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## Monitoring interlaminar crack growth in ceramic matrix composites using electrical resistance

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This letter introduces a method that uses electrical resistance to monitor crack growth during interlaminar fracture testing of woven SiC fiberreinforced SiC matrix composites at room temperature without visual observation. The estimated crack length is in excellent agreement with the measured length after subtracting a constant value of resistance related to the initial stage of crack development. This non-visual monitoring method holds great promise for in situ measurement of crack growth during high-temperature testing of ceramic matrix composites. © 2014 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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Ceramic matrix composites (CMCs) are considered to be one of the most promising materials for high-temperature applications. In particular, woven silicon carbide (SiC) fiber reinforced SiC matrix (SiC/SiC) composites stand out for having good creep and rupture resistance, low density, high toughness, environmental stability and reasonably good thermal conductivity. The mechanical behavior of CMCs under tensile loading has been widely studied [1,2], but there is little work available on other types of damage, such as interlaminar crack growth [3], and their impact on the material's life expectancy. A better understanding of interlaminar crack growth is thus necessary at both room and high temperatures.

The validity of interlaminar fracture tests relies on accurate monitoring of crack growth. Numerous nondestructive testing methods, such as X-ray, ultrasonic C-scan, thermography and eddy current, have been shown to be sensitive to out-of-plane damage (delamination-type cracks) in ceramic matrix composites [4]. However, these techniques either cannot be used for in situ monitoring or do not have the required high-temperature capability at a moderate cost [5]. It has been reported that electrical resistance (ER) provides a sensitive measure of internal damage in CMCs. In melt-infiltrated SiC/SiC composites electrical resistance is particularly sensitive to matrix cracking due to the presence of silicon, which results in low electrical resistivity of the matrix [6]. In this letter, a method is presented to monitor and measure interlaminar crack growth during fracture testing using electrical resistance.

Such a method will be extremely useful in tests where the setup does not allow for visual observation, e.g. during high-temperature testing, because of the limited visual accessibility to the area of interest.

Several methods have been proposed for interlaminar fracture testing of CMCs [7]. Although the double-cantilever-beam (DCB) method is the most widely used, a wedgeloaded DCB was employed here (Fig. 1). In this method, a splitting force is created by inserting a vertically moving wedge in a notch, thereby causing the arms to separate and forcing an interlaminar crack at the sharpest end of the notch. The main advantages of this method are the simplicity of the specimen preparation and the distance between the wedge location and the cracking area, which will be convenient for high-temperature testing. In the present work, an alumina wedge with 18° head angle was used. The wedge was held in the top grips of an MTS machine Model 43, while the specimen was held in the bottom grips over a length of about 5 mm. The wedge was inserted in the specimen's notch at a constant displacement rate of 1 mm  $min^{-1}$  to initiate and propagate the interlaminar crack. Optical microscopy was used to monitor crack growth on the back surface of the specimen. Every  $\sim 2$  s an image was captured by a camera connected to the microscope. The surface was painted with white paint prior to testing in order to help elucidate the crack [3].

The material used in this study was a slurry-cast meltinfiltrated SiC/SiC composite with Tyranno ZMI fibers (Ube Industries, Kyoto, Japan) and a BN interphase. The fiber architecture consisted of eight plies of balanced 2-D woven five-harness satin. The total fiber volume fraction was about 30%, with half of the fibers in the 0° direction and half in the 90° direction. Specimens were 50 mm in

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Figure 1. Schematic of the test setup with the two ER configurations: (a) arm-to-arm and (b) straight. P is the vertical component of the load applied by the wedge, I represents the constant current source (10 mA), V is the voltmeter and AE are the acoustic emission sensors. L, h and tare geometric parameters and a is the crack length.

length, 5 mm in width and 4 mm in thickness. A notch was machined on each specimen in the midplane with a thickness of 0.25 mm and a length equal to about 1/3 of the total specimen length ( $\sim$ 15 mm).

