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MECHANICAL RESPONSE OF CROSS-PLY Si_3N_4/BN FIBROUS MONOLITHS UNDER UNIAXIAL AND BIAXIAL LOADING*

D. Singh, T. A. Cruse, D. J. Hermanson, K. C. Goretta
Energy Technology Division
Argonne National Laboratory, Argonne, IL 60439

F. W. Zok and J. C. McNulty
Materials Department
University of California, Santa Barbara, CA 93106

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ABSTRACT

Mechanical properties of hot-pressed Si_3N_4/BN fibrous monoliths (FMs) were evaluated under ambient conditions in four-point and biaxial flexure modes. Effects of cell orientation, 0°/90° and ±45°, on elastic modulus and fracture strength of the FMs were investigated. Fracture surfaces were examined by scanning electron microscopy.

INTRODUCTION

Ceramic fibrous monoliths (FMs), which generally consist of a strong ceramic cell surrounded by a weaker cell boundary, exhibit graceful failure in flexure [1-5]. FMs are produced from powders by conventional fabrication techniques such as extrusion [1,2]. Several compositions of ceramics and cermets have been processed successfully in fibrous monolithic form [4]. The most thoroughly investigated FM consists of Si_3N_4 cells and a continuous BN cell boundary [3-5]. Through appropriate selection of initial powders and extrusion and hot-pressing parameters, strong and tough final products have been obtained. The high toughness is due primarily to the presence of textured platelike BN grains.

Because of the relatively complex microstructures of FMs, predictive modeling of their mechanical behavior is not trivial. In this regard, a program has been initiated at Argonne National Laboratory (ANL), in collaboration with the University of California at Santa Barbara (UCSB), to conduct mechanical evaluation and modeling of FM response under mechanical loading.

We report here preliminary results from Si_3N_4/BN FM specimens that were tested in uniaxial four-point flexure and biaxial flexure under ambient conditions. Samples with two cell orientations to the loading direction, namely 0°/90° and ±45°, have been tested. Mechanical properties, such as elastic moduli and ultimate strength, were measured and the results were compared with those obtained from existing models.

EXPERIMENTAL DETAILS

Fabrication Procedures

The FMs, fabricated at Advanced Ceramics Research (ACR) in Tucson, AZ, were made from flexible Si_3N_4/BN coextruded green filaments [2] that were 320–330 μm in diameter. They were produced by melt coextrusion of a blend of ≈52 vol.% ceramic powder in an ethylene-based copolymer binder [6]. These coextruded filaments contained ≈82 vol.% core Si_3N_4 material (E-10, Ube Industries, Tokyo, Japan) and ≈18 vol.% BN cladding (HCP Grade, Advanced Ceramics Corporation, Cleveland, OH). The Si_3N_4 was a sinterable composition, 92 wt.% commercial Si_3N_4 powder, 6 wt.% Y_2O_3, and 2 wt.% Al_2O_3.

Sheets of uniaxially aligned green filaments were produced by a winding operation that placed the coextruded filaments side-by-side on a cylindrical mandrel. The filaments were held in place with a spray adhesive that, upon drying, allowed removal of the unidirectional sheets of green FM from the mandrel. The sheets were then stacked to fabricate the specimens; two laminated architectures were fabricated, 0°/90° and ±45° [5,7,8]. The laminates were cut into preforms and warm-pressed at 160°C to produce a solid green panel.

Simple rectangular flat panels (≈152 x 152 x 3 mm) were fabricated for mechanical-property evaluation. The panels underwent a binder pyrolysis step that consisted of slow heating in flowing N_2 to 600°C over a period of 42 h. The Si_3N_4/BN panels were then hot-pressed at 1740°C for 1 h under ≈28 MPa pressure. This procedure yielded FM billets that were >98% of their theoretical density. These billets were distributed to ANL and UCSB.

