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Stress and local texture measurements at the materials science diffractometer STRESS-SPEC at FRM-II

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Abstract. In response to the development of new materials and the application of materials and components in new technologies the direct measurement, calculation and evaluation of textures and residual stresses has gained worldwide significance in recent years. Non-destructive analysis for phase specific residual stresses and textures is only possible by means of diffraction methods. The determination of the local variation of texture for example by inhomogeneous deformation is very important, due to the coherence between the texture and the physical and mechanical properties of materials. The materials science diffractometer STRESS-SPEC which is dedicated to investigate small gauge volumes for stress analyses could also be used for local texture measurements with nearly the same setup.

Introduction

The crystallographic texture was described first by Wassermann [1] for metallic materials and texture analysis was extended to the calculation of the ODF (orientation distribution function) by Bunge [2]. Physical and mechanical properties of polycrystalline materials can be calculated as the texture-weighted average of the single crystal anisotropy [3].
In order to develop modern materials the interest in local inhomogeneity of texture will become more relevant. One technique to measure local texture is by X-ray diffraction [4,5] using a glass capillary slit system. The same technique became available using electrons [6]. The main difference between these is the different orientation and location resolving power. Because of their penetration depth, which for both is in fact very small compared to neutron radiation, it is only possible to measure in two dimensions. In principle one can achieve high penetration depths using high energy synchrotron radiation [7-9] (50 – 150 keV), however, due to the resulting small diffraction angles the definition of the gauge volume is only good

in two dimensions. For a three dimensional texture and strain mapping with a spatial resolution in the millimetre regime neutron radiation is indispensable.

Experimental technique

The new materials science diffractometer STRESS-SPEC at FRM II is designed to be equally applied to texture or residual stress analyses by virtue of its flexible configuration [10,11]. The diffractometer can be easily configured either way due to a highly flexible monochromator setup using three different monochromators: Ge (511), bent silicon (400) and pyrolitic graphite (002). This range of monochromators and the possibility to vary the take-off angles from $2\theta_M$= 30º to 120º allows wavelength adjustment such that measurements can be performed always around a scattering angle of $2\theta \sim 90º$. This is important in order to optimise neutron flux and resolution, especially for stress and local texture analysis. The gauge volume element in that case is cubic (figure 1).

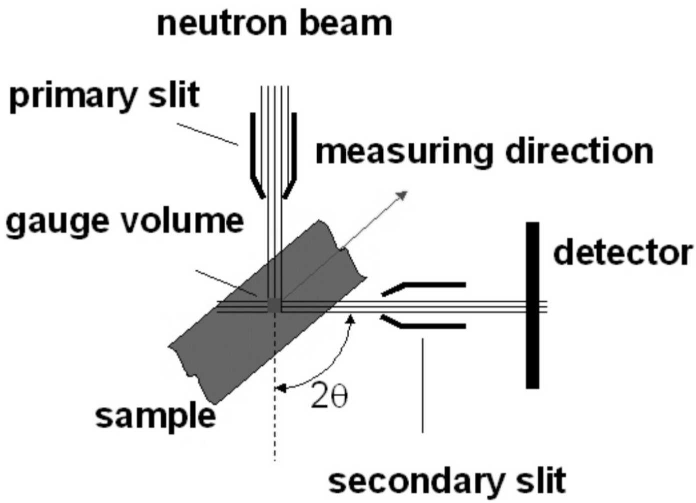

Figure 1. Definition of the gauge volume with a slit system at STRESS-SPEC

A two dimensional position sensitive ^{3}He detector (PSD) is installed as the primary detection option. This detector, developed by EMBL (Grenoble, France), is a multiwire detector with a delay time encoding. It has an active area of 200x200mm^2 with a spatial resolution of about 1.5x1.5 mm^2. The measured efficiency for neutrons of wavelength λ = 1.8 Å is 73%. The high neutron flux at STRESS-SPEC in combination with the area detector decreases the data acquisition time and offers the possibility to investigate local texture using a gauge volume down to 2x2x2 mm^3. For local texture measurement only two changes of the strain setup have to be made. At first an Eulerian cradle with additional (xyz)-translation table [12] has to be mounted in order to be able to detect the (hkl)-reflection under different orientations in

