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X-ray Microdiffraction Studies to Measure Strain Fields in a Metal Matrix Composite

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An x-ray diffraction microscope has been used to map the strain field in a fiber-reinforced composite material. The monochromatic x-ray (11 keV) beam was focused by a phase zone plate to produce a focal spot of 1 x 4 μm^2 on the specimen. The change in the peak position of diffraction patterns due to interatomic spacing change, caused by stress in the sample, was measured by using a two-dimensional CCD detector. The radial residual strain field in the fiber-reinforced composite (SCS-6/Ti-14Al-21Nb) was measured from diffraction patterns with a sensitivity of ~10^{-4} and an average standard deviation of 9.4 x 10^{-5}.

INTRODUCTION

X-ray diffraction studies with the use of synchrotron radiation source have become a very useful tool to understand the interatomic structures of materials. It was recently demonstrated that the high brilliance properties of third-generation synchrotron radiation sources like the Advanced Photon Source (APS) allow one to use a microfocusing technique with a zone plate and thus generate a high monochromatic flux with diameters down to a ~1 μm (1,2). The use of a microfocused x-ray beam for conducting x-ray diffraction studies makes it possible to measure residual strain/stress distributions at microscale levels in composites.

The measurement of strains can be obtained by investigating the relative shift of a peak position of a diffraction pattern. The relative shift is measured from sequential data for different positions of a specimen; this shift is due to interatomic spacing change caused by stress in the specimen. Such a relatively simple measurement provides us with quantitative analysis of the strain distribution in the specimen.

The proposed x-ray diffraction microscope (XDM) used in this work, is a straightforward experimental setup, in which the diffraction patterns are recorded by a CCD camera. We will demonstrate that the importance of XDM at the APS is in allowing a fast measurement and analysis of residual strain distribution through a specimen. The technique permits a precise measurement with a sensitivity of ~10^{-2} degree in diffraction angle (2θ) corresponding to that of ~10^{-4} in strain. The technique is then used to measure the strains near silicon carbide fibers (SCS-6) in a Ti-14Al-21Nb matrix.

EXPERIMENTAL SETUP

The experiment was performed at the 2-ID-D undulator beamline at the Advanced Photon Source (3). Figure 1 shows a schematic view of the experimental setup. A double-crystal monochromator, which is composed of two (111)-Si crystals, was used to select a 11 keV (λ=1.127 Å). A zone plate was used to focus the monochromatic 11 keV x-ray beam ($\Delta\lambda/\lambda = 10^{-4}$) and then produce a focal spot size of ~1 x 4 μm^2 with a flux of 1.3×10^{11} photons/sec. The microfocused, monochromatic 11 keV x-ray beam was used to observe Debye diffraction patterns of the fiber-reinforced composite. The sample was fixed on a two-dimensional (x-y) stage that is attached to a rotational (θ) stage, and a CCD detector is mounted on the 2θ stage. Both θ- and 2θ-stages are concentric, and the center of rotation of the stages is well defined by using the vertical microscope for accurate sample alignment. With the use of horizontal and vertical microscopes, the region of interesting of the sample was positioned at the center of beam and simultaneously at the center of rotation. Further, a Ge detector, which produces a fluorescence spectrum, was used for the more precise determination of the center of a fiber/matrix interface (within a ~5 μm range).