Mechanical-Properties Evaluation

Si_3N_4/BN FM plates with 0°/90° and ±45° orientations were sectioned and tested first in four-point flexure under ambient conditions. Typical dimensions for samples tested at ANL were ≈3 x 4 x 45 mm, with inner and outer loading spans of 15 and 40 mm, respectively. For each specimen type, at least three samples were tested. In addition, strain gauges were attached to tensile surfaces to monitor stress vs. strain. Thus, strains were measured perpendicular to the hot-pressing direction. The tests were conducted at a displacement rate of 1.27 mm/min in an Instron Model 1125 universal tester. Details of the testing of FM samples at UCSB have been presented elsewhere [9]

To investigate flaw sensitivity and failure of FMs under a more-generalized loading configuration, Si_3N_4/BN FMs were also tested under biaxial stress conditions at ANL. Testing was conducted on disk specimens (31.75 mm in diameter and 3 mm in thickness, t) that were placed on a ring support (radius r_l) on one side and loaded transversely by a uniform pressure, p, from the other side. The test cell, a modification of that used by Chao et al. [8], applied load transversely by oil pressure. Each sample and the oil reservoir were separated by a thin metallic membrane. Samples were supported by a ring that consisted of ball bearings around the circumference of the disks (Fig. 1). The ball bearings minimized friction at the loading points. The pressure in the oil reservoir was generated by a piston that was loaded by the Instron crosshead (Fig. 2).

Essentially any controlled loading rate can be achieved. In this configuration, variations of radial (σ_r) and tangential (σ_t) stresses as a function of distance from the center of the disk (r) can be estimated from plate theory for isotropic materials (Eqs. 1 and 2) and can be represented in terms of the maximum equibiaxial stress at the center of a disk (σ_b) and stress gradient parameters α and β (Eqs. 3 and 4).

$$\sigma_r = \sigma_b \left[1 - \alpha \left(\frac{r}{r_1} \right)^2 \right] \tag{1}$$

$$\sigma_t = \sigma_b \left[1 - \beta \left(\frac{r}{r_1} \right)^2 \right] \tag{2}$$

$$\alpha = \frac{3p(3+v)r_1^2}{8t^2\sigma_b} \tag{3}$$

$$\beta = \frac{3p(1+3v)r_1^2}{8t^2\sigma_b} \tag{4}$$

where, σ_b is represented as

$$\sigma_b = \frac{3pr_1^2}{8t^2}\left[2(1-v) + (1+3v)\left(\frac{r_2}{r_1} \right)^2 - 4(1+v)\left(\frac{r_2}{r_1} \right)^2 \ln\left(\frac{r_2}{r_1} \right) \right] + \frac{(3+v)p}{4(1-v)} \tag{5}$$

and r_2 is the radius of the disk sample and v is Poisson's ratio.

Before testing the FMs biaxially, it was necessary to determine the efficacy of the system. Steel and Al_2O_3 disks of known elastic moduli (198 GPa and 368 GPa, respectively [10]) were tested. Strain gauges were attached at the center of the tensile face of each disk, and stress and strain were monitored during testing. Responses were compared with the predictions of plate theory. Figure 3 shows the data of normalized strain as a function of applied stress for the two disks. In addition, theoretical lines of the peak strain (ε_b) as a function of maximum stress at the disk center (σ_b) and elastic modulus (E) have been plotted per

$$\varepsilon_b\, t^2 = [\sigma_b\, (1-v)\, t^2]/E\,. \tag{6}$$

There was excellent correlation between the theoretical lines and experimental data. Moreover, the elastic moduli determined from the experimental data, 205 GPa and 370 GPa for steel and Al_2O_3 samples, respectively, were within 4 and 0.5% of those determined ultrasonically. Thus, it seems that the biaxial system works well and that FMs can be reliably tested.

