ESRF

Experiment title: Development and Exploitation of the ESRF Fast Read-out CCD camera for Protein Cryst allograph Experiments

Experiment number:

MI **118**

Beamline:

BM-14

Date of Experiment:

from: 12th April 1996 to: 14th April 1996

Date of Report:

16th September 1996

Shifts:

9

Local contact (s):

None

Received at ESRF:

17 SEP 1996

Names and affiliations of applicants (*indicates experimentalists):

Andrew Hammersley*, Valerie Biou*(+), L Claustre*(#), Stephen Cusack(#), Ana Gonzalez*, Jean-Claude Labiche*, Sean McSweeney*(#), Jean-Pierre Moy*, Janet Smith(#), Olof Svensson*, Andrew Thompson*(#),

ESRF, BP 220, F-38043 Grenoble, France

+ IBS, 41 Av. des Martyrs, 38042 Grenoble, CEDEX, FRANCE

EMBL, C/O ILL, BP 156, Grenoble 38042, CEDEX 9, FRANCE

Report:

The Detector Group fast read-out ("frelon") CCD camera was installed on BM-14 in place of the standard Princeton Instruments CCD camera, optically coupled to the standard beam-line berylium windowed X-ray image intensifier (XRII). A SDV single channel digital interface was used to read-out into a Sun workstation; control was by spec. The camera in this configuration has a read-out time of roughly 0.3 seconds, so is more than 10 times faster than the standard beam-line system. However, it is slightly more restricted in the dynamic range of the ADC (14-bits) compared to the Princeton system (16-bits).

It is hoped that such a fast read-out system maybe useful in allowing thin "phi-slicing" given that the read-out dead-time over-heads are much lower than the exposure time even for a 0.10 oscillation.

Results

The same 45° of data from the same synthetase crystal were collected in 0.25° and 1° oscillations. The data were corrected for distortions with FIT2D, integrated with DENZO, and scaled with SCALEPACK. The R-merge statistics against resolution are shown in Table 1 for the 1° oscillations. The overall R-factor of 3.7% to 3.0Å and the general high quality of the data are very encouraging, and show the system to be useful for crystallographic measurements.

However, the statistics from the 0.25° oscillation data were "poorer" than for the 1° data, with an overall R-merge of 4.79'0. This is disappointing, in that the advantage of thin "phi-slicing" has not been demonstrated, but closer inspection of the processing output strongly suggests that the integration is not optimum. (The post-refined "mosaic spread" varies enormously from one frame to the next.) From this we conclude that prior to making any assumptions on the success or failure of the thin "phi-slicing", it is necessary to re-integrate using 3-D profile fitting software. (We understand that more recent versions of DENZO/SCALEPACK are available which are able to cope with oscillation ranges smaller than the "mosaic spread", and the data has been made available to the author.)

Recommendations

- Detector prototypes need to be more robust for realistic beam-line testing. A number of problems were encountered which resulted from the camera being a "lab-bench" prototype. The principal problem of over-heating has since been solved. Prior to "full-scale" production it is vital to test equipment on the beam-lines, and prototype systems need to be developed with this in mind.

- A mechanical light shutter needs to be added to the system for crystallographic use. The phosphor continues to glow for 10's of milliseconds after the X-ray shutter is closed. This glow continues during read-out of the CCD leading to vertical asymmetric "tails" from strong peaks, which could seriously affect weak peaks. Whilst a faster output phosphor is available for time resolved applications, this is not necessary for crystallographic applications and results in a loss of resolution. A simple mechanical light shutter, such as those used on commercial systems is needed to solve this problem.

Conclusions

- The frelon camera (used together with the XRII) produces good data.

- The SDV read-out via spec is reliable and well adapted to monochromatic crystallographic measurements.

Table 1: Scalepack R-Merge Statistics from the 1° Synthetase Data

Lower Limit	Upper Limit	Average I	Error	Average stat.	Norm. Chi**2	Linear R-fat	Square R-fat
15.00	7.14	6298.1	237.1	207.5	1.100	.026	.033
7.14	5.77	4310.4	160.7	138.8	.974	.023	.032
5.77	5.08	5855.7	240.8	175.0	.927	.027	.032
5.08	4.63	7864.1	313.1	219.7	1.433	.029	.035
4.63	4.30	9284.5	319.1	263.5	1.715	.031	.039
4.30	4.06	7552.4	329.7	243.5	1.304	.029	.033
4.06	3.86	5674.8	275.3	216.4	1.564	.037	.047
3.86	3.69	5102.1	270.8	220.5	1.574	.041	.051
3.69	3.55	4190.4	261.8	224.2	1.273	.044	.046
3.55	3.43	3531.5	249.8	220.0	1.073	.041	.044
3.43	3.32	2992.7	285.1	230.5	.853	.052	.047
3.32	3.23	2359.8	273.6	236.1	.801	.055	.059
3.23	3.15	2007.1	266.2	238.6	1.040	.074	.081
3.15	3.07	1843.0	272.3	248.3	1.073	.077	.086
3.07	3.00	1643.7	286.2	266.8	1.086	.089	.088
All reflections		4577.7	270.8	224.6	1.183	.037	.041