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Combined X-ray Micro-Diffraction, Radiography and Tomography Analysis of Solid Objects. Detlef Beckers[a], Gabriel Blaj[a], Herbert Pöllmann[b], Klaus Bethke[a], Roger Meier[a], [a]*PANalytical, Almelo, The Netherlands,* [b]*Martin –Luther University of Halle, Mineralogy, Germany*
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In this contribution, we will show examples of the strength of the combination of X-ray (micro-) diffraction, X-ray imaging and Computed Tomography (CT) results on solid objects. New developments – especially in detector technology – allow integration of these techniques on one laboratory X-ray diffraction system. By combining phase analysis with density and microstructure information a complete picture of the sample is obtained. Results can be correlated with macroscopic material properties. Examples from building materials, natural objects and pharmaceutical tablets are shown.

Keywords: Computed tomography, imaging, X-ray diffraction

FA5-MS40-P02

Development of a high brilliance rotating anode dual-wavelength X-ray generator and multi-layer mirror for dual-wavelength. Akihiko Iwata [a,c], Claire Wilson [a], Akihito Yamano [b], Masataka Maeyama[b], [a]*Rigaku European Headquarters, Berlin Germany* [b]*Rigaku Corporation, Tokyo Japan,* [c]*Kyoto University, Kyoto Japan*
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Due to growing demand for the use of different characteristic X-ray wavelengths such as Cu K-alpha and Mo K-alpha for crystal structure determination, dual X-ray source (DS) diffractometers have been widely used lately. However, DS diffractometers use sealed X-ray tube technology, and the X-ray intensity is limited with sealed tube X-ray generators. Still demands for using high intensity and different wavelengths X-ray for structure determination for very tiny crystals and low diffraction efficiency crystals exist. Since the middle of the twenty century, many challenges in developing a rotating anode dual wavelength generator have been overcome to achieve high intensity different characteristic X-ray wavelengths from a single X-ray generator. [1]
We have developed a new rotating anode X-ray generator which is capable of generating two different characteristic X-ray wavelengths without target exchanges. The electron beam can excite the two different target materials plated on a single rotating anode, as the anode can move in the X-ray tube housing under vacuum atmosphere. The newly developed optics unit consists of two confocal multi-layer mirrors, one for Cu K-alpha and the other one for Mo K-alpha, mounted in a single optic component face to face. The optics unit has a unique mechanism to switch between the two different characteristic X-ray wavelengths, details of which will be presented.

[1] Taylor, A.: Journal of Scientific Instruments. 26 (1949) 225.

Keywords: single crystal structure determination, dual-wavelength, high brilliance X-ray generator

FA5-MS40-P03

How tiny is a small crystal today? Holger Ott[a], [a]*Bruker AXS GmbH, Karlsruhe, Germany*
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Instrumentation for single crystal structure analysis has advanced greatly over the last decade. These improvements include the introduction of area detectors, in particular CCD detectors with high dynamic ranges; as well as major software improvements.
For a number of years now systems using a flat graphite monochromator combined with a sealed tube source have been routinely used in laboratories in the western hemisphere. Although rotating anode generators using this monochromator were sparsely installed, they do allow for the measurement of smaller and more weakly diffracting crystals.
The introduction of multilayer mirrors opened the door for rationally designed X-ray optics. Originally these optics were available for wavelengths in the range of 1.54 Å or longer. More accurate control of the deposition process enabled mirrors for shorter wavelengths, down to 0.5 Å. These optics are widely used in microfocus X-ray sources and rotating anode generators using a variety of wavelengths and allow for measurement of tiny crystals. When allied with a high quality goniometer, exhibiting a small sphere of confusion, and a high-end CCD detector the minimum crystal size needed is pushed even further.
The presentation will focus on the recent progress in source development and suggest most suitable source-wavelength combinations based on carefully selected examples.

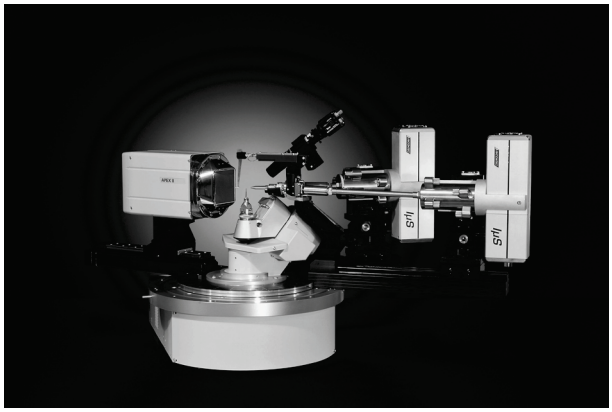

Figure 1: APEX DUO equipped with two micro focus sources

Keywords: Crystal size, Microfocus source, Single-crystal structure

FA5-MS40-P04

On the peak shapes of X-ray micro diffraction. Hejing Wang[a], Jian Zhou[b], [a]*School of Earth and Space Sciences, Peking University, P. R. China,* [b]*Chinese Academy of Geological Sciences, P. R. China*
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Since 1969 Rietveld [1] used the Gaussian function to model X-ray diffraction (XRD) peaks it has been a very common

