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Non-destructive evaluation of strain-stress and texture in materials science by neutrons and hard X-rays

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Abstract

Together with the microstructure residual stresses and crystallographic textures are important parameters in considering the application profile of engineering products. Both residual stress and crystallographic texture are related to identical grain arrangements. That means on one hand texture and strain influence each other and on the other hand both informations contribute to considering anisotropic materials properties, quality of welds, inhomogenous behaviour and so on. Non-destructive evaluations need experimental methods going deeply inside a material. Furthermore, it is necessary to handle complex and heavy samples. Neutrons and high-energy X-rays (about 100keV) have a high penetration power in the cm-range to fulfil these requirements. The instrumentation must be very flexible fitting the set-up for both investigations. The materials science diffractometer STRESS-SPEC at the Forschungsneutronenquelle Heinz Mayer-Leibnitz FRM II at Garching/Germany (neutron) and the HZG materials science beamline Harwi-II at Hasylab-Hamburg/Germany (synchrotron) are able for combined strain profile and texture gradient investigations non-destructively. The newly installed robot at STRESS-SPEC allows a very flexible sample manipulation for both types of experiments. First results are presented. Synchrotron radiations with higher brilliance are favoured for small gauge volume and fast measurements. That opens the field of in situ loading (tension-, compression-, cyclic loading) and in situ welding investigations.

Keywords: residual stress, crystallographic texture, neutron, hard X-rays

1. Introduction

Powder diffraction is an efficient tool to characterize any kind of polycrystalline materials. Standard procedures are available to use the whole diffraction pattern for phase analysis as well as to determine

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crystal structures data, thermal expansion coefficients and lattice dependant elastic constants. In materials science and engineering applications stress-strain state, crystallographic textures and defect characterization is of basic interest for instance to fit application profiles of engineering products, to contribute to live time prediction and failure analysis. All three parameters results from identical grain arrangements of polycrystalline materials. In addition to the classical powder diffraction these materials science aspects can also be investigated by diffraction methods using X-rays, neutron or synchrotron radiation and electron diffraction methods. In this paper a focus is made for crystallographic texture obtained by the intensity of Bragg-reflections and for stress-strain analysis obtained by the shift of Bragg-reflections. The basic principals of the determination of crystallographic textures and residual stresses are published in many text books [1, 2]. Another focus is made on the use of neutron and synchrotron radiation, which have special properties to make them interesting for materials science application [3].

2. Neutron radiation

Neutron radiation for diffraction experiments in materials science application is available in a wavelength range from 0.6Å – 2.6Å, which is comparable to in-house X-ray diffraction by Mo-, Cu- or Cr-tubes. Among others a basic difference is the penetration power, which is for neutrons in the cm range for most materials compared to µm's for conventional X-rays [4]. Thus, neutron diffraction is the main tool for bulk investigations (crystallographic texture and quantitative phase analysis), see Fig. 1a for instance to average over tensile samples, see Fig. 1b, or inhomogeneities after shear, see Fig 1c. Anisotropic absorption is much better to handle than with all other methods. Moreover, bulk measurements require a large enough beam cross section in combination with the high penetration power, available for most neutron materials science diffractometers. This bulk information's can be obtained non-destructively, so that investigations of mechanical tests and microstructure studies can be done on identical samples. Moreover, a non-destructive measurement allows subsequent thermo-mechanical treatments such as annealing and recrystallization as well as cyclic loading. In some cases it makes sense to carry out in situ experiments, such as in situ high temperature or in situ loading [5, 6].

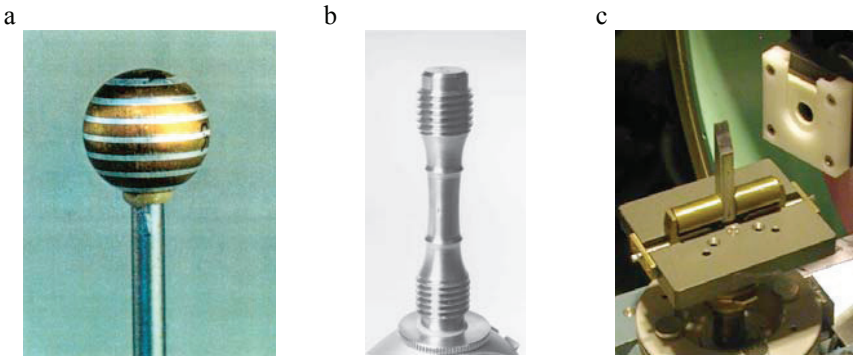

Fig. 1. (a) spherical sample of an Al-Cu composite Ø =10 mm (b) standard tensile sample; (b) ECAP sample at TEX-2

