Original

Staron, P.; Fischer, T.; Keckes, J.; Schratter, S.; Hatzenbichler, T.; Schell, N.; Mueller, M.; Schreyer, A.:

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Materials Science Forum, International Conference on Residual Stresses 9 (2013)
Trans Tech Publications

DOI: 10.4028/www.scientific.net/MSF.768-769.72

Materials Science Forum *Online: 2013-09-23*
ISSN: 1662-9752, Vols. 768-769, pp 72-75
doi:10.4028/www.scientific.net/MSF.768-769.72

Depth-resolved residual stress analysis with high-energy synchrotron X-rays using a conical slit cell

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Keywords: high-energy X-rays; diffraction; conical slits; residual stress

Abstract. A conical slit cell for depth-resolved diffraction of high-energy X-rays was used for residual stress analysis at the high-energy materials science synchrotron beamline HEMS at PETRA III. With a conical slit width of 20 µm and beam cross-sections of 50 µm, a spatial resolution in beam direction of 0.8 mm was achieved. The setup was used for residual stress analysis in a drawn steel wire with 8.3 mm diameter. The residual stress results were in very good agreement with results of a FE simulation.

Introduction

Currently, the most important diffraction techniques for spatially resolved non-destructive residual stress analysis in the interior of a bulk sample are neutron diffraction [1] and high-energy synchrotron X-ray diffraction with a white photon beam [2,3], with each technique having its specific advantages. The advantage of X-rays from a synchrotron source is the high intensity enabling high spatial resolution or fast measurements. The possibility of fast measurements can be used either for producing large two and three-dimensional strain maps or for *in situ* analyses of processes.

In thin sheets, residual stresses can be studied with a monochromatic high-energy X-ray beam penetrating the whole sample, giving information about the residual stress distribution integrated over the thickness. For thicker samples, however, depth-resolved diffraction is required. Depth resolution can be achieved with a conical slit cell (CSC), which had been proposed several years ago [4]; however, this technique apparently has not found widespread application despite its advantages. The CSC has several concentric slits that are focussed on a spot within the sample by their conical shape [5]. With a CSC, complete diffraction rings can be measured with depth resolution, except for small regions where material is required for the mechanical stability of the slits. The analysis of full diffraction rings enables the simultaneous determination of all strain components in the plane. Moreover, also basic information on the texture of the material can be obtained. To achieve depth resolutions well below 1 mm, the slit width as well as the beam cross-section has to be around 20 µm [6]; moreover, the depth resolution also depends on the energy resolution given by the monochromator. Thus, a third-generation synchrotron source with high brilliance is required for the use of a CSC. So far, only few examples for the application of CSC for residual stress and texture analysis can be found in the literature [7, 8, 9].

A CSC is used for residual stress analysis at the beamline HEMS at PETRA III (DESY), run by the Helmholtz-Zentrum Geesthacht [10]. While depth resolutions of a few 100 micrometres can be achieved with narrow beams, the grain size of the studied material often prevents a conventional analysis of diffraction rings with such high resolutions; however, strategies for improving grain statistics can often be applied in such cases. However, in many cases, only moderate depth

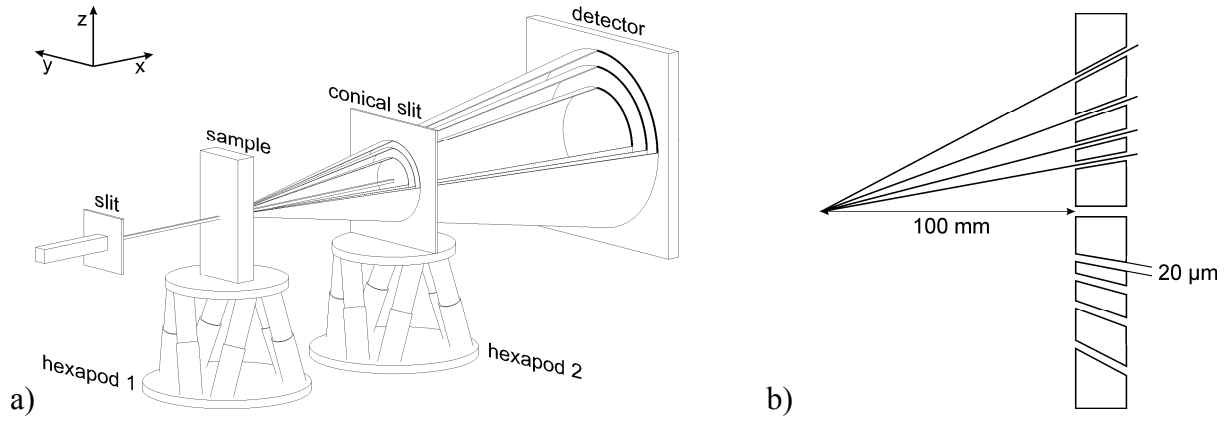

Fig. 1: Sketches of experimental setup (a) and conical slit geometry (b).

resolutions are required, and with a larger beam cross-section, the grain statistics can be improved. The obtained results show that with a beam size of 50 µm and an energy resolution of 0.7% the depth resolution is about 0.7 mm. After a discussion of the depth resolution, an example for residual stress analysis will be presented concerning cold-drawn steel wires.

