

s7.m0.p3 **Structural Studies by the Debye-Scherrer Camera installed at BL02B2, Spring-8.** A. E. Nishibori^a, M. Takata^a, K. Kato^a, Y. Kubota^b, Aoyagi^c, Y. Kuroiwa^c, S. Ikeda^d and M. Sakata^a, ^a*Dept. of Appl. Phys., Nagoya Univ., Nagoya 464-8603, Japan,* ^b*Osaka Women's University, Osaka 590-0035, Japan* ^c*Dept. of Physics, Okayama University, Okayama 700-8530, Japan,* ^d*JASRI, Mikazuki, Sayo 679-5198, Japan.*

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From the materials science view point, it has a fundamental importance to understand the relationships between accurate structures of materials and their physical and/or other properties. For the purpose, it is essential to carry out structural studies at various temperatures. It would be additional benefit if the accurate structure analyses could be done by powder specimens. It is not always easy task to perform a rapid collection of X-ray diffraction data at desired temperatures. A Debye-Scherrer camera, which is now installed at BL02B2, Spring-8, is designed for accurate structure analyses for materials science by taking advantage of third generation SR.

This instrument makes it possible to collect high-angular and high-energy resolution powder data very rapidly, which must contribute to increase the accuracy of structure analysis of crystalline material using powder data¹⁻³. This Debye-Scherrer Camera with radius 286.5mm is available in a wide range of temperatures (15-1000K). The displacive cryostat (~15K) is installed within the ω -stage of this camera. A high temperature gas flow system can be also installed for high-temperature experiments. By using high energy X-ray photons of Spring-8, the effects of absorption become insignificant even for heavy materials involving, e.g., rare-earth metals.

It should be noted that there are many levels of structural studies, for example, to measure just lattice constants, to determine space group by observing superlattice reflections, to refine atomic distances and to obtain accurate electron densities at various temperatures. By doing structural analyses at various levels for some standard materials, extremely high performance of the camera has been proved, particularly for accurate density studies when the data are analyzed by the combination of Rietveld refinements and the Maximum Entropy Method. Such a high performance of the Large Debye-Scherrer Camera at BL02B2 is achieved by taking the advantage of intense, highly parallel and high-energy X-ray beams of Spring-8. The performance at low and high temperature experiments will be also presented.

s7.m0.p4 **A cryo-bench for trapping reaction intermediates in protein crystals.** T. Ursby^{a,b}, X. Vernede^b, P. Charrault^b, B. Gorges^a & D. Bourgeois^{a,b}, *a: ESRF, BP 220, 38043 Grenoble, France, b: LCCP, UPR 9015, IBS, 38027 Grenoble, France.*

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In a collaboration between the Institute of Structural Biology (IBS) in Grenoble and the European Synchrotron Radiation Facility (ESRF), we have set-up a new laboratory to prepare crystals in view of structural determination of reaction intermediates. The systems we are studying include photosensitive proteins and reactions triggered using *e.g.* caged compounds or pH jumps. In general, the major obstacle to a successful experiment is to find conditions where the intermediate can be accumulated to a high proportion. Therefore, the possibility to study the reaction in the crystal prior to the X-ray experiment is vital.

Our main interest is in trapping techniques: a reaction is synchronously initiated and then controlled in such a way that a reaction intermediate along the reaction coordinate accumulates in the crystal. This is typically done by choosing a proper temperature profile, selecting appropriate solvents, or adjusting the pH. The crystal can then be flashed cooled to liquid nitrogen temperature, and transferred to a beamline where diffraction data are collected using standard techniques. However, the set-up is equally suitable for finding conditions to accumulate reaction intermediates either transiently or in a steady state. In this case, the reaction initiation would have to be repeated during the diffraction experiment, performed for example with the Laue technique.

The cryobench includes a mechanical support where a crystal can be mounted and examined visually in a microscope. Absorption and/or fluorescence spectra that witness the accumulation or disappearance of intermediates in the crystal can be recorded with a microspectrophotometer while the reaction is controlled by multiple cooling devices. Reaction triggering can be performed by light pulses from a flash lamp or several lasers. The laboratory is situated adjacent to the experimental hall of the ESRF synchrotron allowing fast access to X-ray facilities for prepared samples.

Several results of biological interest have been obtained with the device, and concern for example photoactive yellow protein, bacteriorhodopsin, nitrite reductase or acetylcholinesterase.

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