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Gradient residual strain and stress distributions in a high pressure torsion deformed iron disk revealed by high energy X-ray diffraction

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ABSTRACT

High energy X-ray diffraction is used to investigate for the first time the distribution of residual X-ray elastic stresses inside a high pressure torsion (HPT) deformed iron disk with a diameter and thickness of ~30 and ~8 mm, respectively. In the experiment, a dedicated conical slit system restricts the diffraction gauge volume in three dimensions to ~0.45 mm^3, which is then used to scan the bulk sample cross-section in a non-invasive manner. Pronounced residuals stress gradients with maximal tensile stresses of ~200 MPa are observed along radial and tangential directions and are correlated to the deformation gradient arising from HPT.

High pressure torsion (HPT) synthesis has opened up the opportunity to obtain deeper insights into the microstructure-dependent behavior of bulk nanomaterials, that are difficult or impossible to produce by other processing techniques [1–3]. In recent years the size of HPT samples has continuously increased and several promising industrial spin-offs have emerged [4–6]. Even though many aspects of microstructure and resulting properties of nanomaterials processed by HPT have been scrutinized [1], the question on the magnitude and gradients of residual stresses in HPT samples has remained fully open. This topic, however, is of great practical importance, due to the impact of stresses on overall mechanical stability and properties of HPT disks. For example, since tensile residual stresses are known to have a negative influence on fatigue behavior [7,8] and stress corrosion cracking [9], their quantification is of paramount importance for possible future application fields of HPT-processed materials.

High energy synchrotron X-ray diffraction (XRD) using photon energies of more than ~50 keV has been routinely used to characterize phases, microstructure and residual stresses in bulk metallic samples. The evaluation of these properties from the experimental data is comparable to the case of the authors' previous work, where a very similar transmission XRD setup was used, although with much smaller samples and lower beam energies [10–15]. Due to the high penetration depths in mm or even cm ranges, it is possible to access volume-representative material properties from sample regions deep under the surface. In the case of samples with gradients of microstructure and/or stresses, however, the elongated gauge volume along the beam path results in volume-averaging of the diffraction information and this effect makes it very challenging to resolve sample inhomogeneities. In order to overcome this restriction, a detection system based on the application of conical slits, placed between sample and two dimensional (2D) detector, has been developed and already successfully applied in several studies [13–17]. The conical slits are machined into a tungsten plate and absorb most of the diffracting signal, but select a small portion of diffraction cone intensities from a gauge volume length of ~0.5–2 mm (along the beam direction), which are then recorded by the detector. By moving the sample in the focus of the conical slits, it is possible to perform scanning diffraction experiments and, in this way, to resolve inhomogeneous sample properties.

In this work, position-resolved high energy synchrotron X-ray diffraction is used to quantify residual stress distributions in an iron disk with a diameter of ~30 mm produced by HPT. The aim is to compare quantitatively stress states at the sample surface and in the center.

For the synchrotron XRD experiment, two disks of pure iron (Armco) with a diameter of 30 mm and an initial thickness of ~11 mm were used. A compositional analysis of the raw sample material yields 0.009 wt% of C, 0.060 wt% of Mn, 0.009 wt% of P, 0.007 wt% of S and the balance fraction of Fe. The two disks were recrystallized at 800 °C for 1 h prior the HPT experiments at room temperature, which applied 1 rotation, corresponding to an equivalent von Mises strain of

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~7.4 at a disk radius of ~15 mm. The process was conducted at a nominal pressure of 2.8 GPa and a deformation speed of 0.07 rotations/min. The anvil geometry is shown in Fig. 1a, where the most significant features are the depth of the depressions with 3.5 mm and the opening angle of the depression of ~40°. This angle is significant, as it allows an easy removal of the sample after the HPT and partly controls the axial homogeneity of the sample [18]. Images of the disks before and after deformation are presented in Fig. 1b (left and center). After deformation, the disk does not possess a strict cylindrical shape (Fig. 1b left) and instead features inclined lateral surfaces (Fig. 1b center) because of the anvils' opening angle. From one HPT disk, thin rods with a diameter of ~1 mm were cut out from various axial and radial positions, as schematically shown in Fig. 1b right. The rods were used to determine unstressed lattice parameters at different disk cross-sectional positions. In this way it was possible to account for axial and radial variations of the materials' unstrained lattice parameter, due to varying defect densities arising from the radius-dependent applied strain.

Residual X-ray elastic strains and residual stresses in the uncut disk were characterized by XRD at the high energy materials science (HEMS) beamline P07 of PETRA III at DESY in Hamburg, which is operated by Helmholtz Zentrum Geesthacht. The disk was irradiated with a monochromatic beam of 87.1 keV photon energy and a cross-section of ~0.5 × 0.5 mm. Debye-Scherrer rings were recorded in transmission geometry using a Perkin Elmer two-dimensional (2D) flat panel detector of 2048 × 2048 pixels with a pixel size of 200 μm. Conical slits were placed between the disk and the detector in order to filter the α-Fe 200 and 211 reflections and to restrict the gauge volume in the direction parallel to the incident beam to a length of ~1.8 mm, resulting in a volume of ~0.45 mm^3 (Fig. 1c). The sample-detector distance was ~1.3 m and a dark current correction was applied to each exposure. A LaB_6 calibration powder was used to determine the exact geometry and distance of the detector with respect to the sample [19].

