A Combined CCD/IP Detection System for Monochromatic XRD Studies at High Pressure and Temperature

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Introduction: High-pressure techniques combined synchrotron radiation has opened a new window to view the Earth we live on. While we try to apply all possible high-pressure/x-ray techniques to study the properties of minerals, quality and resolution of the measurements become an important issue for understanding the minerals. The translating imaging plate (TIP) has largely fulfilled its great potential for the acquisition of high quality monochromatic data, allowing the study of time resolved phenomena at high pressure[1, 2]. This has provided not only the elucidation of phase transition, but also refinement of crystal structure based upon these data[3], thereby allowing the transition to be followed on an atomistic level. Unfortunately, some of the advantages of polychromatic studies, chiefly the excellent collimation, which excludes parastitic scattering from the high pressure vessel, and quasi-real-time feed back of the diffraction data, are lost in the monochromatic IP experiments. The former problem can be solved using special cell design and software technique[4], or a set of radial slits at the price of data collection time[5], but the latter can be problematic. For example, a TIP has a limited translation rang and requires real-time feedback to provide a trigger to activate the plate. The phase transition can then be captured optimally without having to trust to guesswork or several loadings of the high-pressure device. Coupling a charge-coupled device (CCD) detector with the TIP can provide such feedback.

Methods and Materials: The combined CCD/IP system is installed on the bases of previous TIP diffraction system[2] and the large-volume press SAM85 at the X17B beamline. A schematic diagram of the system layout is shown in Figure 1. A Bruker Smart 1 magapixel CCD is introduced on the 2θ goniometer arm replacing the solid state detector (SSD) in the previous system. The active detection area of the CCD is 63 mm x 63 mm square. Sample-to-CCD distance and 2θ of the goniometer are can be optimized to maximize the resolution of the diffraction peaks of interest. The CCD and TIP can be actively used simultaneously: the CCD detects upper part of Debye-Scherrer rings, and the TIP collects the lower part.

Results: The following table lists the experiments that have been carried out with the system.

Experiments with the CCD/IP system

Sample	property studied
Fe2SiO4	phase transition
FexO (0.95<x<0.99)	structure
FeS	phase relation
(Mg,Fe)2SiO4	rheology
MgO	rheology
Al2O3	rheology

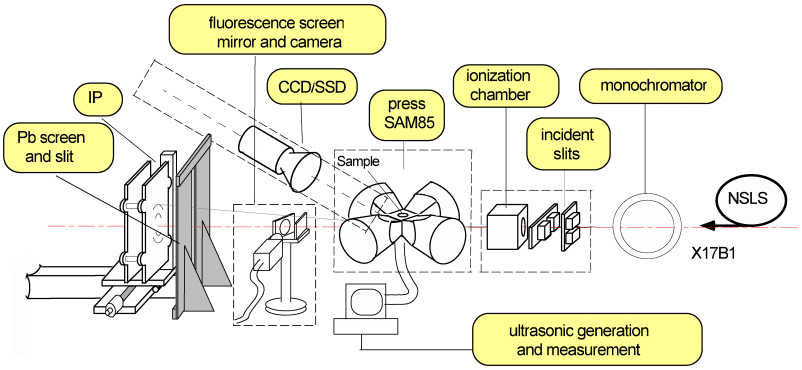

Figure 1. Schematic diagram of the high-pressure x-ray system modified from the previous diffraction system at the X17B1 beamline.

Conclusions: Combination of the CCD and IP detectors yields high quality x-ray diffraction with great experimental convenience for high pressure studies. Quick diffraction feedback enables an efficient time-resolved diffraction measurement; reliable diffraction intensity allows an accurate structure refinement; and high resolution of diffraction pattern rises an opportunity for the rheological measurement at a lower strain level.

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