

Methodological Aspects of the High-energy Synchrotron X-ray Diffraction Technique for Internal Stress Evaluation

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The availability of high-energy X-rays from 3rd-generation synchrotron sources offers new approaches to study internal stresses in the bulk of crystalline materials. A method with a wide application potential is the transmission technique introduced by Daymond and Withers, which however has so far been limited to fine-grained materials. In the present work, the reasons for the currently existing limitations are discussed and a viable strategy to extend the technique to samples with coarser grain sizes is proposed.

Keywords: Internal stress measurement; X-ray diffraction; Monochromatic high-energy X-rays; Probe volume; Grain size

INTRODUCTION

Classical techniques to measure volume-averaged elastic strains in the bulk of crystalline metallic or ceramic materials are based on diffraction of thermal neutrons. While these techniques have been successful for many applications, typical measurement times are long (on the order of hours) due to the low fluxes available at current neutron sources and due to the inherently weak interaction between the neutrons and the specimen material. The availability of high-energy,

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high-flux X-rays from 3rd generation synchrotron research facilities offers alternative approaches to diffraction measurements of internal strains, which do not have these restrictions and are therefore complementary tools to neutron diffraction. An interesting method with a wide application potential is the high-energy X-ray transmission technique first described by Withers and co-workers [1–3]. We have applied and further developed this technique to study the internal load transfer in particle-reinforced copper matrix composites. For details on these investigations we refer to two papers recently published elsewhere [4,5]. The purpose of the present article is to briefly review the current state and the limitations of the technique and to develop guidelines for further improvement.

METHODOLOGY AND LIMITATIONS OF THE TECHNIQUE

The technique is especially suited for characterizing two-dimensional, volume average strains in the bulk of specimens with planar, parallel surfaces (such as, e.g. sheet material or flat dog-bone tensile specimens). A typical experimental setup is schematically shown in Fig. 1. The specimen is irradiated with a parallel, monochromatic beam of high-energy photons. In order for the photons to penetrate several millimeters of materials of technical interest (such as Al-, Fe-, Ni-, or Cu-based alloys), the photon energy must be at least 60 keV, corresponding to a wavelength of about 0.2 \AA . The diffraction geometry is similar to that of a transmission electron microscope in the sense that the wavelength of the high-energy X-rays is considerably smaller than the typical spacings of low-index lattice planes, which, in diffraction condition are thus almost parallel to the incident beam. By evaluating the distortions of the ring-like diffraction patterns recorded on a plane normal to the incident beam, the bulk average strains in the plane perpendicular to the photon beam are obtained. In all experiments described so far in the literature [1–5], complete ring-like diffraction patterns from low-index Bragg reflections were recorded using a high-resolution two-dimensional detector (CCD camera). The maximum allowable distance between the specimen and the detector (camera distance) is dictated by the largest Bragg

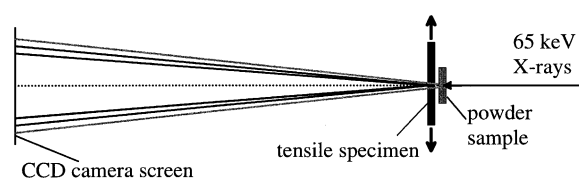


FIGURE 1 Schematic view of the experimental setup for lattice strain measurements using monochromatic, high-energy X-rays.

angle of interest and by the size of the detector. So far, 2D-detectors with screen diameters in the range of 10–20 cm were used and the camera distances lied typically in the range from 0.5 to 1.0 m. In the limited number of studies performed to date, this method has been shown to be applicable to the study of load transfer in metal matrix composites and to the 2D-mapping of local strain variations within the specimen under investigation. As compared to typical neutron diffraction measurements, the X-ray exposure times were at least one order of magnitude shorter although the diffracting volume was typically two to three orders of magnitude smaller. However, due to its novelty and some inherent limitations, this synchrotron X-ray technique has not yet become a general alternative to neutron diffraction measurements. According to our experience, two major issues must be considered for further method development:

Probe Volume and Grain Statistics

The width of a diffraction ring on the detector screen is controlled by the spot diameter of the incident beam and by the specimen thickness. To avoid overlap of neighboring rings and to keep the width-to-diameter ratio of the rings small enough allowing the measurement of ring distortions with sufficient accuracy, the beam must be kept to a diameter considerably lower than 1 mm. In the studies mentioned above, beam diameters of about 200 μm were used, resulting in probe volumes less than 0.1 mm^3 . While such a small probe size is advantageous for high spatial-resolution strain mapping applications, it also limits the technique to fairly fine-grained materials. Only for very fine-grained materials are the diffraction patterns composed of continuous rings. In practice, however, the number of crystallites in diffracting condition is limited and the diffraction rings are grainy, giving rise to poor strain resolution unless sophisticated averaging techniques are applied [2–5]. For a beam diameter of 200 μm , lattice strain measurements accuracies similar to those obtained by standard neutron techniques can, according to our experience, only be performed on materials with grain sizes less than about 10 μm . As many metallic and ceramic materials of technical interest exhibit somewhat larger grain sizes, the scope of the technique would widen substantially if the probe volume could be increased accordingly. This trend is opposite to that occurring in the neutron diffraction community, which is devoting much effort to reducing the probe volume (with a concomitant increase in spatial resolution and measurement time). A viable strategy would be to enlarge the whole experimental setup—including the beam diameter, the camera distance and the diameter of the detector screen, but

