Single crystal diffraction systems based on hybrid pixel array technology

XtaLAB PRO series

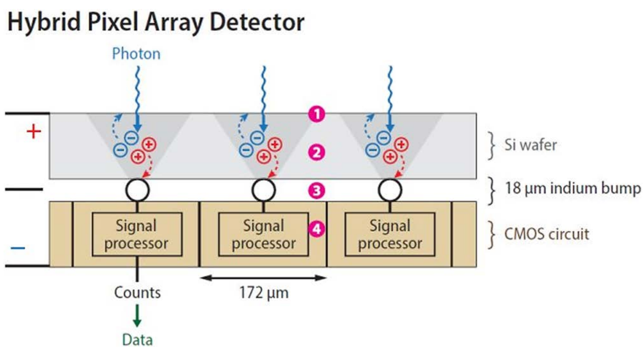
1. Introduction

Rigaku has developed a new series of single crystal diffractometers that address a wide range of sample types, from small molecules to MOFs, to biological macromolecules. The key component that is common for this series of diffractometers is the use of an HPAD (hybrid pixel array detector), a technology that produces an almost perfect detector and greatly expands the capabilities of a single crystal diffractometer in terms of speed of data acquisition and more accurate measurement of weak data. The standard detector in the XtaLAB PRO series is the PILATUS 200K (Fig. 1), a detector that is well proven in the field and based on the same technology adopted by synchrotron beamlines around the world. The outstanding characteristics of these detectors ensure that every XtaLAB PRO diffractometer will perform to produce the best data possible for the X-ray source selected.

Fig. 1. The PILATUS 200K HPAD detector.

2. Why the HPAD outperforms other detectors

There are basically four types of detectors currently being used with single crystal diffractometers. The newest, the HPAD detector (Fig. 2), is a photon counting



1. X-ray photon absorbed in the Si wafer.
2. Electron hole pairs are generated and funneled towards Indium bump.
3. Si wafter is connected to CMOS circuit by Indium bump for effective charge transfer.
4. Pixel signal processing unit, shapes and amplifies then compares vs reference value, if above, it is counted, if below not counted.

Fig. 2. How the HPAD detector works.

detector that differs from the other three detectors in that it directly detects X-ray photons without the intermediate step of converting the photons to light with a phosphor. Without a phosphor and associated substrates, the point spread function of the HPAD detector is defined by the actual pixel size and there is none of the blooming that occurs from the phosphor and substrate.

Imaging plate (IP) based diffractometers have been in production for over 25 years but still are in use because very large apertures can be created, they can be exposed for long periods of time due to the lack of electronic dark current and they are comparatively inexpensive. CCD based detectors are more sensitive than IP based systems but suffer from a reduced dynamic range and errors introduced from the phosphor and glass stub/taper assembly. CMOS based detectors have been introduced more recently and offer a larger aperture than a CCD detector but are intrinsically noisy and still require a phosphor and glass stub assembly for converting X-ray photons to visible light.

Integrating detectors such as CCDs and standard CMOS detectors, have some additional disadvantages. As they use a fiber optic stub (CMOS) or taper (CCD) light loss occurs at the interfaces between the phosphor-stub and stub-sensor as well as in the glass material itself. Diffusion of light in the phosphor and loss of light through the fiber optic stub means CCDs and standard CMOS detectors have a point spread function with longer tails than a Gaussian such that data is spread across many pixels.

As a result of no light diffusion within the detector, HPAD spots have a 'top-hat' point spread function of one pixel rather than the long tail PSF seen with CMOS and CCD detectors.

There are three characteristics of the HPAD detector that make it especially good for the measurement of single crystal diffraction data. First, the readout speed of the detector is so fast that the diffractometer can be run in true shutterless mode. The shutter is opened at the beginning of a scan and the scan axis moves continuously until the scan is finished. Images are constantly being readout during the scan and since the readout time is insignificant they can be treated the same way that single images are normally handled. Shutterless data collection mode leads to huge time savings in data acquisition.

