

**Keywords:** X-ray detector technology, area detectors, X-ray imaging

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**Simple techniques for measuring the performance of 2D X-ray detectors**

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The performance of an X-ray detector depends to a large extent on the signal to noise ratio (SNR). However, this parameter is rather difficult to determine experimentally and thus it is only roughly known (if at all) for many detectors. Here we present a simple experimental technique for determining the quantum gain and the noise of a CCD detector.

In principle the simplest and most direct way to measure the quantum gain of a detector would be a measurement of the response of the detector to a single absorbed X-ray and repeat this measurement a number of times to determine the average gain. However, depending on the X-ray energy and the CCD noise it may be difficult to distinguish reliably a single X-ray “hit” above the detector noise floor, in particular at the rather low energies typically used for X-ray diffraction (e. g. 8-20 keV). Thus, the “classical” approach to measuring the gain is to use an absolutely calibrated X-ray source. However, such sources are often not easily available. Also, the use of an absolutely calibrated source requires that the measurement be corrected for the window transmission and the scintillator absorption efficiencies and these parameters are often not known with precision. Here we describe a simple method for determining the quantum gain without the use of an absolutely calibrated source and also without the need to compensate for absorption in the window and in the scintillator screen. We similarly describe the techniques for determining the total system noise so that the signal-to-noise ratio (SNR) of any given camera can be evaluated.

Specific examples for a Bruker camera will be given, but the techniques can be applied to any detector.

**Keywords:** 2D X-ray detector, quantum gain, signal-to-noise ratio

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**Identification of mineral in thin section by energy-Scanning X-ray diffraction**

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The stationary sample method with polychromatic synchrotron radiation (SR) is advantageous for X-ray diffraction studies with micro-beam on minerals in a thin section. Because the irradiated area of the sample is always the same and fixed, all diffraction spots occur from the same area of the sample. However, their cell parameters, which are necessary for identification of minerals, cannot be directly obtained by this method. In order to obtain the cell parameters of the sample in a thin section even in the case of the sample stationary method, we applied energy scanning of micro-beam of monochromatic SR [1]. In the present study, an unusual mineral in a thin section of Apollo 16

lunar sample (60016) was identified using this method.

We employed the intense X-ray source of SPring-8. At the beam line 37XU the undulator is installed, and a Kirkpatrick and Baez mirror is equipped upstream of the sample giving the beam size of 0.7(V) x 2(H)  $\mu\text{m}^2$  on the sample position in the thin section. Diffraction spots can be measured on the two-dimensional detector (X-ray flat panel sensor, Hamamatsu Photonics). The 60016 thin section was attached onto the sample holder, and the target silicate grain in the thin section was adjusted on the micro-beam position under an optical microscope. We applied energies of incident X-ray from 30 to 20 KeV ( $\lambda=0.4133\text{-}0.6199 \text{ \AA}$ ) with the increment of 40 eV step with each exposure time being 0.5 seconds.

We obtained 17 diffraction spots in energy range above. The energy of each diffraction spot was chosen to give maximum intensity of the spot. The positions ( $x, y$ ) of the diffraction spots at their maximum intensities were determined as those of the top of the diffraction profile fitting along  $x$  and  $y$  with the asymmetric Gaussian function. The reciprocal lattice vectors are calculated by the energies and the positions on the detector of the diffracted X-ray. The vectors calculated by the differences of these vectors are also used for reciprocal lattice construction. One of the domains was found as a result of analysis in a way similar to the indexing method commonly used for the four circle diffractometer, and the 7 spots obtained could be indexed by an olivine single crystal. The obtained cell parameters are  $a=4.751(8)$ ,  $b=10.199(2)$ , and  $c=5.998(4)\text{ \AA}$ . These values suggest that the olivine crystal analyzed is close to near end-member forsterite composition whose Fo composition is higher than Fo<sub>95</sub> [e.g., 2]. In lunar samples, forsteritic olivine is rare. Because this olivine crystal is present in a clast showing a chondrule-like texture, its forsteritic composition suggests derivation from a chondrite that impacted onto the lunar surface. This study demonstrates that the stationary sample method with energy scanning of micro-beam of monochromatic SR is a powerful tool for the non-destructive X-ray diffraction analysis of small extraterrestrial minerals in thin sections.

