X-RAY STRESS MEASUREMENT IN GRAPHITE/EPOXY COMPOSITES

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ABSTRACT

Small amounts (< 2 mg/cm^2) of Al and Nb filler powders were incorporated between the first and second plies of 6-ply unidirectional graphite/epoxy laminates. Cu Kα_1 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.

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 at 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 filler particles. 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/cm^2) 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 (Fansteel 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 cms 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 gage 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 Kα_1 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.

Diffracted 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=ψ=o and ϕ=o, ψ=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=ψ=o with applied tensile stress σ_1* 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 $\bar{\epsilon}_{1f}$ and normal to the laminate $\bar{\epsilon}_{3f}$ were calculated from the X-ray peak shifts below the yield point for the second loading. The results are shown in Figs.

TABLE I

Stress Sensitivities and Hahn Factors of Fillers

Filler and Loading Cycle	$(d\bar{\varepsilon}_{1f}/d\sigma_1^*)/(d\varepsilon_1^*/d\sigma_1^*)$ $(MPa)^{-1}$	η_1	E_f (10^5 MPa)	ν_f	E_{11}^* (10^5 MPa)	$S(10^{-4}$ deg 2θ/MPa) Experiment	Theory (Eq. 3)
Al							
1st loading	---	---	.7034	.347	1.06	8.39	---
2nd loading	.267	.184	.7034	.347	1.06	8.65	6.54
Nb							
1st loading	---	---	1.035	.38	1.20	4.12	---
2nd loading	.154	.22	1.035	.38	1.20	3.72	3.50

E_f and ν_f data for Al from ref. (4), for Nb from ASM Metals Handbook, 8th ed., 1961, Vol. 1, p. 1202.

4 and 5 for Al and Nb respectively. Longitudinal and transverse strain gage strains ε_1^* and ε_2^* divided by ten are shown for comparison. The data fall on straight lines within experimental error. The ratios of the slopes; $(d\bar{\varepsilon}_{1f}/d\sigma_1^*)/(d\varepsilon_1^*/d\sigma_1^*)$ in these figures are given in Table I.

It has been proposed by Hahn[7] that in a particulate composite the mean matrix stress, $\bar{\sigma}_{1m}$ is simply proportional to the mean filler stress σ_{im} with η the proportionality constant. For the isotropic case,

$$\bar{\sigma}_{im} = \eta\, \bar{\sigma}_{if}, \qquad i = 1\text{-}6 \tag{1}$$

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

$$\eta_1 \cong \frac{\varepsilon_1^*}{\bar{\varepsilon}_{1f}} \frac{E_m}{E_f} \tag{2}$$

where E_m is the Young's modulus of the matrix, taken as 3.45×10^3 MPa (0.5×10^6 psi) and E_f is the Young's modulus of the filler. Values of η_1 calculated from eq. (2) using the ratio of slopes in Table I are given in Table I. Knowing η_1 and assuming that σ_1^* is the only non-zero stress, an expression for the stress sensitivity, S, can be derived.[2]

$$S = \frac{[\Delta 2\theta]_{\phi=\psi=o}}{\sigma_1^*} = \frac{2\,\nu_f\, E_m}{E_f\, \eta_1\, E_{11}^*} \cdot \mathrm{Tan}[\theta]_{\phi=\psi=o} \quad (\mathrm{rad/MPa}) \tag{3}$$

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 θ and a small filler modulus and Hahn factor. Al and Nb possess the highest S values of candidate fillers tested to date with $CuK\alpha_1$ radiation.

Residual Stresses - The principal residual strains in the filler particles in cured laminates after 1/2 yr storage under ambient conditions are listed in Table II. The principal stresses in the fillers calculated from the strains assuming isotropic filler properties are also shown. The standard deviations in residual filler stresses obtained from duplicate measurements were ± 5.7 MPa for Al and ± 47 MPa for Nb. The larger standard deviation with the Nb filler is due to the larger particle size and fewer particles of the Nb powder used which resulted in a more non-uniform distribution of diffracting particles in the irradiated area and a greater standard deviation in 2θ measurements than with Al.

TABLE II

Residual Strains and Stresses in Filler Particles in Unidirectional Laminates

Filler	Microstrains ε_1	ε_2	ε_3	Stresses (MPa) σ_1	σ_2	σ_3
Al	439	- 56	-218	27	3.5 - 4.2	
Nb	573	-307	-286	40.6	-25.4	-23.8

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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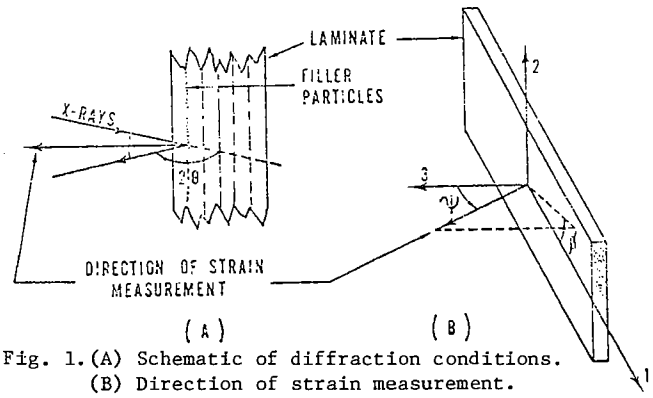

Fig. 1. (A) Schematic of diffraction conditions. (B) Direction of strain measurement.