DISCLAIMER

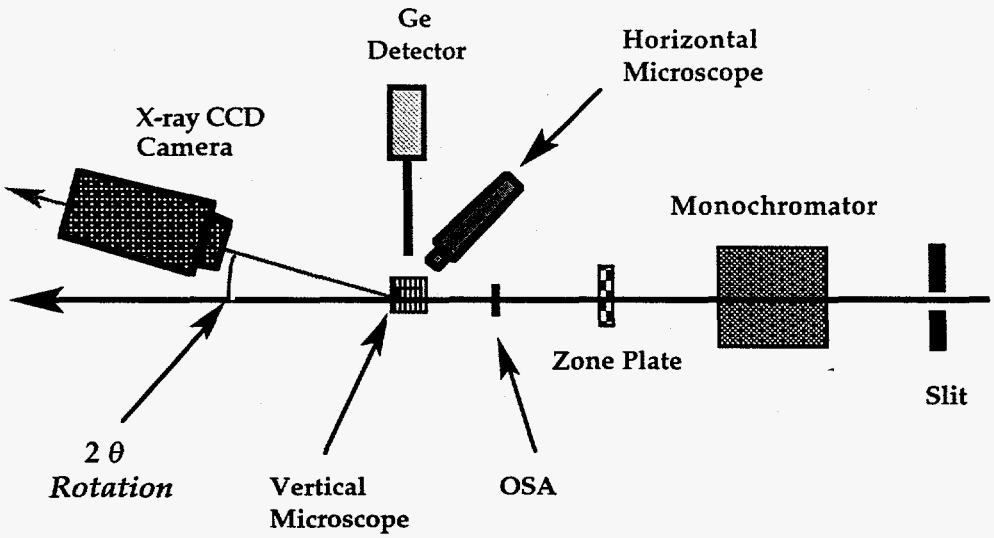

FIGURE 1. Schematic of experimental setup: The x-ray (11 keV) beam is selected by a double-crystal monochromator and focused by a zone plate that has a 54 cm focal length at 11 keV. An order sorting aperture (OSA) selects only the first-order focused beam and then produces a focal spot size of ~1 μm.

The sample for residual strain measurement was a fiber-reinforced composite (SCS-6/Ti-14Al-21Nb) (4). The composite was fabricated by hot isostatic pressing consolidation of an intermetallic Ti-14Al-21Nb (wt%) alloy with silicon carbide fibers of 140 μm diameter. Controlled cooling was done at less than 2°C/minute from the hold temperature of 950°C, and then the composite was cooled to ambient at ~20°C per minute under a pressure of 205 MPa. The sample, as shown in figure 2, was positioned on the sample stage in order to make the orientation of silicon carbide fibers parallel to the beam direction.

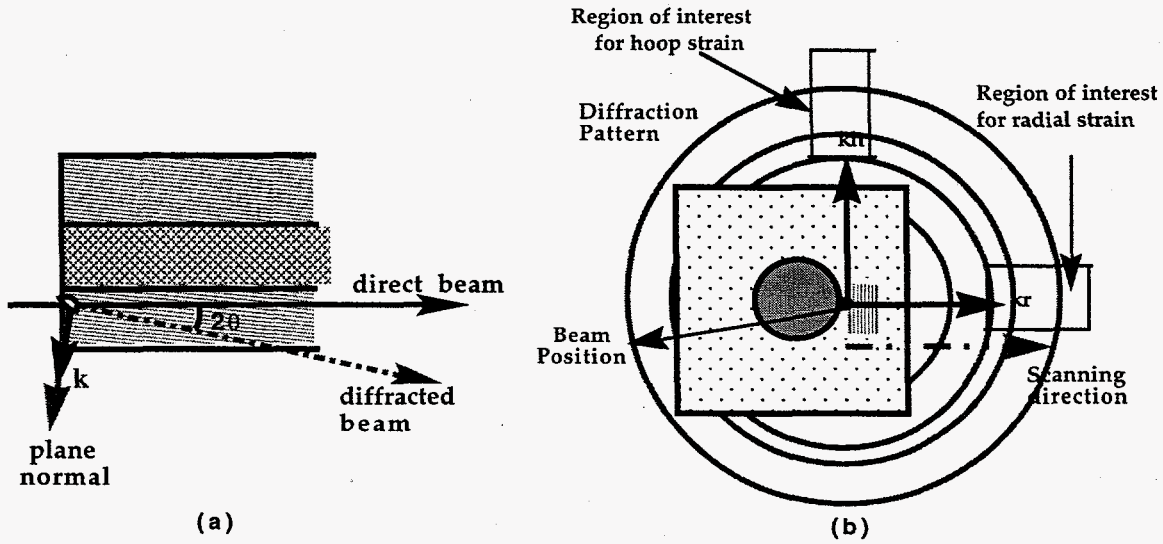

FIGURE 2. Orientation of sample and scanning direction: The sample is oriented parallel to the direction of the beam. For measurement of the radial strain gradient, the sample was scanned in the horizontal direction with a 10 μm step size from the center of the fiber/matrix interface. (a) is the side view of the sample orientation. (b) is the front view of the sample orientation. The experiment was perform by horizontal scanning of the sample to measure the gradient of radial strain.