reciprocal space. The second change includes the sample detector distance, which should be as close small as possible (0.75m in our case) to maximise the available χ-space.
On the basis of the small sample detector distance it is possible to detect 15° section of a diffraction cone at scattering angle $2\theta = 90°$. This allows a complete pole figure to be measured in just 6 tilting steps in χ, compared to 19 steps using a conventional single detector setup. Under these conditions every detector image is divided into three equal γ-sections as shown in figure 2.

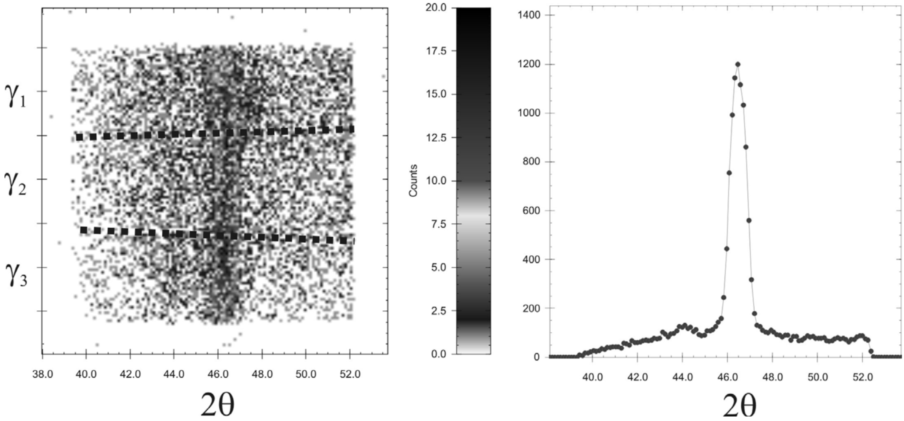

Figure 2. Definition of the γ–sections and the diffraction diagram of a detector image

For each section a diffraction diagram has to be determined, in which for every detected (hkl)-reflection the integral intensity can be extracted. This is automatically done by a software package developed for pole figure calculation of diffraction images from STRESS-SPEC [12]. The algorithm for the pole figure calculation is based on a software package [13] developed for synchrotron texture analysis.

Strain measurement

Here we present exemplarily some of the results of measurements on a single bead on plate weld within the framework of the European Network on Neutron Techniques Standardisation for Structural Integrity (NET) [14] in order to demonstrate the reliability and accuracy of the instrument for residual stress analysis by comparing results made previously from other neutron diffraction facilities using a Bayesian statistical approach [15, 16].

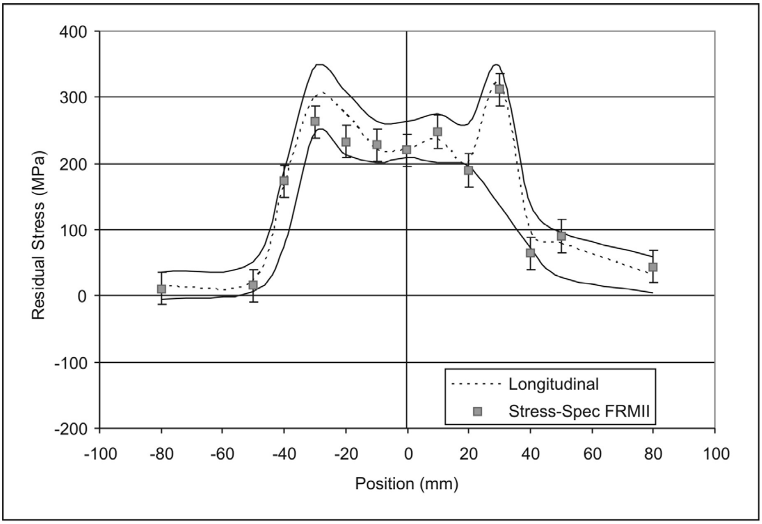

Figure 3. STRESS-SPEC residual stress data (longitudinal direction) from the scan along the weld line compared to Bayesian average (dashed line) with upper and lower limits.

