X-RAY STRESS ANALYSIS OF DAMAGE EVOLUTION IN Ti-SiC UNIDIRECTIONAL FIBER COMPOSITES

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ABSTRACT
A composite’s local response to initial damage under stress is the primary micromechanical process determining its fracture toughness, strength, and lifetime. Through the use of high energy X-ray microdiffraction, the elastic lattice strains of both phases in a Ti-SiC composite were revealed providing the in-situ load transfer under applied tensile stress at the scale of the microstructure. To understand the damage evolution, the measured strains were compared to those predicted by a modified shear lag model. Comparisons between the model and the data demonstrated the importance of accounting for the matrix axial and shear stiffness, provided an optimal stiffness ratio for load transfer and planar interpretation of the geometry in the composite, showed the matrix within and around the damage zone sustained axial load, and highlighted matrix yielding observed in the composite. It was also shown that an area detector is essential in such a study as it provides multiaxial strain data and helps eliminate the ‘graininess’ problem.

INTRODUCTION
Load transfer from a broken fiber to the rest of a composite is one of the fundamental micromechanical processes determining composite strength, lifetime and fracture toughness. It is a complex process that depends on fiber/matrix interface properties, the constitutive behavior of matrix and fibers, the geometric arrangement of fibers, fiber volume fraction, and fiber strength distribution. This process is further complicated since the in-situ mechanical properties of the constituents are significantly different from those of their monolithic forms [1-4]. In composites, the macroscopic stress-strain curves obtained by conventional means result from the co-deformation of the individual phases making it impossible to determine the phase-specific in-situ constitutive behavior. Typical composite deformation includes: collective nucleation and evolution of damage, fiber fractures, matrix fractures and plasticity, as well as interface separation and sliding.

To predict the strength and lifetime of a fiber composite, the load transfer from broken fibers to the surrounding intact material must be understood. This requires accurate determination of stress-strain evolution at the scale of microstructure - usually on the order of the fiber diameter. In special cases, this has been achieved using optical methods such as micro-Raman and piezospectroscopy [4-8]. These studies provided valuable insight about fiber strains in damaged composites at length scales approaching several µm. However, in most of these studies either the matrix could not be characterized, or only shallow surface regions were investigated.
The following describes the use of X-ray microdiffraction to determine the phase specific *in-situ* load transfer and damage evolution under applied tensile stress in a metal matrix composite (MMC). Synchrotron X-rays were required to obtain the intensity to reduce the beam size below fiber diameter (140 µm) while maintaining sufficient diffraction statistics and strain resolution in a reasonable time. No other technique, including neutron diffraction, could provide the phase specific, spatially resolved strain information so crucial to understanding MMC deformation. Strains from the matrix and fibers were compared to predictions by a general micromechanics model [9]. This “matrix stiffness shear lag” (MSSL) model accounts for the linear elastic co-deformation of fiber and matrix in a wide variety of unidirectional fiber composites containing any configuration of multiple fractures. To the authors’ knowledge, this is the first damage evolution study conducted on a continuous fiber MMC where both matrix and fibers are investigated simultaneously at the microstructure scale.

**EXPERIMENTAL PROCEDURE**

*Sample Preparation*

![Diagram of sample and loading geometry](image)

Figure 1. (a) Schematic of sample and loading geometry. (b) Idealized crack geometry for the specific model under consideration [9].

The composite system consisted of a single row of unidirectional SiC fibers (Textron SCS-6, diameter, \(D_f = 140 \) µm) in a Ti-6Al-4V matrix, manufactured into a 0.2 mm thick 16 mm wide laminate by 3M Corp. (St. Paul, MN 55144). The fibers were uniformly spaced with an average center-to-center distance of 240 µm. The average fiber area fraction was 32%. Two damaged composites were investigated. In the first one, HF acid was used to partially expose several fibers; then one fiber was broken with a sharp tip. The damage in the second composite was created using wire electro discharge machining (EDM): a small hole was cut by plunging the wire into the center of the composite. The remaining specimen dimensions were (Figure 1(a)): thickness, \(t = 0.20 \) mm, gage length, \(L_g = 26.00 \) mm, and gage width, \(W_g = 10.25 \) mm for the first
composite and $W_g = 7$ mm for the second one. On the surface, a set of strain gages measured the applied macroscopic strain in the longitudinal direction, parallel to the fibers (Figure 1(a)).

**X-Ray Diffraction Analysis**

Both composites were examined at beamline 1-ID-C (SRI-CAT, Sector 1) of the Advanced Photon Source (APS), Argonne National Laboratory. For the first specimen, 25 keV X-rays were used (wavelength, $\lambda = 0.497$ Å) with a scintillator detector equipped with a Si (111) analyzer crystal. The study of the second composite employed 65 keV X-rays ($\lambda = 0.190$ Å) and a digital image plate. A Si powder (NIST Standard Reference Material 640a) was attached to each sample as an internal standard minimizing systematic errors such as the displacement error [10]. The Si powder was also used to calibrate the image plate [11].

