The Multiple-Detector System at the beamline B2: Efficiency and Applications of the system

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Introduction

Synchrotron radiation sources have become indispensable for high resolution powder diffraction experiments and the number of researchers using synchrotron radiation facilities is increasing steadily. To increase the efficiency of the measurements we have designed and manufactured a multiple-detector system with four analyser units and four scintillation counters. The four analyser diffractometers are based on the Cox [1] parallel beam geometry. The efficiency of the system is determined by the net measuring time. The parameters the angular overlap and the angular width of the scans are the essential factors controlling the netmeasuring time. Different scan configurations will be discussed which provide for a maximum angular overlap of the scans (gain factors up to 3.6) [2] or a maximum width of the angular range. With interval scans reflections are detected only once by one of the four counters: so the gain factor can increased further. The powder diffraction pattern of Si-Al_2O_3-NaCl (Fig.2) took 36h22´ with a single detector. With the multiple-detector-system the measurment has done within 15h11´. Using 11 interval scans the measuring time is reduced to 5h30´. This is a gain factor over 7.

Efficiency of the system

Parameter: angular overlap and angular width

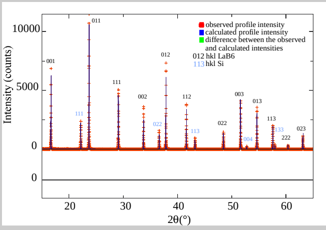
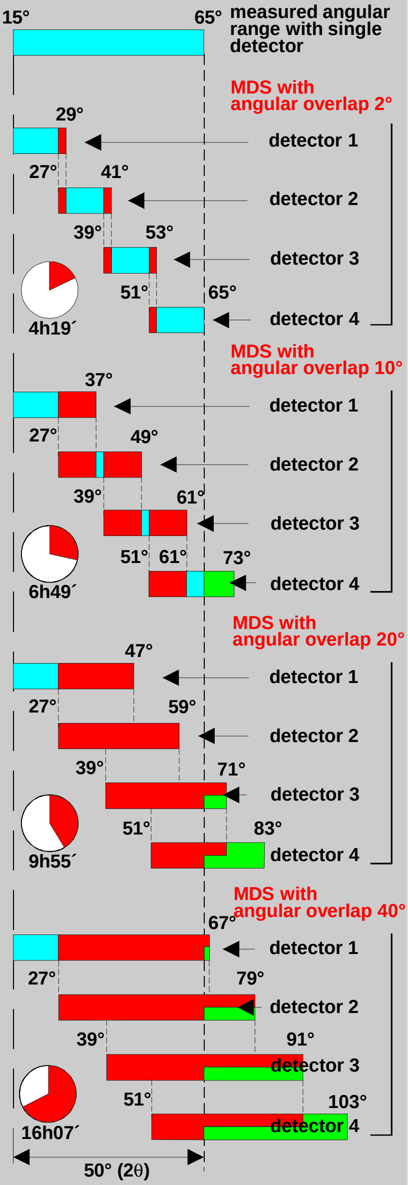


Fig.1: Rietveld refinement of LaB6+Si pattern using a single detector. Red crosses represent the observed profile intensity, blue lines the calculated profile intensity and the difference between the observed and calculated intensities is given at the bottom (green line).

- LaB6+Si-standard at RT
- angular range: 15°- 65°(2θ)
- step width: 0.008°
- data points: 6251
- measuring time: 15h30´



Depending on the measuring job the user can decide whether he wants a short measuring session which requires a small angular overlap, or to extend the measuring range which requires a large angular overlap. In both cases he will profit of the efficiency of the system (Tab.1).

entire angular range in °(2theta)	angular range of detector 1 in °(2theta)	angular overlap in °(2theta)	gain factor compared with single detector	gained angular range compared with single detector in %
15 - 65	15 - 29	2	3.57	-
15 - 68	15 - 32	5	2.94	5.67
15 - 73	15 - 37	10	2.27	13.79
15 - 83	15 - 47	20	1.56	26.47
15 - 93	15 - 57	30	1.19	35.89
15 - 101	15 - 65	38	1.00	41.87
15 - 103	15 - 67	40	0.96	43.18

***Tab.1:** Overview of the gain factors of differ-ent scan configurations. The configurations of the marked rows are shown on the right hand side.*

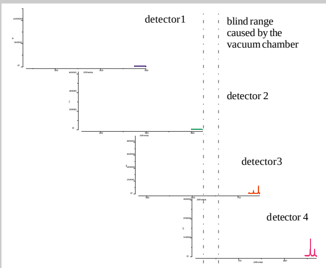


***Fig.2:** Powder diffraction pattern of Si-Al_2O_3-NaCl (capillary 1 mm, RT, λ = 1.2067Å, $\Delta 2\theta$ 23.5° - 87°, step width 0.004°, net measuring time **15h11´**). Scan with 11 intervals reduces the measuring time to **5h30´** (with single detector **36h22´**). Use the transparency to imitate the intervals and the detector window!*