Conical slits

Geometry and adjustment. The used CSC had seven conical slits with different radii that can be used for materials with cubic crystal structure. The width of the conical slits was 20 µm, the focus distance was 100 mm. The CSC was made of 2 mm thick Tungsten alloy. The ring radii were chosen such that a set of diffraction cones can pass the slits at a suitable photon energy (63.4 keV for Fe (Fig. 1).

The CSC was mounted on a hexapod for easy adjustment (Fig. 1). The hexapod allows rotation around arbitrary axes. In the first step, the centre of the conical ring slits has to be placed in the centre of the beam by shifting the CSC horizontally and vertically. In the second step, a thin sample is placed at the focus distance and the rotation of the CSC around *y* and *z*-axis is optimized. Finally, the position of the sample with respect to the gauge volume is determined precisely by scanning the sample in beam direction (*x*) and using an area detector for recording the diffraction rings.

Depth resolution. The depth resolution is the most important parameter for the use of the CSC. It was measured for the Fe (110) and (200) reflections by scanning a 0.5 mm thick pure Fe sample through the focus of the CSC. The intensity on the diffraction rings was integrated and plotted as a function of sample position. The full width at half maximum (FWHM) of the intensity curve was

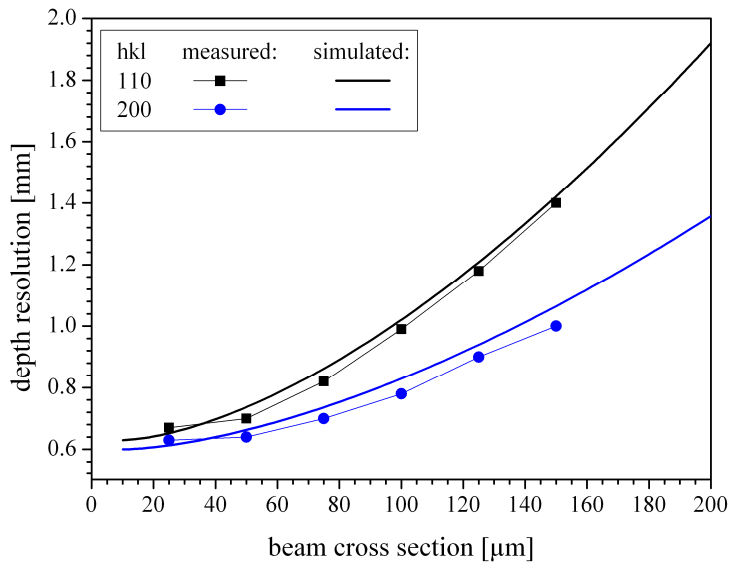

Fig. 2: Depth resolution as a function of beam size for an energy resolution of 0.7% for the Fe (110) and (200) reflections. The beam width is the edge length of the square beam cross-section. The measured values were deconvoluted with a step function representing the sample thickness of 0.5 mm.

used to define the depth resolution (Fig. 2**).** The achieved depth resolution is between 0.6 and 1.4 mm for beam sizes between 50 and 150 µm. When several rings are used simultaneously, it is important that the difference in the positions of the focal spots for these rings is small. For the two analyzed reflections, the difference was 24 µm.

Below a beam size of 50 µm, the depth resolution did not decrease further. The reason is that the depth resolution is limited by the energy resolution given by the monochromator (in this case a double crystal monochromator with elastically bent Si (111) crystals). Further improvement of depth resolution can be achieved by reducing the energy bandwidth. Details can be learned, e.g., from a numerical simulation and will be published elsewhere.

Residual stress in a drawn steel wire

A drawn ferritic steel wire made of a special alloy for cold forging applications with a diameter of 8.3 mm was characterized using the conical slit procedure. The motivation was to obtain experimental residual stress distribution across the wire and compare the experimental values with the results from a FE simulation of the drawing process [11].

An X-ray energy of 77.5 keV was used. The cross section of the X-ray beam was 50 µm × 50 µm and the depth resolution was 0.8 mm. The grain size of the material was small enough to yield homogeneous diffraction rings with this gauge volume. Strain measurements were carried out along one diameter for two different sample orientations (Fig. 3a). In the first orientation, axial and radial strains were measured; in the second orientation, axial and tangential strains were determined. Axial strains obtained from both measurement strategies were within experimental errors of less than 10%. Although the gauge volumes were very different for the two orientations because of the elongated shape of the gauge volume, values for three mutually perpendicular directions could be determined in this way and, thus, distribution of triaxial residual stresses could be evaluated. Stress-free reference values were measured in small cubes cut from the wire.

