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High-pressure two-dimensional angle-dispersive x-ray diffraction measurement system using a Kawai-type multianvil press at SPring-8

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Abstract. We have demonstrated the feasibility of a two-dimensional angle-dispersive x-ray diffraction (2D-ADXD) measurement system using a Kawai-type multianvil press at SPring-8. By taking advantage of the x-ray-transparent anvil and energy-dispersive x-ray diffraction (EDXD) techniques, high-pressure 2D-ADXD measurements of KCl were performed up to 9.9 GPa. Entire Debye-Scherrer rings were obtained, and the B1–B2 phase change of KCl was clearly observed at 2.3 GPa. The developed 2D-ADXD measurement system enabled us to obtain enough high-quality diffraction data to precisely determine and refine the structure of KCl at high-pressure.

1. Introduction
High-pressure x-ray diffraction is an indispensable measuring technique for understanding the structures and properties of condensed matter. Synchrotron radiation provides super-intense x-rays; thus x-ray diffraction data can be obtained by using various types of high-pressure apparatuses, such as the large-volume press (LVP) and diamond anvil cell (DAC). Owing to the nature of the anvil material and geometrical restrictions, the energy-dispersive x-ray diffraction (EDXD) technique with the LVP is widely used to determine the high-pressure phase and unit cell volume. The bending magnet beamline (BL04B1) at the SPring-8 synchrotron radiation facility in Japan is designed for high-pressure and high-temperature research using the LVP, which employs a Kawai-type high-pressure module consisting of eight inner anvils surrounded by six outer anvils [1]. A wide range of high-energy white beams pass through the small gaps between the inner and outer anvils, and the EDXD pattern can be obtained by a Ge solid state detector (SSD) at a fixed small 2\(\theta\) angle (<15°). However, the occurrence of crystal grain growth due to high-pressure and high-temperature often breaks the homogeneous powder diffraction condition and induces loss of the diffraction peaks derived from a spotty Debye-Scherrer ring. This has been a serious problem for high-pressure and high-temperature 1D-x-ray diffraction using the LVP (e.g., [2]). In the EDXD technique, only a limited part of a Debye ring is observed; therefore observation of a whole Debye ring is desired to overcome the problems from grain growth.

Furthermore, recent technological developments on Kawai-type high-pressure modules have been remarkable, and inner anvils of various materials, such as sintered diamond with cobalt (Co-SD), sintered diamond with silicon (Si-SD), cubic boron nitride (cBN), and diamond-SiC (d-SiC) composite, have become available along with the conventional tungsten carbide (WC) anvil. In particular, Si-SD,
cBN, and d-SiC anvils have not only higher pressure generation than WC anvil but also higher x-ray transparency: about 40% of white x-rays within an energy range of 20–150 keV can pass through a 14-mm-thick cubic anvil. Thus, by placing these x-ray-transparent anvils on the scattered x-ray beam positions in a Kawai-type high-pressure module, the observed 2θ angle can be extended and a wider d-spacing range of diffraction data can be obtained by using Si-SD [3], cBN [4], or d-SiC [5] cubes. Moreover, Kubo et al. (2008) obtained the Debye-Scherrer ring of Si up to 15 GPa, combining the monochromatic beam and Si-SD cubes with 10-mm edge length at the Advanced Photon Source (APS), GSECARS, in the United States [6]. Although only the 1D-EDXD method has been used previously on a Kawai-type LVP at the BL04B1 beamline of SPring-8, we installed a monochromator and set up a high-pressure 2D-ADXD measurement system combined with the x-ray-transparent anvil. Moreover, by a combination of the EDXD and the ADXD techniques, high-quality diffraction patterns can be obtained from the entire Debye-Scherrer ring. In this paper, we describe high-pressure 2D-ADXD measurements of KCl using a Kawai-type LVP up to 9.9 GPa.

2. Experimental setup

2.1. Design of the high-pressure 2D-ADXD system

The high-pressure 2D-ADXD system (Fig. 1) installed at the BL04B1 beamline consists of a compact water-cooled double-crystal monochromator, a SPEED-1500 module, and an imaging plate (IP) detector on the upstream side. An incident white x-ray beam without any focusing device is monochromatized by the first and second silicon (111) crystals with an 8-mm vertical offset from the incident beam (Fig. 1(a)). The exit monochromatic beam is tunable in the energy range from 30 to 60 keV. The beam is typically collimated to 0.2 x 0.2 mm² (horizontal (H) x vertical (V) direction) by the slits, and its intensity is constantly monitored by an ion chamber. When the measurement is changed to the EDXD mode, the first-silicon crystal is moved down to pass through the direct white x-ray beam.

The Kawai-type high-pressure module of the SPEED-1500 was improved to extend the observable solid 2θ angle, and a half-cut wedge-shaped groove (±10°) was formed on one side of two downstream outer anvils (Fig. 1(b)). In addition, two downstream inner WC anvils were replaced by cBN anvils, and then the scattered x-rays were transmitted into the cBN cubes. The 2D-ADXD pattern was recorded by a flat imaging plate detector (200(H) x 250(V) mm², FUJIFILM Co., Ltd., Tokyo, Japan), which was placed at a distance of 520 mm from the sample center (Fig. 1(c)). The data recorded on the IP were scanned and digitalized by a conventional IP reader (BAS-2500, FUJIFILM Co., Ltd.) with a pixel size of 0.1(H) x 0.1(V) mm².