A second advantage of the high penetration power is that one can see deep inside the material. The two principal arrangements of bulk and local measurements are shown in Figure 2. Local investigations have been firstly implemented for residual stress determinations to describe stress profiles. The gauge volume, which can be set up by slit systems (see Fig. 2b), depends on neutron flux of the instrument and scattering properties of the investigated material. For strain profiles a gauge volume of 1x1x1mm³ is common to get on one hand a sufficient peak to background ratio and on the other a reasonable total counting for strain

mapping of a welded sample. Due to the high number of up to 32400 pole figure points necessary for quantitative texture analysis longtime a restriction of texture gradient investigations exists by available beam time. Recently build new materials science diffractometers or improved instruments allow local texture investigations with gauge volume of 2x2x2mm³ in reasonable time for many materials.

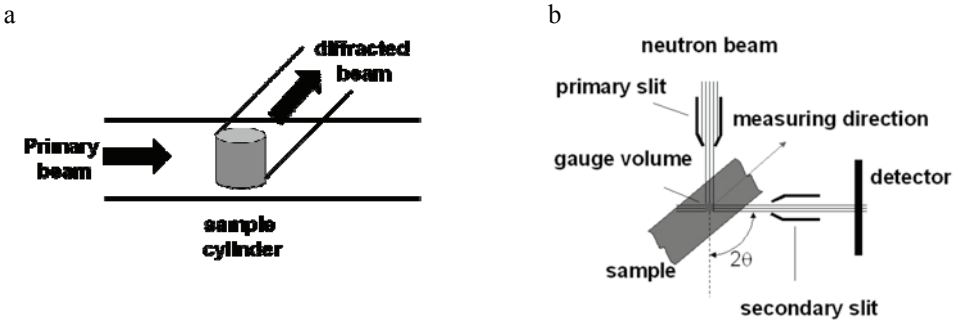

Fig. 2. (a) Bulk method (after Tobisch and Bunge [7]; (b) local investigations (after Choi et al. [8])

In addition to the penetration power, the main advantage of neutron diffraction for local measurements is an optimized gauge volume at 90° in 2θ (Fig. 2b). The 90° arrangement is the best compromise to keep the gauge volume as constant as possible during sample rotation and sample tilt.

The three main disadvantages of neutron diffraction for stress-strain and crystallographic texture determination are:

- The number of available neutron sources
- Insufficient number of instruments
- Much to less available beam time

3. Hard X-rays

There is not a strict classification but energies higher than 50 keV are considered as hard X-rays. That means hard X-rays can be generated firstly by tungsten X-ray tubes, secondly by storage rings such as Doris III and Petra III at Desy/Hamburg, Bessy II at HZG/Berlin or the ESRF at Grenoble and thirdly by linear accelerators such as the X-ray free electron laser XFEL at Desy/Hamburg [9]. In materials science hard X-rays by conventional instruments are mostly restricted to material testing. Standard material testing devices operate up to 450 keV due to their application field for non-destructive testing (NDT). Computer tomography is an efficient tool in many industrial applications. Some tests have been published using monochromatic 70keV W-Kα for texture analysis [10] and an energy dispersive method obtained by a tungsten tube for in situ quality control in Al-sheet production [11].

Material science diffractometers at storage rings operate with up to 200keV that means they cover a wavelength between 0.248Å - 50keV and 0,062Å – 200keV with is much shorter than for neutrons and conventional X-rays (Cu Kα 1.5418Å). These high energies are needed to get a similar penetration power than neutrons. Due to the Bragg's law short wavelength will end up with small scattering angles. All needed reflections are within 10° of 2θ which makes area detectors very efficient because complete Debye-Scherrer cones are obtained. In Figure 3a the principal set up is shown using monochromatic radiation.

