

MS44-2-1 A rigid compact multi-analyzer system for accurate powder diffraction analysis in the laboratory and/or on a synchrotron source to extract high-resolution and low-noise patterns
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Abstract

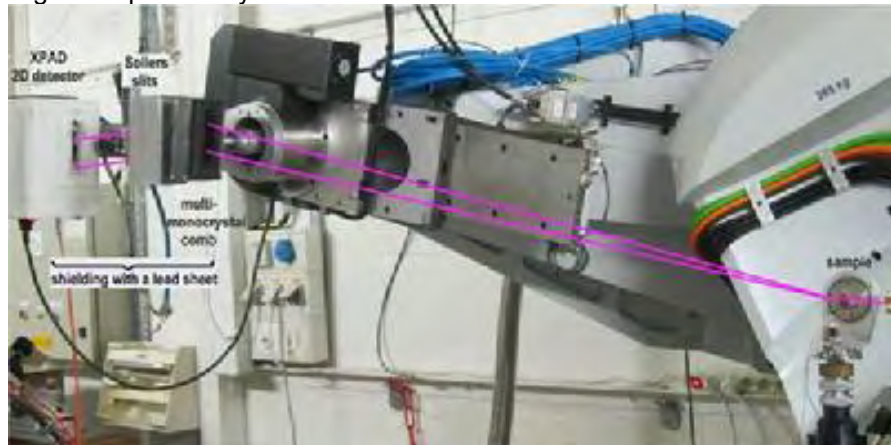
Diffraction instruments using analyzer-filtering have existed since the 1990s for Synchrotron Radiation (SR) sources, (but are hardly practicable on laboratory sources), they have an efficiency that makes them used on the world's SR sources [1-6]. We present the realization/efficiency of a small rigid-compact-multi-analyzer system which allows to filter in parallel 20-50-100 measurements on SR sources, but also on laboratory X-ray sources (AgK α). This filtering is carried out by a diffractive filtering curved surface consisting of either a crystal, a thin layer, or multiple crystals supported on a fixed and rigid log-spiral curved surface. The geometry of this surface is calculated to allow filtration over a large angular range (2θ) and using an X-ray spectral range to be usable by various sources [7]. The rigid-compact-multi-analyzer block completes and makes full use of the qualities of new "pixel" detectors which have a very low intrinsic noise [6]. When used together, they can drastically exceed the measurement detection thresholds (from 2-1% to 0.1%) allowing to detect minor phases in studies of heterogeneous "real" materials.

The tested rigid-compact-multi-analyzer system consists of a rigid-compact-multi-analyzer comb that contain 20-50 Si(111) single crystals and an associated block of 20-50 Soller slits. The angle between each crystal being 0.1°, its measuring range is 2-5°. This geometry has been calculated to operate an X-ray source having an energy ranging from 22KeV to 46KeV. Figure 1 shows a picture of the system on D2AM diffractometer at the ESRF. Figure 2 shows the total diagram (and the corresponding residual noise) of a LaB6 sample measured at 22KeV (AgK α); contributions of each analyzer is also shown for the low angle (110) LaB6 reflection; the peaks have a FWHM: 0.008°; the signal-to-noise ratio is 1/1000. The Rietveld refinement of these LaB6 diagram gives Bragg Rf 1.544 and Rf 1.046. The strong diffraction peak has an intensity of 60,000 cps while the residual noise is 60cps! (i.e. 3.5 to 2.5 cps. per analysis channel!) [8]. Using the same set-up and alignment, we collected data of some complex heterogeneous samples of the PatrimAlp culture heritage program [9]. Thanks to this set-up, we can quantify their components, the width of the lines of each phase is only related to the crystallinity of the phases and the fluorescence is suppressed. After the measurements of these "real" samples, we re-measured the LaB6 reference, without modifying the settings, we found the same intensities and profiles as before.

References

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Rigid-compact-analyzer-comb on D2AM diffractometer



LaB6 pattern using filtering by a 20 crystal comb