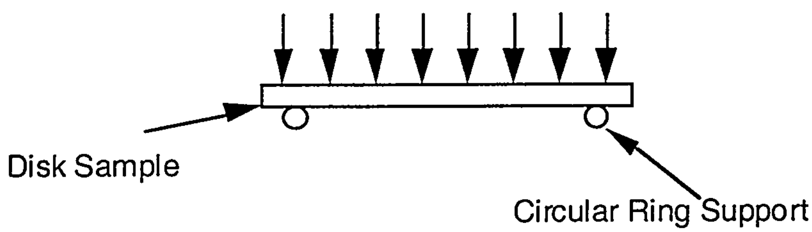

Figure 1. Schematic diagram of loading configuration in biaxial testing.

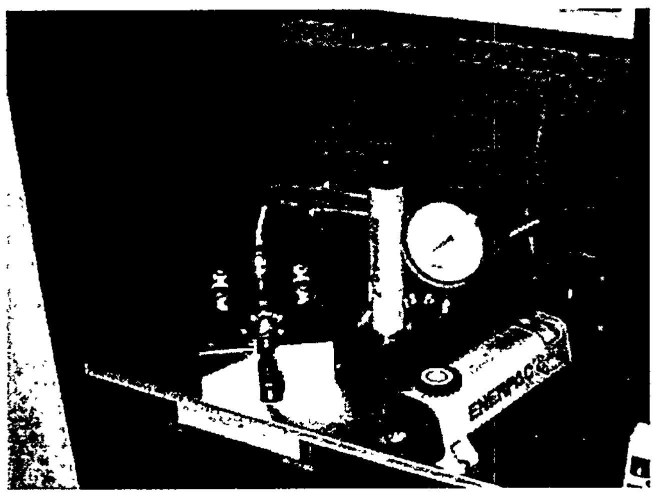

Figure 2. Experimental set-up for biaxial test system.

RESULTS AND DISCUSSION

Typical stress-strain responses obtained for the Si_3N_4/BN FMs, tested in uniaxial and biaxial modes, are shown in Fig. 4. For all samples tested, failure strains were 1000–2500 $\mu\varepsilon$. In addition, all samples exhibited an initial linear stress-strain relationship, followed by deviation from linearity, which may indicate accumulation of damage in the FMs. In general, deviation from linearity occurred at stress levels from 15 to 250 MPa. Elastic moduli for the various FMs were determined from the slope of the linear region of stress-strain plots. Fractures strengths were determined from the peak load.

Analysis of the mechanical-property data in Tables 1 and 2 suggests that there is a distinct difference in the fracture strengths that is based on the testing mode. The biaxial tests exhibit higher strengths than those of the uniaxial tests for both orientations (0°/90° and ±45°) and for billets from both ANL and UCSB. However, one exception is the uniaxial strength data for the 0°/90° sample tested at ANL. Qualitatively, the higher strengths observed for biaxial tests can be explained by the larger effective volume fraction of cells under maximum tension in the uniaxial samples.

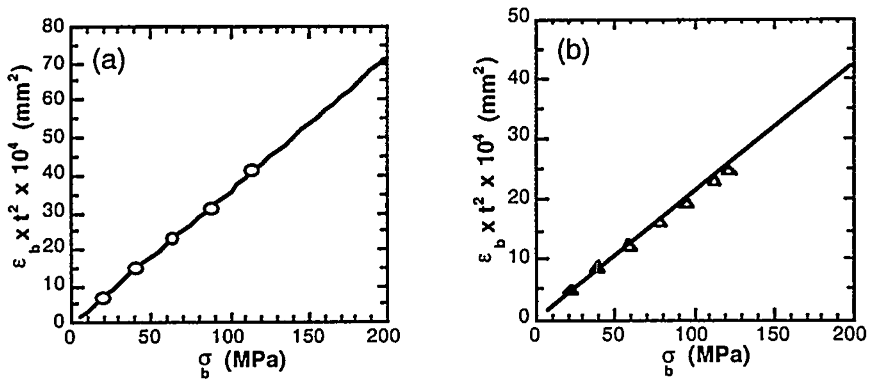

Figure 3. Measured and predicted normalized strain-applied stress for (a) 304 stainless steel and (b) Al_2O_3 samples in biaxial flexure mode

For the biaxial tests, fracture strength is expected to be independent of the orientation of the FMs. This was observed for the 0°/90° and ±45° samples from UCSB billets, which exhibited strengths of 302 ± 32 MPa and 326 ± 18 MPa, respectively. However, the strengths of the ANL 0°/90° and ±45° specimens were 307 ± 27 and 212 ± 35 MPa, respectively. The reason for this discrepancy is not clear at this time, but is probably related to processing variations.