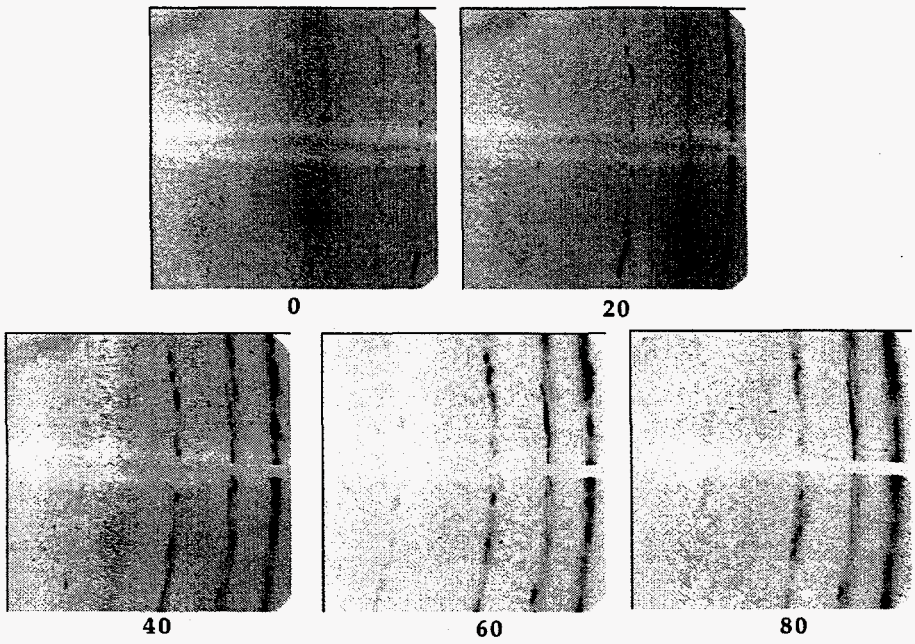

FIGURE 3. Examples of diffraction patterns: Diffraction patterns were taken at every 10 μm movement of the sample from the center of the interface by the CCD camera, which was located at 25 degrees in 2θ and at 282 mm distance from the sample.

For the measurement of strain gradient, the sample was scanned in the horizontal direction with a 10 μm step size from the center of a fiber/matrix interface. Diffraction patterns of the composite were recorded on the cryogenically cooled CCD camera. The CCD camera, which has a 1024 x 1024 pixel format with pixel dimensions of 19 x 19 μm^2, is coupled with a tapered-fiber glass that has a demagnification of 3. The CCD camera was located at 25 degrees in 2θ and at 282 mm distance from the center of rotation of the 2θ-stage (i.e., the sample), and hereby the sensitivity of strain measurement is ~3.85×10^{-4}/pixel. Acquisition time for each picture was 20 seconds.

RESULTS AND DISCUSSION

The pictures of diffraction patterns at different matrix positions are shown as examples in figure 3. For each picture, the relative pixel shift of diffraction was averaged and converted into the change in Bragg reflection (2θ=φ) by multiplying with a geometric conversion factor of 0.0105 degree/pixel. Thereafter, the sequential relative strain at a certain distance from the center of interface can be calculated by

$$\Delta d/d_o = -\Delta\phi/2 \cot\left(\phi/2\right), \qquad \Delta\phi = 0.0105 \times \Delta p \tag{1}$$

where Δp is a number of pixels and Δφ is a change in 2θ.