During the scanning experiments, the uncut disk was oriented with its axial direction parallel to the incident X-ray beam direction and was scanned axially (a) and radially (r) (Fig. 1c) with steps of 1.4 and 1 mm. Special care was taken to ascertain that the gauge volume was fully inside the sample at all times, so that edge effects would not influence the measured X-ray elastic strains. Additionally to the HPT disk, the reference sample rods (Fig. 1b) were scanned in order to obtain 2D distribution of unstressed lattice parameters $d_o(r,a)$ across the disk cross-

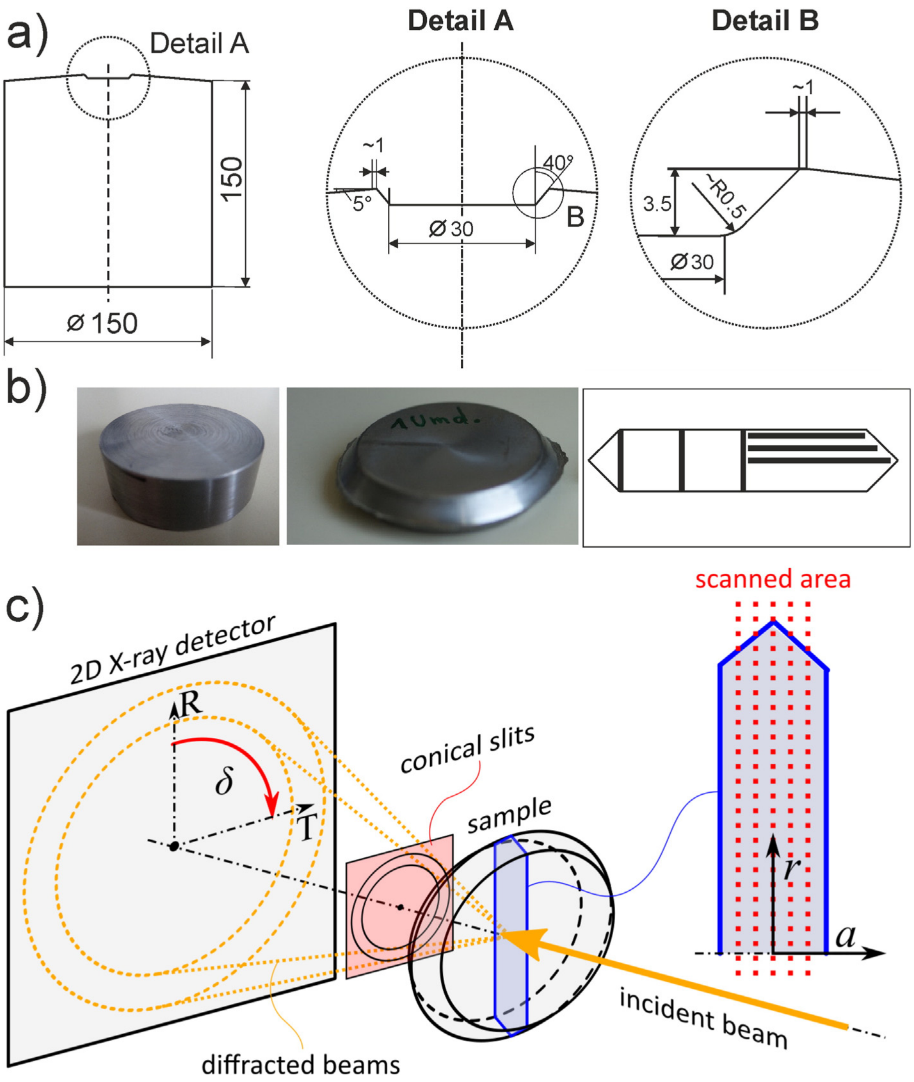

Fig. 1. (a) The geometry of the anvils used for the HPT processing. The depression has a diameter of 30 mm and a depth of 3.5 mm. The transition from the depression to the top of the anvil is realized with an opening angle of ~40°. (b) The geometry of the processed HPT disks before deformation (30 × 11 mm with a strict cylindrical shape, left) and after deformation (height reduction to ~8 mm, adopting the shape of the anvil depression, middle) and the cross-sectional positions of the extracted rods (right) used for determination of the unstressed lattice parameter. (c) A schematic drawing of the experimental setup used at the synchrotron beamline. The HPT disk was scanned relative to the gauge volume determined by the conical slits' focus in order to determine lattice parameters $d_R(r,a)$ and $d_T(r,a)$ within one half of the sample cross-section by fitting Debye-Scherrer ring positions $2\theta_R(r,a)$ and $2\theta_T(r,a)$ for δ values of 0° and 90°, respectively.