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In-situ analysis of crystallographic texture using high-energy X-rays

H.-G. Brokmeier[1,2,*], S.B. Yi[2], B. Schwebke[1,2] and J. Homeyer[3]

[1] Institute of Materials Science and Engineering TU Clausthal, Agricolastr. 6, D-38678 Clausthal- Zellerfeld
[2] GKSS Forschungszentrum, Max-Planck-Str. 1, D-21502 Geesthacht, Germany
[3] Hasylab at Desy, Notkestraße 85, 22607 Hamburg, Germany
[*] brokmeier@gkss.de.

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Abstract. Hard X-rays with energies higher than 50 keV are characterized by there high penetration length in most materials. In the case of 100 keV to 200 keV X-rays the penetration length is in the same order as for thermal neutrons. Texture development belongs to one of the major characteristics of materials processing such as deformation and recrystallization. Using two examples a brief description is given for in-situ experiments carried out at the high energy beam line BW5. In contrast to in-situ strain measurements texture measurements are time consuming, so that the number of this type of experiments is still low. In order to describe the influence of the initial texture on the materials anisotropy texture simulations (VPSC model) and in- situ texture analyses were combined. Loading experiments comparable to standard material tests (stress-strain-curve) were carried out on magnesium-alloys in tension as well as in compression using a 20 kN loading device. Due to the high penetration length of 100 keV X-rays DIN 50125 samples of 6mm in diameter were used in transmission mode. In addition to standard texture measurements at the initial state and after failure up to five points at the known stress strain curve were chosen for in-situ texture measurements. An image plate detector covers a set of complete Debye-Scherrer cones, so that only ω-scans are necessary to get complete pole figures and the whole experiment is fast. It is even fast enough to perform high temperature experiments, which are very sensitive time dependent reactions. Two types of furnaces are available, one which can be combined with the loading device and a stand alone furnace. In many materials a phase and texture transition takes place during processing. The second example describes our first in-situ high temperature experiment on a steel sample to investigate the phase and texture transition (ferrite – austenite).

Introduction

The classification of high energy X-rays ranges from about 50 kev up to 450 keV. With increasing keV the penetration length increases also. Standard material testing devices operate from 120 keV to 450 keV due to their application field for non-destructive testing (NDT),

such as failure analysis in pipelines, quality control in wheel rims etc., A much higher photon flux combined with a fantastic brilliance have photons produced in a storage ring. Compared to material testing tubes with its main application for radiography or tomography, synchrotron radiation was used intensively for diffraction experiments [1-3]. In the case of 100 keV to 200 keV X-rays the penetration length is in the same order as for thermal neutrons, which are well known in materials science applications (texture and strain analyses) as high penetrating radiation. Table 1 shows a comparison of the penetration length for some common metals of three typical radiations (CuKα – 1,5418Å, 100 keV synchrotron radiations – 0,124Å, 200 keV synchrotron radiation – 0.062 Å) [4]. The penetration length is given in cm for a loss of 50% of primary beam intensity.

Table 1: Penetration length of different radiations for some metals

	Cu-Kα 1.54 Å	100 keV synchrotron 0.124 Å	200 keV synchrotron 0.062 Å
magnesium	0.0140 cm	3.4 cm	4.2 cm
aluminium	0.0053 cm	1.5 cm	2.7 cm
copper	0.0015 cm	0.2 cm	0.5 cm
titanium	0.0011 cm	0.6 cm	1.3 cm
lead	0.0003 cm	0.03 cm	0.04 cm

In engineering materials science characterization is mainly done by stress strain curves, by micro hardness measurements and by analysis optical microstructures. One goal to implement diffraction methods in these material characterizations is to work with identical sample geometries prescribed by an industrial norm. Typical tensile samples are in the cm-range, so that on one hand a high penetrating beam is required. On the other hand the reactions inside the sample are fast during thermal treatment or during tension, so that fast measurements are necessary. Therefore, high energy X-rays from a storage ring are excellent for in-situ studies of strains and textures, for temperature dependent phase transitions and recrystallization phenomena.

Experimental equipment

The working horse for the in-situ experiments, presented in this paper, was the high energy beam line BW5 at Hasylab - DESY. A new materials science beamline (Harwi II) operated by the GKSS-Research Center will complete the high energy instrumentation at Hasylab - Desy. Using a two-dimensional image plate detector (MAR345), one second is a typical exposure time for one shoot. Due to the high energy and the low wavelength one can work with small scattering angles. Consequently a set of complete Debye – Scherrer cones are detected simultaneously as shown in figure 1 for a powder sample. In figure 1a one can see an image plate picture of Mg taken in 5 sec. covering a 2-θ range of 6°. The powder pattern includes 15 complete Debye-Scherrer cones, which were integrated and plotted in a sum-diffraction spectrum. An excellent agreement between measured and calculated diffraction pattern was obtained. It should be noticed that a glass cylinder of 8 mm in diameter was filled with Mg-powder without any preferred orientation.