The results in Fig. 3b indicate pronounced residual stresses in all three directions. All stress components show a compression inside the wire and a tension in the surface region. The largest stresses occur in axial direction from +400 MPa close to the surface to –500 MPa in the centre of the wire. The very typical stress gradients across the diameter of the cold drawn wire are a consequence of the complex elasto-plastic deformation during the drawing process resulting in a

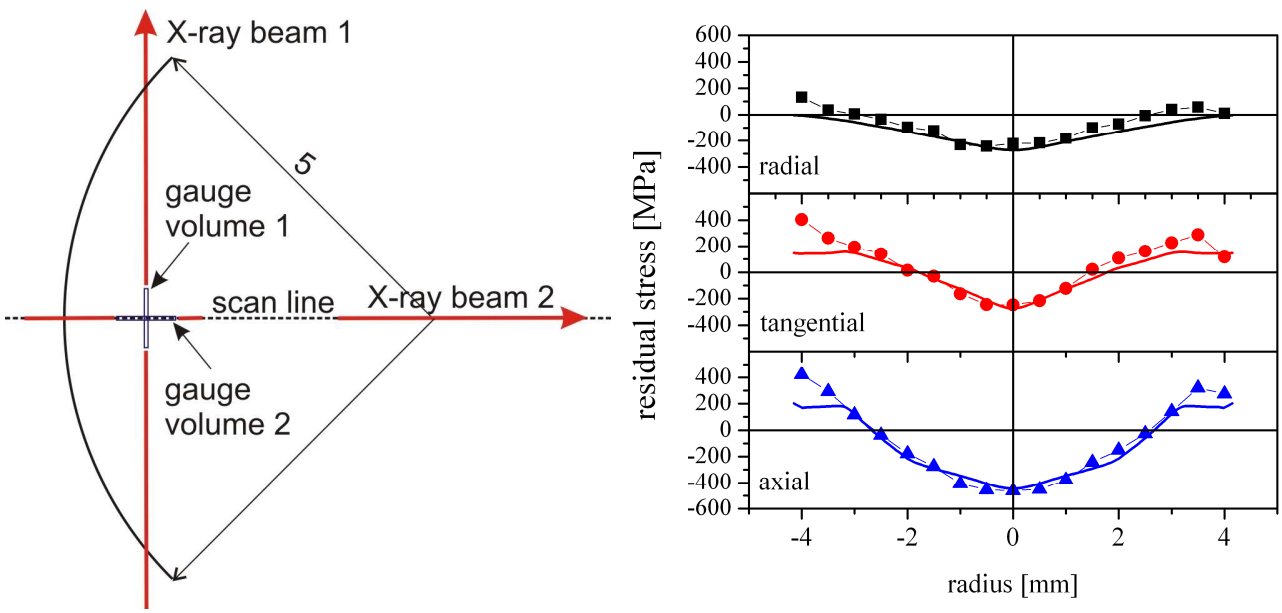

Fig. 3: a) Sample orientation with respect to the X-ray beam during the two measurements along one scan line (only a part of the wire cross-section is drawn). b) Residual stresses calculated from the measurements (symbols) in comparison with FE model predictions (solid lines).

inhomogeneous strain distribution over the wire radius. The residual stress distributions measured in radial, tangential, and axial directions are in a very good agreement with the FE predictions (Fig. 3b) [11].

Conclusions

A conical slit cell was used for depth-resolved residual stress analysis at a synchrotron high-energy X-ray beamline. The experimental set-up is relatively simple when a hexapod is used for the adjustment of the conical slit. With a conical slit size of 20 µm and a beam cross-section of 50 µm × 50 µm a depth resolution of 0.8 mm was achieved for the Fe (211) reflection with a bent Si double crystal monochromator with an energy resolution of about 0.7%.

This opens up the possibility of depth-resolved residual stress analysis with monochromatic high-energy synchrotron X-rays. The depth resolution can be tuned with the beam cross-section from a few 100 µm to a few mm, depending on the energy resolution given by the monochromator.

Since beam intensities are much higher than at neutron instruments, a large number of points can be measured within short time. Thus, two or three-dimensional strain and stress maps can be produced, e.g. for comparison with predictions of finite element model simulations. However, the possibility of easy access to three orthogonal directions is missing for large samples. Moreover, in many cases measures have to be taken to improve the grain statistics. Consequently, neutrons and high-energy X-rays offer complementary capabilities.

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