In situ imaging and strain determination during fracture in a SiC/SiC ceramic matrix composite

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A combined imaging and microdiffraction technique using high-energy synchrotron X-rays is described and used to reveal microstructure, damage and strain evolution around notches in SiC/SiC composites. This technique allows for monitoring the material for cracks while loading and mapping the strain distribution in fibers and matrix with a resolution of tens of microns. We show that at current resolutions this technique is capable of measuring the strain distribution near crack tips in ceramic matrix composites and observe load transfer effects.

Ceramic fiber-reinforced ceramic matrix composites (CMCs) are currently being developed for several high-performance applications at elevated temperatures [1–3]. Such applications are realizable because reinforcement by brittle fibers of a brittle matrix, when properly designed, imparts flaw tolerance and toughness to the system. A variety of constituent phases, including oxides, carbides, nitrides and borides, fiber architectures and microstructural design allow for a variety of CMCs with tailored properties to be manufactured [4]. Despite the number of potential applications of CMCs, lack of experimental data at service conditions and limited in situ studies make it very difficult to predict in-service lifetimes for these materials. There is a need, therefore, for experimental setups that allow for the study of fracture processes in conditions similar to those found in service, in real time, where the strain field can be determined while the fracture progression is observed.

Several synchrotron X-ray studies in composites have been performed previously, by diffraction, radiography, tomography [5] or a combination of them [6]. Most attention has been devoted to unidirectional metal matrix composites containing aligned ceramic monofilaments (>100 μm), since the diameter of the reinforcing phase is well over the typical spatial resolution in microdiffraction setups allowing for determination of strain on individual monofilaments [7,8], as well as fiber bridging and sliding stresses [9,10]. Current generation CMCs, alternatively, are reinforced by yarns of fibers ~10 μm in diameter woven at right angles, and strain determination in a statistically significant number of individual fibers is impossible with current setups. Crack propagation and crack patterns have also been studied using imaging techniques in the synchrotron, but these experiments are most often carried out ex situ, since the sample is usually pre-cracked and then imaged, and the process is repeated several times (e.g. in Ref. [6]). Thus, in this work we propose an experimental set-up that allows both imaging and diffraction to be performed sequentially and in situ during loading, examining composites with reinforcements typical in size for current CMCs.

The composite studied was a melt-infiltrated SiC/SiC composite. First, a preform was obtained by stacking...
pieces of two-dimensional woven five harness, balanced satin cloth of Sylramic(c)-iBN fibers. This preform was CVI infiltrated with SiC, slurry infiltrated with particulate α-SiC and finally melt-infiltrated with Si to fill the remaining porosity; details of its production can be found elsewhere [11]. Panels 2 and 4 mm thick were studied (see Fig. S1 in the supplementary materials). X-ray diffraction confirmed that the material is made mostly of β-SiC from the fibers and CVI coating, with α-SiC as the main constituent of the melt-infiltrated matrix, which contained small amounts of Si from the melt infiltration process (see fig. s2 of supplementary material). Coupons measuring 12.7 mm × 22.5 mm were cut using a low speed diamond blade with their sides parallel to the 0/90° directions of the fibers. Two holes, 4.5 mm in diameter and 18 mm apart, were drilled in the coupons using an ultrasound-assisted drill press to apply a tensile load to the specimens using a clevis-and-pin mechanism. Two notches 4.6 mm deep were cut into the sample using a low-speed wafering blade with a thickness of 0.3 mm; notch roots were semicircular and their radius was confirmed to be ~0.15 mm by scanning electron microscopy observations.

In situ diffraction and radiography experiments were conducted at ambient temperature at beamline 1-ID of the Advanced Photon Source of Argonne National Laboratory. The setup is depicted schematically in Figure 1. High-brilliance X-rays with an energy of 70 keV were produced by a Laue monochromator providing an incident beam of ~1012 photons s⁻¹ mm⁻². Two sets of motorized W slits served to produce a rectangular beam that illuminated the sample. Load was applied using a universal testing machine mounted on a motorized stage that allowed for sample translation in three dimensions.