Legend of colours:

- angular range measured by one detector
- angular overlap of several detectors
- gained angular range compared with single detector

Applications of the system

α-quartz measured with channel-cut analyser

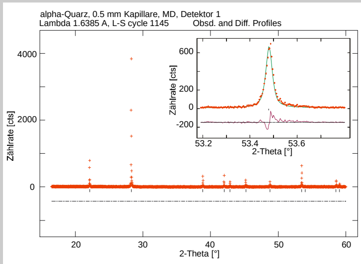


Fig.3: Powder diffraction pattern of alpha-quartz using detector 1. (capillary 0.5 mm, RT, wave-length 1.6385Å).

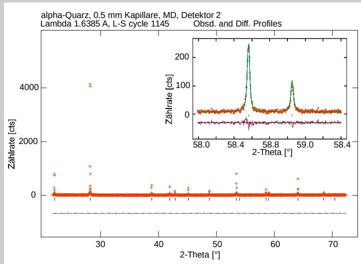


Fig.4: Powder diffraction pattern of alpha-quartz using detector 2. (capillary 0.5 mm, RT, wave-length 1.6385Å).

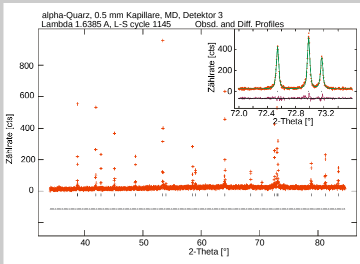


Fig.5: Powder diffraction pattern of alpha-quartz using detector 3. (capillary 0.5 mm, RT, wave-length 1.6385Å).

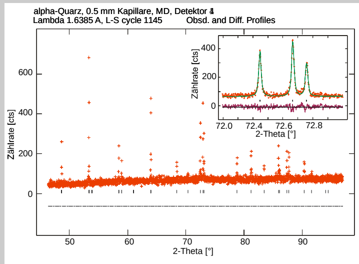


Fig.6: Powder diffraction pattern of alpha-quartz using detector 4. (capillary 0.5 mm, RT, wave-length 1.6385Å).

Tab.2: Results of the Rietveld refinement of alpha-quartz.

The Rietveld refinements are done simultaneously with four data sets (48754 data points). The powder data statistics and the atom parameters deliver good results. The measurement took 9 hours. The parameters are: angular range 9.5° - 97° (2θ), step width 0.004°, wavelength 1.6385 Å, RT.

	atom parameters			detector	wRp	Rp	scale
atom	x	y	z	1	0.2906	0.1864	20.1410
Si	0.470659	0.0	0.33333	sigmas			0.132686
sigmas	0.000287	0.0	0.0	2	0.2774	0.1653	25.7719
shft /esd	0.0	0.0	0.0	sigmas			0.169484
O	0.414298	0.266065	0.213704	3	0.2081	0.1502	29.8938
sigmas	0.000303	0.000330	0.000295	sigmas			0.301823
shft / esd	0.0	0.0	0.0	4	0.1244	0.0990	23.0161
							0.268586
				total	0.1935	0.1284	

Measurements with vacuum chamber (CeO_2), λ=2.2Å

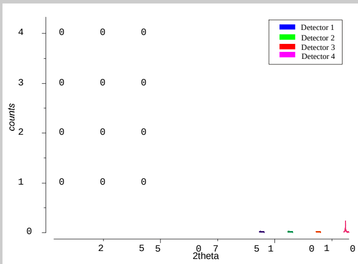


Fig.7: Powder diffraction pattern of CeO_2 (capillary 1mm, CeO_2 NIST 674a, RT, wavelength 2.2Å, vacuum chamber).

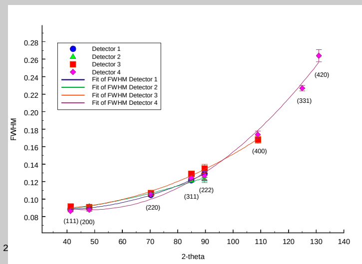


Fig.8: Variations of FWHM of CeO_2 with 2θ for data sets obtained using four detectors.

Acknowledgement

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Reference

[1] D.E. Cox, J.B. Hastings, W. Thomlinson, C.T. Prewitt. Nuclear Instruments and Methods in Physics Research, 208:573-578, 1983
[2] W.-H. Kaps, J.Ihringer, W.Prandl, M. Schilling, The Multiple-Detector System for the powder diffractometer at beamline B2 (HASYLAB), 2nd International SLS Workshop on Synchrotron Radiation, Brunnen, Schweiz, 26.10.-30.10.1999