The specimens were mounted in a load frame perpendicular to the X-ray beam and examined in transmission mode. In this orientation, the diffraction vector lied nearly parallel to the plane of the composites. The X-ray energies were chosen to assure sufficient penetration so that the collected data represented the through-thickness average. Due to the one-dimensional nature of the scintillator detector, only the longitudinal (or axial) strains could be measured in the first composite [12,13]. On the other hand, the image plate provided the axial, transverse, and shear strain in the plane of the second composite according to [14]:

$$a^2 \varepsilon_{11} + 2ab\varepsilon_{12} + b^2 \varepsilon_{22} + 2ac\varepsilon_{13} + 2bc\varepsilon_{23} + c^2 \varepsilon_{33} = \ln \left( \frac{\sin(\theta_0)}{\sin(\theta)} \right)$$

where, $a = \sin \theta$, $b = -\cos \eta \cos \theta$, $c = -\sin \eta \cos \theta$, and $\ln(\sin \theta_0 / \sin \theta)$ represents the diffraction cone distortion for a particular $(2\theta, \eta)$ position ($\eta$ is the azimuthal angle). A biaxial strain state was assumed so the $\varepsilon_{11}$ strain component was along the fiber axis. A computer program was written capable of performing a least square fit of this strain equation to the diffraction rings. The diffraction rings were fit using a pseudo-voigt peak profile in $2\theta$ over 120 azimuthal divisions of $\eta$. Automation of the strain analysis allowed a large array of positions to be analyzed at multiple positions around the damage zone in the second composite [12,15].

The location of the buried fibers around the damage region in each specimen was determined with absorption contrast exploiting the large difference in the absorption coefficients of the matrix and fibers. A Si diode, which monitored the transmitted beam intensity, provided the contrast in this case. This way, it was possible to accurately position the sample coordinate system with respect to the laboratory system.

Damage evolution under tensile stress in both composites was observed with a $90 \times 90 \mu m^2$ beam. This spot size was smaller than either an individual fiber diameter or an inter-fiber matrix region ($W = 100 \mu m$). The X-ray spot was translated in 280 µm steps along fiber axis in the first composite. The four nearest fibers adjacent to the initially broken fiber (nos. = +1, +2, -1, -2), the broken fiber itself (no. = 0), and the intervening matrix regions (i.e., five fibers and four matrix regions - see Figure 1(b)) were scanned along the fiber axes for a distance of 10 fiber diameters in each direction away from the break. Additionally, at a significant distance from the break (1.89 mm from the center of the nearest scanned fiber around the damage region), one fiber and its adjacent matrix region were scanned to obtain a measure of the *in-situ* applied far-field strain in the sample. In the second composite, ten fibers, labeled “A” through “J”, around
the damage zone and the intervening matrix columns, labeled “a” through “j”, were investigated (these were labeled differently to avoid confusion with the first composite). In this case, the step size was 75 µm, again along fiber axis, and covered a distance of 825 µm. The far-field strain locations in the second composite were +/-1425 µm in each direction away from the hole.

Elastic lattice strains in the matrix and fibers of both specimens were obtained by tracking the position of one reflection from the dominant phases in each: (10·2) from α-Ti and (220) from β-SiC. Previous investigations [16] had showed that these reflections were good representatives of the bulk average in each phase.

The first composite was stressed in tension and strain measurements were performed at 90, 420 and 530 MPa. The second composite was subjected to a tensile loading-unloading cycle up to 850 MPa. In this case, in addition to total strain at maximum load, residual strains before and after loading were also determined.

MICROMECHANICS MODEL

As will be shown below, the diffraction data obtained from both composites are detailed enough to be used to validate a mechanics model that simulates damage evolution in fiber composites. A recent model [9] based on the shear lag concept was chosen for this purpose. Called the “matrix stiffness shear lag” (MSSL) model it quickly computes the stresses and displacements in unidirectional fiber composites in response to multiple fiber and matrix breaks. Unlike previous models, here both the fibers and matrix are able to sustain longitudinal load. Other significant advantages of the MSSL model are: computation is tied to the amount of damage, rather than the entire volume considered; discretization is unnecessary, removing any uncertainties associated with meshing; and solutions are provided for any arrangement of fiber and matrix fractures.

