The Multiple-Detector System for the powder diffractometer at beamline B2 (HASYLAB)

W.-H. Kaps, J.Ihringer, W.Prandl, M.Schilling

Institut für Kristallographie der Universität Tübingen, Charlottenstr.33, D-72070 Tübingen
www.uni-tuebingen.de/uni/pki   e-mail: wolf-hendrik.kaps@uni-tuebingen.de

Introduction

Beam time at synchrotron radiation sources is expensive and the number of researchers using synchrotron radiation facilities is increasing steadily: therefore the measurements have to be highly efficient. We have designed and manufactured a multiple-detector system with four flat Si(111) crystal analysers and four scintillation counters. Using the small divergence of the incident beam theBragg reflection angles (2θ) are measured with highest angular resolution and independent of the sample position. Depending on the angular overlap of the scans gain factors up to 3.6 have been obtained.

Experimental Design

The four analyser diffractometers are based on the Cox [1] parallel beam geometry (Fig. 3). This compact unit consists of a three-stage rack 480mm long and 350mm high (Fig. 1). Its base plate is fixed with the 2θ goniometer (880mm in diameter) of the Huber diffractometer. The middle stage carries the crystal analysers and detectors. The top plate holds the analyser shielding houses. A single stepping motor simultaneously adjusts all crystal analysers and a Heidenhain encoder is reading the angles. In addition the relative positions of the angles can be set by individual drives. Due to the identical design and the simultaneous control of the four analyser diffractometers data corrections are not needed.

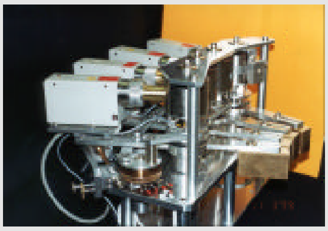

Fig.1: The compact unit of the multiple-detector system. The middle stage carries the crystal analysers and detectors. The top plate holds the analyser shielding houses.

The multiple-detector system is constructed for measurements in a wavelength range of 0.5< λ <1.7Å. Cross talk between the entrance and the exit of the analysers at short wavelengths is eliminated by adjustable lead shields (Fig. 2). They operate up to a distance to the crystal of 4mm and are adjustable with a precision of 0.01mm. Special analyser shielding houses adapted to the beam path enhance the typical peak / background ratio to 300 to 1. The detectors are connected with these houses and ensure by their movement that only the incident beam as well as the diffracted beam can pass the analyser unit.

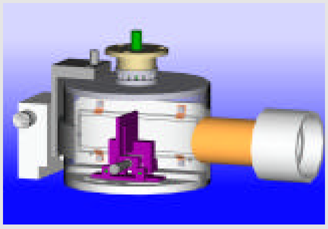

Fig.2: Detailed view of one analyser shielding house (transparent) and the adjustable lead shield (magenta). The flat Si(111) crystal is fixed with small copper springs. Outside the analyser shielding house an additional lead shield covers the entrance opening. In front of it a symmetrical slit restricts the incident

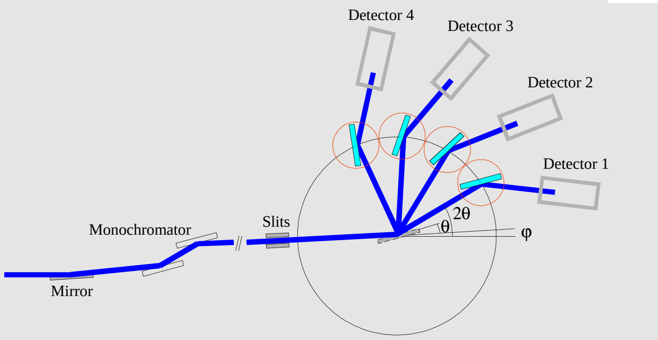

Fig.3: Schematic diagram of the experimetal configuration for synchrotron the X-ray powder diffractometer at beamline B2 (HASYLAB / DESY) with mirror, monochromator and the multi-detector system.

