



Application Report XRD 35

D8 DISCOVER Plus

- High-Resolution Powder Diffraction for Lattice-Distortion and Micro-Structure Analysis

The D8 DISCOVER Plus equipped with the ATLAS goniometer, high-efficiency turbo X-ray source (TXS-HE) and TRIO optics is a versatile solution for high-resolution powder diffraction. Users benefit from the outstanding goniometer accuracy as well as from the intensity of the 6 kW rotating anode in combination with the two-bounce primary monochromator that provides pure $K\alpha_1$ peak profiles.

High intensity, parallel beam geometry in reflection mode is useful for measuring samples with uneven surfaces or sample height displacements (e.g. non-ambient or sample preparation induced) which can shift diffraction patterns to high or to low angles

when using focusing geometry. The parallel-beam geometry is insensitive to sample height displacement and therefore perfect to accurately determine lattice parameters from peak positions. This is a prerequisite for the accurate study of lattice distortions in crystalline materials.

The below example demonstrates the ability of the D8 DISCOVER Plus to collect high-resolution powder diffraction. We measured extremely small lattice distortions in the order of 0.01%. Data from different samples can directly be compared because potential sample preparation artefacts are fully eliminated by the parallel beam geometry.

Microstructure of Undoped and Lithium Doped Lead Stannate

Current research aims at understanding how stereo-chemically active lone electron pairs influence structure/property relationships in stannates, that are considered model substances for the study of long after-glow phosphores.

Samples of undoped and Li-doped lead stannate (Pb_2SnO_4) were measured with a D8 DISCOVER Plus, equipped with the TXS-HE rotating anode X-ray source and the primary TRIO optics. A Göbel mirror and 2-bounce Ge channel-cut monochromator delivered pure $\text{K}\alpha_1$ radiation for high-resolution powder diffraction. More experimental details are found in Table 1.

Geometry	Parallel beam, 560 mm
Generator	45 kV, 120 mA
Radiation	Cu, $\text{K}\alpha_1$
Detector	LYNXEYE XE
Divergence Slit	0.1 mm
Soller Slits	1.5°
Scan Range	12 – 140° 2Theta
Step Size	Variable
Time/Step	Variable

Table 1: Experiment conditions of the rotating anode D8 DISCOVER Plus diffractometer.

Lead stannate and its Li doped derivative both crystallize in the orthorhombic space group Pbam . The unit cell is pseudo-tetragonal with $a \approx b > c$. Lattice parameters were determined from the full diffraction patterns by fitting an hkl-phase in DIFFRAC.TOPAS V5 (Figure 1).

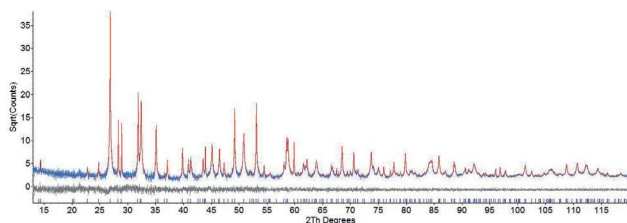


Figure 1: DIFFRAC.TOPAS chart showing very good agreement between the hkl-model fit and experimental data on sqrt-intensity scale. No deviations are found larger than the statistical noise as indicated by the difference curve in the lower part.

Microstructure parameters such as crystallite size and micro-strain were derived applying the Fundamental Parameters model and convolution based peak profiles in the double Voigt approach as implemented in TOPAS. Anisotropy of the peak half-widths was considered by the Stephens model for orthorhombic symmetry. Results are summarized in Table 2.

	Pb_2SnO_4	$\text{Li:Pb}_2\text{SnO}_4$	Lattice distortion $ \Delta d/d $ / %
a / Å	8.7375(3)	8.7391(4)	0.018
b / Å	8.7411(3)	8.7419(4)	0.009
c / Å	6.3100(2)	6.3106(2)	0.008
Crystallite size, LVol-IB / nm	311(27)	344(29)	
Micro-strain, ϵ_0 / %	0.018(1)	0.016(1)	
Axial components of the Stephens strain anisotropy			
S400	3(1)	26(5)	
S040	129(29)	283(44)	
S004	0.5(6)	3(2)	

Table 2: Lattice and microstructure parameters of lead stannate (Pb_2SnO_4) and Li doped lead stannate ($\text{Li:Pb}_2\text{SnO}_4$).

The intercalation of Li into the crystal structure of lead stannate results in an expansion of the unit cell. The related lattice distortion is about twice as large in a-direction compared to the b- and c-directions, respectively. Within the estimated standard deviations the intercalation does neither influence the crystallite size nor the micro-strain significantly. Both crystal structures exhibit significantly anisotropic micro-strain, which is dominant along the b lattice direction (see Figure 2). Apparently, the Li intercalation increases the strain anisotropy. That effect does not result in a related lattice relaxation along the b direction.

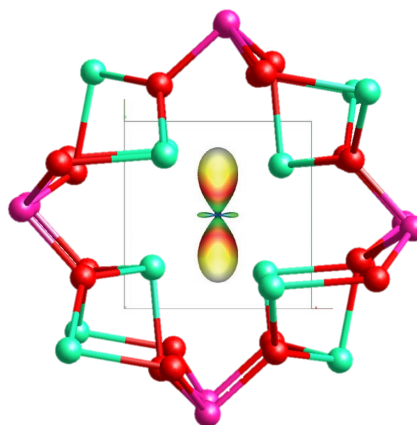


Figure 2: Ball and Stick structural model of lead stannate, Pb_2SnO_6 , projected on the ab lattice plane. Overlaid in the same orientation is the normal plot of anisotropic strain as refined in DIFFRAC.TOPAS V5.

Acknowledgement: Thanks are due to Prof. Bjoern Winkler and Dominik Spahr from the Crystallography Institute of the Johann Wolfgang Goethe-University Frankfurt am Main, Germany, for providing the samples.

