Next generation X-ray detectors for in-house XRD

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A novel type X-ray detector, called PILATUS, has been developed at the Paul Scherrer Institut in Switzerland during the last decade. PILATUS detectors are two-dimensional hybrid pixel array detectors, which operate in single-photon counting mode. PILATUS detectors feature a very wide dynamic range (1 : 1 000 000), very short readout time (<3.0 ms), no readout noise, and very high counting rate ($>2\times10^6$ counts/s/pixel). In addition, a lower energy threshold can be set in order to suppress fluorescence background from the sample, thus a very good signal-to-noise ratio is achieved. The combination of these features for area detectors is unique and thus the PILATUS detectors are considered to be the next generation X-ray detectors. The basic building block of all the detectors is the PILATUS module having an active area of 83.8×33.5 mm^2. The PILATUS 100K is a complete detector system with one module. PILATUS detector systems can have other configurations, including large area systems consisting of 20 to 60 modules that can cover up to an area of 431×448 mm^2. Such large systems are mainly used for macromolecular structure determination, such as protein crystallography and small angle X-ray scattering. The PILATUS 100K detector can be easily adapted to many systems; the single-module detector is integrated to an in-house X-ray diffraction (XRD) system. Examples of XRD measurements with the PILATUS 100K detector are given. © *2008 International Centre for Diffraction Data.*
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Key words: hybrid-pixel X-ray detector, radiation hard design, dynamic range, readout time, signal-to-noise ratio

I. INTRODUCTION

In order to take an X-ray image, various types of area detectors are developed and used. These include X-ray film, imaging plate, charge coupled device (CCD), multi-wire proportional counter, and flat panel. All of these detectors have good features as well as drawbacks. Analogue X-ray film has been used to take X-ray images since X-rays were discovered by Wilhelm Conrad Röentgen in 1895, and it is still widely used in various fields. The X-ray film is inexpensive and easy to obtain in large areas, but the film requires development and printing processes that are the same for conventional optical film, and the obtained image has to be digitized in order to analyze the intensity data using a computer. An imaging plate (IP) can be described as a substitution of the analogue film. IP does not require a dark room for image readout, but it takes about 1 min to read a 300×300 mm^2 image. A CCD detector is an order of magnitude faster than IP. However, the read-out time is in the order of seconds, typically a few seconds for $2k\times2k$ format, and the X-ray source must be blocked by a mechanical shutter during the readout process.

All integrating area detectors are suffering from different sources of noise. The CCD detector has dark current and readout noise and therefore needs to be cooled to reduce the dark current for long-time exposures. The CCD detector could be used for direct X-ray detection. However, the typical thickness of a few tens of microns is not sufficient enough to absorb X-rays. A fluorescence screen, such as GdO_x film, is usually used to convert X-rays into visible light. The conversion efficiency of such screens is typically 40% to 60% and some portion of the incoming X-rays is lost.

A multi-wire proportional counter (MWPC) has the capability of single photon counting, and it can improve peak to background ratios significantly. The MWPC with a large detection area can easily be manufactured, and it has been widely used in many laboratories. The only problem is the counting rate. Slow ion transfer speed is a main cause of the counting rate limitation. The conventional MWPC has a global counting rate limitation of below 10^5 counts/s.

A solid state area detector with a high counting rate, low background noise, and single photon counting capability is a long-time dream for many X-ray scientists. A long-time investigation and utilization of cutting edge technology at the Paul Scherrer Institut (PSI) makes this dream come true (Brönnimann *et al.*, 1998, 2006).

