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E3: Residual Stress Neutron Diffractometer at BER II

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Abstract: The E3 residual stress neutron diffractometer operated at Helmholtz-Zentrum Berlin (HZB) is designed for studies in material science and engineering applications. Recent upgrade activities have made the instrument faster and more adaptable to different types of measurement. Thus, E3 has become more attractive to a broad user community, including industry, and increased substantially its scientific output.

1 Introduction

Neutron diffraction provides an attractive tool for investigations in fundamental research as well as for industrial applications. The large penetration depth within the bulk and the strong scattering power of many materials are advantageous features to probe crystallographic properties non-destructively with neutrons. Hence, utilizing a diffractometer allows the study of lattice strains, phase transitions and preferred crystallographic orientations.

The neutron wavelength λ, applied for such investigations, is of the order of the interatomic distances d^{hkl}. For polycrystalline engineering materials, for example, coherent elastic neutron scattering at angles of $2\theta^{hkl}$ will occur if Bragg's law is fulfilled:

$$n\lambda = 2d^{hkl} sin\theta^{hkl} \quad (1)$$

where the *hkl* Miller indices denote the selected lattice plane of the crystal and n=1,2,3... is the order of the measured lattice reflection, i.e. the Bragg peak.

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2 Residual Stresses

The large penetration depth in combination with the choice of a specific wavelength offers measurements with scattering angles 2θ close to 90° and thus with virtually cubic gauge volumes. Residual stress analysis (RSA) with angular-dispersive neutron diffraction is indeed usually restricted to measurements of a single lattice reflection (depending on scattering angles and detector size), but for many engineering applications this information is sufficient (Staron, 2008).

The RSA utilizes the impact of stresses inside a component (also applied stresses) on the crystal lattice of the material which leads to elastic lattice strains, i.e. lattice spacing variations, which can be determined by measuring the shift of the Bragg angle θ^{hkl}.

The lattice strain ε in the direction of the scattering vector, i.e. normal to the reflecting lattice plane *hkl* of the specimen's crystal structure, is the lattice spacing variation λd over a stress-free reference d_0, but is also determined by means of the measured lattice reflection position 2θ (scattering angle):

$$\varepsilon = \frac{\Delta d}{d_0} = \frac{sin\theta_0}{sin\theta} - 1 \tag{2}$$

Thus, the strain can be determined without the instrument's neutron wavelength, whose value would have to be determined experimentally (and with sufficient precision) in order to transform the measured peak position into a lattice spacing d.

Due to the access to three mutually orthogonal strain directions ε_1,ε_2,ε_3, even in the bulk of the specimen, one is able to determine the stress state in an isotropic polycrystalline sample in one of these directions i=1,2,3:

$$\sigma_i = \frac{E}{1+v}\varepsilon_i + \frac{vE}{(1+v)(1-2v)}(\varepsilon_1 + \varepsilon_2 + \varepsilon_3) \tag{3}$$

The modulus of elasticity E (also known Young's modulus) and the Poisson's ratio v depend on the *hkl* lattice plane selected for the measurements.

Residual stress neutron diffraction measurements are an essential tool for a broad range of engineering applications and fundamental research questions. The RSA in welding components, for example, is of great interest, because induced residual stress can decrease their load carrying capacity and/or their lifetime. Moreover, with neutrons the access to the interior of samples allows the verification of finite element models (FEM) that are typically used as a prediction tool in many industrial applications.

3 E3 instrument layout

The E3 neutron diffractometer at the BER II research reactor is designed for angular-dispersive strain and stress analysis of simple geometric samples as well as for industrial applications and heavy and large components of complex shape. The instrument is located at beam port T2 and employs a horizontally bent and vertically focusing perfect single crystal blades Si (100) monochromator (Wimpory et al., 2008) that supplies neutrons with a wavelength of about 0.1471 nm.

The diffractometer itself consists of two big circles with a diameter of 800 mm each in order to rotate the specimen setup on top (Ω) and rotate the detector around the table and along the scattering angles (2θ) within a range of approx. $35° \leq 2\theta \leq 110°$. The detector measures scattered neutrons over an area of 300 mm × 300 mm by means of ionization of ^{3}He gas. For the analysis of the captured detector images, the StressTex program (Randau et al., 2011) dedicated for stress and texture analyses is available. A schematic drawing of the instrument arrangement is shown in Figure 1.

