Residual Stress Analysis of Graphite/Polymer Composites
using the Concept of Metallic Inclusions

By

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ABSTRACT

Metal particles (Al, Ag, Nb) were embedded between the first and second plies of 6-ply unidirectional and 4-ply 8-harness satin weave cloth carbon/polyimide laminates, as strain sensors for the determination of residual and applied stresses by x-ray diffraction. Specimens containing the particles, each at two concentration levels, were fabricated at NASA Lewis Research Center with the collaboration of Mr. M. Castelli. XRD measurements were made using a Siemens D500 diffractometer with pseudo parallel-beam optics a solid state detector and Cu K\(\alpha\) radiation. Specimens were subjected to bending loads while irradiated, using a 4-point bending device mounted on the D500 goniometer.

Finite Element calculations were performed on a specimen with an isolated particle located at half the distance between neutral axis and the surface of the specimen for the 4-ply laminate and two thirds the distance for the 6-ply laminate. ANSYS v.5.2 was used with tetrahedral Solid92 elements. Eshelby calculations were done using the Eshelby tensor for a spherical inclusion embedded in an infinite homogeneous anisotropic matrix, the known strain matrix for bending and the matrices for thermal expansion of the composite and the metal inclusion.

Strains and stresses obtained by XRD in the embedded particles were sensitive to the residual stresses in the as cured laminates and responded linearly to stresses applied by 4-point bending, qualitatively as expected. Quantitative agreement between the XRD, FEM and Eshelby strains was reasonably although differences where somewhat larger than the XRD error bars. Agreement between the stresses was considerably poorer. Stress transfer factors between the applied stress and the stresses in the particles were measured experimentally and allowed calculation of the residual stresses in the matrix adjacent to the particles.

INTRODUCTION

Aerospace structural components made of carbon fiber-polyimide are vulnerable to combined effects of environment, applied forces and residual stresses, the latter being unavoidably present due to the dissimilar fiber and matrix properties and to the fabrication process. As a consequence, components can fail at lower than expected loads. Since residual strains and stresses in composites are not always easy to model, their experimental measurement is desirable. Such

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measurements on polymer matrix composites have been made successfully by measuring lattice spacings by x-ray diffraction from embedded filler particles or from the matrix if it is semi-crystalline [1-14].

For a single (crystalline) inclusion in an amorphous matrix, knowledge of the stress state inside the inclusion allows us to obtain information about the applied stress that produced that stress state inside the inclusion using the stress transfer coefficient [15]. A stress transfer factor connects the stress states in the matrix and the inclusion.

If both phases, matrix and inclusion, are simultaneously under an externally applied stress, the response in each phase should be a function of this stress. A linear relationship was postulated by Hahn [16]. Assuming an isotropic composite of small particles embedded in an amorphous matrix, the stresses in each phase due to the externally applied stress are related as follows

\[ \sigma_{im} = \eta \cdot \sigma_{ip} \quad \text{for } i=1, 2, \ldots, 6 \quad (1) \]

where \( \sigma_{im} \) is the stress in the “i” direction in the matrix phase, \( \sigma_{ip} \) is the stress inside the particle in the “i” direction.

Equation (1) is reasonably acceptable from both experimental and theoretical points of view. If equation (1) is solved for \( \sigma_{ip} \), assuming \( \sigma_{im}=\sigma_{iun} \), which is the component along the direction of the applied stress, we get a convenient representation of the three stress components inside the inclusion as a function of one stress component in the matrix:

\[ \sigma_{ip} = k_i \sigma_{im} \quad (2) \]

where \( i=1, 2, 3 \), and \( k_i \) is the stress transfer factor for the “i” direction. For a single spherical inclusion in an isotropic matrix, this factor has been calculated to be about two times the uniaxially applied external stress in the stress direction, and minus 0.3 times the applied stress in the direction transverse to the applied stress using the Eshelby method [15]. The stress transfer factor is independent of the particle Poisson’s ratio in the range .2 to .4 for shear modulus ratio \( G_{\text{matrix}}/G_{\text{particle}} < 10^{-2} \). If an external (or internal) force is applied to a composite containing many small spherical inclusions, a similar stress transfer factor should be expected [15, 17].

