

## MS43 Combining x-ray diffraction and other techniques for in situ and operando studies

Chairs: Helmut Ehrenberg, François Fauth

### MS43-P1 Advances in the X-ray Rotation-Tilt Technique

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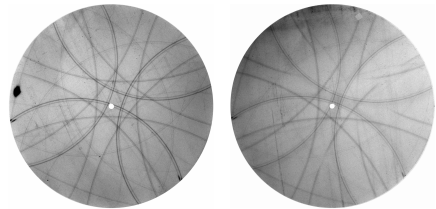
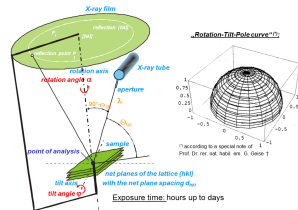
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The X-Ray Rotation Tilt technique (XRRT) [1-2] is an X-ray microdiffraction method in which X-ray interference patterns are produced by directing a monochromatic X-ray or synchrotron beam of high intensity and small diameter onto a monocrystalline region of a sample. Each diffracting lattice plane defines a set of concentric cones with a half opening angle equal to  $90^\circ - Q_{\text{hkl}}$ , whereas  $Q_{\text{hkl}}$  denotes the BRAGG angle. If the incident monochromatic X-ray beam coincides with a generatrix of the interference cone, then the diffracted line on the diametrically opposite side of the cone can generate a small spot on a detector. By moving the sample in a specific way (several rotation-tilt movements whilst the rotation of the sample and detector is constrained to one synchronous rotation around a common axis) it is possible to accumulate such spots on a detector system (e.g. X-ray film, image plate or CCD area detector) which faces the sample (Fig. 1 top). These reflections in entirety form the so-called XRRT lines on the detector. Fig. 1 (bottom) shows an example of an XRRT pattern.

This technique can be applied to a wide range of analytical problems in materials diagnostics, e.g. high-accuracy determination of crystallographic orientations and lattice constants, determination of dislocation density and decrease in symmetry, precision determination of residual stress/strain and phase identification in micro regions. The applicability of the method will be demonstrated by highlighting the results of some selected examples measured on functional materials. For example, the measurement of the transition from the cubic high temperature phase of Barium titanate to the tetragonal room temperature phase shows clearly a well-marked decrease of symmetry in the XRRT-Patterns in Fig. 1 (bottom). By using a new tool based on focal curves it is now possible to automatically determine the orientation and position of the sample from one single pattern. In this example, it permits the determination of the lattice constants of the two  $\text{BaTiO}_3$ -Phases and the direction of the strain during the structural phase transition (Fig. 1 bottom right). This new tool also facilitates the evaluation of XRRT-Patterns of more complex crystal structures, where a manual indexation of the reflections is usually not feasible.

[1] Ullrich, H.-J.; Greiner, W.: Patent WP 139 671, Berlin 1977

[2] Bauch, J.; Ullrich, H.-J.; Reiche, D.: Acta Cryst. Vol. A55 (1999), Supplement



**Figure 1.** Principle of the X-ray-Rotation-Tilt Method (top). XRRT pattern of  $\text{BaTiO}_3$  at  $20^\circ\text{C}$  (bottom left) and at  $130^\circ\text{C}$  (bottom right). The white dot marks the rotation center which is close to the 100-pole.

**Keywords:** X-ray Microdiffraction, X-ray Rotation-Tilt Technique, crystallographic orientation, lattice constants, dislocation density, residual stress/strain