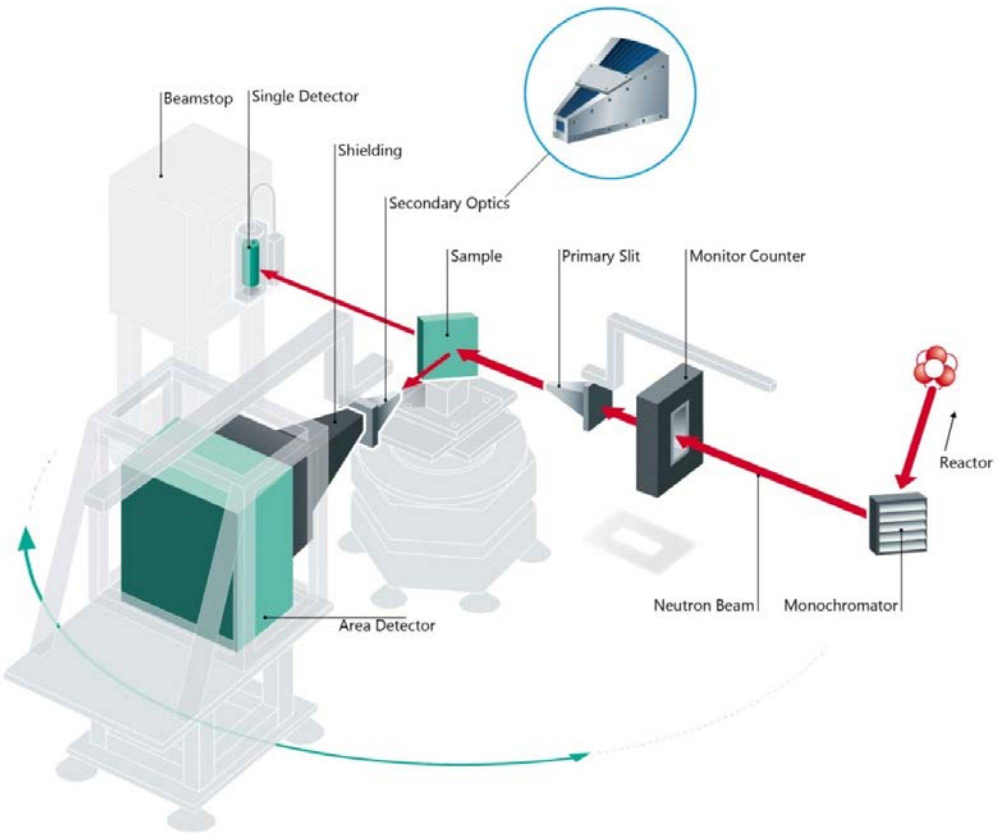

Figure 1: Schematic drawing of the E3 residual stress neutron diffractometer.

For sample positioning an x-y-z translation stage with a maximum travel range of 250 mm on each axis (vertically and horizontally) is placed on top of the Ω-table. This setup is able to carry loads of up to 300 kg and, thus, makes measurements with large and heavy components possible. Figure 2a shows an example of the instrument setup with a 250 x 350 mm^2 weld plate. Furthermore, a range of equipment is available, such as a goniometer table (χ) for heavy samples (up to 50 kg) with the ability to tilt the samples by 90° (Figure 2b) is used to measure three perpendicular sample orientations without user interaction. The goniometer is also used in conjunction with another rotation stage (ϕ) to allow investigations of preferred crystallographic orientations, i.e. texture.

E3 is further able to utilize the HZB central sample environment, such as high-temperature furnaces (Figure 3a) and cryostats for a total temperature range of 1.5 K to 1800 K in order to perform in-situ material investigations. In addition, two dedicated load frames are available for tension and compression tests with a load capacity of up to 50 kN (Hoelzel et al., 2013) and a torsion option for up 12 Nm (Woracek et al., 2011). The first load frame is shown in Figure 3b. The neutron beam size can be adjusted horizontally and vertically by a motorized primary slit in a range from 0-10 mm and 0-20 mm respectively. On the secondary side, a resizable matchstick slit or an oscillating radial collimator with a FWHM of about 2 mm are used to define the gauge volume inside the sample.
The instrument hardware is driven with the in-house development CARESS. This program system also prepares scans, acquires and protocols the measured data and provides interfaces to further control sample environment components, such as the third-party devices mentioned above. A summary of the technical specifications of the E3 neutron diffractometer is listed in Table 1.

