X-Ray Stress Measurement in Graphite/Epoxy Composites

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Abstract
Small amounts (< 2 mg/cm²) of Al and Nb filler powders were incorporated between the first and second plies of 6-ply unidirectional graphite/epoxy laminates. Cu Ka₁ X-rays were diffracted from specific crystallographic planes in these fillers: 333 + 511 for Al and 411 + 330 for Nb, giving peaks in the back reflection region. The peak positions shifted linearly with stress applied to the laminates in the fiber direction and had stress sensitivities of 8.52 and 3.92 x 10⁻⁴ deg 2θ/MPa for Al and Nb respectively. Elastic strains in the filler particles measured by X-rays were found to be proportional to the corresponding composite strains measured by strain gages, in agreement with the model of H. T. Hahn. Residual strains and stresses in filler particles were also measured.

Keywords
Nondestructive Evaluation

Disciplines
Materials Science and Engineering
X-RAY STRESS MEASUREMENT IN GRAPHITE/EPOXY COMPOSITES

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ABSTRACT

Small amounts (< 2 mg/cm²) of Al and Nb filler powders were incorporated between the first and second plies of 6-ply unidirectional graphite/epoxy laminates. Cu Ka X-rays were diffracted from specific crystallographic planes in these fillers: 333 + 511 for Al and 411 + 330 for Nb, giving peaks in the back reflection region. The peak positions shifted linearly with stress applied to the laminates in the fiber direction and had stress sensitivities of 8.52 and 3.92 x 10⁻⁴ deg°/MPa for Al and Nb respectively. Elastic strains in the filler particles measured by X-rays were found to be proportional to the corresponding composite strains measured by strain gages, in agreement with the model of H. T. Hahn. Residual strains and stresses in filler particles were also measured.

INTRODUCTION

It has recently been shown that information about residual and applied stresses in non-crystalline polymeric materials including polymer matrix composites can be obtained by X-ray diffraction from small amounts of crystalline fillers incorporated into the polymer. (1-3) The method has the advantage that the filler particles can be placed at varying depths below the sample surface and can be chosen so as to give sharp, well-resolved diffraction peaks at large diffraction angles. The disadvantage is that relations must be established between the principal elastic strains measured in the filler particles and the stresses in the composite. Thus far these relations have been found to be linear with several fillers at stresses below the yield point of the surrounding matrix. We present here experimental evidence for such linear behavior using two metallic fillers: Al and Nb having promising properties for technological applications.

EXPERIMENTAL PROCEDURE

Al and Nb metal powders of -325 mesh were spread in a thin layer (< 2 mg/cm²) between the first and second plies of uncured 6 ply unidirectional graphite/epoxy laminates (Fiberite T-300/934, Fiberite Corp., Winona, Minn.). The Nb powder (Pansteel Corp., No. Chicago, Ill.) was vacuum annealed 1 hr at 1300°C to sharpen the X-ray peaks prior to use. Spreading of the powders was done as uniformly as possible by rubbing the powders onto the surface of one prepreg ply and shaking off particles which did not adhere. The laminates were laid up in 7.62 x 30.48 cm stacks and cured using the recommended procedures for this prepreg. SEM and energy dispersive X-ray analysis of cross-sections of cured laminates showed that transverse migration of filler particles into adjacent plies during curing was negligible.

Longitudinal tensile samples 15.25 x 1.905 x .089 cm thick were cut from the cured laminates, tapered aluminum (2014-T6) end-tabs were applied and a strain gage (2 element, 90° stacked) attached near the center of the gauge area of each sample. Samples were held in clevis grips in a small tensile frame mounted on a Siemens diffractometer as described elsewhere. (2)