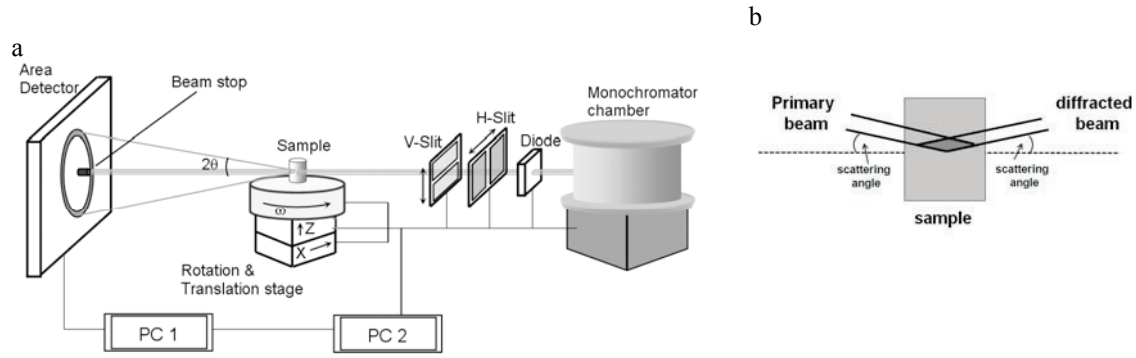

Fig. 3. (a) principal beam path at a hard synchrotron diffractometer (b) gauge volume in case of low scattering angle

In addition to the wavelength two essential differences between hard X-rays and neutrons, important for texture and stress-strain analysis, are firstly:

- Hard X-rays from a synchrotron have very high brilliance
- Beam size is much smaller than for neutron diffraction

All properties together allow firstly the investigation of large samples as well as very small samples, secondly very fast measurements and thirdly the gauge volume can be very small. That means, strain mapping and texture gradient analysis is normally performed with much smaller gauge volume which can go down to the µm-range. Limitation is the balance between the needed grain statistics and the wished strain profile. High brilliance makes synchrotron radiation a perfect tool for in situ investigations. Loading devices are meanwhile available at all materials science beam lines to carry out in situ experiments for applied stress evaluations and texture developments under load. In situ welding investigations were carried out at the HZG-beamline Harwi II (Flexistir for friction stir welding).

Common use is the energy dispersive method to determine stress depth profiles after surface treatments such as ball burnishing or shot peening, see in detail by Genzel [12]. Monochromatic beams are used for instance to measure texture gradients of severe plastic deformed samples, of tube walls and of welded samples and stress strain mapping of welds. Figure 4 shows some samples.

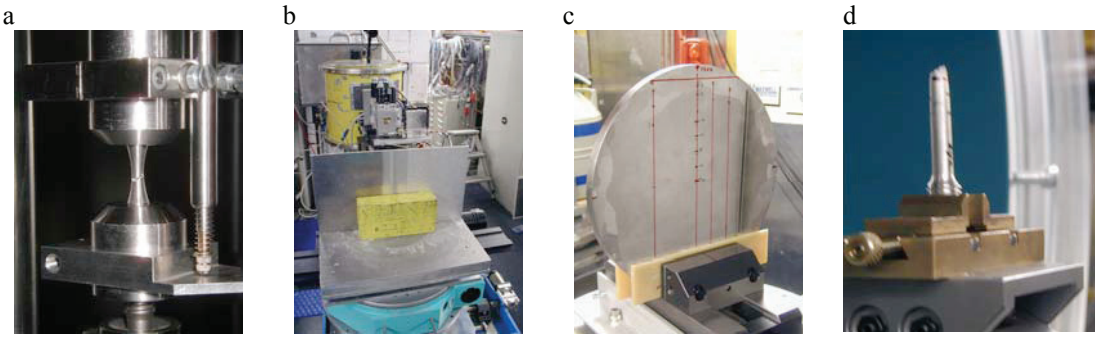

Fig. 4. (a) in situ tensile sample (b) LBW sample of Al (c) disc sample of 300mm Ø (d) texture gradient after failure

In hard X-ray diffraction the disadvantages for stress-strain and crystallographic texture determination are similar to neutron diffraction:

- Restriction in number of available sources and instruments
- Availability and efficiency of beam time
- Necessity to improve detector quality and data treatment (high sped investigations)

4. Instrumentation

Longtime instruments for stress-strain analysis and crystallographic texture analysis have been separated in two machines due to different requirements on resolution and beam flux. Recently developed instruments allow the combination quite well or make a more simple set up adjustment possible. Materials science diffractometers are available meanwhile at all large scale facilities express the high demand for these investigations including bio-, nano- and light weight and energy storage materials. Instruments for energy dispersive methods and for angular dispersive methods are available for neutron and hard X-ay diffraction.