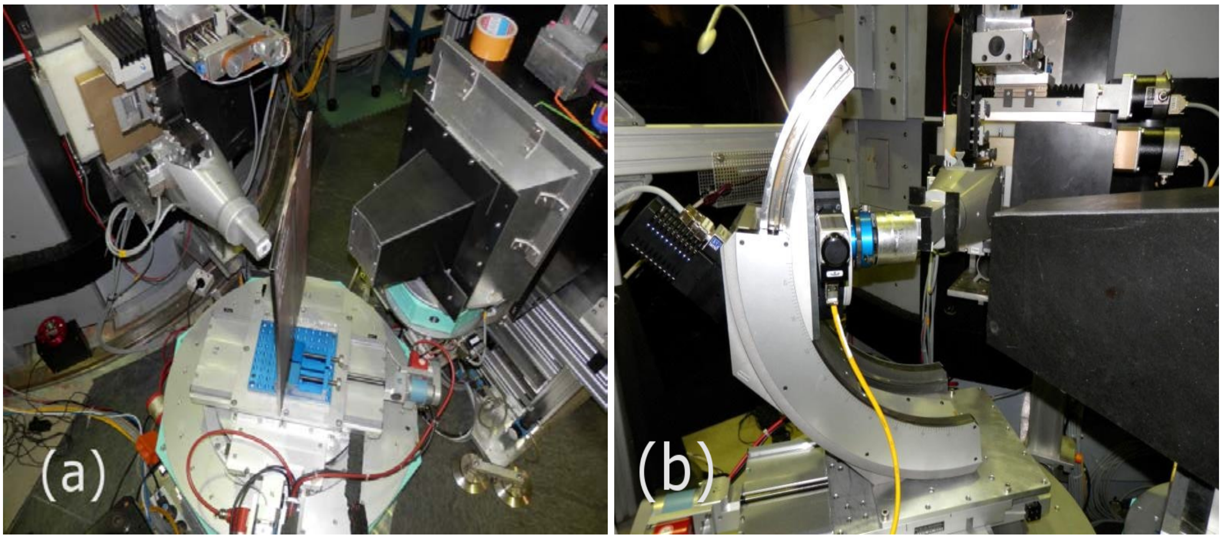

Figure 2: (a) Photograph of E3 with x-y-z table on top of the diffractometer circles for the positioning of a large 40×50 cm^2 weld plate. On the left: the motorized primary slit. On the right: the detector housing with an oscillating radial collimator in front of it. (b) Application of the goniometer table as a tilt stage in order to access three orthogonal sample orientations for RSA.

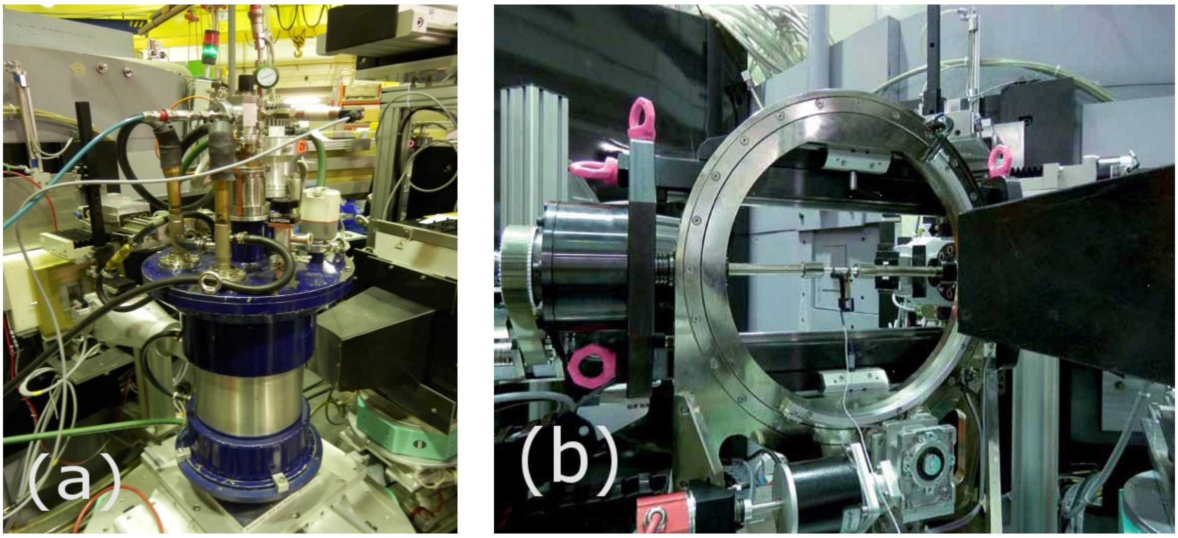

Figure 3: High-temperature furnace setup on E3 (a). A rotatable load frame for tensile and compressive testing (b).

