

align the detector so that one edge of the sensor lies very close to the beam. A special detector was manufactured by Rad-Icon Imaging Corp to pass X-rays between two detector sensors, which were separated by about 3 mm. With this detector, it is possible to record a two-dimensional wide-angle diffraction pattern while another detector at the downstream end of the camera records a small-angle diffraction pattern. An active-pixel CMOS detector from Hamamatsu Photonics is also suitable for this purpose.

[1] Yagi N., Yamamoto M., Uesugi K., Inoue K., *AIP Conference Proceeding*, 2004, **705**, 344. [2] Yagi N., Yamamoto M., Uesugi K., Inoue K., *J. Synchrotron Rad.* 2004, **11**, 347.

**Keywords:** detector properties, synchrotron radiation, small-angle X-ray scattering

#### P.01.10.5

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#### 1- and 2D Detectors and Sample Fluorescence in XRD

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In the last decade, electronic 1- and 2-dimensional X-ray detectors have replaced scintillation detectors and X-ray films for both Single Crystal, and Powder Diffraction. For many applications, these detectors bring orders of magnitude increase in data collection efficiency, and as a result of that, they allow us to increase productivity and/or collect data with a higher accuracy within a reasonable time frame.

A consequence of these more 'open' detection systems is that they are more susceptible to scattered radiation, and also to photons with a wavelength differing from the chosen characteristic radiation, such as K-Beta radiation and sample fluorescence. This is especially visible in powder XRD as the traditional Bragg-Brentano diffractometer geometry includes a focusing diffracted beam monochromator, which takes out a lot of unwanted radiation.

For high-speed X-ray detectors, a focusing diffracted beam monochromator does not exist. This does not mean, however, that it is not possible to record diffractograms with a good peak-to-background ratio. Especially with sample fluorescence, there are a lot of options for obtaining a good result and this contribution is aimed to help selecting the best one. Measurements were analyzed which were carried out on samples of the first row of transition metals, ranging from Ti to Ga as a function of the following variables: tube anode material, use of incident or diffracted beam monochromators, use of incident or diffracted beam beta filters, Pulse Height Discrimination settings, and generator settings.

**Keywords:** X-ray detectors, X-ray powder diffraction techniques, experimental design

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#### Use of a Single Crystal Diffractometer and CCD Area Detector for Phase Identification

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CCD-based single crystal X-ray diffractometers are widely used in the fields of chemistry, materials science, biology and mineralogy for crystal structure determination. However, CCD detectors have not yet been widely utilized in the field of powder diffraction.

A new Phase Identification option has now been integrated into the Bruker APEX2 software suite, so that the same hardware and software may be used for both structure determination and powder diffraction measurements. This module will be offered as an optional add-on feature for the small molecule single crystal instruments using the APEX2 (Version 2.0) software suite. For more specialized applications, data may also be exported to the Bruker DIFFRAC<sup>plus</sup> (EVA & TOPAS) programs.

The powder diffraction option is intended to supplement the primary use of a single-crystal diffractometer to verify that the sample is a single phase or polymorph and that the analyzed single crystal

specimen is representative of the bulk sample.

Various examples of powder diffraction patterns collected on Bruker Kappa APEX II and SMART APEX II instruments equipped with Cu- or Mo-wavelength X-ray sources will be presented.

**Keywords:** powder diffraction, CCD detectors, software

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#### IP Slant-incidence Correction for Accurate Structure Factor Measurements

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Slant incident X-rays on IP give higher intensity than normal incident ones. The correction for the effect was first proposed by considering incomplete absorption of X-rays in the phosphor layer of IP[1]. We proposed a correction factor  $\cos i$  taking into account the absorption of emitted luminescence by the phosphor layer[2]. However they are not good enough at higher slant angles. This inhibits us to develop highly accurate IP devices, although IP has very high potential for accurate structure factor measurements.

The  $\cos v$  formalism was improved by carefully evaluating the accessible area of emitted light by the optical system of the IP readout system, BAS2500(Fuji Film) and applied it to 4f-electron density measurement of CeB<sub>6</sub> by VCIP method. The ratio of the difference of observed and calculated intensities to calculated one were plotted against  $i$  for all the observed reflections, exhibiting excellent coincidence over the observed  $i$  range from 0 to 55°. Actually R factors without correction, with correction employing  $\cos i$  formalism and using the new method were 4.5, 2.9 and 1.9%, respectively. 4f-electron deformation density in CeB<sub>6</sub> appeared only after the new correction, which is similar to the one by four-circle diffractometer[3]. The method can be easily extended to the other IP readout systems.

[1] Zaleski J., Coppens P., *J. Appl. Cryst.*, 1998, **31**, 302. [2] Zhurova E. A., Zhurov V.V., Tanaka K., *Acta Cryst.* 1999, **B55**, 917. [3] Tanaka K., Onuki Y., *Acta Cryst.*, **B58**, 423.

**Keywords:** image plate, slant-incidence, 4f-electron density

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#### XPAD: A Pixel Detector for Material Sciences

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Currently available 2D detectors do not make full use of the high flux and high brilliance of third generation synchrotron sources. For this reason numerous experiments are still performed using slits and photomultipliers that allow only point detection. At the present time, the 2D detectors in most common use are CCD cameras with indirect photon detection.

The XPAD photon counting detector has been developed for materials science and small angle scattering experiments similar to those performed on the CRG-D2AM beamline at ESRF. At the time, its prototype is built of 8 modules of 8 chips for a total area of about 6.8x6.8 mm<sup>2</sup> and 200x192 pixels.

Recent results of powder diffraction of CaSrX zeolite [1] have proved that such 2D detectors present a new opportunity to improve the quality of our measurements. SAXS results will also be presented and compared to CCD ones.

[1] Basolo S., Berar J.-F., Boudet N., Breugnon P., Caillot B., Clemens J.-C., Delpierre P., Dinkespiler B., Koudobine I., Meessen Ch., Menouni M., Mouget Ch., Pangaud P., Potheau R., Vigeolas E., accepted in IEEE Trans. Nucl. Sci., conference IEEE-2004, Rome.

**Keywords:** detector development, synchrotron radiation, materials science