

Development of a 6-8-2 type multi-anvil apparatus and its application: pressure-induced phase transitions in GeO₂

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We have developed a 6-8-2 type multi-anvil apparatus (6-8-2 MA system, Figure 1) [1, 2] aiming to take advantages of both Kawai-type multi-anvil apparatus (KMA) and diamond anvil cell (DAC). By using the new 6-8-2 MA system, pressures higher than 110 GPa and temperatures up to 1500 K are successfully produced in much larger sample volumes than DAC. Here we present a recent application of the 6-8-2 MA system for high-pressure mineral science study. GeO₂ is an analog of SiO₂, which is an important mineral among many planets including the Earth. Therefore, understanding the high pressure-temperature phase relation of GeO₂ is a fundamental issue. GeO₂ has many polymorphs, but some of them are observed only by the experiments using DAC [3-5]. Thus, we conducted 6-8-2 MA experiments to investigate the stability and phase relation of GeO₂ polymorphs. In situ X-ray diffraction experiments were carried out using a KMA installed at SPring-8, BL04B1. The 6-8-2 MA system was adopted as high pressure cell. Nano-polycrystalline diamond (NPD, synthesized at GRC [6]) and single crystal diamond (SCD, Ib) were used as the third-stage anvil material. Rutile-type GeO₂ synthesized from Trigonal type (alpha-Quartz type, 4N) at high pressure-temperature was used as a starting material. The generated pressures were estimated from the unit-cell volume of Au. Pressures exceeding about 90 GPa and was successfully generated in a run M714. In the compression process of M714, rutile-type GeO₂ transformed to CaCl₂-type. Upon heating the sample to about 1000 K, the CaCl₂-type phase gradually transformed to alpha-PbO₂-type. This sequence is in agreement with previous studies on GeO₂ using DAC. Ono et al. (2003) shows that the transform across the alpha-PbO₂ and pyrite-type structures has a positive dP/dT slope, but our results suggest that this phase boundary is not necessarily positive. In comparison with the DAC experiments by Ono et al. (2003), our 6-8-2 MA experiments should have produced more homogenous distribution of temperature and pressure, but lower temperature. At low temperatures, materials are often compressed in a metastable state, and some kinetics effect may need to be considered in our experiments. Note that this is the first observation of this phase transition in GeO₂ using multi-anvil apparatus.