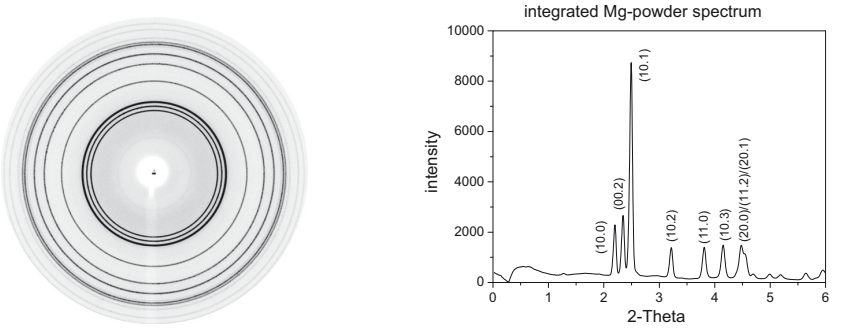

Figure 1. Powder pattern of a Mg-powder a) Image plate picture taken in 5 sec b) Sum spectra integrated over complete Debye- Scherrer cones

BW5 is flexible in changing the energy between 50 keV and 110 keV as well as the sample to detector distance in a wide range. In our case, we have used mainly a sample to detector distance of about 100 cm and a wavelength of 0.124 Å. In order to perform in-situ experiments the sample stage was completed by a loading device and/or a furnace. The set up including the loading device mounted on a rotating table and an image plate detector is shown in figure 2.

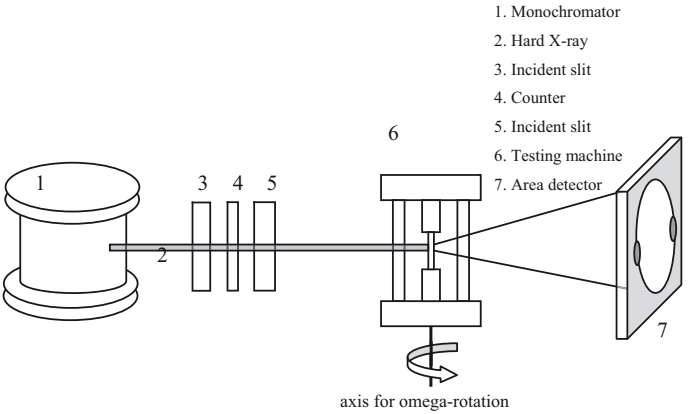

Figure 2. Set up of the synchrotron instrument including a loading device at sample position

The loading device has a maximum capacity of 20 kN in tension and in compression [5]. Both types of test samples round with threads of 4mm, 6mm, 8mm and 10mm and flat samples are in use. In parallel to the loading experiment the extension and the loading power were registered. Three different types of furnaces have been tested. First a mirror furnace of ILL-style with a temperature up to 1600°C was tested [6]. Due to the specification for neutron diffraction at TEX-2, this furnace works but was not optimal. A second furnace shown in figure 3b has a glass dome and a heating system up to 1000°C. The light inside the furnace

results from heating a steel sample in the FCC region. A third furnace (see figure 3c) can be combined with the loading device shown in figure 3a. These equipments allow us to investigate lattice thermal expansion, lattice dependent strains, phase transformations, residual and thermal strains, microstructure evolutions and texture evolutions.

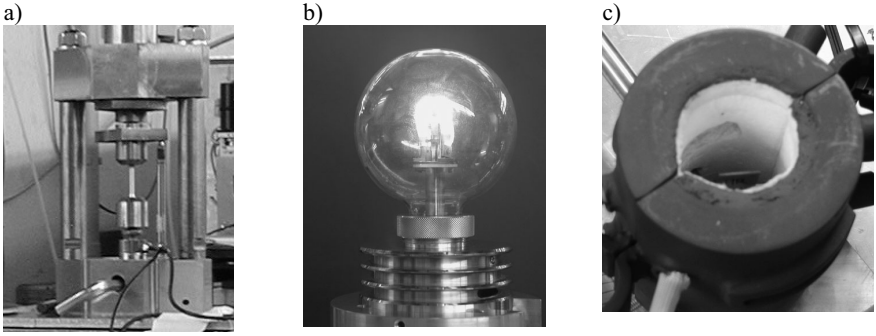

Figure 3. Equipments for in-situ experiments a) 20 kN loading device b) dome furnace c) cylindrical furnace for the loading device [7]

Texture evolution during tension

Depending upon load the tensile sample is in the elastic, the elastic-plastic or the plastic region. Macroscopically one can recognize the elongation during tension or the shortening during compression. Figure 4 shows three stress-strain curves of a bar extruded Mg-alloys. The three tensile samples were cut in 0°, in 45° and in 90° to the extrusion direction with 6mm in diameter. Consequently, the crystallographic textures of the three samples before tension were different. In order to describe the texture evolution, the resulting materials properties and the deformation history, the texture was measured before failure, at four positions at the stress strain curve and after failure.