The principle of the method is shown in Fig. 1. X-radiation is incident on the laminate sample near the strain gage. Cu Ka X-rays diffracted by specific crystallographic planes in the filler particles (333 + 511 for Al and 411 + 330 for Nb) pass through a monochromator into a detector. Strain in the particles appears as a shift in the diffracted peak positions as required by the Bragg law. Diffraction peak positions were determined using a standard procedure. (4) The upper 1/3-1/4 of each peak was step-scanned and a parabola fitted to the points by least squares. The apex of the parabola was taken as the peak position. The direction of strain measurement in the diffraction method is defined by the angles φ and ψ relative to the laminate axes 1, 2, 3 (Fig. 1). The three principal filler particle strains, assumed parallel to the laminate axes, are readily obtained from peak position measurements using 3 different incident beam directions and the peak position for an unstressed powder sample of the filler. Equations for calculating the filler strains were presented elsewhere. (5) For the present applied stress experiments, only two directions of strain measurement were needed: φ = 90 and φ = 45. For residual stresses, the direction φ = 90, ψ = 45 was added.

RESULTS AND DISCUSSION

Applied Stresses - The shift in the diffraction angle, 2θ, at φ = 90° with applied tensile stress εₚ in the fiber direction is shown in Figs. 2 and 3 for laminates containing Al and Nb respectively. The breaks in these curves at 150 and 650 MPa respectively on the first loading are thought to be due to yielding in the filler particles. If the samples are loaded above these filler yield points, then on release of the load the 2θ values are lower than before the first loading (Figs. 2 and 3) indicating that a compressive strain contribution remains after unloading. Possible reasons for this contribution are that the surrounding matrix is compressing the yielded particles in the fiber direction on unloading or that pseudo macrostresses are present in the particles themselves, resulting from the plastic deformation. (6) The slopes of the lines below the yield point in Figs. 2 and 3 are termed the stress sensitivities, S, of the filler and are listed in Table I.

The mean filler strains in the fiber direction ε₁f and normal to the laminate ε₃f were calculated from the X-ray peak shifts below the yield point for the second loading. The results are shown in Figs.
TABLE I

<table>
<thead>
<tr>
<th>Filler and Loading Cycle</th>
<th>((\frac{d\sigma_{1f}}{d\sigma_{1f}^<em>}})/(\frac{d\sigma_{1m}}{d\sigma_{1m}^</em>}})) (MPa)^{-1}</th>
<th>(\eta_1)</th>
<th>(E_f) (10^6 MPa)</th>
<th>(\nu_f) (10^5 MPa)</th>
<th>(E_{11}^*) (10^5 MPa)</th>
<th>(S) (10^{-6} deg 20/MPa)</th>
<th>Experiment Theory (Eq. 3)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Al</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1st loading</td>
<td>---</td>
<td>---</td>
<td>.7034</td>
<td>.347</td>
<td>1.06</td>
<td>8.39</td>
<td>---</td>
</tr>
<tr>
<td>2nd loading</td>
<td>.267</td>
<td>.184</td>
<td>.7034</td>
<td>.347</td>
<td>1.06</td>
<td>8.65</td>
<td>6.54</td>
</tr>
<tr>
<td><strong>Nb</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1st loading</td>
<td>---</td>
<td>---</td>
<td>1.035</td>
<td>.38</td>
<td>1.20</td>
<td>4.12</td>
<td>---</td>
</tr>
<tr>
<td>2nd loading</td>
<td>.154</td>
<td>.22</td>
<td>1.035</td>
<td>.38</td>
<td>1.20</td>
<td>3.72</td>
<td>3.50</td>
</tr>
</tbody>
</table>


4 and 5 for Al and Nb respectively. Longitudinal and transverse strain gage strains \(\varepsilon_{1f}^\ast\) and \(\varepsilon_{2f}^\ast\) divided by ten are shown for comparison. The data fall on straight lines within experimental error. The ratios of the slopes; \((\frac{d\sigma_{1f}}{d\sigma_{1f}^*}})/(\frac{d\sigma_{1m}}{d\sigma_{1m}^*}})\) in these figures are given in Table I.

