

Nondestructive Strain Tensor Scanning within Samples of Cylindrical Symmetry

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Introduction

Recently, high-energy synchrotron radiation has been employed for strain scanning within polycrystalline bulk materials, a field so far dominated by neutron diffraction (see, e.g., Ref. 1). The unique features pertaining to high-energy synchrotron radiation are as follows: (a) micrometer-sized, intense beams that can be prepared by focusing [2], (b) small scattering angles, and (c) penetration power that is strongly dependent on the atomic number of the sample elements. Thus, it becomes feasible to map strain fields with unprecedented narrow spatial resolution. This report deals with the measurement of “macro” strain (i.e., averages over a sufficiently large number of grains with parallel reciprocal lattice vectors). Therefore, the gauge volume must encompass an even larger number of grains, since only a fraction of the illuminated grains will be in diffraction position. An insufficiently small number of grains within the gauge volume will result in two problems with different natures. First, a systematic experimental error may arise if the diffracting grains are not distributed homogeneously throughout the gauge volume (in the extreme case, there may be no diffracting grain). Second, the interpretation of the measured strain may be biased by intergranular contributions. It is therefore of great interest to experimentally average over symmetry equivalent sample volumes. A large area of applications is 1-D strain gradients, such as the shot-peened cylindrical samples investigated here, where the strain state depends on only the radial distance from the sample surface. High spatial resolution is required parallel to the strain gradient, but experimental averaging should take place over planes perpendicular to the gradient.

Methods and Materials

Triangulation refers to defining a gauge length parallel to the incident x-ray beam by inserting a slit into the diffracted beam (see Fig. 1). The direction of the diffracted beam is defined by its position on the 2-D detector and by the fixed slit position. Therefore, an intersection volume is defined with the incident beam. A detailed discussion of the geometrical implications was published [3]. The gauge length depends on the width of the incident beam, the slit aperture, and the scattering angle. Because of the small scattering angles, the gauge length Δ_{\parallel} is typically 10 times the slit aperture.

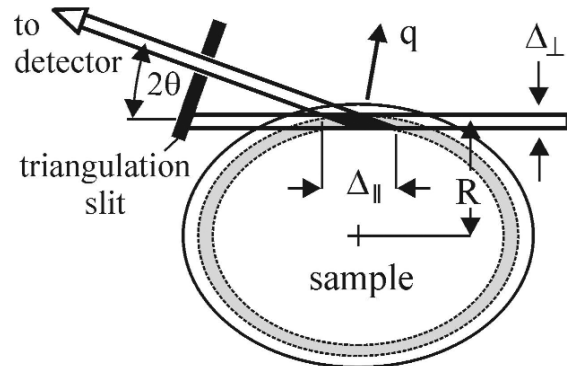


FIG. 1. Definition of a longitudinal gauge length Δ_{\parallel} by triangulation of the diffracted beam. The intersection of the scattering plane with the cylindrical sample becomes an ellipse if the cylinder axis is not perpendicular to the scattering plane.

The determination of the strain tensor components requires peak shift measurements in at least six independent directions. In the present case, where the diffraction plane was fixed as defined by the triangulation slit, the sample needs to be oriented in at least six directions. At all sample orientations, a narrow beam dimension must be parallel to the direction of the strain gradient to preserve spatial resolution. This requires that the incident beam be confined in two dimensions, resulting in a narrow spot. The spot diameter defines the best achievable spatial resolution. For the cylindrical sample geometry investigated here, the scattering vector can be made parallel to both the radial and axial directions. However, the hoop direction can be approached only as it results in extensive path length within the sample and therefore prohibitive attenuation.

The largest gauge volume compatible with the above-described constraints is cigar shaped, with one long dimension and two short dimensions. This matches the elongated gauge volumes due to the small scattering angles. The effective number of grains within this prescribed gauge volume can be increased by several orders of magnitude by a 2-D sample oscillation. In the case of a cylindrical sample geometry, the strain state should depend only on the radial position within the sample, and the integration

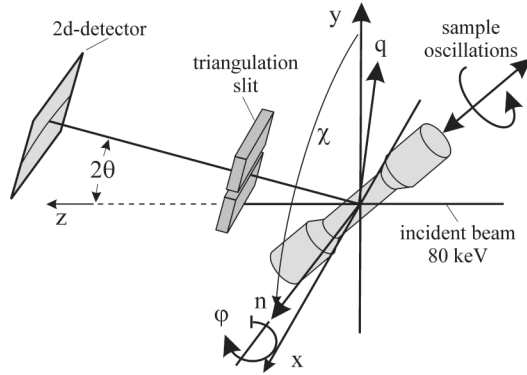


FIG. 2. Sketch of the experimental setup. n indicates the surface normal (radial direction) for the actual gauge volume, and χ and ϕ define the sample orientation.

surfaces are therefore given by hollow, coaxial cylinders. These surfaces can be densely covered by rotating the sample around the cylinder axis and translating the sample parallel to the axis with appropriate speed. Keeping the illuminated gauge volume narrow and oscillating the sample dramatically increases the effective density of diffracting grains. Errors due to an inhomogeneous coverage of the gauge volume are therefore suppressed more efficiently than by increasing the illuminated gauge volume [4].

The samples were of cylindrical geometry, with a 1-in.-long by 1/4-in.-diameter central region that was shot-peened and 1/2-in.-diameter end pieces. The front sides had blind center holes from the lathe machining. Here we report on a steel sample of proprietary composition. The samples were mounted in a custom-designed rotation device and gripped by the center holes. The eccentricity of the shot-peened region was found to be below 20 μm . The rotation device was mounted on a xyz translation stage within a Huber three-circle diffractometer. One of the translations was aligned parallel to the sample axis and used for sample oscillation.