4.1. The material science diffractometer Stress-Spec at FRM II/Garching-Germany

Stress-Spec the materials science diffractometer at the Forschungsneutronenquelle Heinz Maier-Leibnit FRM II at Garching, is dedicated for combined analysis for residual stress investigations and crystallographic textures. Specifications are high neutron flux, monochromatic beam, very flexible in wavelength set up, heavy basement for residual stress investigations, Eulerian cradle for standard texture work and a Stäubli RX160 robot for texture and strain mapping as well as for sample changing. For more details in instrumentation, basic principal and examples see Hofmann et al. [13] for strain measurements and Brokmeier et al. [14] for texture measurement.

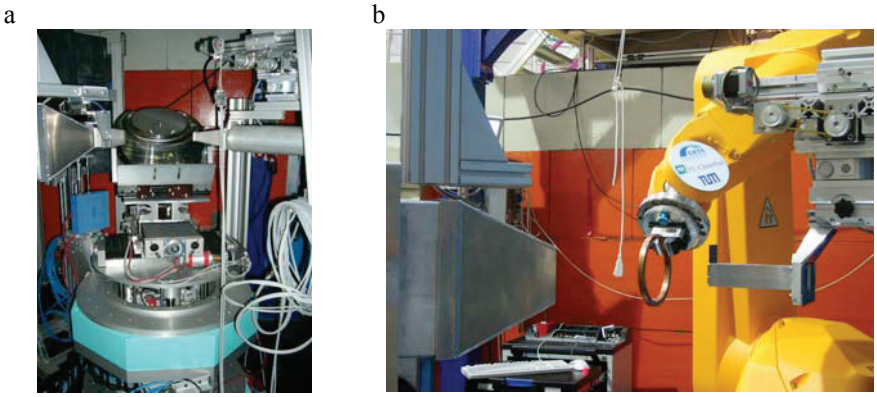

Fig. 4. (a) Stress.Spec sample stage for stress analysis (b) Stress-Spec robot manipulating a Cu-ring for texture analysis

4.2. The HZG-material science diffractometer Harwi-II@Doris III/Hasylab-Hamburg/Germany

The high energy materials science beamline Harwi-II is equipped for synchrotron tomography and diffraction. A high photon flux up 120 keV, a very flexible sample stage for various set ups, different types of area detectors and a great variability in sample to detector distance (0.5m – 10m) allow to optimize the beamline for high resolution scattering (strain mapping), for fast experiments (in situ

investigation), for industrial application (up to 600kg weight). An essentials part of total beamtime is for residual stress-strain and crystallographic texture measurements. Due to the characteristics of the storage ring Doris III Harwi II (length of 289m) has a medium brilliance which make Harwi II ideal for medium grain size materials for stress profile studies and texture gradient investigations with beams between 500x500µm² and 2x2mm². Moreover, Harwi II offers a number of equipments for in situ measurements such as 100 kN and 20 kN loading device, furnaces, friction stir welding device (FLEXISTIR) and dilatometer.

4.3. The HZG- material science diffractometer HEMS@Petra III/Hasylab-Hamburg/Germany

The upgraded storage ring Petra III (length of 2304m) has a much higher brilliance than Doris III. Higher brilliance results in higher photon flux even for higher energies. In addition to Harwi II, higher brilliance results in much higher resolution and faster measurements. Therefore, different experiment hutches are installed firstly with a general purpose diffractometer for strain and texture mapping of fine grained specimen, secondly an experiment station having a heavy load hexapod up to 1t for heavy industrial samples and thirdly a 3D-XRD microstructure mapper on the single grain level [15].

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