The second characteristic of the HPAD detector that makes shutterless data collection a routine technique is the high dynamic range which means that rescans due to intense reflections are not necessary. With a CCD detector, the shutter is opened and closed with each image and you have to wait as the image is read out. If there is an overload (due to the restricted dynamic range) then a rescan has to occur. In addition to the speed improvement, shutterless data collection eliminates errors that are associated with the opening and closing of shutters as well as the ramping up and ramping down of scan axes.

The third important characteristic of the HPAD detector is the fact that the noise level is essentially zero (no detector is perfect and so it is not correct to call it a noiseless detector, but the HPAD is the closest thing to a noiseless detector available today). A detector with extremely low noise is important because it means that you can measure weak reflections more accurately since backgrounds are as low as possible. Any crystal will have a combination of relatively weak and strong reflections but crystals that most benefit from the accurate measurement of weak reflections are crystals that scatter poorly. With an HPAD detector, the low noise characteristic means that you can expose a crystal for significantly longer periods of time than either a CCD or a CMOS based detector. There is no significant build up of noise with the HPAD detector and you can measure diffraction from poorly diffracting crystals that would be swamped with electronic noise in other types of detectors.

3. X-ray Source Configurations

While the HPAD detector is the common element of the XtaLAB PRO series, the selection of X-ray sources is quite diverse and allows one to select an X-ray source configuration based on the radiation type or types as well as the level of flux desired.

The standard configuration for the XtaLAB PRO (Fig. 3) includes a Cu MicroMax-003 microfocus sealed tube source and a 3 kW Mo standard sealed tube source with a SHINE (curved graphite monochromator) optic. The selection of a microfocus Cu source and a standard focus Mo source is based on the difference in efficiency between Cu and Mo radiations when coupling micro focal spots with multilater optics. Figure 4 shows the relative flux through a $100\,\mu$m aperture at the crystal position for both a standard sealed tube Cu source with graphite monochromator and a microfocus Cu X-ray source with a multilayer optic. The approximately 12-fold improvement in flux at the sample makes the use of the Cu microfocus X-ray source an easy decision.

Figure 5 shows the relative flux through a $100\,\mu$m aperture at the crystal position for a standard sealed tube Mo source with graphite monochromator, a standard sealed tube Mo source with a curved graphite

Fig. 3. The standard configuration of the XtaLAB PRO series includes a PILATUS 200K detector, a kappa goniometer, a Cu microfocus sealed tube source (MicroMax-003) and a 3 kW Mo sealed tube source.

monochromator (SHINE optic) and a microfocus Mo X-ray source with a multilayer optic. It is easy to see that the standard Mo source with the SHINE optic produces equivalent X-ray flux at the sample as the microfocus Mo source. The decision to include a standard Mo X-ray source with the XtaLAB PRO standard model is based on the wide availability and ease of replacement of standard Mo X-ray tubes. While the XtaLAB PRO can be equipped with a Cu and Mo microfocus source, the performance will not be improved and, in fact, for larger crystals, the microfocus Mo source produces less flux at the sample than the standard Mo sealed tube source with a SHINE optic.

Beyond the standard configuration of the XtaLAB PRO, it is possible to increase the flux at the sample significantly by utilizing a rotating anode based X-ray source and Rigaku offers a number of models and configurations to choose from.

The MicroMax-007HF is a microfocus rotating anode generator and the most popular rotating anode source utilized for single crystal analysis around the world. It is available in both a single wavelength configuration as well as a unique double wavelength configuration. In the double wavelength configurations (Mo/Cu or Cu/Cr), the wavelength to be used can be selected automatically.

For even more flux at the sample, the FR-X microfocus rotating anode generator is available and is the most powerful rotating anode X-ray source for single crystal analysis available today.

These two rotating anode sources are well proven in the field and offer a low-maintenance regimen compared to rotating anodes of the past. While the ongoing maintenance of a rotating anode generator is more than that of a sealed tube generator, the increase in flux at the sample is significantly higher and allows the researcher to measure samples that could only previously be measured at a synchrotron.

Figures 7 and 8 show the relative flux through a 100 μm aperture at the crystal position for a standard sealed tube source, a microfocus sealed tube source, the MicroMax-007HF, and the FR-X for Cu and Mo radiation respectively.