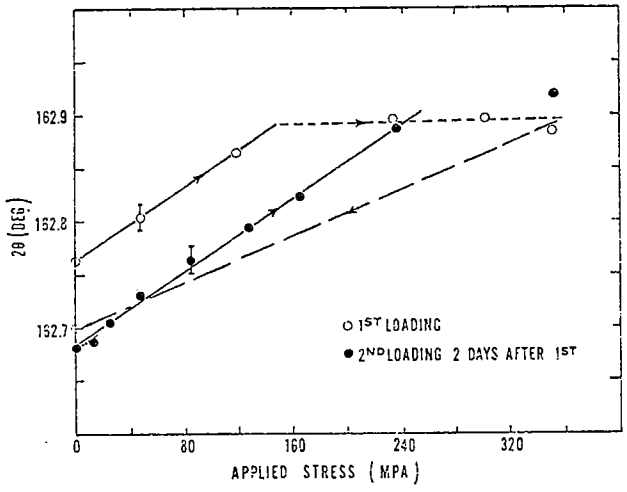

Fig. 2. Linear increase in 333+511 X-ray peak position with tensile stress applied in fiber direction of a 0° laminate containing Al particles. (ϕ=ψ=o. CuKα_1 radiation). Error bars in this and subsequent figures are ± one standard deviation.

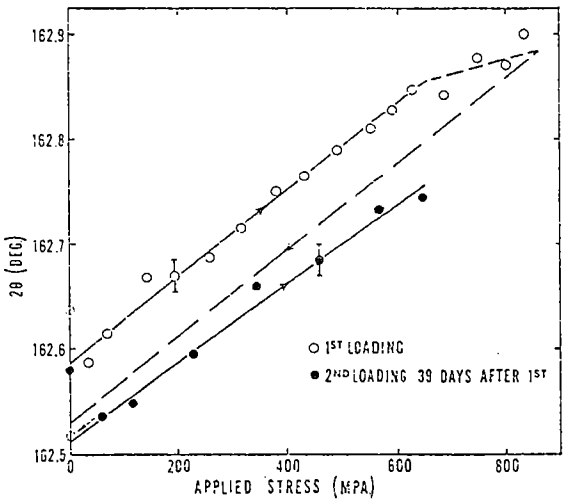

Fig. 3. Linear increase in 411+330 X-ray peak position with applied tensile stress in a 0° laminate containing Nb particles. Conditions are similar to Fig. 2.

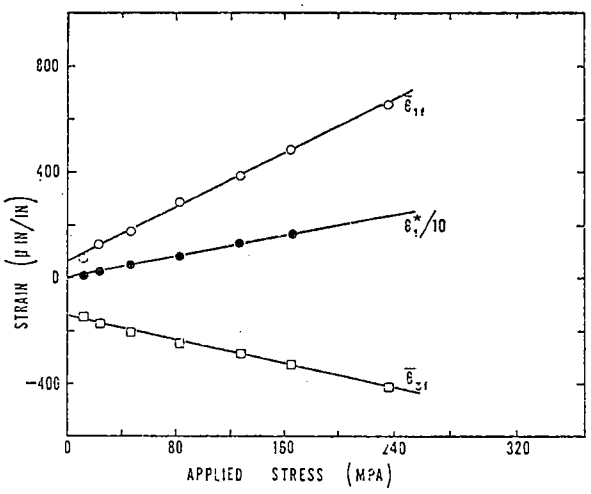

Fig. 4. Linear change in X-ray strains, $\bar{\epsilon}_{1f}$ and $\bar{\epsilon}_{3f}$ with applied stress in the Al containing laminate (2nd loading). Strain gage strain is also shown.

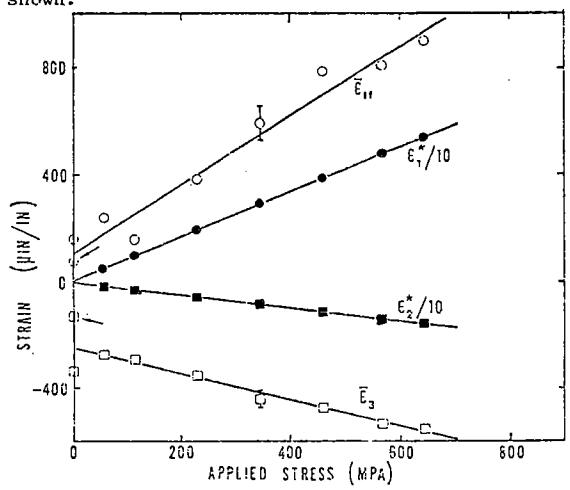

Fig. 5. Linear change in X-ray strains, $\bar{\epsilon}_{1f}$ and $\bar{\epsilon}_{3f}$ with applied stress in the Nb containing laminate (2nd loading). Strain gage strain is also shown.