Diffraction on heavy samples at STRESS-SPEC using a robot system

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Abstract. The material science diffractometer STRESS-SPEC has high flux and a high flexible monochromator arrangement to optimize the needed wavelength. Many specific sample handling stages and sample environments are available. One of them is a Stäubli RX 160 robot with nominal load capacity of 20 kg and more freedom for texture mapping than the Huber 512 Eulerian type cradle. Demonstration experiments of non-destructive pole figures and strain measurements of Cu-tube segments weighing 12 kg weight and 250 mm in length and 140 mm diameter have been carried out. The residual strains measured by the robot and by the XYZ-stage fit quite well, that means the robot is reliable for strain measurements. The texture of the Cu-tube has dominant recrystallization texture components represented by the cube and the rotated cube.

1. Introduction
Neutron diffraction is well established for many kinds of investigations in materials science. For crystallographic texture investigation neutrons are favored for bulk information [1], but local studies are needed for residual stress profiles [2]. The beam port STRESS-SPEC at Heinz Maier-Leibnitz Zentrum (MLZ) at Garching/Germany is designed as a materials science diffractometer, which is able to offer strain [3] and texture analysis [4]. Both investigations can be done because of a comparably high neutron flux and a large flexibility in choosing the optimized wavelength for strain (2θ=90° arrangement) and texture (high intensity for low (hkl)) research.

STRESS-SPEC is equipped with a large sample stage, composed of XYZ-stage and an ω-rotary table for strain scanning. This stage can handle samples up to 300 kg. For pole figure measurements a Huber 512 type Eulerian cradle with an asymmetric φ-table must be installed on top due to the need to tilt and rotate the sample. Texture mapping is strongly limited by sample weight as well as by sample movement. Some investigations have been done with complex geometries, such as large tubes, shaft, turbine blade, etc. [5] with manual sample positioning and automatic pole figure scanning. To overcome these limitations a Stäubli RX160 robot was installed at STRESS-SPEC. The working range for strain scanning and texture mapping is within a circle of 500 mm (safety radius) and the present limitation is given by a nominal load of 20 kg. The big brother Stäubli RX170 with a nominal load of 30kg can be used only for low Bragg angles due to restricted space at STRESS-SPEC, and is not suitable for residual strain measurements. The aim of this work is to illustrate the capabilities and advantages of the RX160 robot in diffraction analysis of heavy samples, giving an example of texture and strain analysis of a 12 kg Cu-tube segment.

2. Experimental procedure
2.1. Sample description
The test sample was an industrial produced seamless SF-copper tube of 140 mm outer diameter, 10 mm wall thickness and 1m length. The chemical composition of the SF-Cu tube...
is: 0.0168% P, 0.0041% Pb, 0.005% Al, 0.0016 Ni and the rest is Cu. The average grain size obtained by optical microscopy was about 76 µm. A tube segment of 250 mm length was cut

2.2. Three dimensional laser scanner
For both pole figure and residual strain measurements, the sample within the sample holder must be scanned by a 3D laser scanner as shown in fig.1 a,d. The scanner captures the profiles generated by the interaction of a projected laser and the target topography by using triangulation. In the next step the coordinates for all sample positions for texture mapping and strain scanning were defined. An in house developed mathematical library was used to extract the numerical values for the selected points (X, Y, Z, RX, RY and RZ with respect to robot plate) using Geomagic Studio software and then implemented in the robot software. Sample including sample holder were mounted on the robot arm (fig. 1b).

2.3. Pole figure measurement by the Robot
The pole figure measurements were carried out using a dual wavelength of $\lambda_{PG(004)} = 1.55$ Å and $\lambda_{PG(006)} = 1.03$ Å to save beam time. STRESS-SPEC is equipped with an area detector of 300x300 mm², so that the two wavelengths result in three Cu pole figures, namely Cu (111) and Cu (200) with 1.55 Å, and Cu (220) with 1.03 Å, see fig 1c. Depending on the $2\theta$ angle ($2\theta = 47^\circ$) and the sample detector distance (1065 mm) the sample was tilted five times in $\chi$. For each $\chi$-position a continuous scanning along $\phi$ followed and an automatic data collection every 5° was performed. Continuous scanning, which is very easy with the robot, has the advantages of reducing counting time and collecting information from all crystallites in the gauge volume. The disadvantage is the averaging over 5° in $\phi$, not useful for sharp textures. The results obtained are incomplete pole figures, due to heavy absorption along the length of the sample (250 mm). As an example, the texture from the central part of the tube wall at the 90° position was obtained using a primary slit of 5 mm in diameter and a radial collimator (5 x 5 mm²) as secondary slit. The exposure time for each detector image (5° rotation in $\phi$) was 2 min. The software package StressTextureCalculator [6] has been used to extract pole figure data from area detector data using the mathematical formalism of Bunge and Klein [7].