For radiographic imaging, the incident beam was opened to 2.5 mm (H) × 1 mm (V). Transmitted images were recorded with an in-line LaG scintillator crystal, 45° mirror and a CCD camera oriented at 90° to the incident beam. A 5× objective was used with the 4 MP camera, providing a nominal resolution of 1.5 μm pixel⁻¹. To correct for uneven background illumination, bright-field exposures were acquired by moving the sample out of the beam periodically during the course of the experiments. Radiographic images were median-filtered to remove salt-and-pepper noise, cropped, rotated, aligned and finally divided by the bright-field exposures to remove the background. Radiographs were acquired at a rate of approximately 1 s⁻¹ while the load was increased in constant displacement rate mode, resulting in a nominal strain rate of ~2 × 10⁻⁴ s⁻¹. Since the illuminated area was slightly smaller than the distance between notch roots, the sample was translated horizontally so that radiographs of the areas around both notches were taken alternately, and this translation accounted for most of the acquisition time. Once an interesting feature such as a crack was observed, loading was stopped and the slits were brought progressively closer, in an iterative fashion, until the beam reached the desired size, and the feature of interest was in the beam for strain measurements. Once strain maps were acquired, loading and imaging of the sample was resumed.

Strain in the composite as a function of applied load was measured using X-ray microdiffraction. An amorphous Si area detector (41 × 41 cm², 200 μm pixel size) was placed 1.14 m from the sample, and a CeO₂ standard was used to accurately calibrate for sample-to-detector distance, detector tilt and beam center coordinates. In our setup, the CCD and area detectors were mounted on motorized stages, and were inserted and retracted as needed for imaging and diffraction, respectively.

Forward-scattered diffraction formed Debye rings in the detector, which were fitted to pseudo-Voigt peak profiles to determine the peak centers as a function of azimuth. Strain was then calculated using:

$$ e = \frac{r_0 - r(\eta)}{r_0} $$

where \( r(\eta) \) is the ring radius at angle \( \eta \) and \( r_0 = r(\eta^*) \) is the stress-free radius, which is the measured radius at the strain-free angle \( \eta^* \), calculated from the Poisson’s ratio of the material assuming a purely uniaxial stress using:

$$ v = \frac{\sin^2 \eta^*}{1 - \sin^2 \eta} $$

A value of \( v = 0.17 \) for both α and β-SiC was used for strain determination. This value is the mean of Voigt and Reuss averages from single crystal data, and has been used successfully in the past for similar SiC/SiC composites [12]. Eq. (1), therefore, allows us to calculate the average strain tensor in the diffracted volume, projected along the beam direction. One reflection from each phase, (311) for β-SiC and (109) for α-SiC, was used for strain determination. Strains were mapped by shifting the sample at constant intervals along the x and y directions. Typical low-resolution maps comprising the whole region between notches used a beam size of 200 μm (horizontal) × 100 μm (vertical) and consisted of 21 × 10 points, respectively. The exposure time was 1 s, and five exposures from the same volume were averaged together for a higher signal-to-noise ratio; this resulted in ~25–30 min per map when the time needed for sample translation was added. Load–displacement curves were recorded during experiments and showed a typical non-linear behavior characteristic of these composites [3]. A small amount of relaxation was observed while the crosshead displacement was stopped for diffraction mapping, evidenced as a small decrease in load (see Fig. S3 of the supplementary material). Figure 2 shows one of the acquired strain maps and strain profiles at three different loads in the 2 mm thick panel. In

![Figure 1. Experimental setup used in beamline 1-ID (not to scale). See text for a description.](image-url)
Figure 2a strain profiles across the load bearing area are shown for axial, transverse and shear strain components; it can be seen that the latter was negligible, thus confirming that sample alignment was reasonably good during the experiments. Strains concentrate near the notches, as expected, and increase with increasing load. At the highest load, the sample response was just beyond the linear elastic region, and the axial strain profiles are comparable to those previously determined by Gyekenyesi and Morschler [13] by in situ thermoelastic stress analysis under cyclic loading. Further insight can be gained by observing the maps of the axial strain components, depicted in Figure 2b. Strain concentration was observed near the notches, as expected, but in regions emanating at 45° angles from the notch tip and at opposite angles from each other. This matched the crack propagation patterns observed during radiography, especially at higher loads, where the cracks were more easily seen. Inhomogeneities in the strain maps were observed and attributed to the presence of pores and voids that not only affect the stress field, but also result a smaller amount of material in the diffracted volume.

