

The Multiple-Detector System for the powder diffractometer at beamline B2: The modular system of the analyser units

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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. The normal setting at the beamline B2 in HASYLAB is a single detector with analyser crystal or soller slits. To increase the efficiency of the measurements we have designed and manufactured the multiple-detector system with four analyser units and four scintillation counters. The four analyser diffractometers are based on the Cox [1] parallel beam geometry. Using the small divergence of the incident beam the Bragg reflection angles are measured with highest angular resolution and independent of the sample position. The modular system of these analyser units consists in their exchangeable crystal analysers. The user can choose between two settings: four flat Si(111) crystal analysers (Fig. 1-3) or four Ge(111) channel-cut crystal analysers (Fig. 4-6). Using the multiple-detector system with the vacuum chamber the channel-cut crystal analysers enable measurements up to a wavelength of 2.5Å.

Experimental Design



Flat Si(111) crystal analyser unit

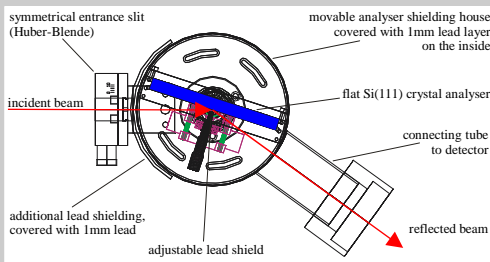


Fig.1: The four flat Si(111) crystal analysers are designed for a wavelength range from 0.5Å to 1.7Å. The dimensions of the crystals are 75mm in length, 25mm high and 6mm thick. Adjustable lead shields eliminated cross talk between the entrance and the exit of the analysers at short wavelengths. Outside the analyser shielding house an additional lead shield covers the entrance opening. In front of it a symmetrical slit restricts the incident beam.

Ge(111) channel-cut crystal analyser unit

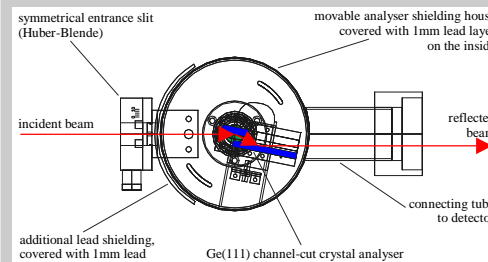


Fig.4: The channel-cut crystal is made of a monolithic Ge crystal. The (+n, -n) arrangement is designed for the symmetrical reflection on the Bragg plane [111] in a wavelength range from 1.2Å to 2.5Å. The crystal is 42mm long and 30mm high. The (+n) crystal is only 14mm long and the (-n) crystal is 35mm in length. The channel is 2.5mm wide. The main property of the channel-cut analyser in the (+n, -n) arrangement is the small shift between the incident and reflected beam.

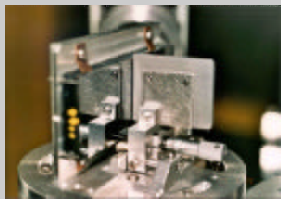


Fig.2: Open analyser unit with flat Si(111) crystal analyser and adjustable lead shield in front of it.

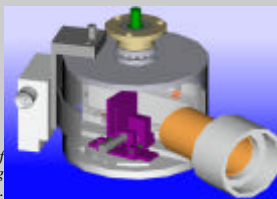


Fig.3: Detailed view of one analyser shielding house (transparent).

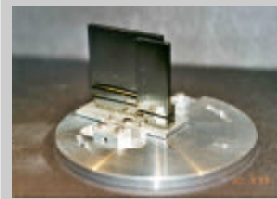


Fig.5: The Ge(111) channel-cut crystal analyser is mounted on the modular part of the analyser unit. A small adjustable plate carries the crystal.

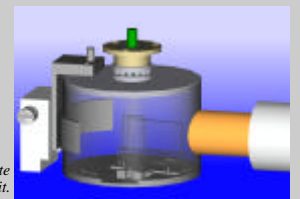


Fig.6: Complete analyser unit.

