of Radiation Damage to Ceramic Composite Interfacial Strength," Journal of Nuclear Materials [191-194] 1992, pp. 566-570.

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[4] J. Lipowitz, *et al.*, Ceramic Engineering and Science Proceedings, **16** (1995) 55.

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