The experiments were performed at APS beamline station 1-ID-C, and the setup is sketched in Fig. 2. The energy of the incident x-ray beam was 80.7 keV, and the lateral beam size was confined by slits to $30 \times 30 \mu\text{m}$. A 50- μm -wide open triangulation slit was placed 33 mm behind the sample. A 2-D detector (Princeton charge-coupled device [CCD]) with 1242×1154 pixels 22.5 μm in size was installed 687 mm behind the slit. A short sample-to-slit distance and a large slit-to-detector distance minimize geometrical artifacts of the triangulation technique, as described previously [3].

The data presented here were collected for the [321] reflection at a scattering angle of $2\theta = 11.5^\circ$. Sixteen different sample orientations were measured,

incrementing χ in 10° steps from 0 to 90° at $\phi = 0^\circ$, and incrementing ϕ from 0 to 45° in steps of 7.5° at $\phi = 90^\circ$. At $\chi = 90^\circ$ and $\phi = \theta$, the scattering vector is parallel to the sample axis, and the sample axis is perpendicular to the vertical diffraction plane at $\chi = \phi = 0^\circ$ (compare with Fig. 2). At each sample orientation, depth profiles were measured. For each depth below the sample surface, the 2-D detector was exposed for 15 seconds while the sample was oscillated at speeds of 2 turns per second and 4 mm per 15 seconds.

Results

The peak intensities as a function of depth are smooth curves, indicating successful grain averaging even in the bulk where the grain size is about 100 μm .

The atomic lattice spacings as a function of the sample orientation (χ, ϕ) were evaluated from the 2θ center positions of the diffraction peaks by applying Bragg's law. Lattice strains were calculated by using the bulk lattice spacing, which is available because of the employed oscillation strategy, as a strain-free reference. The geometrical coefficients for least-square fitting of the strain-tensor components to the experimentally available directional lattice strains were taken from Ref. 5. One shear component, ϵ_{13} , had to be constrained to zero for the least-square fitting. The remaining five depth-dependent strain tensor components are plotted in Fig. 3. The depth profiles show the qualitative features that are known from the analysis of residual strain fields resulting from shot-peening, namely: (a) in-plane compression and radial tension near the surface; (b) formation of a well-defined, plastically deformed surface layer (note the steep strain gradients between 200 and 400 μm below the surface); and (c) small shear components. The experimental finding of negligible shear components ϵ_{12} and ϵ_{23} justifies the assumption on ϵ_{13} . The existence of a plastically deformed surface layer also shows as increased width of the diffraction peaks. The extensive

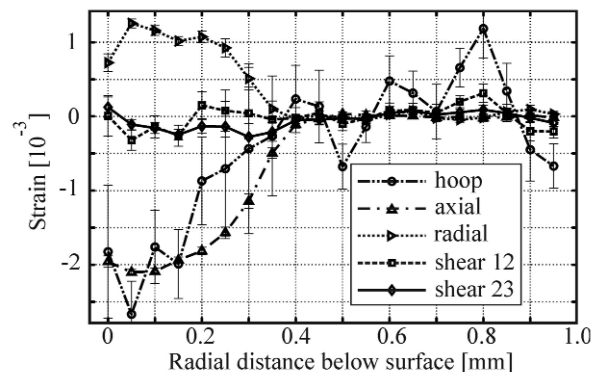


FIG. 3. Strain tensor components extracted from the experimental data while it was assumed that the shear 13 component is zero.

scatter and error bars for the hoop component result from the lack of measurement of appropriate sample orientations.

Discussion

We have presented a strain-scanning technique that enables measurement of the complete strain tensor within the bulk of components over almost arbitrary gauge volumes. The technique has been applied to shot-peened steel samples with cylindrical cross sections. By rotating the sample around and translating it parallel to the cylinder axis, gauge volumes of the shapes of the hollow cylinders have been produced, which are ideally matched to the sample symmetry. The depth resolution is given by the wall thickness of the hollow cylinders, which, in turn, is given by the beam diameter. A depth resolution of 30 μm has been achieved in the present study. Grain averaging has been attained even within the large grained interior of the sample.

Acknowledgments

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References

- [1] P.J. Withers, M. Preuss, P.J. Webster, D.J. Hughes, and A.M. Korsunsky, *Mater. Sci. Forum* **404-407**, 1-12 (2002).
- [2] U. Lienert, C. Schulze, V. Honkimaki, T. Tschentscher, S. Garbe, O. Hignette, A. Horsewell, M. Lingham, H.F. Poulsen, N.B. Thomsen, and E. Ziegler, *J. Synchrotron Radiat.* **5**, 226-231 (1998).
- [3] U. Lienert, R. Martins, S. Grigull, M. Pinkerton, H.F. Poulsen, and A. Kvik, *Mater. Res. Soc. Symp. Proc.* **590**, 241-246 (2000).
- [4] A. Wanner and D.C. Dunand, *J. Neutron Res.* **9**, 495 (2001).
- [5] B.B. He and K.L. Smith, *Proceedings of the Society for Experimental Mechanics Spring Conference on Experimental Applied Mechanics* (Houston, Texas, 1-3 June 1998), p. 217.
- [6] S.F. Nielsen, A. Wolf, H.F. Poulsen, M. Ohler, U. Lienert, and R.A. Owen, *J. Synchrotron Radiat.* **7**, 103-109 (2000).