For the case of a fiber composite containing particles between plies, the relation between the externally (or internally) applied stress and the average stress in the particles depends on many factors and is not easily modeled. Hence, it is necessary to measure this relation experimentally to provide guidance for the modeling.

To make such measurements, it is necessary to apply known stresses to a specimen and to measure simultaneously the strains and stresses in the particles. This is conveniently done by using a 4 point bending device which attaches to an x-ray goniometer. Such measurements must necessarily be made simultaneously on many particles in order to obtain sufficient diffracted intensity.

One can then compare the results of Eshelby and finite element modeling with the diffraction measurements to better understand the stress transfer process and to provide a method for determining residual stresses. In this work, residual stresses are defined as those present at room temperature after curing and before external loads are applied.
SPECIMENS

In the present study, three types of metal powders Al, Ag, Nb, were used as filler particles and were placed between the 1st and 2nd plies of 6-ply unidirectional and 4 ply cloth carbon/polyimide laminates during lay-up. This was done by uniformly painting on a slurry of the metal powders onto the first green ply then placing the 2nd and subsequent plies on top of this layer. Two concentrations of powders were used as indicated in Table I.

Table I.a Unidirectional specimens: Nominal Powder Concentrations and Particle Sizes

<table>
<thead>
<tr>
<th>Specimen Name</th>
<th>Filler Type</th>
<th>Nominal Particle Size (μm)</th>
<th>Nominal Particle Concentration mg/cm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-1U</td>
<td>Al</td>
<td>1-5 μm</td>
<td>2.3</td>
</tr>
<tr>
<td>Al-2U</td>
<td>Al</td>
<td>1-5 μm</td>
<td>4.6</td>
</tr>
<tr>
<td>Ag-1U</td>
<td>Ag</td>
<td>1-5 μm</td>
<td>1.9</td>
</tr>
<tr>
<td>Ag-2U</td>
<td>Ag</td>
<td>1-5 μm</td>
<td>5.6</td>
</tr>
<tr>
<td>Nb-1U</td>
<td>Nb</td>
<td>1-5 μm</td>
<td>0.8</td>
</tr>
<tr>
<td>Nb-2U</td>
<td>Nb</td>
<td>1-5 μm</td>
<td>2.3</td>
</tr>
<tr>
<td>o-U</td>
<td>none</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table I.b Cloth specimens: Nominal Powder Concentrations and Particle Sizes

<table>
<thead>
<tr>
<th>Specimen Name</th>
<th>Filler Type</th>
<th>Nominal Particle Size (μm)</th>
<th>Nominal Particle Concentration mg/cm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-1C</td>
<td>Al</td>
<td>1-5 μm</td>
<td>2.3</td>
</tr>
<tr>
<td>Al-2C</td>
<td>Al</td>
<td>1-5 μm</td>
<td>4.6</td>
</tr>
<tr>
<td>Ag-1C</td>
<td>Ag</td>
<td>1-5 μm</td>
<td>1.9</td>
</tr>
<tr>
<td>Ag-2C</td>
<td>Ag</td>
<td>1-5 μm</td>
<td>5.6</td>
</tr>
<tr>
<td>Nb-1C</td>
<td>Nb</td>
<td>1-5 μm</td>
<td>0.8</td>
</tr>
<tr>
<td>Nb-2C</td>
<td>Nb</td>
<td>1-5 μm</td>
<td>2.3</td>
</tr>
<tr>
<td>o-C</td>
<td>none</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

These concentrations were selected after preliminary experiments and calculations, so as to provide sufficient diffracted intensity to permit x-ray strain measurements. Specimens were prepared at OAI/NASA Lewis Research Center with the assistance and collaboration of Mr. Michael Castelli.