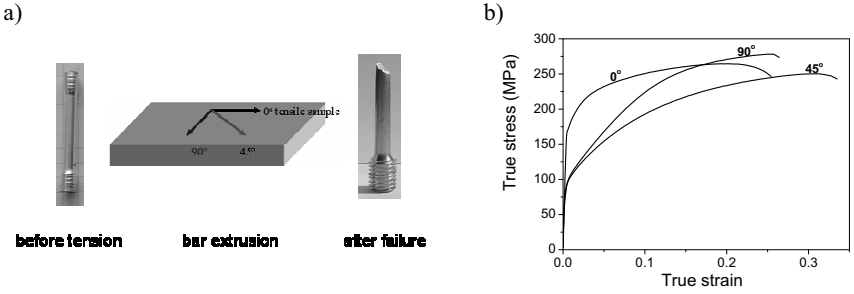

Figure 4. a) Texture samples b) Stress strain curves for 0°, 45°and 90° to extrusion direction

Image plate pictures as shown in figure 1a give only a small part of each pole figure, as already described at earlier times for film techniques [8], the first area detectors. 19 rotations around ω (see figure 2) describe a complete pole figure in about 45min -60min. Together with a texture simulation using the viscoplastic self consistent method (VPSC), the influence of the initial texture on the activation of different glide systems and on twinning was calculated. Detailed description of investigations on Mg-AZ31 and on Mg-AM20 were published elsewhere [9, 10]. Based on this results new measurements are planed for in-situ high temperature loading and for cyclic loading. Moreover, a new 100 kN loading device will be installed in future at the GKSS-high energy beam line Harwi-II.

Texture evolution during heating

The majority of all processing lines include high temperature treatment such as recovery, recrystallization or phase transition. Thus high temperature phase analysis by X-rays or neutrons is very common. Due to a much longer total counting time high temperature texture analysis is restricted. New instrumentations on high flux beams offers the possibility to measure complete textures in less than 60 min and in the case of high pole figure symmetries in 60 sec. The furnace shown in figure 3b was used to measure textures of shape memory alloys and in steel. In the case of the steel experiment a set of only 9 image plate pictures with different ω were taken for the different temperatures with 1 sec. exposure time for each picture. The reduced number of image plate pictures is possible because of the orthorhombic texture symmetry. The program package MAUD (Materials Analysis Using Diffraction) [11] offers on one hand the determination of crystallographic and microstructure data (volume fraction, grain size and strain) using a Rietveld refinement and on the other hand a quantitative texture analyse using a WIMV texture program. Thus we were able to determine the thermal expansion coefficients of α-ferrite, the phase transition (α-ferrite to γ-austenite), the volume fractions in the two phase region and the texture transition. In figure 5 an example is shown for the determination of the volume fractions and the lattice constants in the two phase region α-ferrite/ γ-austenite [12]. Moreover, the texture transition of α-ferrite at room temperature to the high temperature phase γ-austenite was shown. The texture agrees with the Kurdjumov-Sachs orientation relationship for BCC ↔ FCC transformation, $(110)_{BCC}$ //$(111)_{FCC}$.

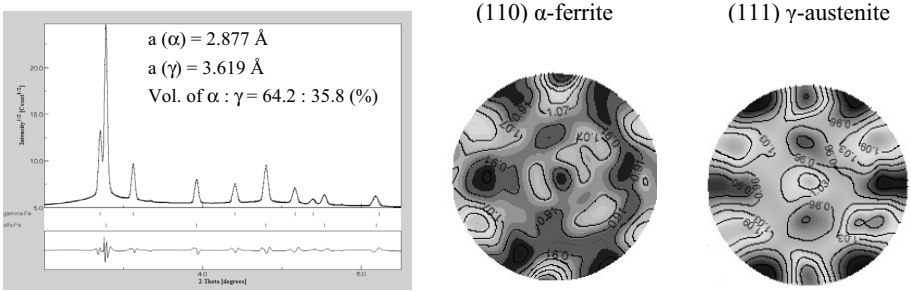

Figure 5. High temperature investigations on steel; lattice constants and volume fractions of α-ferrite and γ-austenite in the two phase region; (110) pole figure of α-ferrite at room temperature compared (111) pole figure of γ-austenite at high temperatures.

Concluding remarks

The combination of the high energy X-rays (50 keV – 200keV) with a loading device or a furnace offers excellent possibilities to perform in-situ experiment in a rather short timescale. Examples on magnesium have demonstrated that in-situ texture analysis give important input for texture simulations and flow field calculations. To verify texture simulations not only for the texture type but also for the texture sharpness and to calculate materials properties in-situ experiment can be important. Further instrumental improvements for in-situ high temperature loading, for cyclic loading and for a 100 kN instrument at Harwi-II (Hasylab) will offer new possibilities. Fast texture measurements using a furnace also allow studies of texture transitions in-situ. Combined Rietveld refinement and quantitative texture analysis lead to texture corrections in quantitative phase analysis and to quantitative texture results for all temperatures up to 1000°C. Taking the pole figure symmetry into account we were able to analyse the quantitative texture of ferrite and austenite in the two-phased region in about 10 min. Further developments of new furnaces and faster detectors will provide a great potential for in-situ experimentation using high energy X-rays.

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