PARALLEL BEAM METHODS IN POWDER DIFFRACTION AND
TEXTURE IN THE LABORATORY.

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ABSTRACT
The availability of Polycapillary X-ray Optical devices has allowed the production of large area
quasi-parallel X-ray beams with substantial gain in X-ray flux and which can be used with
conventional laboratory sources.
The Polycapillary Optics are provided by XOS Inc. and collect X-rays diverging from the point
focus of a conventional XRD tube, over a solid angle of 4.1 degrees. A parallel beam is
produced, with a beam divergence of 0.22 degrees, at 30% efficiency over an area of 10x10mm.
Both axial and planar directions are equally collimated.
The diffractometer must be capable of adjustment for Source to Theta axis distances around
250mm and have a parallel collimating and crystal monochromator device in front of the
detector, which matches the beam divergence of the Polycapillary Optic. A soller slit collimator
and LiF flat crystal combination is used. This diffractometer also includes a Eulerian cradle for
the texture work, and is the “MMA”, manufactured by Diffraction Technology.
Applications of the Polycapillary Optic fitted to the “MMA” for powder diffraction and texture,
will be discussed and compared with conventional parafocussing or Bragg-Brentano geometry.

INTRODUCTION
The purpose of this study was to examine the place of parallel beam methods in routine
laboratory X-ray analytical work. The unique abilities of the quasi-parallel beam produced by
polycapillary optics [1 – 3] have been reported previously [4 – 6] and this study examines in
more detail the particularly significant areas of improvement over focussing or “Bragg-Brentano”
geometry for qualitative identification, other Powder Diﬀraction and Texture (Pole Figures)
applications. The Diffractometer used was a “MMA” Mini-Materials Analyser from Diffraction
Technology, which has a convenient geometrical layout for positioning the X-ray source at an
extended distance from the goniometer axis to accommodate the optic. It also has an Eulerian
cradle for texture and a “Glancing Incidence” attachment (secondary collimator, LiF analysing
crystal and detector) for thin film work. The Polycapillary Optic used was a model X-8-L10
From X-ray Optical Systems Inc., providing a 10 x 10 mm. quasi-parallel beam from a solid
angle of collection of 4.1 degrees. The source was a conventional laboratory normal focus (1mm x 10mm) copper anode tube.

QUALITATIVE PHASE ID
The characteristics of Powder Diﬀraction patterns taken with Parallel Beam geometry have been
reported elsewhere [4,5]. The method gives precise peak positions, unaffected by sample
displacement which are suitable for search/match against the ICDD PDF Database [6]. A
minimum of sample Preparation is needed, as angular, irregularly shaped or rough samples can
be placed in the beam — all that is required is that they have a suitable surface area exposed to the primary beam and on the secondary beam side that the detector is sufficiently large. Fig 1 shows a piece of aluminum tube mounted on the stage of the Eulerian cradle. With this sample stage, actually designed for metal samples for texture, the sample stub is adjustable for height with a friction fit. The sample can be stuck to the stub using an adhesive putty. Fig 2 shows the pattern with “PDF” lines superimposed.

Other applications are:
- The surface of Bauxite Pisoliths, the 6 – 8 mm pebbles are glued to a piece of board.
- Fibrous cellulose - again, a sample simply glued to a piece of board,
- Surface of as-sawn blocks of rocks, such as Silcretes

Fig 3 shows a pattern from the Bauxite pisoliths, after background stripping, with Database lines superimposed. This surface composition also reveals extreme preferred orientation.