Fig.1 shows an example of Ag particles between plies. The particles on the surface are brighter than those deeper down, hence they are relatively far apart to be approximated by a single inclusion FEM or Eshelby model.
EXPERIMENTAL SETUP

For this work, a four-point bending device was built to fit onto the x-ray goniometer of a Siemens D500 diffractometer without affecting its alignment or the x-ray optics (Fig. 2a, b, c, d.). Fig. 2a shows the device installed. Fig. 2b shows the special holder to which the device is attached. A top view of the device is shown unloaded in Fig. 2c, and loaded, Fig. 2d. The device makes it possible to move the curved specimen surface so that the 0/2θ axis of the goniometer lies in this surface, thus avoiding displacement errors. The parallel-beam optics of the diffractometer removes remaining displacement and transparency errors. The 4 pins are free to rotate inside special bushings and also inside roller bearings in order to minimize friction forces at the specimen contact points, which may perturb the uniform stress field between the inner pins. The displacement of the outer pins is controlled by the micrometer that can be seen in the center of the device, (Fig. 2a, c). The inner and outer pin spacings were 12.5 and 40 mm respectively.

The main feature of this device is that the strains induced in the laminate are proportional to the outer pin displacements which are very well controlled by the micrometer. The device can be rotated 90 degrees in order to collect data for both strains $\varepsilon_{\theta=0,\psi}$, $\varepsilon_{\theta=90,\psi}$, where $\theta$ and $\psi$ are defined in Fig. 3. The device allows strain gages to be present on the specimen and the measurements are easy to perform.

The diffractometer optics consisted of a 0.1 degree divergence slit, a radial divergence limiting Soller slit with a divergence of 0.15 degrees on the diffracted beam side and a Peltier-cooled Si(Li) detector. Measurements were made with CuKα radiation and the (422), (422) and (321) reflections of Al, Ag and Nb respectively.
SINE SQUARED PSI METHOD

Hauk [14] gives formulas for determining the principal stresses $\sigma_1, \sigma_2, \sigma_3$ in polycrystalline specimens while neglecting the shear stresses $\sigma_{12}, \sigma_{23}, \sigma_{31}$. Use of these formulas is justifiable because of the low magnitude of the shear stresses in our specimens. Here directions 1, 2 and 3 inside the inclusion correspond to the directions $x, y$ and $z$, respectively. Assuming random orientation of the particles and knowing the x-ray elastic constants (XEC) and $d_{0}$, the stress-free lattice spacing $d_0$ (which is readily measured on the filler powder), the strain measured in the direction defined by $\phi$ and $\psi$ (Fig.3) is given, in terms of the principal stresses, by:

$$\varepsilon_{\phi,\psi} = \frac{d_{\phi,\psi} - d_0}{d_0} = \frac{1}{2} \delta^{(1)}_{i,j} \left( \sigma_1 \cos^2 \phi \sin^2 \psi + \sigma_2 \sin^2 \phi \sin^2 \psi + \sigma_3 \cos^2 \psi \right) \delta^{(2)}_{i,j} \left( \sigma_1 + \sigma_2 + \sigma_3 \right)$$

(3)
and in terms of the principal strains, \( \varepsilon_1, \varepsilon_2, \varepsilon_3 \), by:

\[
\varepsilon_{\psi,\varphi} = \varepsilon_1 \cos^2 \varphi \sin^2 \psi + \varepsilon_2 \sin^2 \varphi \sin^2 \psi + \varepsilon_3 \cos^2 \psi
\]  

(4)

If measurements of the filler lattice spacing \( d_{\psi,\varphi} \) for a back-reflection peak hkl are made at \( \varphi = 0 \) and various values of \( \psi \), eq.(3) can be written:

\[
\sigma_1 - \sigma_3 = \frac{1}{\frac{1}{2} S_{2\theta}^{hkl}} \frac{\partial \varepsilon_{\psi=0,\varphi}}{\partial \sin^2 \psi}
\]  

(5)

where \( \frac{\partial \varepsilon_{\psi=0,\varphi}}{\partial \sin^2 \psi} \) is the slope of the \( \varepsilon_{\psi=0,\varphi} \) versus \( \sin^2 \psi \) plot.