Beam tube	T2
Collimation	Open
Monochromator / take-off angle	Si (100), double focusing / 65°
Wavelength / Flux	0.1471 nm / ~0.5 − 1 × 10^7 n/cm^2/s
Range of scattering angles	$35° \leq 2\theta \leq 110°$
FWHM standard powder	~0.3 (at 2θ = 80°)
Detector	position-sensitive ^{3}He area detector 30 × 30 cm^2
Resolution	$\Delta d/d \approx 1.4 \cdot 10^{-3}$
Sample to detector distance	600 mm to 1300 mm
Beam size at sample	0..10 × 0..20 mm^2
Maximum sample size	~0.5 m diameter
Scan range	• max. 250 mm (sample position) • ~$35° \leq 2\Theta \leq 110°$ (scattering angle)
Instrument options	• texture option • variable slit systems • radial collimator
Sample environment	• x-y-z table for max. 300 kg • goniometer table • load frames (tension, compression, torsion) • cryostats and furnaces
Software	StressTex (analysis), CARESS (instr. control)

Table 1: Technical data of E3.

4 Applications

The flexible setup allows for a broad range of applications. Investigations on welds (Hensel et al., 2014; Kromm, 2014) and the FEM verifications (Hemmesi et al., 2014) have been mentioned already. Below, a selection of further applications is listed:

- Phase distribution measurements on metastable 304L stainless steel samples exhibiting the transformation induced plasticity (TRIP) effect after tensile and torsional deformation were performed to obtain reference neutron diffraction results for the evaluation of imaging experiments (Woracek et al., 2014).
- Near-surface measurements using a partially emerging gauge volume can be performed on E3. With paying attention to artificial peak shifts the gap from neutron in-depth measurements to surface-zone investigations with X-rays can be bridged (R. C. Wimpory et al., 2011).
- Plasma-facing, but also heat-extracting divertor components developed as interlayer materials for the new ITER fusion reactor have been studied in order to find matrix alloys, fiber materials and an optimal interface design to achieve high mechanical strength and small thermal expansion mismatch for long-term stability (Schöbel et al., 2011).
- E3 regularly takes part in round robin activities to check and compare against other neutron instruments and other non-destructive and destructive techniques and, thus, develop and offer reliable concepts for industry-relevant residual stress applications (Smith et al., 2010).
- Single crystal samples have also been measured using a cryo-furnace at different temperature conditions in order to analyze both structural and magnetic phase transition temperatures (Chmielus et al., 2010; Rolfs et al., 2010).
- By supplying reference neutron diffraction results for residual stress and texture applications, E3 takes part in method developments such as the Bragg edge imaging concept (Boin et al., 2011; Strobl et al., 2011).

5 Recent upgrade activities

In order to meet the growing demand for neutron beam time, E3 is constantly being upgraded. Since the installation of a new monochromator in 2007, the instrument has become much faster and more attractive for the user community (R. Wimpory et al., 2008). Further upgrade activities have significantly increased the range of applications and improved the experiment performance (Boin & Wimpory, 2014):

- A set of perfectly bent Si (100) crystals providing a neutron wavelength of 0.1471 nm focusses on the sample. Thus, the diffractometer has become faster and more adaptable to different types of measurement.
- A new motor control system and detector electronics have been implemented providing a reliable and modular interface between instrument and the CARESS control software making the instrument more flexible for applications with third-party devices.
- An oscillating radial collimator secondary optic has been implemented to improve the instrument resolution, especially at interfaces and for in-depth measurements of complex-shaped samples.
- An open tilt stage to measure three mutually orthogonal strain directions within one sample alignment is available for measurements without user interaction. The same is possible with a new custom-developed stress rig for tension and compression experiments with loads of up to 50 kN (Hoelzel et al., 2013).
- E3 is now equipped with a new primary slit device to change the neutron beam size without instrument re-calibration (in both vertical and horizontal directions) and, thus, offers new types of on-the-fly measurements, such as the influence of grain sizes on peak shifts (Boin & Wimpory, 2014).
- A new laser scanner system is to be implemented on the E3 diffractometer to make instrument (re-) calibration and sample alignment much faster and more precise.

6 Summary

E3 is part of a complementary suite of HZB instruments (including X-ray and synchrotron diffractometers) for microstructural material investigations for fundamental and industry-near research. Being on a medium-flux neutron source (BER II) could be a limiting factor but recent activities have shown that E3 can compete with instruments on higher-flux sources and thus offer neutrons for a broad range of applications to an increasing user community.

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