Strain evolution after fiber failure in a single-fiber metal matrix composite under cyclic loading

Jay C. Hanan a,b,∗, Sivasambu Mahesh b, Ersan Üstündag a, Irene J. Beyerlein c, Geoffrey A. Swift a, Bjørn Clausen a, Donald W. Brown b, Mark A.M. Bourke b

a Department of Materials Science, California Institute of Technology, Pasadena, CA 91125, USA
b Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM 87545, USA
c Theoretical Division, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

Abstract

The evolution of in situ elastic strain with cyclic tensile loading in each phase of a single Al2O3-fiber/aluminum-matrix composite was studied using neutron diffraction (ND). An analytical model appropriate for metal matrix composites (MMCs) was developed to connect the measured axial strain evolution in each phase with the possible micromechanical events that could occur during loading at room temperature: fiber fracture, interfacial slipping, and matrix plastic deformation. Model interpretation showed that the elastic strain evolution in the fiber and matrix was governed by fiber fracture and interface slipping and not by plastic deformation of the matrix, whereas the macroscopic stress–strain response of the composite was influenced by all three. The combined single-fiber composite model and ND experiment introduces a new and quick engineering approach for qualifying the micromechanical response in MMCs due to cyclic loading and fiber fracture.

© 2005 Elsevier B.V. All rights reserved.

Keywords: Metal matrix composites; Neutron diffraction; Micromechanical events; Interface shear; Composite deformation; Fiber failure

1. Introduction

Nearly all predictive models for fiber composite failure, regardless of their level of sophistication, require knowledge of the deformation behavior of the composite phases. The deformation of individual phases in composites can differ significantly from their respective monolithic behavior. Consider a metal matrix composite (MMC) with ceramic fibers. Any differences between the in situ and ex situ deformation of the metal matrix could arise because of microstructural constraints, localized strain gradients as a result of phase boundaries or defects, thermal residual stresses, and changes in the microstructure by virtue of composite fabrication and consolidation. One example of this is provided by recent work by He et al. [1]. They showed that in Al2O3/Al composites, the in situ matrix yield strength was significantly higher than the monolithic value and increased with decreasing local fiber spacing. This distinctive in situ behavior was attributed to reduced grain size and increased dislocation densities near the fiber surface. Likewise, the ex situ versus in situ fiber strength could be different. A degradation of in situ fiber strength, e.g. lower average strength and higher variability could be the result of fabrication-induced defects. Lastly, such MMCs have an interface region, an interphase, or a reaction zone between the fiber and matrix materials. It is important to consider this region as a third constituent owning properties distinct from the fiber and matrix. Naturally, the in situ behavior of an interface cannot be determined and in situ characterization necessarily involves inverse or iterative methods using an interpretive model.

The failure of fiber-reinforced composites is largely dominated by the nucleation and interaction of fiber fractures. Local deformation in response to a fiber fracture may involve matrix plasticity, interfacial debonding and slipping.
matrix creep or a combination of these mechanisms. Such complex cases have been analyzed in a number of polymer matrix composites [2,3], using micro Raman spectroscopy and in metal matrix composites (MMCs), using chromium fluorescence peizospectroscopy [1]. There are, however, very few complete in situ studies of damage evolution in MMCs where both the matrix and fibers could be investigated (see e.g. [4–6]). Spectroscopy techniques are not ideal for MMCs because micro Raman spectroscopy [7] requires transparent matrices and peizospectroscopy [8] is a surface technique. X-ray and neutron diffraction are more advantageous for damage evolution studies in MMCs as they provide in situ strain data from all phases, and especially for neutrons, are much more penetrating (in the order of cm).

A single-fiber composite, containing at least one fiber fracture, can be an excellent tool for characterizing and understanding various damage evolution mechanisms. More traditional characterization techniques, which use such specimens are fiber pull-out, push-out, or fragmentation tests. The latter typically involves a rather complex statistical analysis for interpretation [9]. Fiber pull-out (or push-out) specimens often contain a large volume fraction of matrix (approximately 80–99%), much larger than that found in real composites.

Therefore, microstructural constraints mentioned earlier may not exist in such specimens. Furthermore, in most of the above-mentioned tests substantial statistical variation in the estimates of the interfacial shear stress renders some of the results inconclusive.