Results

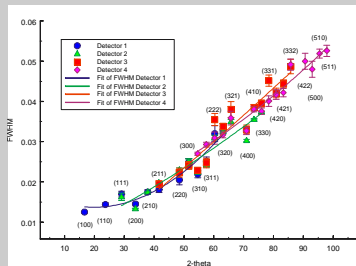


Fig.7: Variations of FWHM of LaB₆ with 2θ for data sets obtained using four detectors (flat specimen, LaB₆, NIST 660, RT, wavelength 1.20672Å, fit is done with CMPR).

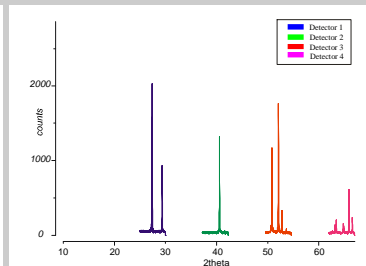


Fig.8: Powder diffraction pattern of Si - Al₂O₃ - NaCl collected using the multiple-detector system (capillary 1mm with Si - Al₂O₃ - NaCl, RT, wavelength 1.20672Å).

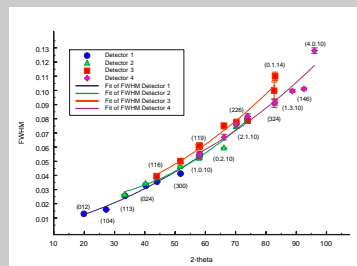


Fig.11: Variations of FWHM of Al₂O₃ with 2θ for data sets obtained using four detectors (flat specimen, Al₂O₃, NIST 1976, RT, wavelength 1.2Å, fit is done with CMPR).

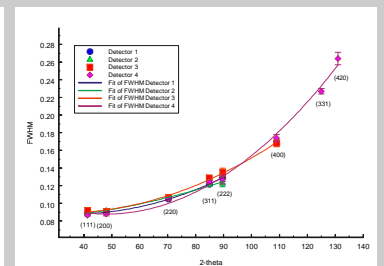


Fig.12: Variations of FWHM of CeO₂ with 2θ for data sets obtained using four detectors (capillary 1mm, CeO₂, NIST 674a, RT, wavelength 2.2Å, vacuum chamber, fit is done with CMPR).

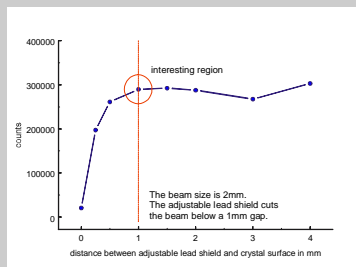


Fig.9: Variations of cts with distance between adjustable lead shield and crystal surface. The primary beam ($\lambda=1.668\text{\AA}$) is reflected by the Si(111) crystal analyser and collected with detector 1.

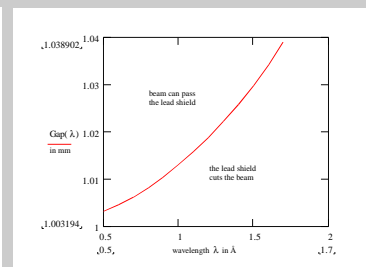


Fig.10: Calculated data for the distance between adjustable lead shield and crystal surface (gap). The lead shields operate with a precision of 0.01mm.

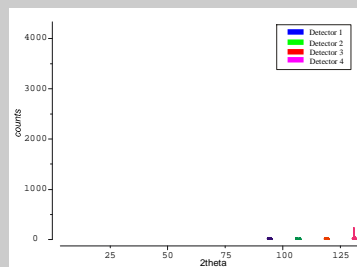


Fig.13: Powder diffraction pattern of CeO₂ wcollected using the multiple-detector system (capillary 1mm, CeO₂, NIST 674a, RT, wavelength 2.2Å, vacuum chamber).

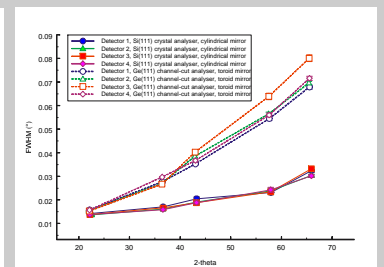


Fig.14: Variations of FWHM of Si with 2θ for data sets obtained using four detectors (flat specimen, RT, wavelength 1.2Å, fit is done with CMPR).

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