II. PILATUS DETECTOR

The name PILATUS is an abbreviation for **Pi**xel **A**pparat**u**s for the **S**LS (Swiss Light Source). Currently, PILATUS has three variations, namely 6M, 2M, and 100K. The PILATUS 6M covers an area of 424×435 mm^2, and is

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Figure 1. PILATUS 100K.

dedicated for protein crystallography. The PILATUS 2M is slightly smaller (252×268 mm^2) and designed for small angle X-ray scattering (SAXS). The PILATUS 100K (Figure 1) consists of one PILATUS II single module with detective area of 83.8.5 mm^2. For many experiments, a detector with this size is quite sufficient. With its outside dimensions of $256 \times 140 \times 85$ mm^3 and a weight of 4 kg, it can be integrated easily into an in-house laboratory X-ray apparatus.

A. History

The development of large area hybrid pixel detector started at the Paul Scherrer Institute in 1997. This detector was aimed to be used for protein crystallography. The specification of this detector was discussed thoroughly, and single-photon counting capability and fast data readout feature were desired. The single-photon counting capability reduces background noises, and thus data quality is improved. The fast readout feature reduces damage of protein crystals. In 2001, the first generation of PILATUS chip using DMILL's 0.8μ process was established. Then the first large area single photon counting pixel detector, the PILATUS 1M detector, was constructed and used to collect single crystal data in 2003. The second generation PILATUS chip (PILATUS II chip) was submitted in 2004 and showed greatly improved performance. Table I shows the specifications of the PILATUS I and II chips. The PILATUS 6M detector is based on this chip and was constructed in 2006. In parallel, the developers founded DECTRIS Ltd., a spin-off company of PSI in order to commercialize the PILATUS detector technology. With the PILATUS 6M detector, regular user operation was started at the SLS in the spring of 2007.

TABLE I. Properties of PILATUS I and II chips (Kraft, 2005).

Properties	PILATUS I	PILATUS II
Technology	DMILL	UMC 0.25
Active area	10.1×17.7 mm^2	10.45×17.54 mm^2
Number of pixels	$44 \times 78 = 3432$	$60 \times 97 = 5820$
Pixel size	217×217 μm^2	172×172 μm^2
Counter (pixel)	15 bit	20 bit
Count rate	<10 kHz/pixel	Up to 3 MHz/pixel

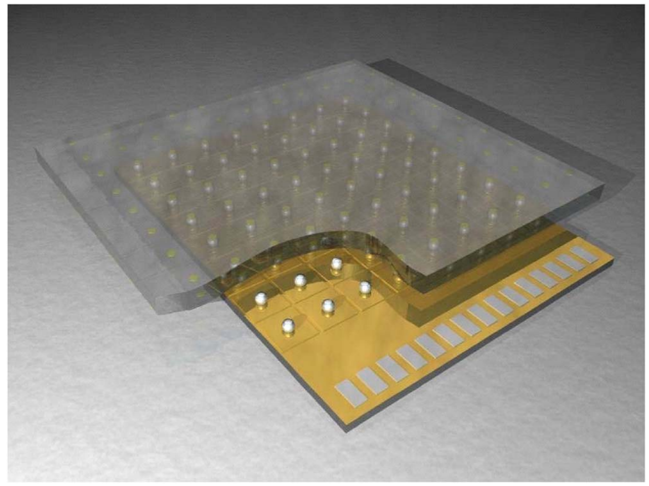

Figure 2. (Color online) Hybrid detector scheme.

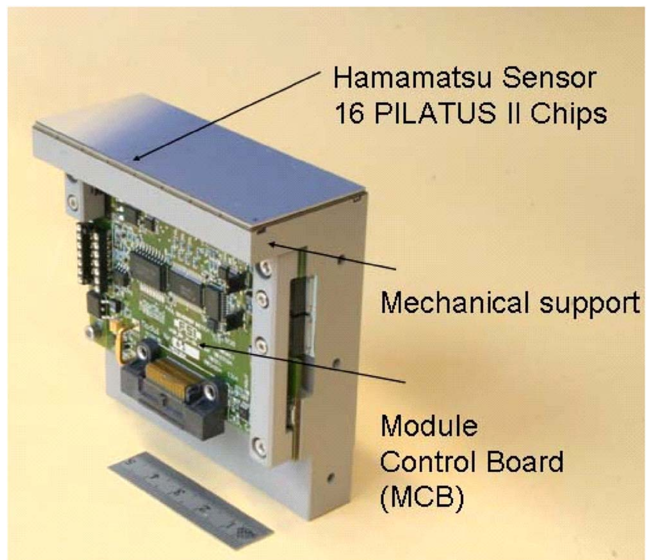

Figure 3. (Color online) PILATUS II module.