![Figure 1](image.png)

Figure 1. The high-pressure 2D-ADXD system installed at the BL04B1 beamline of SPring-8 consists of (a) a compact water-cooled double-crystal monochromator, (b) a Kawai-type module of SPEED-1500, and (c) the imaging plate (IP) detector.
2.2. Experimental procedure
To obtain $d$-values from the IP data, both the monochromatic beam energy and the sample–detector distance must be determined before the 2D-ADXD measurement by measuring a standard sample (e.g., CeO$_2$) whose $d$-values are well known. In this system, we use CeO$_2$ as the standard sample to calibrate the energy of the monochromatic beam and to determine the sample–detector distance. For calibration of the monochromatic beam energy, CeO$_2$ is measured using the monochromatic beam and the Ge-SSD, which is preliminarily calibrated by using other standard metals. For determination of the sample–detector (IP) distance, we chose the EDXD mode using the white beam and the Ge-SSD. When the Ge-SSD is set on a horizontal goniometer at a fixed $2\theta$ angle, the position of the diffraction center of EDXD can be determined from the intensity profile of CeO$_2$ by scanning the sample position along the direct white beam direction. If the incident white and monochromatic beam position is moved only along the $z$-axis direction, the position of the diffraction center of EDXD is not changed in either the EDXD or ADXD modes. The sample–detector distance can then be determined by adjusting the sample position to the diffraction center of EDXD. Even if the sample position is moved by changing the sample or deforming the sample container by compression, the sample position can always be adjusted by an EDXD measurement of the sample. The recorded Debye-Scherrer rings were successfully converted to 1D-diffraction data by integrating them over the entire 360° azimuth angle using WINPPIP software [7]. Figure 2 compares 1D-diffraction patterns of San Carlos olivine obtained by the EDXD and ADXD methods.

The pre-sintered powder sample was packed into a cylindrical BN chamber with 1.2-mm inner diameter and 1.1-mm length. EDXD was performed using a high-energy white beam (0.05(H) x 0.1(V) mm$^2$) with an energy range of 10–150 keV at an accumulation time of 200 s (Fig. 2(a)). Diffracted x-rays were collimated to 0.05(H) x 2(V) mm$^2$ using a collimator and detected at a fixed $2\theta = 6^\circ$. An ADXD measurement was also performed, and Debye-Scherrer rings of more than 1.5 Å on the IP detector were obtained at an exposure time of 20 min. The incident monochromatic beam was collimated to 0.2(H) x 0.2(V) mm$^2$ with energy of 40.03 keV, and the sample–detector distance was 519.1 mm, which was determined within an accuracy of 0.1 mm by scanning the intensity profile of standard CeO$_2$ using EDXD. Figure 2(b) shows a converted 1D-diffraction pattern from the Debye-Scherrer rings integrated over the entire 360° azimuth angle by using WINPPIP. This figure demonstrates that ADXD allows us to obtain almost the same quality diffraction pattern as that obtained by using EDXD.

Figure 2. Comparison of 1D-diffraction patterns of San Carlos olivine by the EDXD and ADXD methods. (a) The EDXD pattern was obtained by using a Ge-SSD with a 4096 multichannel analyzer at an accumulation time of 200 s. (b) The ADXD pattern was obtained by using a flat IP detector (200(H) x 250(H) mm$^2$) at an exposure time of 20 min.
3. 2D-ADXD measurements of KCl at high-pressure

High-pressure 2D-ADXD measurements of KCl were performed by using SPEED-1500. cBN cubes with 14-mm edge length were used for the two downstream inner anvils by combining the six WC cubes in the Kawai-type high-pressure module, each of the cubes had a triangular truncation with an edge length of 5 mm. A cylindrical KCl sample with 2.1-mm inner diameter and 1.5-mm length was put into the octahedral 5% Cr$_2$O$_3$-doped MgO pressure medium with 10-mm edge length. To prevent a background peak from the gasket and pressure medium, a boron-epoxy (BE) x-ray window, made from a mixture of amorphous boron and epoxy resin, was placed along the beam path. Details of the sample assembly are described in Nishihara et al. (2009) [8]. Diffraction data of KCl were derived from the IP detector during compression up to 9.9 GPa by each exposure time of 20 min (Fig. 3).

![Figure 3](image)

**Figure 3.** (a) 2D-ADXD patterns of KCl at 1.9 GPa (B1 phase) and 9.9 GPa (B2 phase), each at an exposure time of 20 min. (b) Changes in the 1D-diffraction patterns of KCl with increasing pressure, which were converted from the obtained entire Debye-Scherrer rings by using WINPIP.

The energy of the monochromatic beam (0.2(H) x 0.2(V) mm$^2$) and the sample–detector distance were calibrated by CeO$_2$ to 50.07 keV and 517.4 mm, respectively. Due to the use of the BE x-ray window, very clear Debye-Scherrer rings were obtained and the B1–B2 phase transition was observed by compression (Fig. 3(a)). Figure 3(b) shows the changes in the KCl diffraction patterns with increasing pressure. Pressures were determined from the equation of state for KCl [9]. The B1–B2 phase change occurred from 2.3 GPa and was completely finished at 2.9 GPa. Vaidya & Kennedy (1971) [10] reported the B1–B2 phase transition at 1.7–2.1 GPa. Although the small difference in the transition pressure may be caused by reaction kinetics and the different sample assembly, our result is consistent with the result of Vaidya & Kennedy (1971).

We have demonstrated the feasibility of the 2D-ADXD measurement system using the Kawai-type multianvil press installed at SPring-8. By taking advantage of the x-ray transparent anvil and the EDXD techniques, entire Debye-Scherrer rings were obtained, and the B1–B2 phase change of KCl was clearly observed at 2.3 GPa. The developed 2D-ADXD measurement system will be a powerful tool for phase determination (crystal/melt), structure refinement (Rietveld refinement), or stress measurement (lattice distortion analysis from the entire azimuth angle) under high-pressure conditions.
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