If measurements of \( \varepsilon_{\psi,\varphi} \) are made at \( \varphi = 90 \) and various \( \psi \) values, eq.(3) yields:

\[
\sigma_2 - \sigma_3 = \frac{1}{\frac{1}{2} S_{2\theta}^{hkl}} \frac{\partial \varepsilon_{\psi=90,\varphi}}{\partial \sin^2 \psi}
\]  

(6)

where \( \frac{\partial \varepsilon_{\psi=90,\varphi}}{\partial \sin^2 \psi} \) is the slope of the \( \varepsilon_{\psi=90,\varphi} \) versus \( \sin^2 \psi \) plot.

Also \( \sigma_3 \) is given by:

\[
\sigma_3 = \frac{d_{\psi=90,\varphi} - d_0}{d_0} - S_{1}^{hkl} \left( \sigma_1 + \sigma_2 \right)
\]  

(7)

The principal stresses are then obtained from eqns.5-7. The principal strains are similarly obtained from \( \sin^2 \psi \) plots using eq.(4). This method is only valid if the \( \sin^2 \psi \) plots are non-oscillatory. These plots were generally linear if pin displacements were moderate (<4 mm). An example is shown in Fig.4.
Sir

Fig. 4. $\varepsilon_{\psi=0,\psi}$ as a function of $\sin^2\psi$ for specimen Ag-2U, outer pin displacement $\delta=2$ mm.

Dölle [17] describes a method to get the X-ray elastic constants (XEC) using single crystal elastic constants $S_{11}$, $S_{12}$, and $S_{66}$ for cubic symmetry. Using this method, the average of Voigt and Reuss models of the XECs were calculated for the 3 powders and the reflections used. Table II shows the $S_{11}$, $S_{12}$ and $S_{66}$ values used [18], the calculated XECs in column 5 and 6, and the bulk mechanical values in columns 7 and 8. The calculated and bulk mechanical values agree quite well.

Table II Single crystal and x-ray elastic constants for the powder fillers used, in units of 1/TPa.

<table>
<thead>
<tr>
<th>Filler(hkl)</th>
<th>$S_{11}$</th>
<th>$S_{44}$</th>
<th>$S_{12}$</th>
<th>$S_{1}(hkl)$</th>
<th>$\frac{1}{2}S_{2}(hkl)$</th>
<th>$S_{1}=-\nu/E$</th>
<th>$\frac{1}{2}S_{2}=(1+\nu)/E$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al(422)</td>
<td>15.8</td>
<td>35.8</td>
<td>-5.8</td>
<td>-4.9</td>
<td>19.0</td>
<td>-5.0</td>
<td>19.5</td>
</tr>
<tr>
<td>Ag(422)</td>
<td>22.9</td>
<td>22.4</td>
<td>-9.8</td>
<td>-4.2</td>
<td>15.9</td>
<td>-4.4</td>
<td>16.5</td>
</tr>
<tr>
<td>Nb(321)</td>
<td>6.6</td>
<td>34.8</td>
<td>-2.33</td>
<td>-4.0</td>
<td>13.9</td>
<td>-3.5</td>
<td>12.9</td>
</tr>
</tbody>
</table>

Using the calculated XECs in Table II, one can get the stress transfer factors as is shown in the next section.

DETERMINING STRESS TRANSFER FACTORS

The stress measured in the direction $\varphi\psi$ is given by:

$$\sigma_{\varphi\psi} = \sigma_1 \cos^2 \varphi \sin^2 \psi + \sigma_2 \sin^2 \varphi \sin^2 \psi + \sigma_3 \cos^2 \psi$$

(8)
and the strain $\varepsilon_{\varphi\psi}$, by eq(4).

For the four-point bending device, the displacement, $\delta$, is the independent variable. In order to get the transfer factors, we have to transform the stresses in equation (8) to functions of $\delta$ and get the appropriate slopes that contain the parameters of interest. The stresses inside the inclusion are influenced by the stresses inside the fibers as well as inside the matrix. There is a stress transfer factor, $k$, of the matrix stresses to the inclusion stresses. For inclusions uniformly distributed in a homogeneous matrix, there are two stress transfer factors, one parallel to the applied stress and the other perpendicular to it [15].