Fig 1 Aluminum Tube mounted on stage of Eulerian cradle

Fig 2 Aluminum tube pattern with PDF lines superimposed
OTHER POWDER DIFFRACTION

The beam divergence characteristics of the Polycapillary Optic match well to the mosaicity of "real-world" samples such as natural materials, minerals etc. The beam divergence of 3.8 mrad (0.22 deg) is set by the critical angle for total external reflection of 8keV X-rays from the inner glass walls of the capillaries. The last reflection on the inside of the capillary tube can depart from the axis of the optic by this angle. Hence, to make an X-ray optical beam path matched to the optic, the secondary Soller Slt collimator and the FWHM of the secondary analyzing crystal must match this divergence, for optimal performance. The Soller collimator in the "MMA" has an acceptance angle of 0.34 deg., which has been found to give an optimum balance between resolution and intensity. The LiF(200) analysing crystal is of the type used in wavelength dispersive X-ray spectrometers and has a surface mosaic treatment which gives it a peak FWHM of approximately 0.2 deg. Matching of beam components enhances the sensitivity of the system. The corollary of the beam divergence is an increase in FWHM of peaks in a powder pattern. Whereas a typical standard Bragg-Brentano powder diffractometer has an FWHM of 0.15 deg, we have found an FWHM of 0.27 deg. for the same sample for the parallel beam geometry. Fig 4 shows overlapping scans from 60 deg. to 100 deg. 2Theta, run in parallel beam mode and in Bragg- Brentano focusing mode with a radius of 240mm. (FWHM 0.09 deg.) on a Silicon standard. Most diffractometers cannot achieve this radius and resolution so the difference is exaggerated, nevertheless, it provides a valuable direct comparison.
TEXTURE

One of the limitations inherent in pole figure data collection using a conventional Schulz collimator is the extreme defocusing which occurs at high Chi angles (The Euler angle notation is used here, Altitude is Chi, Azimuth is Phi). In an attempt to minimize this, the Schulz collimator restricts the irradiated area to 1mm wide by 5-6 mm high. This also reduces the intensity by reducing the number of crystallites contributing and makes mechanical oscillation of the sample necessary to enhance the crystallite statistics. A further limitation is the poor FWHM due to the use of the point focus of the X-ray tube.

When a parallel beam geometry using a polycapillary optic is employed, these limitations are significantly reduced. The irradiated area is 10mm wide and its height is dictated by the Theta angle, but it is of the order of 20 times greater than with the Schulz collimator, this gives generally a 20 fold increase in intensity and consequently faster data collection with better statistics (and with immunity to height variation problems). Further, the FWHM remains constant at around 0.27 deg with all Chi angles.

In addition, due to the larger irradiated area, higher angle peaks are much more intense, so the weighting factors which usually have to be applied to these peaks are reduced (they can be eliminated). The immediate result of the better geometry and the better counting statistics is better fits of Harmonic Coefficients to produce Orientation Distribution Functions (ODFs) by the method of Bunge [7].

Fig 5 shows Parallel beam Pole Figures collected on rolled Aluminum sheet.
These Pole Figures were collected with 2 sec. Count times using 30kV, 20mA power.
Fig 5 Series of Pole Figures on Rolled Al Sheet Collected in Parallel Beam Geometry

The effect of the parallel beam of 10mm x 10mm size and the use of the secondary Soller slit collimator was investigated. Three X-ray optical configurations were used – (1) Classical focusing system with Schulz collimator, (2) Parallel beam system with single lateral Soller slit collimator (aligned with 0 deg. Chi), and (3) Parallel beam system with crossed Soller slit made by attaching two half length collimators in line, rotated 90 deg. to each other.

For each of these geometries, a series of scans on the Al (111) peak at 38.5 deg 2Theta for Cu Kα was run and the increase in peak FWHM and fall-off in intensity with Chi were logged. FWHM for the Focussing Geometry was between 0.83 and 0.89 deg.
FWHM for the Single Collimator was between 0.26 and 0.27 deg.
FWHM for the Crossed Collimator was between 0.24 and 0.25 deg.

(a) Single Collimator, parallel geometry
(b) Crossed Collimator, parallel geometry

Fig 6 Comparison of Collimators in parallel geometry with focusing geometry
CONCLUSIONS

We have shown possible applications of parallel beam geometry for everyday life powder diffraction applications. The coupling of a polycapillary optic to the Mini Materials Analyser (MMA), provides an economical robust diffraction system with the same capabilities as larger more cost intensive diffraction systems used for routine powder diffraction measurements. It can find its way into Industrial labs for process control and variations of the system can be thought of for in-line process control.

REFERENCES

[6] ICDD, 12 Campus Boulevard, Newtown Square, PA, 19073-3237, U.S.A.