There are a few analytical models, which analyze interface or matrix damage from a single broken fiber under cyclic loading [10,11]. However, these models are based on the shear lag concept, which assumes that the matrix deforms only in shear. While quite promising for composites with high-fiber volume fractions (over 60%), they are not applicable for the composite considered in this study (which had 33% fiber volume fraction). In this case, and for MMCs in general, it is essential that the axial deformation of the matrix is included in the analysis. The impact of residual stresses on local deformation, propagation and shrinkage of the plastic regions around the break, and macroscopic composite deformation will largely be governed by both shear and axial deformation in the matrix. Therefore, for reliable characterization of the interface and matrix properties, the interpretative model must allow for a combination of interface slip and matrix axial deformation.

The present work is concerned with characterizing the in situ matrix and interface deformation in a single-fiber MMC using neutron diffraction (ND). We studied the effects of in situ fiber fracture, matrix plasticity and interface slip on the elastic strain evolution observed with ND in both the fiber and matrix. The ND data were complemented with macroscopic stress–strain measurements which described the overall composite behavior. A new approach was developed that relates ND data to a simple, analytical model which tracks damage evolution in an Al₂O₃-fiber/Al-matrix composite under cyclic loading. The model presented here did consider the axial deformation of the matrix, which was necessary for the correct interpretation of the deformation mechanisms.

2. Experimental procedure

2.1. Sample processing

A model composite comprised of a single, polycrystalline Al₂O₃ fiber (4.75 mm diameter, from Coors Ceramics, Golden, CO, USA) and an Al matrix were prepared by casting. The Al alloy used was 6061 (ESPI Metals, Agoura Hills, CA, USA). The manufacturer reported an average grain size of 7 µm in the fiber. The average grain size in the matrix was determined to be 21 µm using the linear intercept method [12,13].

In order to ensure a fiber fracture at the center of the gage section during subsequent loading, a 0.4 mm notch was cut around the circumference of the fiber to a depth of 1.1 mm using a diamond saw. The sample was cast in a 304 stainless steel mold under vacuum after purging the mold with argon. The mold was machined to hold the alumina fiber vertically in a tube furnace while the Al melted around the fiber. Following 30 min at 800 °C, the entire mold was quenched in water at room temperature. A cylindrical tensile sample was then machined from the cast form. The final dimensions of the composite gave a 30 mm long gage length with an 8.25 mm gage diameter and 76 mm total sample length (Fig. 1). These dimensions yielded 33% fiber volume fraction for this specimen in the gage section.

To verify the fiber and interface integrity following casting and machining, X-ray radiography was performed using a Philips Paws-O-Mat at 65 kV and 7.5 mA. It was shown that the matrix did not contain noticeable voids within an estimated resolution of 30 µm and the fiber was aligned with

![Fig. 1. Schematic of the single-fiber composite test during neutron diffraction. Longitudinal strains measured in the \(2\theta = -90^\circ\) detector bank were integrated over the volume irradiated by the incident neutron beam.](image)
ter failure and final residual strain values were recorded: by diffraction. The sample was maintained in position af-
it its threaded section and outside the gage region sampled
the fifth cycle, the specimen failed near the shoulder of
measurements were conducted during these cycles. During
unloaded to 3 MPa. Several neutron
volume is illustrated in Fig. 2 as the grey shaded area.

2.2. Neutron diffraction experiments

Mechanical loading with simultaneous neutron diffrac-
tion was performed using the Neutron Powder Diffractometer
(NPD) and the Spectrometer for Materials Research at Tem-
perature and Stress (SMARTS) [14], both at the Los Alamos
Neutron Science Center (LANSCE). Special hydraulic load frames stressed the samples in 10–20 MPa intervals for ap-
proximately, 45 min per loading step starting at 3 MPa.

Five tensile loading–unloading cycles were applied to the
composite. In the first cycle, it was loaded to 80 MPa and
loaded to 3 MPa. In the subsequent cycles, it was loaded
to 100 MPa and unloaded back to 3 MPa. Several neutron
measurements were conducted during these cycles. During
the fifth cycle, the specimen failed near the shoulder of
that its threaded section and outside the gage region sampled
by diffraction. The sample was maintained in position af-
ter failure and final residual strain values were recorded:
+600 × 10^-6 in the fiber and −740 × 10^-6 in the matrix.

Post-mortem X-ray radiography of the composite showed
that the fiber had also broken at the notch in the middle of
the gage section.

