

Developments toward standardization of protein crystallography experimental station at bending-magnet beamlines

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For the purpose of commissioning of JASRI/NASDA joint protein crystallography system called MIPS, Microgravitatorial and Industrial Protein structure analysis System, fundamental data regarding to the diffraction experiment and fluorescence absorption spectrum have done successfully. The result provides the fundamental information on the genome beamline of RIKEN in the commissioning. We checked the accuracy of energy selection and the level of the fix exit from the viewpoint of protein crystallography especially focused on the MAD method. Because the achievement of the commissioning of double crystal monochromater have some variation among the beamline, deviation can be seen from the use side. The standardization and consistent achievement will be achieved via the improved user satisfactory.

Two research groups including Dr. Chen Chun-Jung from SRRRC, Dr. Yen-Chywan Liaw and his student participated in this visit. The proteins and experiments performed at BL38B1 are listed in the Table 1.

Overall result for this visit is successful. The long-run data collection at 1.54 Å showed the radiation damage on one of three disulfide bonds of insulin crystals in a comparison of the data at 0.907 Å. These systematic sulfur SAD studies will enhance our understanding on the strategy of data collection. The diffraction result from Upps, a very small crystal, gives a very good diffraction pattern and a very good Rmerge.

The NDK (Nucleotide diphosphate kinase) crystal diffracts up to 2.0 Å while the original in-house data was collected only to

2.8 Å resolution. The thioesterase S10G experiment also gave good data. A high-resolution data set of the monoclinic form volvatoxin was successfully collected up to 1.9 Å resolution.

Table 1 Results data collection.

| Protein | Experiment | Resolution / Å | Result |
|-------------------|-----------------|----------------|---------------|
| NDK | Good Data | 2.0 | Good data |
| transglutaminase | Good Data | - | Crystal small |
| Insulin | Damage effect | 1.8 | Positive |
| Volvatoxin Mono | High resolution | 1.9 | Good data |
| Volvatoxin Tetra | High resolution | - | No Beam Time |
| Volvatoxin Mono | Br MAD | 2.5 | |
| Volvatoxin Tetra | Br MAD | 2.8 | |
| Thioesterase S10G | Good data | 1.9 | Good data |
| Upps | Good data | 3.0 | Good data |

The energy fluorescence scans for Br is very successful. A clear absorption edge was obtained. Four MAD data sets for four different soaking conditions were obtained. The data sets are still under analysis. However, since the data collection time for this experiment is very time consuming. We have no chance to collect data for half of our crystals.

Rare Gas Mixtures Excited with Synchrotron Radiation X-ray Beam

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1. Introduction

Ionization columns produced with X-ray beams in gases have a lateral extension that is basically determined by the beam size and the range of the electrons generated. The electron and ion densities in the columns could reach "quasi-plasma" state, when the third generation of synchrotron radiation X-ray beams are employed [1]. From the viewpoint of radiation physics, it is of important to explore the nature of ionization column in "quasi-plasma" state, *i.e.*, the spatial-temporal distributions of electrons and ions in the columns.

2. Multistep Parallel Plate Chamber

In order to experimentally investigate ionization columns produced with X-ray beams in rare gas mixtures, we have constructed a multistep parallel plate chamber (hereafter referred to as MPPC), which consists of a drift volume and a detection volume (Fig.1).

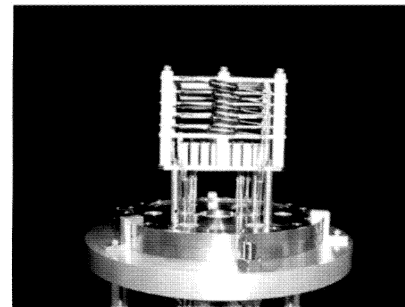


Fig. 1 The drift and the detection volumes configured with multiple electrodes of the MPPC. The entire electrode structure is positioned onto a stainless flange to be enveloped with a vacuum vessel.

The charge carriers (either electrons or ions)

generated along an incident X-ray beam in the drift volume would drift towards the detection volume, where eight anodes are placed to collect the carriers. The MPPC also has two optical windows; one for an image intensifier visualizing the optical image of the incident X-ray beam, and the other for a photomultiplier observing the intensity of the primary and secondary scintillation induced in the rare gas mixture filled. The MPPC thus has several operation modes such as ionization chamber mode, proportional counter mode, scintillation counter mode, proportional scintillation counter mode, and luminescence imaging mode.

3. Experimental Results

The MPPC was operated under the irradiation of synchrotron radiation X-ray beams at BL38B1 June 2001 as the first time. Since the beamline provides a moderated intensity of X-ray beams, it was best suited for the MPPC at its early stage to verify its performance. The MPPC functioned in the all modes mentioned above, and succeeded in visualizing the ionization column image induced with 20 keV X-ray beam in argon gas at 1 atm (Fig.2). The data analysis is currently in progress to extract further information.

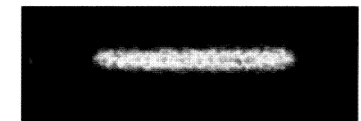


Fig. 2 The observed ionization column produced with 20keV X-ray beam in 1 atm argon gas. The visualized part of the column is 90 mm long in the present case.

Ref. [1] M. Suzuki and H. Minchira, Nuclear Viewpoints Vol. 45 No.2 (1999)62.