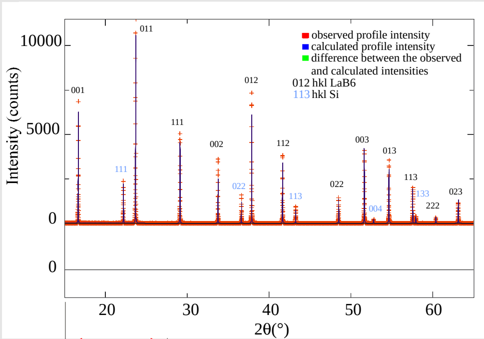

*Fig.5: Rietveld refinement of LaB6 + Si pattern using the **detector 1**. Red crosses represent the observed profile intensity, blue lines the calculated profile intensity and the difference between the observed and calculated intensities is given at the bottom of each diagram (green line).*

Offset Detector 2 ca. 12°

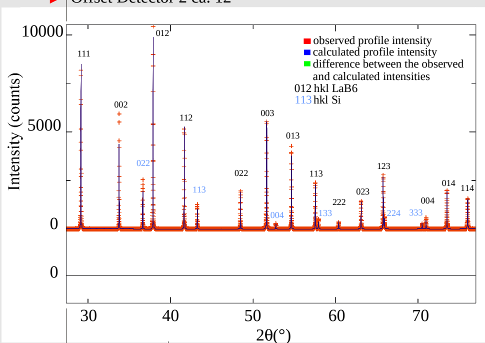

*Fig.6: Rietveld refinement of LaB6 + Si pattern / **detector 2**.*

Offset Detector 3 ca. 12°

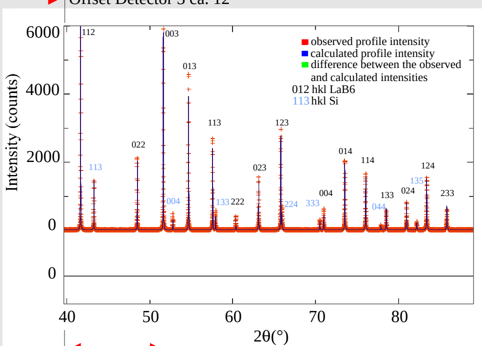

*Fig.7: Rietveld refinement of LaB6 + Si pattern / **detector 3**.*

Offset Detector 4 ca. 12°

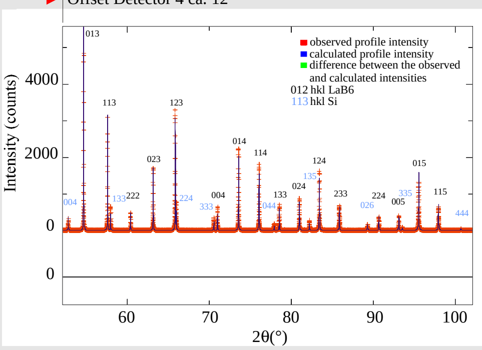

*Fig.8: Rietveld refinement of LaB6 + Si pattern / **detector 4**.*

Results

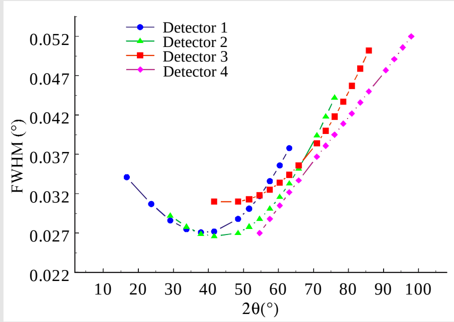

Fig.4: Variations of FWHM of LaB6 with 2θ for data sets obtained using four detectors.

	Rp	Rwp	Rexp	Gof	Nsigma	Bragg
Det 1	17.028	22.688	13.435	1.688	146.276	9.36
Det 2	15.402	21.279	12.750	1.668	140.586	5.71
Det 3	15.243	21.087	13.795	1.528	104.965	7.75
Det 4	16.086	20.941	14.341	1.460	89.438	10.82

***Table 1:** Results of the Rietveld refinement of LaB6 + Si pattern.*

We acknowledge financial support by the BMBF

Reference

[1] D.E. Cox, J.B. Hastings, W. Thomlinson, C.T. Prewitt. Nuclear Instruments and Methods in Physics Research, 208:573-578, 1983

Institut für Kristallographie   **Universität Tübingen**