Figure 5 presents the failure modes for the 0°/90° and ±45° samples tested in uniaxial modes. Extensive delamination and crack deflection, as observed by scanning electron microscopy (SEM), were evident in the 0°/90° and ±45° FMs, but the failure origins could not be unambiguously identified. Figure 6 shows the fracture surface of a nominally ±45° specimen that was tested biaxially. As expected, the failures appeared to originate at the center of the tensile surfaces of disks and much delamination was observed. In most cases, the biaxially tested specimens fractured into four approximately equal quarters, from an apparent combination of tensile and shear stresses, as indicated by the fracture of the cells and the substantial delamination.

The ultimate strength of the ±45° specimens exhibited billet-to-billet variations. In both testing modes, strengths were higher for the specimens prepared from the UCSB billet. This difference can clearly be attributed to processing variations.

Table 1. Properties of 0°/90°Si_3N_4/BN FMs tested in uniaxial and biaxial modes.

Test Mode	Billet Source	Test Location	Measured E (GPa)	Strength (MPa)
Uniaxial	ANL	ANL	158 ± 34	379 ± 86
Uniaxial	UCSB	UCSB	180	236 ± 20
Biaxial	ANL	ANL	165 ± 20	307 ± 27
Biaxial	UCSB	ANL	188	302 ± 32