Figure 4 presents a radial residual strain profile in the Ti-14Al-21Nb matrix as a function of distance from the fiber/matrix interface. A solid line represents the strain calculated by the general form of the residual radial strain from the prediction of a simple cylinder elastic model (5), given by

$$\sigma_{radial} = A\left[1 - b^2/r^2\right], \tag{2}$$

where b is the radius of the matrix sleeve, r is a distance in the radial direction, and A is a constant determined by the characterization of a sample. The radial residual strain developed in the matrix was measured from diffraction patterns with a sensitivity of ~10^{-4} and an average standard deviation of 9.4 x 10^{-5}. Reasonable agreement between measured and calculated strains is shown, but the slight conflict, close to the interface, might be caused by relaxation, by plasticity, and (or) by radial cracking and spalling upon sample cooling (4,6).

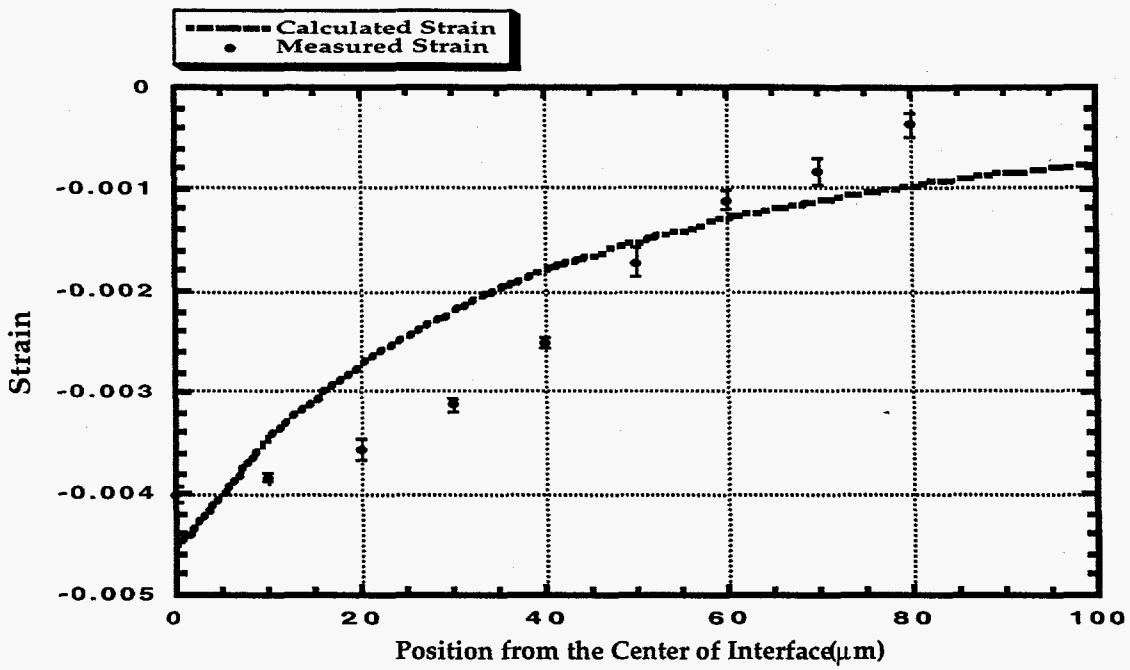

FIGURE 4. Profile of radial residual strain as a function of distance from the fiber/matrix interface: The measured radial residual strains (i.e., solid dots) in Ti-14Al-21Nb matrix have reasonable agreement with the calculated values (i.e., dotted line)

CONCLUSION

The XDM at the 2-ID-D beamline is a straightforward experimental setup, which has the capability to acquire diffraction patterns quickly with a highly brilliant monochromatic x-ray beam. In addition, the microfocusing method using a zone plate enables the XDM to produce a high spatial resolution of ~1 μm in measurements. The XDM allows one to quickly measure and analyze diffraction patterns to study lattice structures and understand physical phenomena inside materials.

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