Tensile stress during diffraction was also applied to the
reference fiber sample to observe its mechanical response in
monolithic form. The fiber was stressed in 20 MPa intervals
up to 160 MPa while recording neutron diffraction patterns
at the same time. The tensile stress–strain curve of the refer-
ence matrix sample was determined at Caltech using a screw-
driven load frame. The axial macroscopic (total) strain in all
three samples was monitored with a 25 mm gage length ex-
tensometer. Results showed that the matrix was linear elastic
with a Young’s modulus of 70 GPa until it reached its initial
yield point of 80 MPa. Thereafter, the stiffness was reduced
to 6 GPa. The fiber Young’s modulus was measured to be
330 GPa.

Using time-of-flight neutron diffraction, the bulk average
elastic lattice strain in the specimens was obtained in both
the longitudinal and transverse directions simultaneously. A
schematic revealing the loading and diffraction geometries
is shown in Fig. 1. As indicated by the orientation of the
scattering (Q) vector, data from the 2θ = 90° bank provided
longitudinal (axial) strain information from both the fiber and

the matrix. It is important to note that the measured strains
were an average over the 14 mm neutron gage length created
by a 10 mm wide neutron beam, which illuminated the entire
cross-section of the composite at an angle of 45°. This gage
volume is illustrated in Fig. 2 as the grey shaded area.

The diffraction data were analyzed using the Rietveld
method [15,16]. Comparison of the initial values of the lat-
tice parameters of each phase of the composite to those ob-
tained from the two reference monolithic samples showed
that the thermal residual strains were relaxed in the compos-
ite to within 200 × 10^-6 strain. This value is typical for strain
error in neutron diffraction experiments that involve sample
changes and is attributed to the sample displacement error
[17]. Therefore, the reference point for the lattice strains due
to applied loading was taken to be the nominally zero load
position (at 3 MPa applied stress). The elastic strain in the Al
matrix was calculated from changes in its lattice parameter.
The cubic crystal structure of Al (space group Fm3m, face
centered cubic), its relatively low elastic anisotropy (α = 2),
and the effective averaging done by the whole-pattern Rie-
tveld refinement justified this approach. However, the alpha
alumina fiber had trigonal crystal structure (space group R-
3c, corundum) and was somewhat textured. This led to an
anisotropic (though linear) response by its a- and c-lattice
parameters to applied loading. Furthermore, the relatively
low intensity of 00l type reflections led to higher fitting er-
rors in the c-lattice parameter. Therefore, the polycrystalline
average of the Young’s modulus of the fiber (α = 330 GPa, de-
termined by the extensometer during diffraction experiments
on the reference fiber) was used to “adjust” the lattice strain
exhibited by the a-lattice parameter so that it would represent
the polycrystalline average. This approach was taken due to
lack of detailed information about the texture in the fiber and
to avoid making sweeping assumptions about load sharing
among various lattice planes of alumina (see [18] for a re-
aplated discussion).
3. Experimental results

Fig. 3 exhibits the axial lattice strain evolution in the composite during several loading–unloading cycles. The residual strains shown in this figure (at the end of each unloading cycle) experience a gradual evolution in both the matrix and the fiber. This evolution is especially pronounced in the first three cycles and tends to saturate in the last two. This suggests that inelastic deformation no longer evolves beyond cycle 3.

Another important observation from Fig. 3 is the change in the apparent (diffraction) moduli of each phase given by the slope of applied composite stress versus elastic strain plots. To clearly see where these changes initiate, Fig. 4 shows the lattice strain evolution for just the loading portion of the first cycle. Even at the beginning, both phases evolve separately but appear to exhibit similar diffraction moduli until 60 MPa. Above this applied stress, the diffraction moduli of the matrix and fiber deviate further. As shown in Fig. 3, the matrix diffraction modulus decreases continuously and saturates around 74 GPa in cycles 3–5. At the same time, the fiber diffraction modulus increases from cycle 1 to 2 and does not change significantly in the remaining cycles until failure.

4. Analysis

The purpose of the following analysis is to associate the experimental strain data with a model that describes the mechanical behavior and interactions of the fiber, matrix, and interface. Since X-ray radiography of the composite did not reveal any voiding or cracking in the matrix, it was assumed that the most important aspects to consider in the model were fiber fracture, matrix plasticity and interfacial shear response. The preliminary analysis in Section 4.1 shows that the fiber–matrix interface was not intact during the cyclic loading. Based on this result, a simple analytical model with an elastic fiber, a slipping interface, and an elastoplastic matrix was developed and will be described in Section 4.2.