The MSSL model considers a planar, two-dimensional composite with a single row of aligned, parallel and continuous fibers (as shown in Figure 1(b)). The analysis assumes the thickness, \( t \), in the out-of-plane direction is the same for the fibers and matrix. The width of each matrix region is \( W \), and the width of the fibers is \( D \). The transverse displacements of the system and the shear deformation of the fibers are neglected. Both the fiber and matrix are assumed to be linear elastic and well bonded. Therefore, the relevant material properties are the longitudinal Young's moduli of the fibers and matrix, \( E_f \) (= 393 GPa) and \( E_m \) (= 125 GPa), respectively, and the longitudinal shear modulus of the matrix, \( G_m \) (= 48 GPa). As indicated in Figure 1(b), both the fiber and matrix are subjected to a uniform applied stress \( \sigma \), and thus the same far-field strain \( \varepsilon \).

This model provides the average longitudinal strain over the \( n \)th fiber cross-section, \( \varepsilon_f^m(x) \), and average matrix longitudinal strain \( \varepsilon_m^m(x) \) across the \( n \)th matrix region \( Wt \).

The critical parameters of the MSSL model are the longitudinal stiffness ratio (\( \rho \)) and the shear lag decay length (\( \delta \)) given by:

\[
\rho = \frac{E_m A_m}{E_f A_f} \quad \text{and} \quad \delta = \sqrt{\frac{E_f A_f W}{G_m t}}
\]

where, \( A_f \) is the cross-sectional area of the fiber, and \( A_m \) is the cross-sectional area of the matrix between two consecutive fibers. The stiffness ratio \( \rho \) can range from 0 to infinity, but it is usually between 0 and 1 for most MMCs [9]. In this study, the ‘optimum’ value of \( \rho \) for the Ti-SiC composites was found to be around 0.290 [13,15]. The shear lag decay length can be used to
normalize the axial coordinate \((x\) in Figure 1(b)) as \(\xi = x/\delta\). Finally, it is worth mentioning that the MSSL model distinguishes between two different cases of crack configuration: a transverse matrix crack extending from the fiber break to the next fiber, case (ii), versus no crack extension from the fiber break into the matrix, case (i), as illustrated in Figure 1(b). Additional details about the model and its application to the MMC studied here are described in references [9,12,13,15].

RESULTS AND DISCUSSION

Strain data obtained from both composites allowed the monitoring of damage evolution \textit{in-situ}. This way, it was possible to investigate the deformation of a fiber composite, for the first time, at the scale of its microstructure by collecting strain data from all of its constituents. Both composites started with a single broken fiber, but experienced additional fiber fracture during loading. Fiber strain data from both composites clearly exhibited this evolution of damage. Fiber strain data also showed good correspondence with the predictions of the MSSL model.

The strain data obtained from some of the fibers in the first composite (investigated using a scintillator detector) are shown in Figure 2. Before comparison, the initial residual strains were subtracted from the total strains since the MSSL model does not consider them. Fiber 0 was broken initially; fiber +1 broke during the test. Both appear to follow the model predictions relatively well (Figure 2(a)). From these two fibers alone, it is difficult to determine which case of the crack configuration is more ‘realistic’. This information is better displayed by the first intact fibers in Figure 2(b). It is clear that the model prediction for the intact matrix case (i) is a closer fit to the experimental data. This result suggests that the matrix at the crack tips carries significant load. Unfortunately, the matrix strain did not possess the resolution to confirm this conclusion. Figure 3(a) shows the matrix strains between two intact fibers (-2 and -1). Not only the data points are quite scarce, but the error bars fluctuate enormously, too. The reason behind

![Figure 2](image2.png)

Figure 2. Comparison of strains from the MSSL model predictions (case (i)-intact matrix - the black line, case (ii)-broken matrix - the grey line) and XRD data from fibers (symbols) in the \textit{first} composite: (a) The two broken fibers; (b) first intact fibers. Strains were normalized with respect to the averaged applied far-field value. The expansion of the profile for fiber 0 is due to the width of initial damage around the fiber. The typical error bar plotted in (a) was determined by a 95% confidence limit to the center of peak position.
this observation is the “graininess” problem in the matrix. Figure 3(b) shows the location of the diffraction grains for the α-Ti (11·2) reflection (a similar map was also obtained for the (10·2) reflection). Since the matrix grain size is large compared to the sampling volume, very few grains are favorably oriented for diffraction into the scintillator (point) detector. This is not observed for the fibers since their grain size is about 200 nm. In conclusion, the point detector was seen to be insufficient for the proper characterization of the matrix. This led to the use of the area detector for the second composite. Since complete Debye-Scherrer rings could be collected with this detector (compared to only a narrow portion captured by the point detector), more matrix grains could be sampled allowing the collection of continuous strain maps.

Figure 3. (a) Comparison of normalized strains from the MSSL model predictions (case (i)-intact matrix) and XRD data from matrix (symbols) in the first composite. The region between two intact fibers (-2 and -1) is shown. The applied tensile stress was around 440 MPa. (b) Map of α-Ti (11·2) reflection indicating the location of diffracting α-Ti grains in the first composite. The intensity of the (11·2) reflection was superimposed on an absorption contrast map to show the location of fibers. With a Ti grain size of about 29 µm, few grains are favorably oriented for diffraction at a given θ angle.