2.4. Strain measurement by the robot
The strain measurements were carried out using a neutron wavelength of 1.53 Å obtained from a Si(400) monochromator. A rectangular primary slit 2 x 2 mm² made of cadmium and a radial collimator (2 x 2 mm²), acting as a secondary slit, were used. The sample to detector distance was 1065 mm and the primary slit to sample distance was 50 mm. To measure the peak position of Cu(311) for strain analysis the detector position was $2\theta = 89.6^\circ$. The strains at sample positions 0°, 90° and 180° across the thickness (at 6 positions) for every direction (axial, radial and hoop) were measured. The exposure time for every measuring point was 5 min. The strains obtained by the robot were compared with those obtained using the XYZ-stage at the identical sample positions. The same experimental conditions have been applied except that 8 positions across the thickness were measured when using the XYZ-stage. The exposure time for measurement was 2 min (because of limited beam time) for every step. It should be mentioned that the configuration for the axial strain was adjusted manually. Figure 1e and f shows the axial configuration for strain measurement using the robot and the XYZ-stage.
The Orientation Distribution Function (ODF) was calculated by the iterative series expansion method using incomplete (111), (200) and (220) pole figures. The degree of series expansion was $L_{\text{max}}=22$. The sample symmetry of the ODF-calculation was triclinic. For the ODF results of face centered cubic (fcc) materials sections $\phi_2=0^\circ$ and $\phi_2=45^\circ$ have been used to describe the texture components because most of them are visible as shown in fig. 2. The texture in this Cu tube is characterized by 6 components. The recrystallization components [8, 11] are cube $\{001\}<100>$, rotated cube $\text{C}_{\text{RD}} \{013\}<100>$, Goss$\{110\}<001>$. Rot $\text{C}_{\text{RD}}$ was described as recrystallization twins, which have been observed in Cu during static and dynamic recrystallization. The deformation components [8, 11] are copper $\{112\}<111>$, brass $\{110\}<112>$, and S $\{123\}<643>$ also known as $\beta$-fiber. The dominant components are the rotated cube $\text{C}_{\text{RD}} \{013\}<100>$ with an intensity of 11 multiples of random distribution (mrd) and the cube $\{001\}<100>$ with 8.7 mrd. This copper tube was made by hot deformation which results in recrystallization during and after extrusion.

Fig. 3 shows comparisons for the axial and radial strains at the 90° position for both the robot and the XYZ-stage measurements. They are in good agreement. The axial strain varies from tension in outer region to compression in inner region and the radial strain profile goes from compression in the outer region to tension in inner region.
4. Conclusion
The high penetration power of neutrons and sample manipulation by the Stäubli RX160 robot combine well for local and non-destructive texture and residual strain measurements. The texture of the investigated tube shows a combination of deformation represented by the β fiber and recrystallization textures represented by the rotated cube C_{RD} {013} <100> and cube {001} <100>. The robot can handle larger samples than the Eulerian cradle. The residual strain measurements using the robot show a good agreement with the strains determined using the XYZ-stage. The positioning of the robot was sufficiently good for strain analysis. A great advantage was that all the three strain directions can be measured with identical set up, being not necessary to change the sample alignment manually.

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References
[1] Brokmeier H.-G. 2006 Physica B: Condensed Matter 385, 623
[2] Lorentzen T., Hutchings M.T., Withers P.J. 2005 Introduction to Characterization of Residual Stress by neutron Diffraction (Taylor and Francis, London)
[3] Hofmann M., Robelo-Kornmeier J., Garbe U., Wimpory R.C., Repper J., Seidl G.A., Brokmeier H.-G., Schneider R. 2007 Neutron News 18, 27
[4] Brokmeier H.-G., Gan W. M., Randau C., Rebelo-Kornmeier J., Hofmann M. 2011 Nuclear Instruments and Methods in Physics A 642, 87.
[5] Brokmeier H.-G., Randau C., Gan W.M., Hofmann M., Lippmann T., Schell N. 2012 Mater. Sci. Forum. 702-703, 499.
[6] Randau C., Garbe U., Brokmeier H.-G. 2011 J. Appl. Crystallogr. 44, pp. 641-646.
[7] Bunge H. J., Klein H. 1996 Z. Metallkunde. 87, 465.
[8] Lücke K. 1984 Proc. of 7th International Conference on Textures of Materials, Netherlands Society for Materials Science, 195.
[9] Wasserman G. 1963 Z. Metallkd. 54, 61
[10] O. Engler: Acta mater. Vol. 48, P4827 (2000).
[11] Humphreys F. J. and Hatherly M. 2004 Recrystallization and related annealing phenomena (Amsterdam, Elsevier), chap.3, p. 67 and chap.12, p.379.

Fig. 3 Comparision between the measurements accomplished using the robot and the XYZ-stage at 90° (left) axial strain and (right) radial strain (OD: outside diameter and ID: inside diameter)