For a non-homogeneous matrix, $k$ can be direction dependent. An Eshelby method can be applied, Withers [19], even if the system is heterogeneous [20]. Using appropriate transformations, the non-homogeneous case can be reduced to an homogeneous one by using the principle of equivalent inclusion and traction forces at the interface. Therefore, we can consider the stress transfer factor as a matrix with three components along the first diagonal.

$$k = \begin{pmatrix} k_1 & 0 & 0 \\ 0 & k_2 & 0 \\ 0 & 0 & k_3 \end{pmatrix}$$  \hspace{1cm} (9)

The stresses inside the inclusion can be expressed as follows:

$$\sigma_{\varphi\psi} = \sigma_{\varphi\psi}^{\text{Residual}} + k E_m \chi_\delta$$  \hspace{1cm} (10)

where $\chi_\delta$ is given by the following matrix:

$$\chi_\delta = \begin{pmatrix} 2z\delta & 0 & 0 \\ \frac{a(a+L)}{2z\nu_{12}\delta} & 0 & 0 \\ 0 & \frac{-2z\nu_{12}\delta}{a(a+L)} & 0 \end{pmatrix}$$  \hspace{1cm} (11)

and the constant, $\sigma_{\varphi\psi}^{\text{Residual}}$ is arbitrary at this point, because only the derivatives of these quantities are involved in eqs(5) and (6). $E_m$ is Young’s modulus for the matrix, $a$ is the distance between the inner and outer pin and $L$ is the distance between the two inner pins.

This matrix (11) has a non-zero term in the middle, due to the observation that $\varepsilon_{22}$ is a little bit different than zero in FEM simulations (the real situation is between the theory of plates and of beams) and because the Poisson’s ratio is no longer isotropic in real situations. This formulation gives a general approach by considering an anisotropic interaction between the components of a laminate. The factors, $\nu_{12}$, and $\nu_{13}$ were introduced for convenience. For the sake of derivation of stress transfer formulae, the $\sigma_{\varphi\psi}^{\text{Residual}}$ constants are neglected at this point since, for a given $\varphi$ and $\psi$, they do not vary with $\delta$. 

Substituting eq. (10) in (8) at the appropriate $\varphi$ and $\psi$ values gives expressions for $\sigma_1$, $\sigma_2$, and $\sigma_3$, the principal stresses in the inclusion as functions of $\delta$:

$$\sigma_1 = 2k_1 E_m z \frac{\delta}{a(a + L)}$$  \hspace{1cm} (12)

$$\sigma_2 = -2k_2 E_m v_{12} z \frac{\delta}{a(a + L)}$$  \hspace{1cm} (13)

$$\sigma_3 = -2k_3 E_m v_{13} z \frac{\delta}{a(a + L)}$$  \hspace{1cm} (14)

These values substituted in (3) give the strains as a function of XEC, direction $\varphi$, $\psi$, stress transfer factors, geometry of the sample, etc.

$$\varepsilon_{\varphi\psi} = -\frac{E_m z \delta}{a(a + L)} (-k_1 S_2^{hh} \cos^2 \varphi \sin^2 \psi + k_2 v_{12} S_2^{hh} \sin^2 \varphi \sin^2 \psi + k_3 v_{12} S_1^{hh} + 2k_2 v_{12} S_1^{hh} + 2k_3 v_{13} S_1^{hh})$$  \hspace{1cm} (15)

The double derivative of the $\varepsilon_{\varphi\psi}$ expression with respect with $\sin^2 \psi$ and $\delta$ variables, gives equations (16) and (18), and the first derivative with respect to $\delta$, gives equations (17) and (19). Eq. (16)-(19) can be solved for $k_1$, $k_2$ and $k_3$.