The area detector provided a wealth of information about the two-dimensional strain tensor from both components of the second composite. The total elastic strain (residual plus applied) for the matrix is shown in strain maps (Figures 4 and 5) for the three elements of the strain tensor of the matrix: axial (ε_{11}), transverse (ε_{22}), and shear (ε_{12}). The influence of the hole on the applied strains is apparent in each map. Fiber D was broken at the beginning, whereas fiber E broke before reaching 850 MPa. As a result, the matrix regions between these fibers are relaxed compared to the rest. The axial strains also show where stress concentration occurred in the intact matrix beside the hole. The shear strain (Figure 5) also reveals the stress gradients near the hole. Two distinct regions of matrix are represented in each map: matrix between fibers and matrix at fibers (i.e., on each side of fiber surface). The position of the fiber within the matrix is most apparent in the transverse strains. The transverse strains in the matrix were tensile near the fibers and compressive between the fibers (Figure 5). It is also clear that there are large local strain variations in the matrix. These variations can reach 50% and are due to the elastic and plastic anisotropy of the matrix [16].

To view the fiber and matrix strains on a common scale, the strains were normalized with respect to the far-field strain in each fiber or matrix column. The resulting strains are visible as a combined fiber-matrix strain map (Figure 4(b)). The first intact fiber shows the greatest response to damage in the composite with strains up to 1.4 times the far-field strain. The matrix
Figure 4. Contour maps of elastic axial strains in the second composite at 850 MPa: (a) total strains in the Ti matrix; (b) normalized strain in the composite. The fiber positions are labeled and separated from the “matrix only” columns by dashed grid lines. The dashed white lines (b) highlight the tendency of the matrix to transfer the stress concentration away from the break plane.

Figure 5. Contour maps of the total elastic strains in the matrix of the second composite: (a) transverse strains; (b) shear strains. The arrows in (b) indicate load transfer directions away from the damage zone.

also shows regions of significant stress concentration with several regions approaching 1.3 times the far-field strain. As seen in Figure 4(b), the matrix transfers the stress concentration away from the break plane in approximately diagonal directions. This suggests (as expected) load transfer via plastic deformation in the matrix.

The strain results from the second composite are compared to the MSSL model predictions in Figure 6. Since the model does not consider plastic strains, however, the unloading strain data were used in this comparison. It was shown independently that unloading is largely elastic in this composite [12], whereas loading up to 850 MPa did induce slight plasticity in the matrix. For the same reason, the residual strains were also subtracted. The data fit the model rather well for both fibers and matrix. The crack configuration considered here was again for the intact matrix at crack tips (case (i)). However, instead of the infinitely thin crack shown in Figure 1(b),
nine breaks were assumed in order to approximate the size of the hole. It is clearly observed that strains relax in the broken regions for both matrix and fibers. The strain (and stress) concentrations in the first intact fibers are about 1.4, and those in the second intact fibers are about 1.1. Note that the error bars for the fiber data are smaller than the symbol size, while those for the matrix are both smaller and more consistent compared to those shown in Figure 3(a). Of the two stiffness ratios considered, $\rho = 0.290$ appears to fit the data better. This fit helped establish the ‘appropriate’ geometry of the composite with respect to the model’s assumptions. Namely, by averaging the distance between the fibers for $W$ and reducing the thickness in the model to the average thickness of the fiber, the model approaches the actual composite’s geometry better. These results essentially prove that the MSSL model is valid when only elastic deformation occurs in each component of a fiber composite. However, it needs significant improvement to account for residual strains and plastic deformation so that it can perform a more realistic simulation of damage evolution in MMCs. This is currently under consideration and the results will be reported in a future article.

CONCLUSIONS

X-ray microdiffraction was used to monitor strains from both constituents of a Ti-matrix/SiC-fiber composite during damage evolution under applied stress. This was accomplished for the first time at the microstructure scale (slightly less than fiber diameter). It was shown that an area detector provides significantly more information compared to a point detector. The former also helped solve the ‘graininess’ problem in the matrix. The technique proved valuable for the identification of yielding in the matrix, stress concentration around the damage zone, load
transfer in the matrix, and in the case of the area detector, allowed the measurement of the two-dimensional strain tensor in the matrix and the fibers.

Specifically for the Ti-SiC composite investigated, it was shown that its elastic behavior during damage evolution can be described with a shear-lag-based micromechanics model. Although significant stress/strain variations were found in the matrix (due to its elastic and plastic anisotropy), overall, the behavior of the composite closely followed the model predictions. The model was also found to be deficient as it did not take into account residual stresses/strains and plasticity in the matrix.

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