

# An evaluation of residual stresses in graphite/PMR-15 composites by X-ray diffraction

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## Abstract

In this work, a method based on X-ray diffraction measurements of internal stresses in embedded metallic ellipsoidal inclusions is briefly described. The method has been recently developed for the determination of residual thermal stresses in high temperature graphite/polyimide composites. The effects of external bending loads and aging on the measurements of the internal stress in unidirectional and woven graphite fiber (T650-35)/polyimide (PMR-15) composites were examined in addition to several other factors, which could influence the accuracy of the stress measurements. Such factors as the volume fraction of inclusions, their aspect ratios and the interaction between individual embedded inclusions were also evaluated in this study. It has been shown that despite its complexity the proposed method can be successfully applied to the evaluation of residual stresses in high temperature polymer matrix composites subjected to external loads and aging.

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## 1. Introduction

The evaluation of the residual thermal stresses in polymer matrix composites is not a straightforward task. Several methods have been proposed for the residual stress measurements in the composites, each with its own limitations [1–22]. One of them is based on X-ray diffraction (XRD) measurements of residual strains in crystalline ellipsoidal inclusions embedded in the interlaminar regions of unidirectional and woven polymer matrix composites [8–22]. The residual interlaminar stresses in the composites are then extracted from the XRD data for the inclusions using either analytical or numerical models. The idea of employing embedded metallic inclusions as stress sensors was first proposed

by Predecki and Barrett [8–11] and used to measure residual stresses in unidirectional graphite/epoxy composites with embedded aluminum, silver and niobium inclusions. In their work [8–11] Predecki and Barrett were successful in measuring the strains and stresses in the embedded inclusions including the effect of externally applied mechanical loads. However, they were unable to quantitatively evaluate the stress transfer from the composites to the embedded inclusions, which is critical for the correct determination of the state of stress in the composites.

Fenn et al. [12] used XRD to measure triaxial strains in nickel inclusions embedded in Araldite MY 753 epoxy resin as a function of the volume fraction of inclusions and the polymer curing temperature. The authors observed little difference in measured strains over a range of metallic inclusion volume fractions (10–30%). They also observed that the measured strains inside inclusions for the examined curing temperatures did not coincide with theoretically predicted values. This discrepancy

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was attributed to the lack of the incorporation of temperature dependent resin properties into the model. Meske and Schnack [13,14] investigated strains in silver inclusions embedded between composite layers using XRD. They proposed a new micromechanical approach to study the stress–strain relationship between the measured strain in the inclusions and the stress in the composite. However, their approach was based on the assumption that the inclusions formed a continuous layer in the composite, which is rarely satisfied as revealed by SEM analyses [15,16].

Recently, major research activities have been undertaken [15–22] to further improve the method of Barrett and Predecki and apply it to the measurements of residual interlaminar stresses in advanced high temperature polymer matrix composites based on T650-35 graphite fibers embedded in a PMR-15 polyimide matrix (see Figs. 1(a) and (b)). These composites develop large residual stresses, which can, in some cases, cause premature interlaminar fracture of the composite panels during manufacturing [23–33]. The residual stresses can also greatly affect the mechanical performance of the composites, especially under shear dominated biaxial

loads [24,26,31,33]. Thus, the precise determination of the stresses in these composites was found to be of primary importance. In order to measure the stresses the XRD method of Predecki and Barrett [8–11] was used and significantly improved by adding strong analytical and numerical components [15–22]. Without these two new components, it would not be possible to extract the residual stresses in the composites from the XRD residual stresses in the inclusions for different composite architectures (woven and unidirectional) as a function of manufacturing conditions, external loads and potential in-service aging conditions. The present methodology is based on XRD measurements of residual stresses in embedded inclusions and then on extracting the information about the interlaminar stresses in the composites using the Eshelby/Mori–Tanaka approach [34–36]. Throughout this paper we will refer to this methodology as the XRD–Eshelby/Mori–Tanaka (XRD–E/M–T) method.