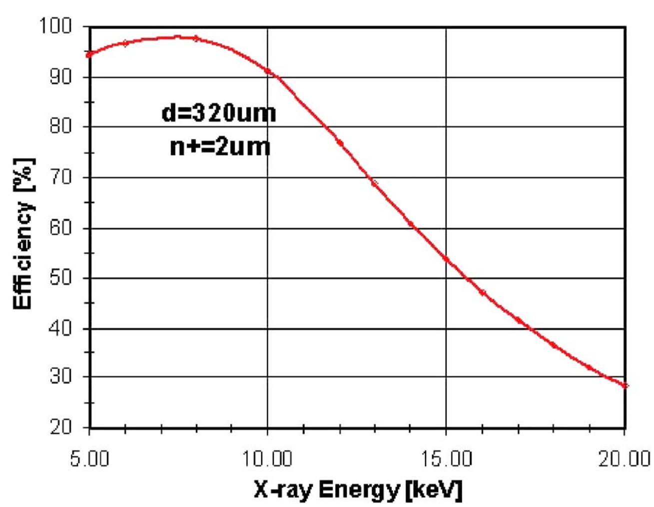

Figure 4. (Color online) PILATUS 100K sensor (detective quantum efficiency).

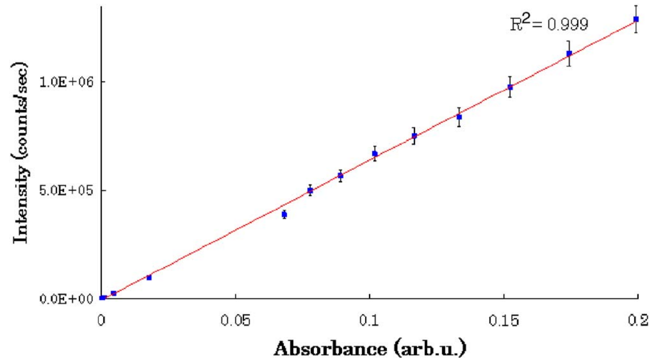

Figure 5. (Color online) Counting rate of single pixel.

B. Technology

The detector consists of a two-dimensional array of pn-diode, usually processed in high resistivity silicon, connected to the readout Application Specific Integrated Circuit (ASIC), which is designed with the latest complementary metal-oxide semiconductor technology. These two parts are connected by small metal balls (bump-bonding). In principle, the sensor can be chosen out of a variety of materials. Hence, this type of detector is often called "hybrid detector" (Figure 2). PILATUS II single module consists of 16 (2×8) PILATUS II readout ASICs which are bump-bonded to a 320-μm-thick monolithic silicon sensor. The assembly is connected to a mechanical support carrying the module control board (Figure 3). After assembling, the detector is calibrated by exposing it to a homogeneous field of X-rays, and the trim bit of each pixel is adjusted in order to have a uniform threshold (Brönnimann *et al.*, 2000).

For X-ray energies above 8 keV, X-rays penetrate the sensor and a certain dose is deposited in the readout chip. Therefore a radiation hard design is applied and the PILATUS II chip can tolerate more than 30 Mrad dose.