The material data used in the following calculations are shown in Table 1. The alumina fiber was assumed to be elastic throughout, with a modulus of 330 GPa, as measured in the tension test of the reference fiber. A linear hardening law was fit to the matrix alloy’s monolithic stress–strain curve in tension (fit not shown). The initial yield point was 80 MPa and the resulting values for the stiffness of the monolithic matrix in the elastic ($E_m$) and plastic ($E'_m$) regions are listed.

In what follows, relative displacement of the fiber and matrix across the interface will be called interfacial slipping.

4.1. Intact interface

This section establishes that the fiber–matrix interface in the composite specimen was not intact, i.e., it experienced slipping during tensile loading. An estimate of the uniform stresses and strains far from specimen ends in the fiber ($\sigma_r$, $\epsilon_r$) and matrix ($\sigma_m$, $\epsilon_m$), when both of these are

Table 1

| Material parameters for the matrix and interface used in the modeling calculations |
|-----------------------------------|-------------------------------|
| $E_m$ (GPa) | $E'_m$ (GPa) | Yield point (MPa) | Yield point, $\sigma_f$ (in situ) (MPa) |
| 70 | 60 | 80 | 55 | 80 |

The first three were obtained from the tension test of the monolithic matrix sample while the last two are in situ parameters in the composite estimated by the model.
are obtained: 

\[ \varepsilon \] 

respectively. The elastic strain in the fiber (\( \varepsilon_r \)) and matrix (\( \varepsilon_m \)) is given by the rule of mixtures as:

\[ \varepsilon_r = \frac{\sigma_r}{E_r} \]  

and \[ \varepsilon_m = \frac{\sigma_m}{E_m} \]

Here, \( \sigma_r \) is the applied composite stress, while \( A_r \) and \( A_m \) denote the cross-sectional areas of the fiber and matrix, respectively. The elastic strain in the fiber (\( \varepsilon_r^{m.m} \)) and matrix (\( \varepsilon_m^{m.m} \)), however, are equal:

\[ \varepsilon_r^{m.m} = \varepsilon_m^{m.m} = \frac{\varepsilon_r^{f.m} + \varepsilon_m^{f.m}}{E_f} = \frac{\varepsilon_r^{f.m} + \varepsilon_m^{f.m}}{E_m} \]

Chen et al. [19] have studied an alumina fiber/6061 aluminum alloy matrix composite with an intact fiber and a strong interface obtained by a fabrication procedure different from that used in this work. They found that the experimental composite modulus matched those derived from rule of mixtures considerations.

Fig. 4 shows the elastic strain evolution in the present specimen in both phases during cycle 1 along with the rule of mixtures curve. If the fiber were intact, the fiber and matrix were elastic and well-bonded, and the (neutron) gage volume sufficiently removed from the ends of the specimen where load was applied, then the strain in the gage volume would be uniform and the fiber and matrix would experience the same strain as that predicted by the rule of mixtures. However, it is seen that fiber and matrix strains fall to either side of the rule of mixtures line (Fig. 4). Because of this deviation, we can conclude that the fiber and matrix are neither deforming elastically nor well bonded.

Another indication of slipping at the interface is furnished by the discrepancy in the residual strains after specimen fabrication. It was reported in Section 2.2 that the measured average thermal residual strain in the specimens is small, and comparable to the experimental error of measurement. We used an axis-symmetric finite element (FE) calculation to compute the thermal residual state in a specimen cooled by 100 K from a stress-free state. In choosing the stress-free temperature to be 100 K above room temperature, we are guided by the work of Fukui and Watanabe [20] on an alloy similar to the present matrix material (albeit with SiC particle reinforcements). They find that the thermal residual stress in their material is well-modeled by their theory if the stress-free temperature is taken as 140 K above room temperature. In order to be sure of underestimating the residual elastic strains, we round this down to 100 K above room temperature.

In the FE calculation, we assumed an intact elastic fiber, an isotropically hardening matrix whose stress-strain curve is set to the measured bulk response of the matrix material, and a perfectly bonded interface. We took the coefficient of thermal expansion of the aluminum matrix to exceed that of the alumina fiber by 12 \( \times \) 10\(^{-6}\)/K, independent of temperature. The FE results yield elastic strains much more than what is measured. The calculated elastic axial strain at \( r = r_t \) is approximately 790 \( \times \) 10\(^{-6}\), while that near \( r = r_m \) is approximately 330 \( \times \) 10\(^{-6}\). These values remain nearly constant in the neutron gage volume, away from the specimen ends. The gage volume average of the axial elastic strain must fall between these extremes. Since the measured value lies far lower, it suggests that an assumption of the model, viz. compatibility at the interface is being violated in the experiment.