Electrical resistance was measured by a four-point method using an Agilent 34420A meter. During the measurement, a direct current (10 mA) was applied through the outer probes, and the voltage was measured across the two inner probes to monitor the resistance of the material. The advantage of this procedure is that it minimizes the effect of contact resistance on the measurements, since the current through the inner probes should be near zero. Two configurations were considered for probe attachments: arm-to-arm and straight configurations (Fig. 1). In the armto-arm configuration, all probes were attached to the arms with the current passing around the notch, compared to two probes attached to the arms and two connected at the bottom of the specimen for the straight configuration. The arm-to-arm configuration was selected as the method of choice because of its ease of use, accuracy (as will be discussed later) and suitability for high-temperature testing since all the probes would be away from the hot zone. For probe attachments, thin strips of conductive silver paste were applied around the specimen surface. The electrical resistivity of the as-received material was  $0.15 \Omega$ -mm.

Assuming that the axial resistivity of the composite is homogeneous and that the notch is perfectly centered in the middle of the specimen, the resistance will increase as the crack forms and propagates due to the opening of the arms from the ceramic wedge. The total resistance can be expressed as follows for the arm-to-arm configuration:

$$R_{total} = 2\frac{\rho L}{A_{\rm arm}} + 2\frac{\rho a}{A/2} + R_x \tag{1}$$

where  $\rho$  is the axial electrical resistivity of the undamaged composite,  $A_{arm}$  is the cross-sectional area of the arm, Ais the cross-sectional area of the composite, L is the distance from the inner lead to the end of the notch, a is the crack length and  $R_x$  is a constant additional resistance that will be explained later. Solving for the crack length a and substituting the dimensions h (arms thickness), t (specimen thickness) and w (specimen width) for the appropriate areas, the crack length can be estimated using the following equation:

$$a = \frac{wt}{4\rho}(R - R_x) - \frac{Lt}{2h}$$
(2)

For the straight configuration, the total resistance can be expressed as:

$$R_{total} = \frac{\rho L}{A_{\rm arm}} + \frac{\rho a}{A/2} + \frac{\rho [D_{\rm in} - (L+a)]}{A} + R'_{x}$$
(3)

where  $D_{in}$  is the distance between the inner probes. Solving for *a* gives:

$$a = \frac{wt}{\rho} \left( R - R'_x \right) - \frac{Lt}{h} + L - D_{\rm in} \tag{4}$$

Although both methods are valid for estimating crack growth depending on the increasing electrical resistance, the arm-to-arm configuration is more accurate since it has higher sensitivity compared to the straight configuration: from Eqs. (2) and (4), and for a 1 mm increase in crack length, the arm-to-arm configuration shows 75% more sensitivity compared to the straight configuration, using the same values for  $\rho$ , w, t, h and L. Therefore, the arm-to-arm configuration method was used to estimate the crack length. The only misrepresentation accompanied with using the arm-to-arm configuration is that it does not take into account the electrical resistance associated with current going around the notch or the crack tip. However, we considered this resistance to be constant throughout the test and included in the term  $R_x$ .

Modal acoustic emission (AE) was also monitored during tests using two wide band sensors (B1025, high sensitivity from 50 kHz to 2 MHz) placed back to back just above the bottom grips. The AE sensors were clamped to the specimen surface and vacuum grease was used as a coupling agent. The waveforms were recorded by a four-channel fracture wave detector acquisition system (Digital Wave Corporation, Centennial, CO). Software from the same company and an in-house Matlab program were used to filter and analyze the recorded waveforms [8–10].

A typical AE waveform recorded during mechanical test of a CMC specimen consists mostly of the zero-order flexural and extensional modes,  $A_0$  and  $S_0$ , respectively. In the frequency range considered in AE (up to 2 MHz), the high-frequency  $S_0$  mode has a constant velocity greater than that of the lower-frequency  $A_0$  mode. As a result, the recorded waveforms will show an increasing separation between the extensional mode (reaching the sensor first) and the flexural mode (travelling at lower velocity) with increasing propagation distance. During the wedge test, two main types of AE waveforms were recorded: one with clear separation between the high-frequency extensional mode and the low-frequency flexural mode, the other with superimposed low- and high-frequency components (Fig. 2). The first type (Fig. 2a) was associated with chipping, friction and/or grooving at the wedge surface. The corresponding AE waves propagated a significant distance to reach the AE sensors, leading to the clear separation observed between the two modes. This type of waveform, being unrelated to material damage, was filtered out. The second type of waveform (Fig. 2b) originated from sources closer to the sensors, as shown by the shorter extensional region prior to the superimposed modes dominated by the

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