A fully depleted 320-μm-thick silicon sensor absorbs 99% of incident X-ray at 8 keV and 40% at 17 keV (Figure 4). Since most in-house X-ray diffraction experiments are carried out with Cu $K\alpha$ X-rays (8 keV), there is no need to make the silicon sensor thicker or to replace it with other materials such as GaAs or CdTe. For some experiments using Mo $K\alpha$ X-rays (17 keV), the efficiency goes down to less than 40%, but it still provides sufficient performance. Thicker silicon sensors are possible, however it is difficult to make large monolithic sensors with GaAs or CdTe.

C. Performance

Since PILATUS is a single-photon counting digital detector, it is not suffering from dark current or readout noise. For setting the threshold level correctly, PILATUS covered with a lid only counts cosmic background for minutes-long exposure. High counting rate capability was tested by putting

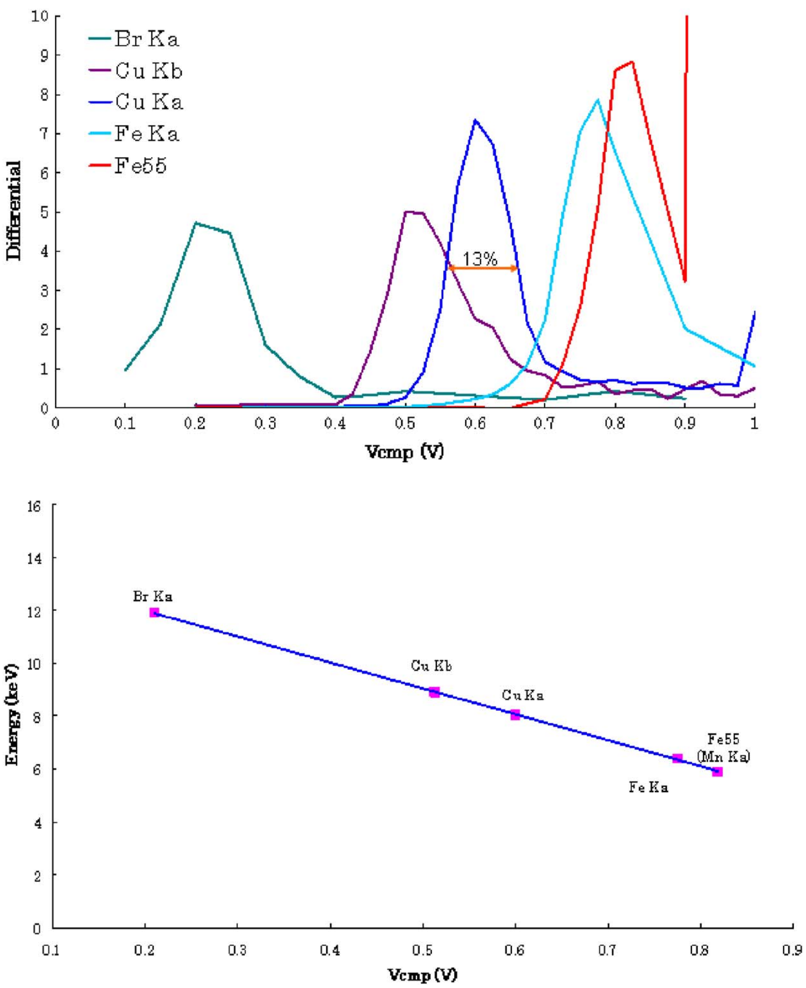

Figure 6. (Color online) Threshold scan result (top: peak width; bottom: energy linearity).

