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Structural and Mechanical Characterization of Hi-Nicalon Fibers

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The need of reinforcements for structural ceramic matrix composites to be used in air at temperatures above 1000°C has encouraged great changes in small-diameter ceramic fibers since their initial development as refractory insulation [1]. A very important type of ceramic fibers for structural reinforcement are the Hi-Nicalon fibers, which are oxygen free SiC fibers that have been commercially produced by an electron beam curing process [1]. The Hi-Nicalon fiber has higher elastic modulus and thermal stability and better creep resistance than the Nicalon fiber. Hi-Nicalon fibers are also highly resistant to oxidation and chemical attack. The performance of any ceramic matrix composites is highly dependent upon the properties of the reinforcement, which make important studying the mechanical behavior of these fibers. The objective of the present work was to evaluate the tensile behavior of single Hi-Nicalon fibers at room temperature using a Universal Fiber Tester [2], equipped with a load cell of 250 g calibrated from 0 to 100 g, with a precision of 0.01 g. The specimen gauge length was 30 mm and the fiber was gripped between two sets of jaws, the strain speed used for the tests was 4.05 x 10^{-3} s^{-1}. Data acquisition used a PC linked to the fiber tester via a National Instrument interface card and WinATS 6.2 software from Sysma. In order to normalize the stress, the diameter of the tested fibers was systematically measured before each test by using a Mitutoyo LSM-500S laser apparatus, with an accuracy of 0.01 μm. Scanning electron microscopy (SEM) characterization was performed using a JEOL JSM7401F microscope operated at 2 kV. Samples were prepared by focused ion beam (FIB) in a JEOL JEM 9320-FIB microscope operated at 30 kV and 25 mA, and analyzed by transmission electron microscopy (TEM) in a JEOL JEM-2200FS microscope operated at 200 kV.

The tensile stress-strain curves of Hi-Nicalon fibers were acquired, which remain linear up to failure. Tensile test results are summarized in Table 1 together with the diameter; they are the average of thirty measurements. The fiber surface before testing seems to be smooth (Fig. 1a), although some of them have flaws coming from the fiber manufacturing process. After the tensile tests the fibers present a classical brittle fracture; images of the fracture surface are shown in Figs. 1b and 1c; there is no change in appearance of the fracture surfaces between the Nicalon and Hi-Nicalon fibers [1]. These brittle fractures, in consort with the linear stress-strain curves, demonstrate that the material strain is everywhere elastic with no plastic yield [3]. The fiber diameter was also measured by SEM in order to calibrate the LSM apparatus, and the result showed that the Hi-Nicalon monofilament family possesses variable diameter along the gauge [4], as can be seen in Fig. 1a. Thin specimens for TEM were prepared by FIB etching (Fig. 1d). Figure 2 presents two high-resolution transmission electron microscopy (HRTEM) images. During their fabrication Hi-Nicalon fibers are pyrolysed up to about 1400°C and so the fiber contains more free carbon, because of this the fiber encloses well ordered ovoid SiC grains, surrounded by Si and C atoms which were not completely crystallized into β-SiC grains. The size of these β-SiC grains were seen to be in the range of 5 to 10 nm.

References:

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Table 1. Mechanical properties of the Hi-Nicalon fibers.

Tensile strength (GPa)	1.9 ± 0.5
Breaking strain (%)	1.0 ± 0.4
Young's modulus (GPa)	204.4 ± 54.2
Diameter (μm)	15.8 ± 0.1

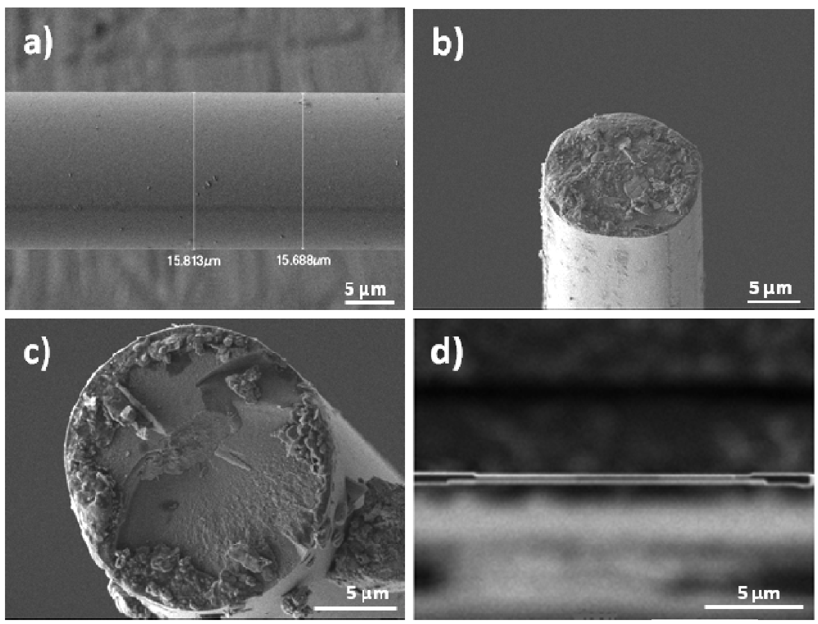

Fig. 1. SEM micrographs of Hi-Nicalon fibers showing (a) the fiber before testing, (b) and (c) the fracture surface, (d) a sample prepared by FIB.

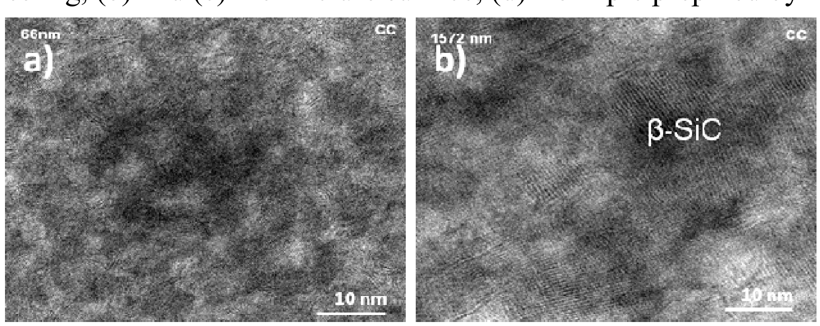

Fig. 2. HRTEM of the as-received Hi-Nicalon fibers, showing β-SiC grains surrounded by poorly organized Si-C phase.