work for XRD analysis using mathematic functions to simulate the XRD peaks and to find the solution for structures. This work is based on i) an XRD peak is consisted of five basic elements [2] such as, peak position, maximum intensity, full width at half maximum, shape coefficient and asymmetry, and the integrated intensity and integrated width can be produced from them; ii) an XRD peak can be well described by those functions, nowadays, such as: the Voigt, the pseudo-Voigt and the Pearson VII functions. Comparing the experimental peaks of normal XRD (line source) with those of micro XRD (point source) it reveals, for the sake of five basic elements, that i) the intensity from micro XRD is as 20-time strong as that of normal XRD on the equivalent conditions, e.g. the same sample from the same illumination area and ii) the resolution of micro XRD is as half low as normal XRD analysis, iii) the peak shapes of micro XRD are super wide and round at top and super narrower at bottom, iv) there is no difference in position between normal and micro XRDs and v) nearly symmetry in shape of the micro XRD peak on the equivalent optic conditions. In the normal XRD system, the fixed divergence optic path causes a different illumination length (area) on lower and higher diffraction angle sides of a reflection that leads the different intensities on two sides of a reflection and thus induces the asymmetry of a peak. Due to nearly no divergence the micro diffraction system produces an equal illumination on both lower and higher diffraction angle sides of a reflection and results in symmetry peak in shape. Depending on the convergence optics the highest resolution of normal XRD reaches a minimum width of 0.045 °Δ2θ, e.g. the resolution of the X'Pert Pro diffractometer. However, for the micro diffraction system, it is not the convergence optics and thus reduces the resolution of a peak. It is deduced from the shape characteristics of a micro diffraction peak that a band of quartz's reflections (212), (023), (301) and their $K\alpha_2$ doublets from normal XRD (in the range of 67-69 °2θ Cukα radiation and often shows five independent peaks) will merge into corresponding four or three or two or even one micro diffraction peak if the peak width of those quartz reflections from normal XRD be >0.0777 or >0.0836 or >0.0861 or >0.1625 °Δ2θ respectively. This is verified by quartz sample measured with the micro diffraction system. It is concluded that the micro diffraction peak possesses a super symmetric Gaussian distribution in shape (shape coefficient Sc_{micro}=1.015; note: Sc_{normal}=0.63-0.94) and with a double equivalent width whilst the normal XRD peak is of an asymmetric shape in different variations from the Gaussian extreme to the Lorentzian extreme mostly.

[1] Rietveld, H.M., *J. Appl. Cryst.*, 1969, 2, 65. [2] Wang, H., Zhou, J., *J. Appl. Cryst.* 2005, 38, 830.

Keywords: peaks, microdiffraction, function

FA5-MS40-P05

Simultaneous imaging of radiographic and crystallographic information.

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Real structure characterisation of bulk single crystals or large grain polycrystals is often made by X-ray diffraction, in which the intensity distribution of the diffracted beam is imaged in back-reflection. The X-ray topographic method delivers information about the distribution of crystals defect in the surface and over a small depth within the sample. In many cases it is also important to investigate the real structure over the whole volume of a single crystal (dislocation density and distribution, small angle grain boundaries, lattice plane distortion, macroscopic and microscopic inclusions). For this transmission methods are suitable only. Due to the typical thickness of a sample hard X-rays or neutron beams can be used only. If a high spatial resolution is desired then it is appropriate to use hard X-rays emitted from a micro or “nano” focus X-ray tube. With this it is possible to observe macroscopic and microscopic defects by X-ray projection microscopy or by micro-computer tomography (µCT). On the other hand, the diffracted hard X-radiation delivers real structure information from the inside of the crystalline sample. A report is given about the parallel measurement of diffraction and shadow microscopy information using divergent hard X-rays [1]. As part of this, the equipment and its components as well as the production of images will be discussed, along with some highlighted measurements. We thank the Deutsche Forschungsgemeinschaft for their sponsorship.

[1] Bauch J., Ullrich H.-J., Böhling M., Lupascu D.C., Deutsche Patentschrift DE 10 2008 008 829

Keywords: X-ray, XRD in transmission, real structure imaging

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Automatic detection and analysis of conic X-ray diffraction lines.

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The presented method demonstrates a first step in the development of a high resolution "Residual stress microscope". It has been implemented but is not exclusively used for the KOSSEL technique and the “X-ray Rotation-Tilt Method” (XRT). Through the implementation of as far as possible automated procedures the presented method allows rapid access to a diverse evaluable data base of many X-ray diffraction images. Thus, there is now the possibility of systematic studies of materials science based basic phenomena, such as smeared or double reflection maxima and local maxima along a diffraction line. An essential component is the fully automatic detection of these reflections in form of conic sections (quadratic curves). This is done with the involvement of modern methods of digital image analysis and processing, for instance the 3D Hough transform. In addition to the detection of the inexact location of diffraction lines, there is also the registration of reflection micro structure with subpixel accuracy and other curve parameters with associated adjustment calculus. The thus obtained data can be used, inter alia, for the calculation and output of the precision strain- and residual stress tensor. We thank the Deutsche Forschungsgemeinschaft for their sponsorship.

[1] Bauch J., Brechbühl J., Ullrich H.-J., Meinl G., Lin H., Kebede W., *Cryst. Res. Technol.* 34(1999)1 [2] Bauch J., Wege S., Böhling M., Ullrich H.-J., *Cryst. Res. Technol.* 39(2004)7 [3] Maurice C., Fortunier R., *Journal of Microscopy*, Vol 230, Pt 3 (2008)