$$\frac{\partial^2 \varepsilon_{\varphi=0,\psi}}{\partial \sin^2 \psi \partial \delta} = E_m z \frac{S_2^{hh} (k_1 + v_{12} k_2)}{a(a + L)}$$  \hspace{1cm} (16)

$$\frac{\partial \varepsilon_{\varphi=00,\psi}}{\partial \delta} = -E_m z \frac{S_2^{hh} k_3 v_{13} - 2S_1^{hh} (k_1 - v_{12} k_2 - v_{13} k_3)}{a(a + L)}$$  \hspace{1cm} (17)

$$\frac{\partial^2 \varepsilon_{\varphi=90,\psi}}{\partial \sin^2 \psi \partial \delta} = -E_m z \frac{S_2^{hh} (v_{12} k_2 - v_{13} k_3)}{a(a + L)}$$  \hspace{1cm} (18)

$$\frac{\partial \varepsilon_{\varphi=90,\psi=0}}{\partial \delta} = -E_m z \frac{S_2^{hh} k_3 v_{13} - 2S_1^{hh} (k_1 - v_{12} k_2 - v_{13} k_3)}{a(a + L)}$$  \hspace{1cm} (19)

Table III shows the values of $k$ thus obtained for the three fillers in unidirectional and cloth laminates:
Table III. Experimental stress transfer factors for the fillers in unidirectional and cloth laminates

<table>
<thead>
<tr>
<th>Stress Transfer Factors</th>
<th>Unidirectional laminate</th>
<th>Cloth ply laminate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ag(422)</td>
<td>Al(422)</td>
</tr>
<tr>
<td>k₁</td>
<td>2.44</td>
<td>1.86</td>
</tr>
<tr>
<td>k₂</td>
<td>-0.47</td>
<td>-1.10</td>
</tr>
<tr>
<td>k₃</td>
<td>-0.77</td>
<td>-0.85</td>
</tr>
</tbody>
</table>

With these constant k values, one can find the stresses in the matrix if the stresses inside the inclusion are known, via eq.(2).

RESULTS AND DISCUSSION

Fig.5 shows schematically how the measured and calculated strains are related as a function $\delta$. $\Delta$ in Fig.5 is the shift due to the multiple inclusion effect and can be formally calculated using Axelsen’s procedure [21]. The values of the shifts in Table IV were obtained by comparing the XRD strains with those obtained by Eshelby and FEM methods.

Fig.6a and Fig.6b show the comparison of FEM and Eshelby models, described elsewhere [22], with XRD measurements from Ag filler. Elastic constants used for generating the FEM and Eshelby strains (Fig.6a) and FEM and Eshelby stresses (Fig.6b) are those in Table II. The error bars in these figures refer to XRD data points only. In general the agreement between the XRD, FEM and Eshelby strains is reasonable although differences are somewhat larger than the error bars. The $\varepsilon_2$ strains are close to zero as expected for plate bending. The agreement between the stresses is considerably poorer and could be due to difficulties in specimen alignment at $\phi=90$ and to assumptions made in models. Yielding of the particles may also be occurring at large $\delta$. 

Fig.5 Schematic diagram for residual strain shifts due to multiple inclusion effect
Table IV Multiple inclusion shifts for Ag and Al for correcting one inclusion FEM and Eshelby model, in unidirectional laminates.