TABLE I Scaling considerations

	<i>Scaling factor for enlargement of the setup by a factor of α</i>	<i>Example: Dimensional changes for $\alpha = 10$, starting from setup described in Ref. [4]</i>
Required lateral resolution of the 2D-detector (pixel size)	α^{-1}	64 \rightarrow 640 μm
Diffraction ring intensity on screen	α^0	–
Spot diameter	α^1	200 $\mu\text{m} \rightarrow$ 2 mm
Width of diffraction ring		200 $\mu\text{m} \rightarrow$ 2 mm
Specimen-to-detector distance		65 cm \rightarrow 6.5 m
Diameter of diffraction ring		~ 10 cm $\rightarrow \sim 1$ m
Diameter of 2D-detector		13 cm \rightarrow 1.3 m
Tolerable grain size of the polycrystalline specimen under investigation	$\alpha^{2/3}$	~ 10 $\mu\text{m} \rightarrow \sim 50$ μm
Spot area	α^2	0.04 mm ² \rightarrow 4 mm ²
Probe volume		0.06 mm ³ \rightarrow 6 mm ³
Area of diffraction ring		~ 63 mm ² $\rightarrow \sim 63$ cm ²

excluding the specimen thickness—by one order of magnitude. The scaling considerations for the corresponding quantities of interest are summarized in Table I. As the probe volume scales with the square of the beam diameter and the number of grains in that volume is inversely proportional to the cube of the grain size, the tolerable grain size could be increased by a factor of $10^{2/3} \approx 5$. In other words, provided the ring diameter and width are increased by a factor of ten, the strain evaluation in a sample with 50 μm grain size would be as accurate as that for a 10 μm sample using the established, smaller-size setup. In preliminary investigations, we have studied the effect of spot size on the structure of the diffraction patterns. The two images shown in Fig. 2(a) and (b) show sections of the (111) diffraction ring from a fine-grain copper sample recorded at the same camera distance of 0.5 m but with beam sizes differing by a factor of 4. It is apparent that, by increasing the spot size, the diffraction ring becomes much smoother, thus increasing the accuracy of the strain measurement. However, the ring also becomes thicker, having the opposite effect, unless the diameter of the diffraction ring is enlarged accordingly by increasing the camera distance. Due to the very small beam divergence from the highly parallel primary beam, the ring thickness is dictated by the beam size and sample thickness, but is unaffected by the camera distance. A practical limitation is the size of the experimental hutch. A camera distance of up to about 10 m and a detector area of up to 2×2 m appears

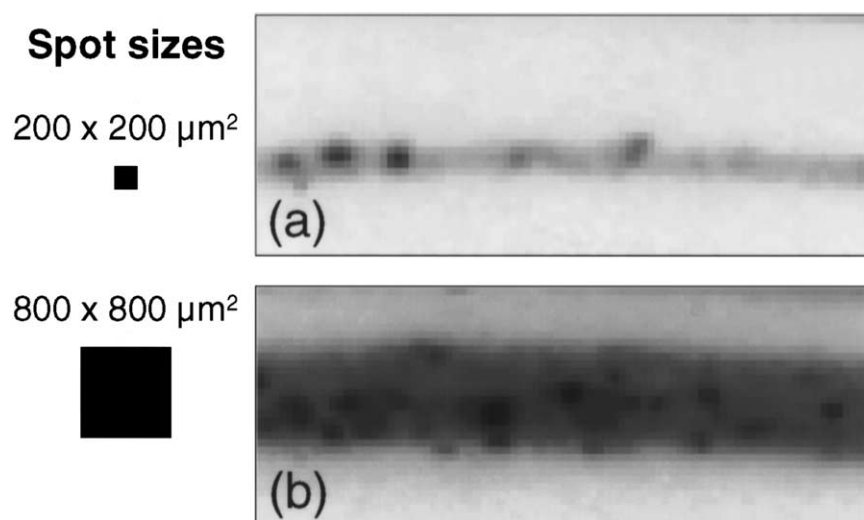


FIGURE 2 Effect of the spot size of the incident X-ray beam on the structure of a diffraction ring.

still feasible as there already exist some experimental hutches with such dimensions at current research facilities. Currently not available, however, are area detectors with sizes approaching the required dimensions. There exist no major engineering obstacles to constructing much larger area detectors than currently available, given that the pixel size could be increased accordingly. For the time being, however, it appears more feasible to use an array of several smaller area detectors mounted on a rigid frame. A possible arrangement of four detectors is shown schematically in Fig. 3. Alternatively, a single detector could be moved to different places to provide sequential measurements, with the limitation that the measurement time is increased and positioning inaccuracies are problematic. The kind of setup shown in Fig. 3 would be ideal to study the longitudinal and transverse lattice strains during an *in situ* uniaxial loading experiment of the kind described in Refs. [1,4,5].

