Static compression of B2 KCl to 230 GPa and its P-V-T equation of state

SHIGEHIKO TATENO1,*, TETSUYA KOMABAYASHI2, KEI HIROSE1,3, NAOHIRA HIRAO4, and YASUO OHISHI4

1Earth-Life Science Institute, Tokyo Institute of Technology, 2-12-1 Ookayama, Meguro, Tokyo 152-8550, Japan
2School of GeoSciences and Centre for Science at Extreme Conditions, University of Edinburgh, Grant Institute, The King’s Buildings, James Hutton Road, Edinburgh EH9 3FE, U.K.
3Department of Earth and Planetary Science, The University of Tokyo, 7-3-1 Hongo, Bunkyo, Tokyo 113-0033, Japan
4Japan Synchrotron Radiation Research Institute, 1-1-1 Kouto, Sayo-cho, Hyogo 679-5198, Japan

ABSTRACT

The pressure-volume-temperature (P-V-T) measurements of the B2 (CsCl-type) phase of KCl were performed at 9–61 GPa/1500–2600 K and up to 229 GPa at room temperature, based on synchrotron X-ray diffraction measurements in a laser-heated diamond-anvil cell (DAC). The nonhydrostatic stress conditions inside the sample chamber were critically evaluated based on the platinum pressure marker. With thermal annealing by laser after each pressure increment, the deviatoric stress was reduced to less than 1% of the sample pressure even at the multi-megabar pressure range. The obtained P-V-T data were fitted to the Vinet equation of state with the Mie-Grüneisen-Debye model for thermal pressure. The thermal pressure of KCl was found to be as small as ~10 GPa even at 3000 K at any given volume, which is only half of that of common pressure markers (i.e. Pt, Au, or MgO). Such a low-thermal pressure validates the use of a KCl pressure medium as a pressure marker at high temperatures.

Keywords: KCl, equation of state, high pressure, DAC

INTRODUCTION

Potassium chloride is often used as a pressure gauge and pressure-transmitting medium in high-pressure experiments using a diamond-anvil cell (DAC) because it hardly reacts with silicates and metals and provides lesser deviatoric stress inside the sample chamber. Moreover, it can be used for very high-temperature experiments such as melting experiments (e.g., Anzellini et al. 2013; Andrault et al. 2014; Morard et al. 2017), because the melting temperature of KCl is much higher than that of NaCl (Boehler et al. 1997). KCl is also useful for synchrotron-based experiments when the X-ray beam is accurately aligned to a heating laser beam spot because an X-ray induces visible fluorescent light in KCl in a wide pressure range, although the diffraction peaks are stronger compared to NaCl.

The B1-phase of KCl with the NaCl-type structure transforms into the B2 (CsCl-type) structure at 2 GPa (Walker et al. 2002). Equations of state (EoS) of B2 KCl have been proposed from experimental studies in a multi-anvil press or DAC (Yagi 1978; Campbell and Heinz 1991; Walker et al. 2002; Dewaele et al. 2012). Cold compression experiments on KCl in a helium pressure medium performed up to 160 GPa reported its highly compressible behavior comparable to solid argon (Dewaele et al. 2012). Walker et al. (2002) reported that B2 KCl exhibits low-thermal expansivity up to 8 GPa and 873 K, which was supported by recent theoretical calculations by Dewaele et al. (2012). The low-thermal expansivity may provide the opportunity for KCl to serve as a practical pressure standard at high temperature even when it is used as a pressure medium; namely, a large temperature gradient, if any, across the pressure medium may not matter when calculating the sample pressure from its P-V-T EoS. Hence, precise evaluation of its thermal EoS is of great importance for high P-T experiments in the DAC. Here we present a new EoS for B2-type KCl from our high P-T experiments to 230 GPa/300 K and 60 GPa/2600 K in a laser-heated DAC.

EXPERIMENTAL METHODS

High P-T conditions were generated using laser-heated DAC techniques. Diamond-anvil cells with a caulet size of 300, 120, or 40 μm were used depending on the target pressure. The starting material was powder of KCl (Wako Pure Chemical Industries, Ltd., 99.5% purity) that was mixed with platinum black that served as an internal pressure standard and laser absorber. The sample mixture was loaded into a hole drilled in a Re-gasket together with insulation layers. We used SiO2 glass (runs 1 and 2) or argon (runs 5 and 6) for thermal insulation. Argon was cryogenically loaded into the sample chamber. The sample assembly was then dried by leaving the cell in a vacuum oven at 393 K for >1 h prior to pressurizing, and flushed with argon gas when the oven was opened. The sample pressure was calculated from the unit-cell volume of Pt based on the EoS proposed by Sokolova et al. (2016).

Angle-dispersive XRD measurements were conducted at BL10XU, SPring-8 (Ohishi et al. 2008). XRD patterns were collected on an imaging plate (Rigaku RU-8 AXIS IV). The typical exposure time was 2 min. Monochromatic incident X-rays were focused by stacked compound refractive lenses and collimated to an area of approximately 6 μm full-width at half maximum (FWHM) at the sample position. The wavelength was precisely determined during each beamtime using a CeO2 standard: 0.4133–0.4135 Å (~30 keV). Two-dimensional XRD images were integrated over the Debye-Scherrer rings using the IPAnalyzer program (Soto et al. 2010) to produce conventional one-dimensional diffraction patterns as a function of 2θ angle. The obtained peak profiles and backgrounds were fit to pseudo-Voigt line shapes within the software package of PDindexer (Soto et al. 2010). The lattice parameters were obtained by a least-squares fit of peak positions. The unit-cell volumes were determined by averaging lattice parameters from 3–6 peaks and 2–5 peaks for Pt and B2 KCl, respectively after careful selection based on stress analysis (see below). Weak or poorly resolved diffraction peaks were not used in volume determination or stress analysis. Heating was performed from both sides of the sample by employing a pair of 100 W single-mode Yb fiber lasers (SP9). Temperatures were measured by a spectroradiometric method (Ohishi et al. 2008). To reduce the radial temperature gradient, we used beam shapers (New focus) that convert a beam with a Gaussian intensity distribution to one with a...