It must also be mentioned that the above residual strain computation could have alternatively been performed using simple concentric cylinder models (see Johnson et al. [21]).
4.2. Slipping interface: elastic strain evolution

In this section, we use a simple analytical model to gain a first understanding of the evolution of elastic (lattice) strains in both the matrix and fiber. A key assumption on the interfacial shear stress profile underlies the simplicity of the model. Below, we describe the model with its assumptions and then examine their validity and consequences. Fig. 2 shows a schematic of the sample geometry around the neutron gage section. The assumed interactions between the fiber and matrix are modeled as shown in the free body diagrams of Fig. 6. Two cases of interest are the events before and after fiber fracture; these occupy respectively, the first and second rows of the figure. The figure also shows two of the unknown parameters in the model: \( \tau_s \) and \( \tau_c \), which quantify the model interfacial shear profile shown. The latter is a function of the applied stress \( \sigma \) while fitting all cycles of the experiment.

The applied stress at the grips is represented by a uniform tensile stress \( \sigma_{imp} = \sigma_{imp}A_1 + \sigma_{imp}A_2 \) acting at each end of the matrix (see Fig. 2). The fiber and matrix are ignored. Therefore, the shear strain arising from the Poisson effect is not considered in comparison to the axial strains due to axial loading.

Using one-dimensional equilibrium considerations along the fiber axis \( r \) direction for the intact fiber case (the first row of Fig. 6), the fiber stress was obtained as:

\[
\sigma_f(z) = \begin{cases} 
\frac{2\tau_s l_s}{r_f} & \text{if } 0 \leq |z| \leq l - l_s \\
\frac{2\tau_s (l - z)}{r_f} & \text{if } l - l_s \leq |z| \leq l 
\end{cases}
\]  

(4)

where \( l \) is the total length of the composite. As marked in Fig. 2, \( l = 38 \text{ mm} \), \( r_f \) and \( r_m \) are the fiber radius and outer radius, respectively, and \( z \) is the fiber axial coordinate defined from the notch. Similarly, the matrix stress was given by:

\[
\sigma_m(z) = \begin{cases} 
\frac{\sigma_{imp} r_m^2 - 2\tau_s r_m r_f}{r_m^2 - r_f^2} & \text{if } 0 \leq |z| \leq l - l_s \\
\frac{\sigma_{imp} r_m^2 - 2\tau_s (l - z) r_f}{r_m^2 - r_f^2} & \text{if } l - l_s \leq |z| \leq l 
\end{cases}
\]  

(5)

Following the same approach for the broken fiber case (the second row of Fig. 6), the fiber and matrix stresses were:

\[
\sigma_f(z) = \begin{cases} 
\frac{2\tau_s c}{r_f} & \text{if } 0 \leq |z| \leq l_s \\
\frac{2\tau_s l_f}{r_f} & \text{if } l_s \leq |z| \leq l - l_s \\
\frac{2\tau_s (l - z)}{r_f} & \text{if } l - l_s \leq |z| \leq l 
\end{cases}
\]  

(6)

\[
\sigma_m(z) = \begin{cases} 
\frac{\sigma_{imp} r_m^2 - 2\tau_s l_f r_f}{r_m^2 - r_f^2} & \text{if } 0 \leq |z| \leq l_s \\
\frac{\sigma_{imp} r_m^2 - 2\tau_s (l - l_s) r_f}{r_m^2 - r_f^2} & \text{if } l_s \leq |z| \leq l - l_s \\
\frac{2\tau_s (l - z)}{r_f} & \text{if } l - l_s \leq |z| \leq l 
\end{cases}
\]
and

\[
\sigma_{el}(z) = \begin{cases} 
\frac{\sigma_c r_m^2 - 2 \tau_c z r_m}{r_m^2 - r_t^2}, & \text{if } |z| \leq l_t \\
\frac{\sigma_c r_m^2 - 2 \tau_c z r_t}{r_m^2 - r_t^2}, & \text{if } l_t \leq |z| \leq l - l_c \\
\frac{\sigma_c r_m^2 - 2 \tau_c (l - z) r_c}{r_m^2 - r_c^2}, & \text{if } l - l_c \leq |z| \leq l 
\end{cases}
\]  

(7)

For both cases, the one-dimensional Hooke’s law gave the elastic strains as:

\[
e_{el}^{f}(z) = \frac{\sigma_c(z)}{E_f} \quad \text{and} \quad e_{el}^{m}(z) = \frac{\sigma_c(z)}{E_m}
\]

(8)

To compare with the neutron measurements, all calculated strains were averaged over the neutron gage volume, which was asymmetric, as illustrated in Fig. 2. Eqs. (4)–(8) involve two unknowns: \(\tau_c\) and \(l_c\). Following the procedure detailed below, \(\tau_c\) and \(l_c(\sigma_c)\) were refined, so that the calculated average matrix strains agreed well with the neutron diffraction data. The success of the model was judged by the agreement between the measured and predicted fiber strains.