Sensitivity to Unintentional Specimen Movement

At present, the high-energy X-ray transmission technique is particularly sensitive to unintentional specimen movements during mechanical or thermal loadings. This sensitivity also complicates potential averaging techniques, where the specimen would be moved perpendicular to the beam to increase the effective

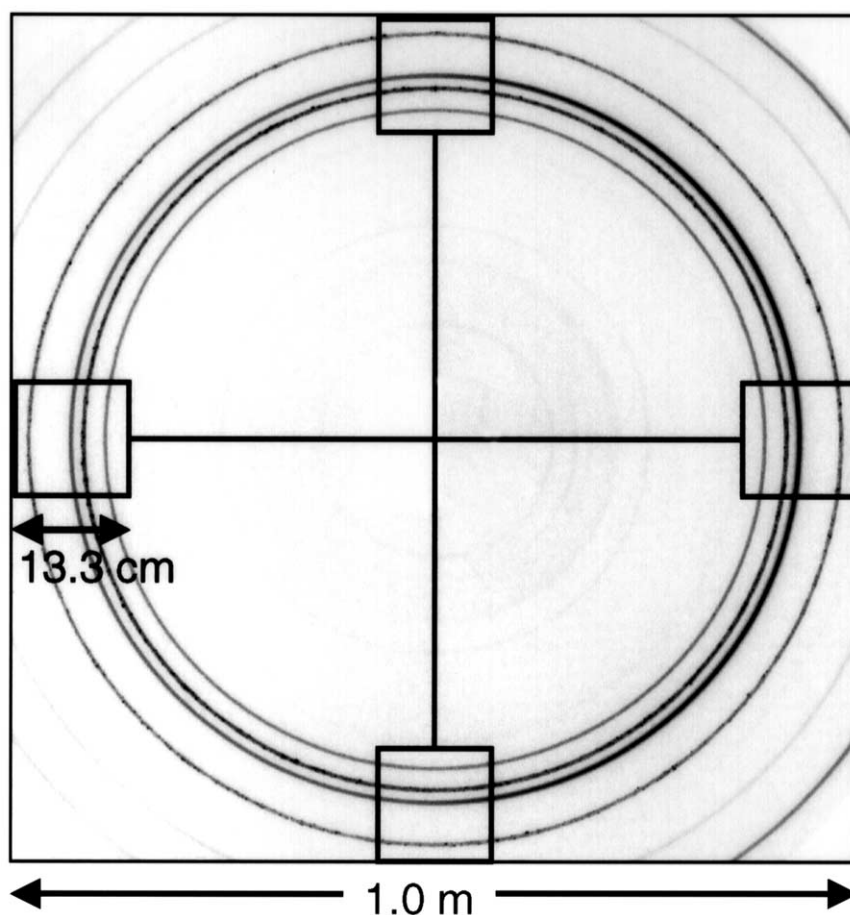


FIGURE 3 In order to increase the probe volume we propose to enlarge the experimental setup by one order of magnitude. As the typical sizes of low-index diffraction rings become too large to fit on the screen of a standard 2D-detector, an array of several detectors must be used in stead. Four detectors arranged in the way shown here would be ideal to study the longitudinal and transverse lattice strains during *in situ* uniaxial loading experiments.

probe volume. If they are uncorrected, these specimen displacements lead to errors in lattice strains, which scale with the ratio of specimen movement to camera distance. Our recent studies have shown that, by using a calibration substance in powder form rigidly attached to the specimen, such errors can largely be eliminated. This strategy is, however, not always practical. Major problems arise, for example, in high-temperature experiments, where it is more difficult to contain and affix the powder and to keep it at the same temperature as the specimen. This problem becomes even worse if varying temperatures come

into play. Hence, enlarging the setup would also reduce the current strong sensitivity to specimen positioning. For a camera distance of 10 m, attaching a calibration powder for correction purposes would become unnecessary provided that, unintentional specimen movements can be held in the limits of about ± 0.5 mm.

CONCLUSIONS

Monochromatic high-energy X-rays can be used to measure internal stresses in the bulk of crystalline materials with higher speed and higher spatial resolution than established neutron diffraction techniques.

Currently, this high-energy X-ray diffraction technique is limited to very fine-grained materials and suffers from a strong sensitivity to unintentional specimen displacements.

These limitations can be addressed by increasing the probe volume substantially. Simple considerations show that this could be accomplished by enlarging the diameter of the incident beam and the camera distance (and thus the detector size) by one order of magnitude.

Acknowledgements

Part of this work has been supported by the Deutsche Forschungsgemeinschaft (DFG) through grant SFB381/A1. The diffraction experiments were performed at the DND-CAT Synchrotron Research Center located at the Advanced Photon Source (APS), whose staff we acknowledge for their excellent technical support. DND-CAT is supported by the E.I. DuPont de Nemours and Co., The Dow Chemical Company, the U.S. National Science Foundation through grant DMR-9304725, and the State of Illinois through grant IBHE HECA NWU 96. Use of the APS was supported by the U.S. Department of Energy under contract no. W-31-102-Eng-38.

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