For Cu radiation, the 7 and 19 fold increases in

MicroMax-007HF FR-X

Fig. 6. Microfocus rotating anode generators MicroMax-007HF and FR-X.

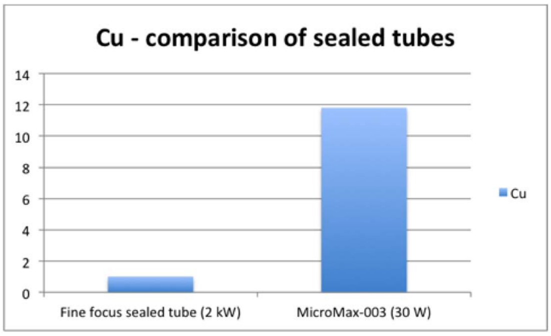


Fig. 4. Relative flux through a 100 μm aperture at the crystal position for sealed tube Cu sources.

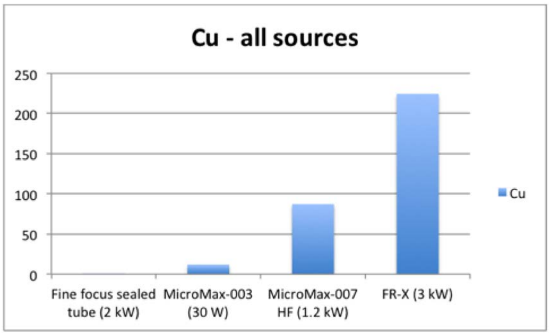


Fig. 7. Relative flux through a 100 μm aperture for all Cu sources.

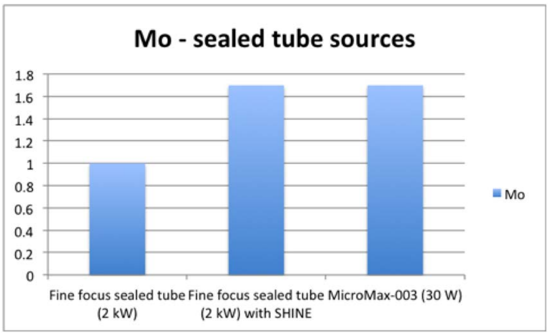


Fig. 5. Relative flux through a 100 μm aperture at the crystal position for sealed tube Mo sources.

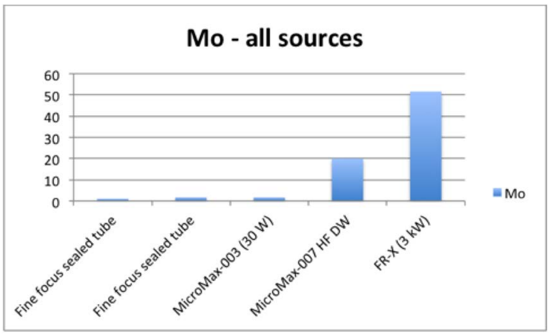


Fig. 8. Relative flux through a 100 μm aperture for all Mo sources.

Fig. 9. The XtaLAB PRO enclosure was designed to allow maximum access to the instrument.

Fig. 10. There is enough room inside the XtaLAB PRO enclosure to place items that can be useful in mounting sensitive crystals quickly.

flux for the MicroMax-007HF and FR-X respectively compared to the MicroMax-003 (microfocus sealed tube) is a clear indication of the performance improvements that are possible when using rotating anode based X-ray sources. It is even more significant for Mo radiation where 12 and 30 fold improvements can be gained from the MicroMax-007HF and FR-X respectively compared to a Mo sealed tube source with a SHINE optic or a Mo microfocus sealed tube source.

4. A Work Environment Designed for Research

The XtaLAB PRO series is housed in a newly developed enclosure (Fig. 9) that was designed to improve the workflow of mounting air-sensitive and temperature sensitive samples. There is space inside the XtaLAB PRO enclosure for a microscope and a dewar (Fig. 10). No matter what type of samples you are working with, the ability to identify and mount crystals in the proximity of the diffractometer can be a true time saver. In the case of air-sensitive and temperature-sensitive crystals, having close proximity of the mounting station to the goniometer can mean the difference between a crystal that diffracts and a crystal that dies.