The agreement between the different facilities is good; with the exception of the weld start position (+30mm) in the longitudinal direction where there is a large uncertainty (figure 3). All facilities used different setups and measured on different diffraction planes. The final residual stress values therefore depend on a number of factors, for instance: gauge volume size, positioning (especially within large strain gradients), texture, grain-size effects, uncertainties in diffractometric used, peak fitting routines used etc. The agreement of the STRESS-SPEC data compared to the average is good, being one of the best agreeing data sets. The average quoted uncertainty for the STRESS-SPEC data is ± 25 MPa [16].

Local texture measurement

First local texture measurements with neutron radiation were carried out by Brokmeier [17] at TEX-2 with a gauge volume of 4x4x4mm³ on a niobium cylinder. The first sample for local texture measurement at STRESS-SPEC was the aluminium VAMAS ring and plug cylinder seen in figure 4. The sample was used as round robin standard for neutron residual stress analysis. The inner cylinder was cooled and than placed in the warmed ring. At the border of the two pieces a well characterised stress pattern is generated. In addition the global texture of this sample was also determined during the stress round robin thus making it also a good test specimen for local texture measurement. Three measurement positions were chosen to measure the (222) pole figure using a gauge volume of 2x2x2mm³. The first point is in the centre of the whole sample giving texture information of the cylinder. Point 3 shows the texture of the ring, at the border between ring and plug (point 2) a mixture of both texture components will be found.

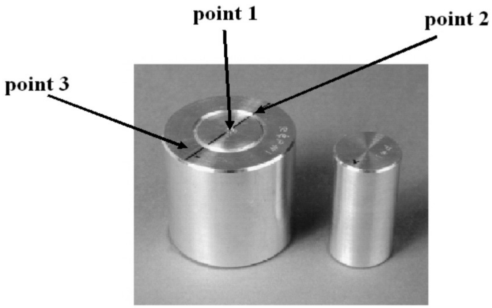

Figure 4: Position of the local pole figure measurement at the VAMAS ring and plug sample

Because of the negligible absorption cross section of aluminium it was possible to get first reliable results for pole figures even without a proper absorption correction. The pole figures in figure 5 observed at the three points show very similar intensity distributions. Comparing the first and the third pole figure one can recognise a small shift of the maxima around the pole figure normal. Pole figure 2 shows a mixture of the texture components in point 1 and 3.

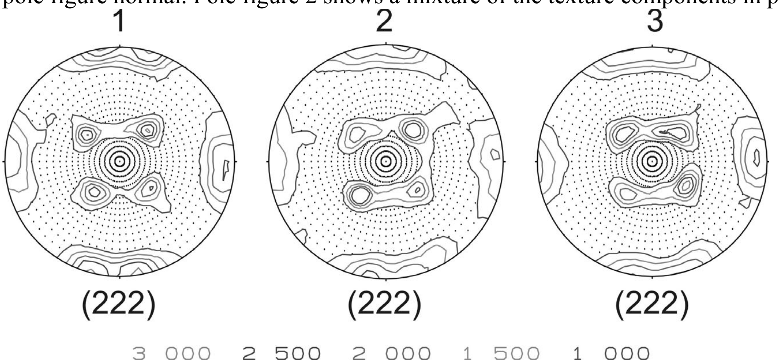

Figure 5. (222)-pole figure of the VAMAS sample measured at three different positions

Conclusions

The established method of strain measurement with neutron radiation at STRESS-SPEC provides a good basis for local texture analysis. Due to the flexible configuration it is possible to reduce the data acquisition time for every local pole figure down to 5 hours. With further planned improvements of the beamline using focussing neutron optics and a larger detection area it is expected to reduce the data collection time considerably so that a 3-dimensional texture mapping of complete workpieces will be feasible.

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