It has been proposed by Hahn(7) that in a particulate composite the mean matrix stress, \(\sigma_{1m}\) is simply proportional to the mean filler stress \(\sigma_{1f}\) with \(\eta_1\) the proportionality constant. For the isotropic case,

\[
\sigma_{1m} = \eta_1 \sigma_{1f}, \quad i = 1-6
\]  

(1)

For a unidirectional composite, if \(\sigma_{1m}\) and \(\sigma_{1f}\) are the only non-zero stress components; it has been shown(2) that at small volume fractions of filler, the Hahn factor

\[
\eta_1 = \frac{\varepsilon_{1f}^\ast}{\varepsilon_{1m}^\ast} = \frac{E_m}{E_f} \quad \eta_1 = \frac{\varepsilon_{1f}^\ast}{\varepsilon_{1m}^\ast} = \frac{E_m}{E_f} \quad \eta_1 = \frac{\varepsilon_{1f}^\ast}{\varepsilon_{1m}^\ast} = \frac{E_m}{E_f}
\]  

(2)

where \(E_m\) is the Young's modulus of the matrix, taken as 3.45 x 10^5 MPa (0.5 x 10^6 psi) and \(E_f\) is the Young's modulus of the filler. Values of \(\eta_1\) calculated from eq. (2) using the ratio of slopes in Table I are given in Table I. Knowing \(\eta_1\) and assuming that \(\sigma_{1f}^\ast\) is the only non-zero stress, an expression for the stress sensitivity, \(S\), can be derived.

\[
S = \frac{(\Delta \phi)}{\phi_{\nu=0}} = \frac{2 \nu_f E_m}{E_f \eta_1 E_f} \cdot \tan(\phi_{\nu=0}) \quad \text{(rad/MPa)}
\]  

(3)

Values of \(S\) calculated from eq. (3) and obtained from the data in Figs. 2 and 3 are compared in Table I. The agreement is reasonable in view of the assumptions required for eq. (3). Equation (3) also predicts approximately a constant slope for the lines in Figs. 2 and 3 below the yield point, as observed. Desirable filler properties for high stress sensitivity are obviously a diffraction peak at large \(\phi\) and a small filler modulus and Hahn factor. Al and Nb possess the highest \(S\) values of candidate fillers tested to date with CuK\(\alpha\) radiation.

Residual Stresses - The principal residual strains

TABLE II

<table>
<thead>
<tr>
<th>Filler</th>
<th>(\varepsilon_1)</th>
<th>(\varepsilon_2)</th>
<th>(\varepsilon_3)</th>
<th>(\sigma_1)</th>
<th>(\sigma_2)</th>
<th>(\sigma_3)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Al</strong></td>
<td>439</td>
<td>-56</td>
<td>-218</td>
<td>27</td>
<td>3.5 - 4.2</td>
<td></td>
</tr>
<tr>
<td><strong>Nb</strong></td>
<td>573</td>
<td>-307</td>
<td>-286</td>
<td>40.6</td>
<td>-25.4 -23.8</td>
<td></td>
</tr>
</tbody>
</table>

The values in Table II require experiments with fillers in neat resin before their significance can be fully assessed; however, they all show the same trend. The stresses and strains in the fiber direction are algebraically the largest, indicating a residual tensile stress in the resin in this direction. The remaining two stresses are significantly smaller and not greatly different from zero. They result from differences in Poisson contraction and thermal expansion coefficient between the resin and the filler.

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REFERENCES


Fig. 1. (A) Schematic of diffraction conditions. (B) Direction of strain measurement.

Fig. 2. Linear increase in 333+511 X-ray peak position with tensile stress applied in fiber direction of a 0° laminate containing Al particles. Conditions are similar to Fig. 1.

Fig. 3. Linear increase in 411+330 X-ray peak position with applied tensile stress in a 0° laminate containing Nb particles. Conditions are similar to Fig. 2.

Fig. 4. Linear change in X-ray strains e1f and e2f with applied stress in the Al containing laminate (2nd loading). Strain gage strain is also shown.

Fig. 5. Linear change in X-ray strains e1f and e2f with applied stress in the Nb containing laminate (2nd loading). Strain gage strain is also shown.