As Eqs. (5), (7), and (8) indicate, the axial strain in the matrix has a linearly decreasing region near the fiber-free surfaces and a region of uniform strain in between. A larger \(\tau_c\) implies a more rapid load transfer between the fiber and matrix, i.e., a smaller \(l_c(\sigma_c)\) for all \(\sigma_c\). For fixed \(\tau_c\) by adjusting \(l_c(\sigma_c)\) it was, therefore, possible to alter the average elastic strain over the neutron gage volume. From Fig. 3, it is observed that the measured, average, elastic fiber strain \(e_{el}^{f} \) is sensitive to \(\sigma_c\). According to Eqs. (4) and (6), however, the model predictions of this strain should be independent of \(\sigma_c\) if \(l_c(\sigma_c)\) were to approach or exceed the neutron gage length of the fiber. This observation supplied us with the following constraint on the choice of \(\tau_c\): \(\tau_c\) was required to be large enough, so that \(l_c(\sigma_c)\) would always be contained within the neutron beam. Therefore, for a given \(\tau_c\) within this range one can choose a suitable \(l_c(\sigma_c)\) to fit the measured matrix strains with the model predictions. The value of \(\tau_c\), and its associated \(l_c(\sigma_c)\) were selected based on the one that best predicted the measured fiber strain.

Next, within this range of \(\tau_c\) an iterative calculation was performed in which \(\tau_c\) was varied to obtain the best agreement between the measured and calculated fiber strains. Note that this was a one-parameter \((\tau_c)\) fit of the fiber profile and did not amount to fitting the fiber curve in the same sense as fitting the matrix curve. Furthermore, \(\tau_c\) can only be varied over a limited range as described above. This involved fitting \(l_c(\sigma_c)\), each time \(\tau_c\) was altered to continue to fit the matrix strains. The calculations showed that the calculated \(e_{el}^{f}\) always exceeded the measured \(e_{el}^{f}\). Therefore, the best choice of \(\tau_c\) was determined by the aforementioned constraint: \(\tau_c = 55\) MPa was the smallest value such that the \(l_c\) remained within the beam.

Figs. 7 and 8 compare the calculated and measured average elastic strains in the first and second loading cycles of the composite, respectively. The prediction used \(\tau_c = 55\) MPa and its associated \(l_c(\sigma_c)\). The \(l_c(\sigma_c)\) used for the fit are shown in Fig. 9. \(l_c(\sigma_c)\) progressively increases with higher applied stress and reaches its maximum value (=9.31 mm) in cycle 3 at \(\sigma_c = 100\) MPa. The residual value of \(l_c(\sigma_c)\) (at \(\sigma_c = 3\) MPa) also increases slowly with successive cycles suggesting a slow degradation of the interface with continued cyclic loading of the composite.

The model is only slightly sensitive to the value of \(\tau_c\). Setting \(\tau_c = 60\) MPa, and refitting \(l_c(\sigma_c)\) so as to align the calculated and measured average matrix strains results in an increase of only \(4 \times 10^{-6}\) in the computed average fiber strain at the peak load of 80 MPa during cycle 1. The corresponding increase in cycle 2 is \(17 \times 10^{-6}\). The computed average fiber

![Fig. 7](image-url)
residual strain after cycle 2 remains unchanged when $\tau_s$ is changed from 55 to 60 MPa.

It is generally accepted that $\tau_s$ may decrease substantially during the first few cycles of loading, as the asperities leading to friction at the interface are ground down during load cycling [22,23]. Using the present simple model, although we are unable to firmly deduce such a lowering of $\tau_s$ with load cycling, or lack thereof, we have grounds to believe that any such reduction is small in our specimen. Because, according to the model, $\tau_s = 55$ MPa leads to a maximum $l_s = 9.31$ mm in the third cycle, that is, only slightly less than quarter the length ($l_s/4$; see Fig. 2) of the composite. Since physically, $l_s \leq l / 4$, the actual $\tau_s$ during the third cycle cannot be much less than 55 MPa. Now, suppose that the value of $\tau_s$ is larger than 55 MPa in the first two cycles. This contradicts the result of the preceding paragraph that the fits using $\tau_s = 60$ MPa are poorer than those using $\tau_s = 55$ MPa (albeit only slightly).

