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Combined materials characterization by area detector investigations using hard X-rays

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Abstract. Materials characterization includes a long list of information’s which are on one hand important for process optimizations and on the other hand key knowledge for application, lifetime and failure predictions. With the help of area detector investigations one is able to get simultaneously all information related to line position, line intensity and line broadening of Bragg reflections. Examples are residual stress profiles, quantitative texture mapping, diffraction elastic constants, lattice dependent stress-strain curves, thermal expansion coefficients and defect densities. A very special case is high energetic synchrotron radiation which allows very fast and complete detection of generalized pole figures, namely intensity pole figures (texture), line broadening pole figures (micro-strain) and line position pole figures (macro-strain) using complete Debye-Scherrer rings.

1. Introduction
It is known that complete diffraction patterns contain a lot of information’s. New technologies in experimental facilities and data analysis open many fields for combined materials characterization. Materials characterization in terms of engineering science and applications means all kinds of structure information needed to predict or explain materials behaviour, critical limits in processing and application and failure analysis [1,2]. Materials characterization by diffraction methods is intensively used since many years. Standard is the measurement of phase composition and phase transition as well as texture and residual stress analysis by X-rays. Less often microstrain and domain size investigations have been reported [3,4] as well as the determination of materials constants such as the diffraction elastic constants, thermal expansion coefficients and lattice dependent yield points. But for all investigations one needs a specific instrumental set-up which optimized for some of the investigations and longtime one uses different instruments for texture and residual stress analysis. The availability of area detectors opens the field to get much information simultaneously. Together with high brilliant synchrotron sources a dynamic materials characterization or a mapping of information becomes possible in reasonable time. Among laboratory X-rays and neutrons, hard X-rays with energies > 50 keV are in use.

2. Hard X-rays
Great advantage of hard X-rays with energies up to 200keV is, among others, the low scattering angle, which means comparably small area detectors can be used to get a set of complete Debye-Scherrer cones without sample movement. Figure 1 shows a typical area detector image with a set of complete Debye-Scherrer rings. As one can see, two different types of information can be extracted. First is the intensity distribution along the 2-θ scale, so that one gets a sum diffraction pattern as integration over complete Debye-Scherrer rings or a set of individual cake diffraction pattern. The sum diffraction pattern is mainly used to identify the quantitative phase distribution and determine crystallographic structures. Cake diffraction pattern give much more details related to specific sample orientations. This may come from sample processing such as rolling with typical mechanical test directions at 0°, 45° and 90° to RD (RD – rolling direction) or by in situ loading tests with cake diffraction

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patterns with $\text{LD} \parallel \text{RD}$ or $\text{LD} \perp \text{RD}$ ($\text{LD} – \text{loading direction}$). The hkl-dependent information contain in much better way the anisotropic behavior of polycrystalline materials. A second kind of data extraction is done to obtain the intensity distribution along Debye-Scherrer rings. This intensity distribution is used to calculate generalized pole figures. Mostly known is the peak intensity pole figure named as crystallographic texture pole figure. With line broadening and peak position pole figures, which can be obtained simultaneously, additional informations are available directly, comparing macro- and micro-strains with texture components.

![Indexing of a diffraction pattern](image)

**Fig. 1:** Various information obtained simultaneously from area detector images

For low scattering angles less than $5^\circ$ in 2-$\theta$ the intensity distribution of the Debye-Scherrer rings are very close to the outer great circle. That has the great advantage that for generalized pole figure scanning only sample rotation is needed but no sample tilt. Another advantage is that one gets from area detector images diffraction patterns to use the $\sin^2 \psi$ – method for in-plane residual stress analysis. Figure 2 shows an area detector image of a rolled Al sample.

![Area detector image of Al (left), intensity distribution of the first Debye-Scherrer ring in pole figure plot (Al (111) – middle), $\omega$-rotation to cover complete pole figures](image)

**Fig. 2:** Area detector image of Al (left), intensity distribution of the first Debye-Scherrer ring in pole figure plot (Al (111) – middle), $\omega$-rotation to cover complete pole figures
For FCC materials, the first Debye-Scherrer ring with the lowest 2-θ value is the (111) reflection. The intensity distribution of the (111) reflection in the pole figure plot indicates already same typical information of the texture. In principle this sole ring on the different images is enough to use sin²ψ – method for residual strain analysis. In this special case the texture is so strong to get sufficient results. In the right figure one can see that an ω-rotation is enough for complete pole figure coverage. Step size in ω must be related to the accuracy of the pole figure. Due to the high brilliance of synchrotron radiation, measurements with ω-rotations of 1° or less can be carried out very fast without problem.

3. Examples
All Experiments were carried out at the high energy beamline HEMS P07B at Petra III/DESY-Hamburg. With energies >80keV sample from mm to cm thickness can be measured in transmission mode using the pin hole method in 1 to 5 sec.

3.1. In situ tensile test on of Ti2.5Cu
In situ experiments on Ti2.5Cu have been used to measure on one hand the texture evolution during tensile deformation and to determine on the other hand polycrystalline X-ray elastic constants, which are microstructure and texture dependent [5]. A loading device, see figure 3 left, has been used to measure continuously diffraction patterns during tensile load. Result was a set of 193 diffraction patterns. The colours in figure 3 middle represent 4 different areas of the stress-strain curve. A typical MAUD-fit of one cake diffraction (±5° || to tensile direction) is shown in figure 3 right.

Fig. 3: In-situ loading (left), loading curve (middle), MAUD-fit of one pattern (right)

The analysis of the elastic area (black points) was carried out to the get lattice dependent diffraction elastic constants (DECs), hkl dependent yield points and Poisson ratios as well as load dependent change of the c/a ratio. As expected the polycrystalline DECs differ from pure Ti and influence the residual stress calculation. A variation of the lattice constants and the c/a ratio between LD and TD indicate the pre-residual stress of the sample originated by processing. The texture undergoes a strong texture change which is discussed in more detail by Maawad et al. [5], see figure 4.

Fig. 4: Ti pole figures before and after load
3.1. Intensity and line broadening pole figure of F138 steel.
The material is F138 austenitic stainless steel, a kind of AISI316LVM, with a moderate strong preferred orientation [6]. For the first test the standard set up for pole figure measurement was used. As shown in figure 1 left from each area detector image one gets easily 360 diffraction patterns, totaling 13320 diffraction patterns in case of 5° rotation in ω. To extract intensity pole figures and line broadening or full width of maximum pole figures (FWHM) we have used STECA [7]. STECA is a software package looking on individual reflections. A key problem is the intensity statistics for strongly textured samples. Due to pole figure areas with less scattered intensities the error of FWHM determination increases. One way to overcome this problem is to improve the counting statistics and another way is to use only those pole figure areas having high enough intensities.

![Image of intensity pole figure and FWHM pole figure](image)

Fig. 5: Area detector image of F138, intensity pole figure, FWHM pole figure

4. Conclusion
Area detector experiments with all kinds of radiation show a high potential to make combined materials characterization [8,9,10]. Key point for these studies is firstly a perfect characterization of the used instrument, which is only roughly needed for texture research only. Second is that combined interpretations require well known gauge volume and sufficient statistics in intensity as well as in detector resolution. In two examples it has been shown that the anisotropy of the materials dominated by texture and microstructure can be understood much better if a combined analyses is done.

References