<table>
<thead>
<tr>
<th>Filler*</th>
<th>Strain shift matrix</th>
<th>Stress shift matrix, MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag</td>
<td>$\varepsilon_{FEM} = \begin{pmatrix} 0 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 1.5 \ 0 &amp; 0 &amp; 0 \end{pmatrix} \times 10^{-4}$</td>
<td>$\sigma_{FEM} = \begin{pmatrix} 41 &amp; 0 &amp; 0 \ 0 &amp; 38 &amp; 0 \ 0 &amp; 0 &amp; 48 \end{pmatrix}$</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_{Esh.} = \begin{pmatrix} 0 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 \end{pmatrix}$</td>
<td>$\sigma_{Esh.} = \begin{pmatrix} 65 &amp; 0 &amp; 0 \ 0 &amp; 45 &amp; 0 \ 0 &amp; 0 &amp; 55 \end{pmatrix}$</td>
</tr>
<tr>
<td>Al</td>
<td>$\varepsilon_{FEM} = \begin{pmatrix} 5 &amp; 0 &amp; 0 \ 0 &amp; 5 &amp; 0 \ 0 &amp; 0 &amp; 2 \end{pmatrix} \times 10^{-4}$</td>
<td>$\sigma_{FEM} = \begin{pmatrix} 15 &amp; 0 &amp; 0 \ 0 &amp; 25 &amp; 0 \ 0 &amp; 0 &amp; 23 \end{pmatrix}$</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_{Esh.} = \begin{pmatrix} 2 &amp; 0 &amp; 0 \ 0 &amp; 0.1 &amp; 0 \ 0 &amp; 0 &amp; -1.4 \end{pmatrix} \times 10^{-4}$</td>
<td>$\sigma_{Esh.} = \begin{pmatrix} 10 &amp; 0 &amp; 0 \ 0 &amp; 15 &amp; 0 \ 0 &amp; 0 &amp; 20 \end{pmatrix}$</td>
</tr>
</tbody>
</table>

Fig. 6a Strains inside Ag inclusions obtained from FEM and Eshelby models and XRD measurements.
Knowing the stress transfer factors, one can calculate the stresses in the matrix, which are not directly accessible through XRD measurements, using eq (2). The stresses, inside and outside the inclusion are described by the following equation:

\[ \sigma_{\text{FEM}}^{\text{radial}}(\text{inclusion}) = k_{\text{XRD}} \sigma_{\text{radial}}^{\text{radial}}(\text{matrix}) \]  

(20)

where the matrix \( k_{\text{XRD}} \) is measured with XRD method described before.

The values thus obtained in Table V, for a unidirectional laminate, are of the same order of magnitude with those given by Hyer [23], for a unidirectional square cross section composite, with graphite fibers in a hexagonal packed array. The higher values for cloth laminates are not well understood and required further experiments.

Table V Local residual stresses in the matrix phase close to the inclusion, assuming perfect bounding at the polyimide/inclusion interface. The units are in MPa.

<table>
<thead>
<tr>
<th>Inclusion</th>
<th>Unidirectional laminate</th>
<th>Cloth laminate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \sigma_{xx} )</td>
<td>( \sigma_{yy} )</td>
</tr>
<tr>
<td>Ag</td>
<td>14</td>
<td>-24</td>
</tr>
<tr>
<td>Al</td>
<td>18</td>
<td>-13.4</td>
</tr>
</tbody>
</table>
The values in Table V were expected to be similar for Ag and Al inclusions since they have similar elastic properties and thermal expansion. It is known that the reproducibility of physical properties of graphite/polyimide laminates is poor - fiber volume fraction cannot be well controlled, and even the thickness of the specimen cannot be kept constant. Therefore, processing-induced residual stresses [21-22], could be a factor in getting different results in Table V. In addition, the high non-uniformity of stresses in these type of laminates may be another factor contributing to the spread of results in Table V.

CONCLUSIONS

Based on results obtained thus far on unidirectional and cloth graphite/polyimide laminates:

- Strains obtained by x-ray diffraction from particles embedded between the 1st and 2nd plies are sensitive to residual stresses in the as-cured laminate.
- These strains respond linearly to stresses applied by 4-point bending, qualitatively as expected.

- Strains and stresses in the filler particles predicted by FEM, and Eshelby models, show the same trends with pin displacement as those measured by XRD. Quantitative agreement is reasonable for the strains but less so for the stresses. Reasons for the differences are not well understood.

- Ag filler was found to give the most linear \( \sin^2 \psi \) plots presumably because of the small size and uniform size distribution of the particles.

- Stress transfer factors between the applied stress and the stresses in the particle were obtained and allowed the residual stresses in the matrix adjacent to the particles to be calculated.

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BIBLIOGRAPHY