

# High-Energy X-ray Diffraction Study of Internal Stresses in Metal Matrix Composites

A. Wanner,<sup>1</sup> D. C. Dunand<sup>2</sup>

<sup>1</sup> Institut für Metallkunde, Universität Stuttgart, Stuttgart, Germany

<sup>2</sup> Department of Materials Science and Engineering, Northwestern University, Evanston, IL, U.S.A.

## Introduction

The load-bearing capacity of a composite is controlled by the load transfer occurring from the soft matrix to the rigid reinforcements. Experimental measurement of load partitioning between the individual phases of composites can give a wealth of information on the micromechanical scenario going on in composites during loading and deformation. The classical method to characterize this load transfer in the bulk of crystalline materials is to measure the lattice strains via diffraction of thermal neutrons. Neutron diffraction techniques, however, require long data-collection times, making them unsuitable for stress changes occurring on times scales of minutes or seconds, which are typical for many thermo-mechanical situations of fundamental and practical interest. The high-energy, high-flux x-ray beams provided by the Advanced Photon Source offer the opportunity to perform internal stress measurements in the bulk of metallic materials with much shorter resolution times. In a series of experiments performed at beamline 5-BM we have improved a novel high-energy x-ray transmission technique introduced by Withers and co-workers<sup>1,2</sup> and applied it to study the internal load transfer and damage in metal matrix composites during plastic deformation.

## Methods and Materials

Dogbone-shaped, 1.5-mm-thick tensile specimens of molybdenum particle reinforced copper matrix composites were irradiated with a monochromatic beam of 65 keV photons and loaded

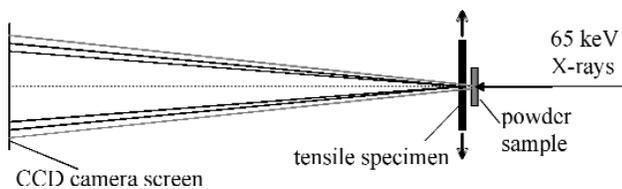


FIG. 1. Sketch of the experimental arrangement.

mechanically *in situ*. The experimental setup is sketched in Fig. 1. Low-index diffraction rings from matrix and reinforcement were recorded using a high-resolution, two-dimensional detector (CCD camera) at exposure times of several minutes (as opposed to several hours typical for neutron experiments). Upon mechanical loading, these rings are slightly distorted due to the lattice strains caused by internal and external stresses. Lattice strain measurements with this technique are, however, made difficult by two effects: (1) a particularly strong sensitivity to unintentional movement of the specimen under investigation, and (2) the graininess of the diffraction rings caused by the relatively small diffracting volume. These issues were investigated in detail and strategies to eliminate or minimize the related measurement errors were developed.

## Results

The effects of specimen movement can largely be eliminated by taking additional diffraction rings into account stemming from a calibration substance (Fe powder) attached to the specimen. The effects of diffraction ring graininess can be minimized by applying a newly developed, sophisticated ellipse fitting procedure<sup>3,4</sup> illustrated in Figs. 2 and 3. The improved measurement technique

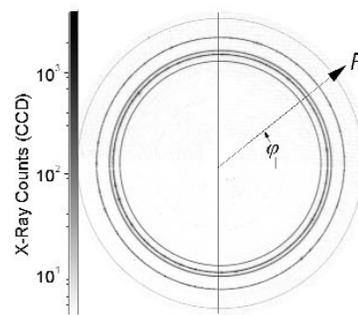


FIG. 2. (a) Concentric diffraction rings stemming from the two components of the composite (Mo and Cu) and the calibration substance (Fe powder).

was applied to study the evolution of elastic phase strains in the copper matrix composites reinforced during uniaxial tensile deformation. The lattice strains in the molybdenum phase were used to estimate the load fraction carried by the reinforcements, which was found to vary significantly in the course of the deformation of the composite. At small applied strains, load transfer from the yielding matrix to the elastic reinforcements was observed. At larger strains (i.e., at about 1-2% total deformation), the load fraction carried by the reinforcements reached a maximum and then decreased continuously (Fig. 4). This reversal of load transfer is attributed to the disintegration of the reinforcement particulates, which consist of fine, sintered molybdenum particles infiltrated by copper.

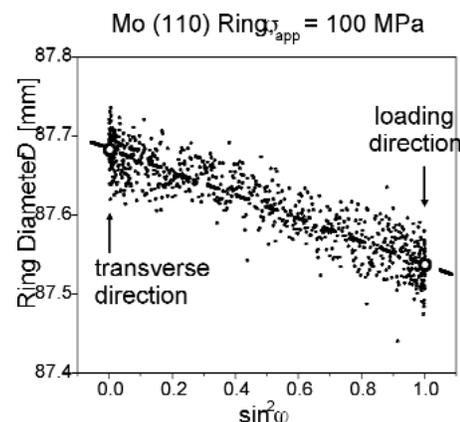


FIG. 3. The minute elliptical distortion of a diffraction ring is evaluated by plotting the ring diameter  $D$  against sine square of the azimuthal angle  $\varphi$ .

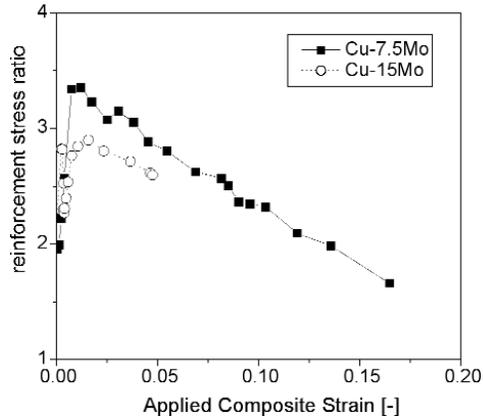


FIG. 4. Plot of stress in the reinforcement phase normalized by the applied stress vs. the applied strain for uniaxial tensile tests on copper reinforced with 7.5 or 15 vol% of molybdenum particulates.

## Discussion

This study emphasizes the fact that high-energy synchrotron x-ray beams are a powerful tool for studying the mechanics of metal matrix composites. This technique has the potential for resolving the time dependency of the load transfer occurring in composites at elevated temperatures under application-like conditions. However, the technique is currently limited to materials with grain sizes  $< 10 \mu\text{m}$ . This limitation can only be addressed by increasing the probe volume substantially. Strategies to accomplish this goal are under development<sup>5</sup> and will be explored in future experiments.

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