Thus, even in the first two cycles, we suspect that $\tau_s$ is not much more than 55 MPa in the experimental specimen.

Push-out and pull-out experiments are commonly used to characterize inter-facial $\tau_s$. While such experiments do not give a clear indication of the shear profile along the interface, an approximation of $\tau_s$ is obtainable by assuming the inter-facial shear profile to be uniform and applying Kelly’s [24] formula:

$$\tau_s \approx \frac{P}{2\pi aH}$$

where $P$ is the peak load applied to the fiber, $a$ its radius, and $H$ its embedded length in the matrix. $\tau_s$ depends sensitively on the fiber and matrix materials, composite-fabrication technique, testing temperature, among others. We are unaware of extensive interfacial studies through pull-out or push-out tests for our composite system. However, a detailed push-out study of SiC fiber–Al matrix interfaces was done by Lu et al [25], who found $\tau_s$ to be a Weibull-distributed random quantity of fairly large variance, with average strength 60.9 or 46.8 MPa, depending on the composite fabrication procedure. Thus, the fit value $\tau_s = 55$ MPa falls within a reasonable range.

The fiber was assumed to fracture between 60 and 80 MPa of cycle 1 in these calculations. While an X-ray radiograph of the specimen after the experiment did reveal a broken fiber, there is some uncertainty as to when exactly it broke during the experiment. Although a lower probability, the fiber might have also broken during cycle 2. If this were the case, then the fiber failure would have occurred between 80 and 100 MPa in this cycle because the sample faced no higher stresses in subsequent cycles. Separate calculations were performed (not shown here) for the second case of fiber fracture, also with $\tau_s = 55$ MPa. The fiber strains were predicted to be within $\pm 20 \times 10^{-6}$ of those estimated for the first case (fiber fracture during cycle 1). In either case, however, the main premise of this study is still valid; strain evolution during the
cyclic loading of this alumina/aluminum composite could be described reasonably well by a slipping interface model.

The success of the present simple model can be judged by comparing the calculated average strains in the fiber with those measured. The model reproduced the fiber elastic strain data within the error of measurement for small applied stresses and captured accurately the residual strain at the end of cycles 1 and 2 (Figs. 7 and 8). There was some discrepancy, however, between the data and model predictions at higher loads (the maximum strain deviation was $118 \times 10^{-6}$ at $\sigma_c = 80$ MPa in cycle 1). These deviations suggest a slight difference between the actual shear profile at the interface and that assumed by the model. Several kinks in the fiber strain data (e.g. at $\sigma_c = 40$ MPa during unload- ing, in both cycles 1 and 2) support this hypothesis. These kinks were likely to be caused by some non-uniform slipping along the interface, contrary to the uniform slipping assumed by the proposed model. In conclusion, it is not possible to rule out some stick-slip-type behavior at the interface. Nonetheless, based on arguments presented earlier (see Section 4.1), it is clear that the composite behavior is better characterized by a uniformly slipping interface than an intact one.

4.3. Slipping interface: plastic strain evolution

For a final validation of the slipping interface model, the total (elastic + plastic) surface strain in the composite as measured by the extensometer was compared to model predictions. Fig. 10 shows the evolution of the total axial surface strain in the matrix as a function of applied composite stress ($\sigma_c$) during cycle 1 (with a maximum stress of 80 MPa) and cycle 2 (with a maximum stress of 100 MPa). In this comparison, the tensile stress–strain curve of the monolithic matrix (not shown) was approximated as two straight lines: linear elasticity with modulus $E_m$ up to $\sigma_f$, followed by linear hardening with modulus $E_{m}^H$:

$$
\sigma_m(z) = \begin{cases} 
E_m\epsilon_{m}^{(z)}, & \text{if } \epsilon_{m}^{(z)} < \epsilon_{f} \\
E_{m}^H\epsilon_{m}^{(z)} + (E_m - E_{m}^H)\epsilon_{f}, & \text{if } \epsilon_{m}^{(z)} \geq \epsilon_{f}
\end{cases}
$$

where $\epsilon_f = \sigma_f/E_m$ is the yield strain, and $\sigma_f$ is the yield stress. Therefore, for $\sigma_m \geq \sigma_f$, the plastic part of the matrix strain during loading over the previous maximum stress is given by

$$
\epsilon_{m}^{pl}(z) = \sigma_m(z) - \sigma_f \left( \frac{1}{E_m^H} - \frac{1}{E_m} \right)
$$

The material parameters obtained by fitting this equation to the stress–strain curve of the monolithic matrix are shown in (Table 1).