[1] K. Hagiya, T. Mikouchi, M.E. Zolensky, K. Ohsumi, Y. Terada, N. Yagi, M. Takata, *73rd Annual Meeting of the Meteoritical Society*, 2010, 5083 [2] S. Akimoto, H. Fujisawa, *Journal of Geophysical Research* 1968, 73, 1467–1479.

**Keywords:** monochromatic, microbeam, Laue

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**The application of vacuum in the elucidation of structural changes**

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The use of non-ambient conditions in order to understand structure/property correlations in the solid state is of great interest to single crystal small molecule crystallographers and chemists alike. The ability to access 3-dimensional information under short experimental time frames allow for almost real time results when combined with in-situ techniques. This combination transfers the rate limiting step from the experiment to the data processing. This approach in-turn converts the perception of a “static” single crystal “snap-shot” technique into a pseudo “dynamic” one which can be to a large range of problems and key materials ranging from sensors to pharmaceuticals.

The development of novel instrumentation and methodologies for the study of small crystals, using in-situ single crystal X-ray diffraction techniques both in-house and at synchrotron radiation facilities are presented.

## Poster Sessions

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**Keywords:** diffraction, environment, in-situ

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#### A Kappa diffractometer for micron sized crystals

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Despite using sub-micrometer precision spindles, diffractometers generally offer a few micrometers stability when equipped with Kappa axis. This limits the minimum affordable crystal size to a few tens micrometers. The experience gained in applications using small crystals and the increased availability of micro-focus beamlines now encourage crystallographers to use much smaller crystals. A number of micro-beam based experimental approaches may help mitigating the radiation damage as a major limiting factor [1], [2], [3]. Techniques are also developing to scan crystals with a tinny beam to find good diffracting areas, and to further collect data at the best places [4].

Supporting this type of applications will require diffractometers with sub-micrometer precision. Moreover, the use of kappa goniometers could be essential to pre-orient micro-crystals in order to minimise radiation damage and to optimise collection of complete data sets over several micro-crystals.

Gravity was found to significantly impact on the stability of centring tables and kappa mechanism associated with Omega spindles set horizontally, thus resulting in poor stability at crystal position. A new Kappa diffractometer equipped with an air bearing goniometer set vertically is currently under development. We expect the precision to

be around 0.3 micrometer for Omega, and the stability better than a micrometer with Kappa. Including all the features present in the most advanced micro-diffractometers, the new device should be suitable for processing micrometer sized crystals. The plans are to equip the EMBL@PETRA3 MX2 beamline in 2012, and to further upgrade the ESRF ID23-2 micro-focus beamline.

[1] Cowan, Nave, *J. Synchrotron Rad.* **2008**, *15*, 458-62. [2] R Moukhametzianov et al., *Acta Cryst.* **2008**, *D64*, 158-166. [3] Sanishvili et al., *Acta Cryst.* **2008**, *D64*, 425-435. [4] M.W. Bowler et al., *Acta Cryst.* **2010**, *D*, submitted

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#### DW varimax rapid II: one source, two wavelengths, power and flexibility

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The drive towards comprehensively equipped analytical facilities, is both an economic necessity and a big opportunity for interdisciplinary research; there is a stringent need for manufactures to provide new tools, optimised for this scenario.

DW Varimax is a confocal, high flux, optics system capable of switching between a range of exchangeable radiations and designed to work with a high-flux source with dual target. The power and flexibility of such source is an ideal match for our Rapid II IP detectors, capable of exploiting its key capabilities.

A wide range of selected advanced applications will be presented, demonstrating the diverse specialist applications for which this machine is optimised.

**Keywords:** charge density, microdiffraction, protein