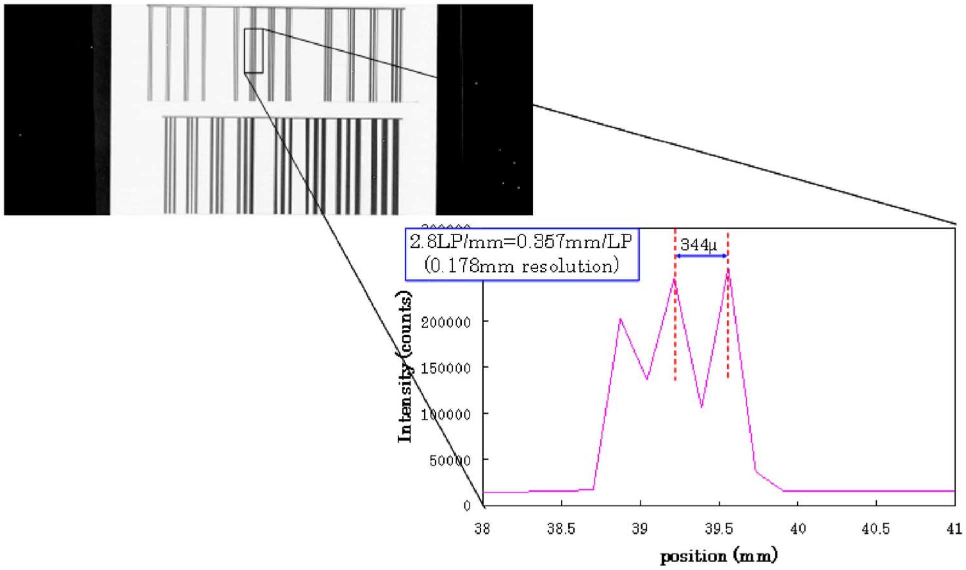

Figure 7. (Color online) Spatial resolution test result.

thin aluminum foils in front of the detector. In Figure 5, the number of photons counted in a second is plotted against the thickness of the absorber. Counting rates beyond 10^6 cps/pixel were confirmed with a laboratory X-ray source. However, since this is a counting detector, count-rate corrections need to be applied, for rates above ~100 kcts/s/pixel. In order to cover different experimental needs, there are three settings of the pixel amplifiers. For standard settings, the dead time measured is set to ~200 ns. For X-rays with energies below 5 keV, high-gain setting may be used and dead time of 300 ns is applied. The low-gain mode can be chosen for very high counting rate measurement with dead time of 129 ns.

PILATUS detectors have a lower threshold level so that it can suppress fluorescence background. X-rays of various energies (5.9, 6.4, 8, 8.9, and 11.9 keV) were irradiated, and output intensity was measured by scanning the threshold level by changing the value of Vcmp. As a result, it is observed that the FWHM of Cu $K\alpha$ X-rays is about 13% (130 mV/90 mV/keV/2.35) and the Vcmp value is linear to input energy (Figure 6).

Because PILATUS is a solid state pixel detector, it is not expected to be affected by parallax like a gas counter. An X-ray test chart with various line spacings was put in front of the detector and irradiated by a micro focus X-ray source with 10 μm focal spot size from a distance of 500 mm. The 2.8 line pair/mm, or 0.357 mm/line pair, feature was resolved with about 50% modulation (Figure 7). This indicates the spatial resolution of this detector is about 178 μm, which is equivalent to the pixel size. Thus the point spread function of this detector is the same as the pixel size.

Figure 8. (Color online) Combination of PILATUS 100K (top left: PILATUS 100K system; top right: PILATUS 100K with XRD-DSC II; bottom left: PILATUS 100K with Nano-Viewer; bottom right: PILATUS 100K with AFC-10).