Microstructural inhomogeneities, which have been shown to be responsible for fracture behavior in these materials, pose particular challenges for quantitative analysis of the local strain field as well as load transfer effects. For instance, melt-infiltrated SiC/SiC composites can be thought of as having a dual matrix, composed of the melt-infiltrated SiC/Si and CVI SiC regions. Cracks have been shown to originate at the former and then propagate to the latter [14]. The presence of this dual matrix also complicates the X-ray diffraction interpretation since the former is composed of α-SiC while the latter is composed of β-SiC and is indistinguishable from the fibers. Nonetheless, with selected assumptions, we can obtain some quantitative information from the measured strain maps and profiles. Since this composite is made of woven fibers, approximately half of the fibers are not bearing any load, so the thickness-averaged strain calculated from the X-ray diffraction patterns, shown in Figure 2a, can be estimated to be half of the strain in the load-bearing fraction of the fibers. The net section sample between the notches is (3.5 × 2.0) mm², which at the highest load corresponds to a net stress of 130 MPa and a linear elastic stress of ~390 MPa since the stress concentration factor is approximately 3.1 in this geometry [15]. This value is well over the proportional limit of 180 MPa measured in these composites, which corresponds to ~700 µε using $E = 260$ GPa [16,17] for the macroscopic Young’s modulus of the composite. Measured, volume-averaged strains in the near-notch region range from 900–1200 µε for the load-bearing fraction of the sample (twice what is observed in Fig. 2a), which means that fiber bridging is already taking place. It has been shown that the stress concentration factor increases with damage progression in these composites [13]; in our case, the average strain in the region between the notches (from $x = 0.5$ mm to $x = 3.0$ mm in Fig. 2) is~300 µε for the load-bearing fibers, in agreement with the predicted elastic strain at 130 MPa (350 µε using $E_{\text{fiber}} = 380$ GPa [18]), and the measured stress concentration factors at the notches are then 3–4, indicating that matrix cracking was already present and that the measurement was performed past the proportional limit.

Figure 3 (video online) shows a sequence of radiographs near a notch in the 4 mm panel composite, where matrix cracks can be seen to start emanating from the notch tip at a load of ~1 kN. It is evident that fibers 90° apart from each other can be easily distinguished in the radiographs. Cracks emanate horizontally at first, but then deviate and branch at a 45° angle in accordance with the strain distribution. Failure of this sample occurred at ~2.2 kN, which corresponds to a net section stress of 160 MPa and a near-notch stress of 480–640 MPa using previously measured SCFs, and extensive fiber pullout was observed post-mortem in the scanning electron microscope (see Fig. S4 of the supplementary material). Crack patterns were easily observed with this technique once cracks of significant length were formed, but cracks of a length comparable to inhomogeneities in the material were difficult to distinguish.

Before failure, a high-resolution strain map of beam-size 100 µm (horizontal) × 50 µm (vertical) and spanning 0.5 mm² was acquired near the notch tip, as shown in Figure 4. The load corresponds to a net section stress of 125 MPa, similar to the highest mapped load for the 2 mm thick sample. Axial strains in both reinforcement (top) and matrix (bottom) are shown, and the mapped area corresponds to the region marked in Figure 3. A region of higher strain in the reinforcement...
and lower strain in the matrix is marked with an arrow. It can be seen that these regions approximately coincide with the pattern of cracks emanating from the notch tip. A load transfer effect is therefore observed, as strain is transferred to the fibers when cracks form in the matrix.

Although, in the present case, both spatial and mesh resolution are probably too low to elucidate the strains in a scale comparable with the features of the crack pattern, a qualitative description of the material’s behavior is provided. A quantitative analysis would require mapping a large area near the notch to ensure that all bridging cracks are accounted for. For instance, in Figure 3, the high strain region emanating from the bottom of the notch could be attributed to a second crack at 45° to the loading direction that could be relaxing the crack above, making unambiguous interpretation difficult. In the near future we anticipate development of stitching techniques for high-energy X-ray tomography, which will allow for volumes of several mm³ to be mapped with micron-level resolution. Additionally, faster diffraction area detectors and beam sizes down to 5 µm, attainable through focusing optics, will allow diffraction information to be captured with similar resolutions. Together, these will provide higher fidelity data, by building off the experimental framework presented here.

Likewise, the setup we describe can easily enable a whole new range of experiments simply by setting the sample in a specially designed furnace, for truly in situ studies of CMCs in real service conditions.

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Figure 3. Representative radiographs near a notch showing cracks emanating from the semicircular notch tip, at increasing loads. Note that the crack pattern matches the regions of higher and lower strains in fibers and matrix, respectively, presented in Figure 4.

Figure 4. High-resolution strain maps of 10 x 10 points spanning 1 mm horizontally and 0.5 mm vertically. The beam size was 100 µm (horizontal) x 50 µm (vertical). Compared to Figure 3, a region of higher-than-average strain in the fibers (top) can be seen to match with the crack pattern, which also coincides with a region of lower-than-average strain in the matrix (bottom).