In this work, we briefly describe the XRD–E/M–T method and present a few key examples of its application to the residual stress determination in the T650-35/PMR-15 composites. New computational results are also presented here to further verify the accuracy of the method and to establish its potential limitations.

## 2. Fundamental basis of the XRD–Eshelby/Mori–Tanaka approach

### 2.1. XRD measurements

The experimental determination of the strains and stresses in Al inclusions embedded in composites was performed in the following way. First, the lattice spacing  $d_0$  between 422 atomic planes for unstressed Al powder was measured using the well-established XRD technique [37–39]. Second, the lattice spacing  $d_{\phi\psi}$  for 422 planes perpendicular to the diffraction vector  $\mathbf{L}_{\phi\psi}$  (see Fig. 2) for the Al inclusions were experimentally obtained using the same XRD technique. It can be shown that if  $\frac{d_{\phi\psi}-d_0}{d_0}$

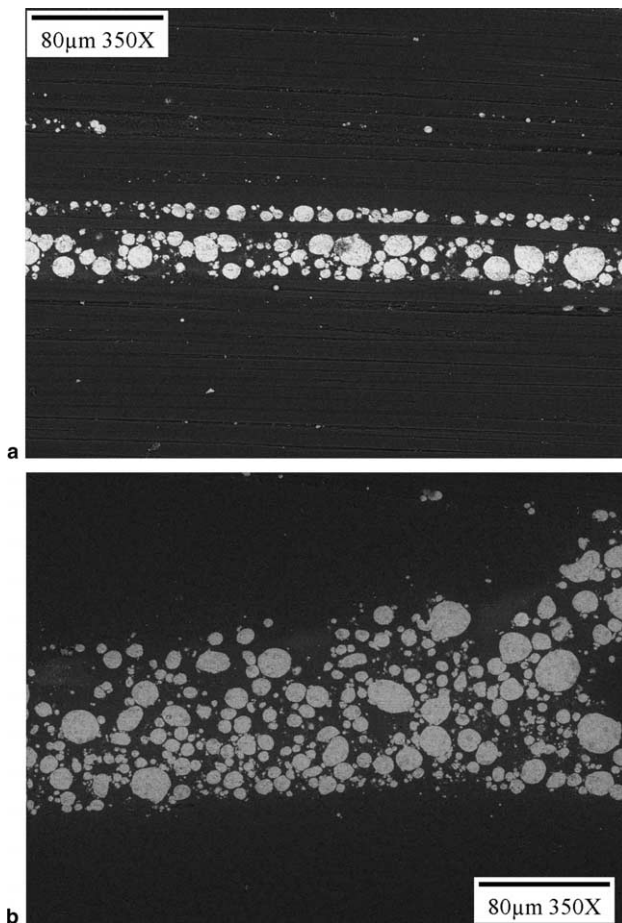


Fig. 1. Al inclusions distributed in the interlaminar region of (a) unidirectional and (b) 8HS woven T650-35/PMR-15 composite.

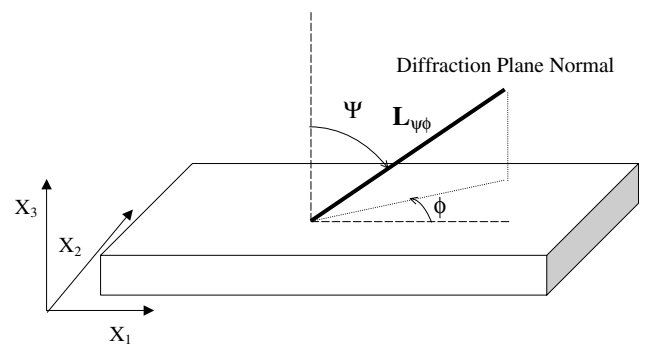


Fig. 2. Definition of angles  $\phi$  and  $\psi$ , and the specimen coordinate system  $x_1, x_2, x_3$ .

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