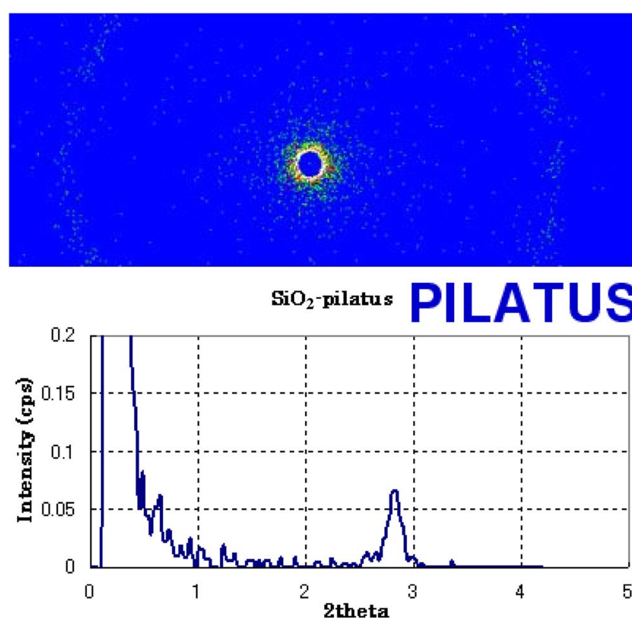

Figure 9. (Color online) SAXS pattern (top) and the annual integration (bottom).

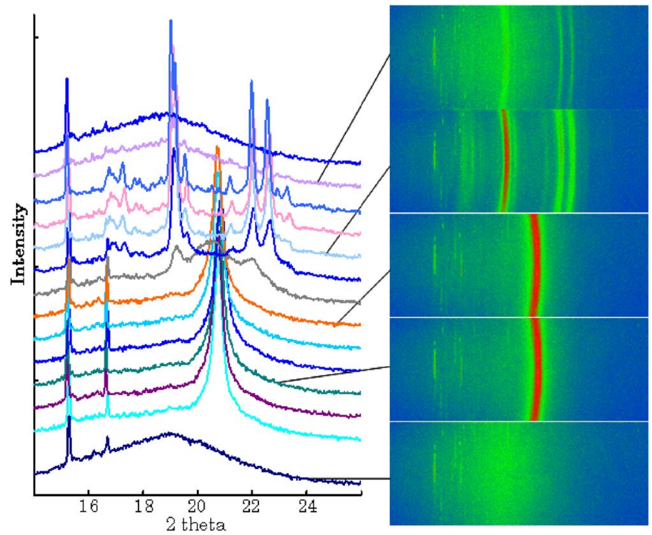

Figure 10. (Color online) Phase transition of Tripalmitin.

D. Applications

A PILATUS 100K detector system was combined with various in-house XRD systems (Figure 8) and tested. The integration to in-house systems was surprisingly easy and the detector started up instantaneously.

One of the most attractive features of the PILATUS 100K is its very low background noise and very high counting rate. It is rare to find these two features combined in conventional X-ray detectors. These two features are particularly good for SAXS measurements: Figure 9 shows a SAXS data of meso-porous silica (SiO_2) measured with a PILATUS 100K detector. The exposure time is 1 s and the intensity of a 60° to 120° arc is accumulated and plotted. The background for the whole data range is almost zero, and the weak scattering peak at $2.8°2\theta$ is clearly visible. According to this data, signals less than 0.05 counts/s can be observed. Thus very faint scattering can be observed by using the PILATUS 100K detector. One of the other advantages of using this detector for SAXS experiments is the parallax-free measurement with good point spread function described below. Using conventional area detectors, such as IP or CCDs, the exposure time needs to be longer and a higher background is usually observed.

One of the other key features of PILATUS is the fast framing rate of 300 Hz. It can collect data without shutter control, minimizing the dead time and it can thus easily be used for dynamical phase transition study, for example experiments as carried out using a small area detector (Taguchi, 2006). The phase transition process of Tripalmitin (neutral lipid) was observed using the XRD and DSC (differential scanning calorimetry) combined apparatus with PILATUS 100K. The sample was heated up to 85 °C and phases were observed in cooling stage (Figure 10). The PILATUS 100K can cover about $17°2\theta$ with angular resolution of 0.0346° at the distance of 285 mm from the specimen.

III. SUMMARY

The PILATUS 100K detector system is combined with in-house XRD systems. It provides new, quality data compared with conventional detectors and will open new frontiers in the field of X-ray diffraction for in-house XRD users as well as synchrotron radiation users.

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