Knowing the matrix stress in the composite ($\sigma_m(z)$) from Eqs. (4) to (7), the plastic part of the matrix strain could be computed from Eq. (11). Here, $\epsilon_{m}^{el}(z)$ was only calculated during loading and above the previous maximum stress. The plastic strain was assumed to remain unchanged during unloading, i.e., the matrix was not expected to yield during this step. Note that in the above $\sigma_f$ can be considered an unknown, allowing for the possibility that the in situ yield stress is different than the ex situ one. The calculated total matrix strain is then $\epsilon_m = \epsilon_{m}^{el} + \epsilon_{m}^{pl}$ as given by Eqs. (8) and (10) above.

The calculated and measured total strain in the matrix (i.e., at the surface of the composite) are compared favorably in Fig. 10. When the in situ matrix yield stress in the composite ($\sigma_f$) was treated as a fitting parameter, a value of 80 MPa gave the best fit. Interestingly, 80 MPa was also the yield point of the monolithic matrix specimen. This result suggests that the matrix had not undergone appreciable in situ hardening during quenching, possibly due to the poor interfacial constraint between fiber and matrix. This is also in agreement with the observation in Section 3 that the thermal residual stress in the matrix after quenching was small. All of these results confirm the poor quality of the fiber–matrix interface in the composite specimen.

5. Conclusions

Combined neutron diffraction and micromechanical modeling allowed the characterization of the in situ failure events occurring in an Al$_2$O$_3$/Al single-fiber composite under cyclic tensile loading. Neutron diffraction provided the necessary strain resolution to deduce the deformation characteristics of each phase. From the in situ strain evolution and the macroscopic stress–strain curve, it was evident that interface slipping, fiber fracture, and matrix yielding evolved during the first two cycles of the five-cycle test, with maximum stresses of 80 and 100 MPa, respectively. Significant changes in the composite’s residual strains were associated with these failure events. Later, cycles with the same maximum stress of 100 MPa showed minimal irreversible deformation. The
results also provided evidence that the fiber-sustained load in spite of a fiber fracture and interface degradation.

An analytical model was used to interpret the in situ interface and matrix behavior from the neutron diffraction results. Consistent with specimen geometry, the model considers a single-fiber composite allowing for axial matrix strain and interfacial slipping with or without a fiber fracture. The analysis accounted for the asymmetric neutron gauge volume, and yielded good agreement with the experimental data. The interpretation of the model suggests that the elastic strain evolution of the fiber and matrix was governed by fiber fracture and interface slipping, whereas the macroscopic stress–strain response of the composite was also governed by the plastic deformation of the matrix. Possibly due to the weak interface the in situ matrix yield stress was the same as that in monolithic form. This suggests that repeating the current studies using single-fiber composite specimens with a strong interface would certainly elucidate differences between the in situ and ex situ yield stresses.

The present analytical model does not account for compatible displacement across part of the interface, which would call for a displacement-based model of matrix plasticity and slip. A model, which accounts for matrix plasticity and slip over part of the interface (including reversals) is needed for a better analysis of the experiment and is presently being developed.

The methodology developed in this study provides a quick, yet powerful engineering tool to investigate the in situ damage evolution in fiber-reinforced composites, especially under cyclic loading. New capabilities now exist with neutron diffraction [14] that allows loading experiments at temperatures reaching 1500 °C. The approach presented here will be invaluable in quantifying matrix and interface behavior in high-temperature regimes. Work is in progress to achieve this and to exploit the new experimental capabilities and the improved methodology described above.

Acknowledgements

Funding was provided by the National Science Foundation (CAREER grant no. DMR-9985264) at Caltech and a Laboratory-Directed Research and Development Project (no. 20000043) at Los Alamos. The neutron diffraction experiments were conducted at the Lujan Center, LANSCE, a national user facility supported by the Department of Energy, Office of Basic Energy Sciences under contract W-7405-ENG-36. Dr. C. Brian Hooper assisted with the radiographic measurements.

References