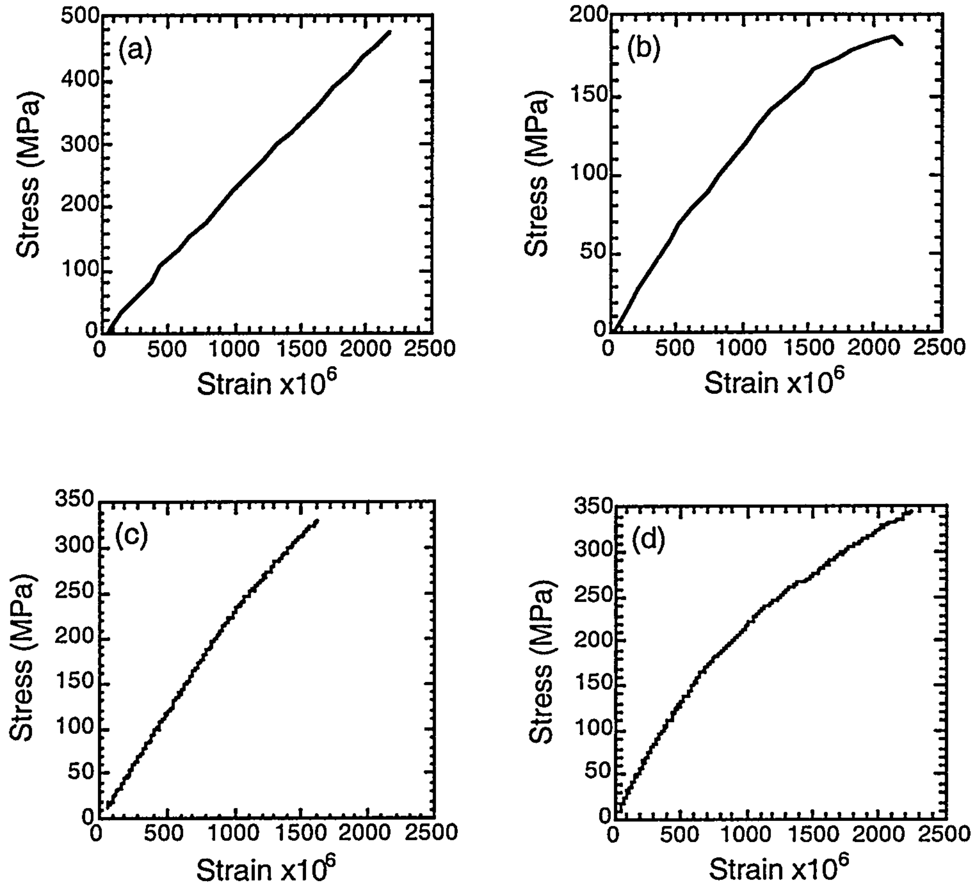

Figure 4. Typical stress-strain data for Si_3N_4/BN FMs tested in uniaxial and biaxial modes.

Table 2. Properties of ±45° Si_3N_4/BN FMs tested in uniaxial and biaxial modes.

Test Mode	Billet Source	Test Location	Measured E (GPa)	Strength (MPa)
Uniaxial	ANL	ANL	92 ± 15	175 ± 13
Uniaxial	UCSB	UCSB	176	227 ± 17
Biaxial	ANL	ANL	181 ± 12	212 ± 35
Biaxial	UCSB	ANL	188 ±25	326 ± 18

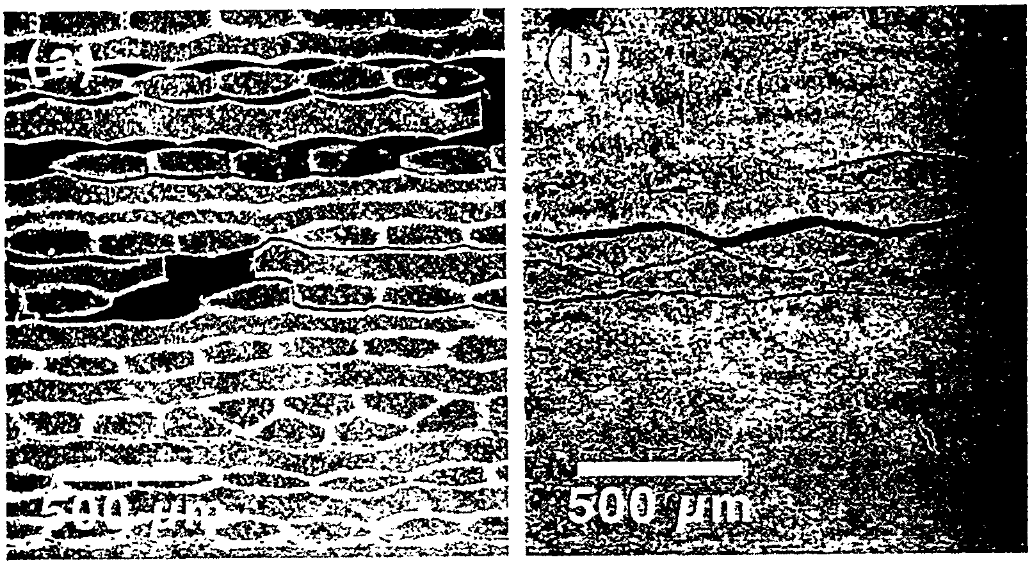

Figure 5. SEM photomicrographs of (a) 0°/90° and (b) ±45° Si_3N_4/BN FMs tested in uniaxial mode, showing delamination and crack deflection.

Figure 6. SEM photomicrograph of tensile fracture surface of ±45° Si_3N_4/BN FM tested in biaxial mode; interlayer delamination is evident

SUMMARY

- Mechanical properties of Si_3N_4/BN FMs with two orientations (0°/90° and ±45°) were tested in uniaxial and biaxial modes under ambient conditions.
- In general, the ultimate strengths and elastic moduli of samples tested biaxially were higher than those of specimens that were tested uniaxially.
- Although the data were fairly consistent, billet-to-billet variations were observed.
- Failure of Si_3N_4/BN FMs under biaxial testing appeared to occur from a combination of tensile and shear modes.

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