High Pressure and Temperature Phase of ZrO2

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Introduction

Zirconia (ZrO_2) has a monoclinic symmetry at ambient conditions and two high pressure polymorphs of orthoI and orthoII have been reported. Figure 1 is the pressure-temperature phase diagram of ZrO_2[1]. In-situ studies have been made at room temperature or, at most, a few hundred degree, while the phase boundary around 1000℃ was determined by synthesis experiments. As shown in this figure, the phase relation at elevated temperature is still unknown. In order to reveal the phase relation and the crystal structures of high-pressure-and-temperature ZrO_2 phases, we have attempted an in situ observation under high temperature and pressure.

Experimental

Experiments were performed by using a 1500ton large volume multi anvil type pressure system (SPEED1500), which is installed at the BL04B1 in SPring8. Details of the high pressure cell assembly are shown elsewhere[2]. Synthesized orthoI powder and a mixture of orthoI and Au powder for pressure markers were separately encased in the pressure medium. Because Zr ions strongly absorb x-ray, the thickness of the sample chamber vertical to the incident x-ray beam was reduced to 800 μm. X-ray data were collected by an energy dispersive method. The incident x-ray beam size was 0.2 mm in a vertical direction and 0.1 mm in a horizontal direction.

Results and Discussion

Dashed lines and allows in Fig. 1 indicate experimental paths along which x-ray diffraction data were collected. The sample was, first, compressed above 20GPa and then annealed at 900℃ for 30 minutes to make whole the sample transform to orthoII phase. Thus prepared orthoII was heated up to 1400 ℃ at 20GPa. No transition was observed but the x-ray data were well indexed as orthoII. On the course of decompression, sample temperature was increased again to 1400℃ for 3 times, at 19, 16 and 14 GPa, respectively. All of the x-ray diffraction patterns obtained during decompression were orthoII. However, when pressure was completely released, the sample was reverted to monoclinic. Present results show remarkable differences in following two points from those reported. First, stability field of orthoII determined by synthesis experiments does not coincide with that observed by in-situ. Secondly, orthoII is quenchable but present orthoII shows reverse transition to monoclinic phase. In order to clear these discrepancies, further investigation by in situ observations is needed.

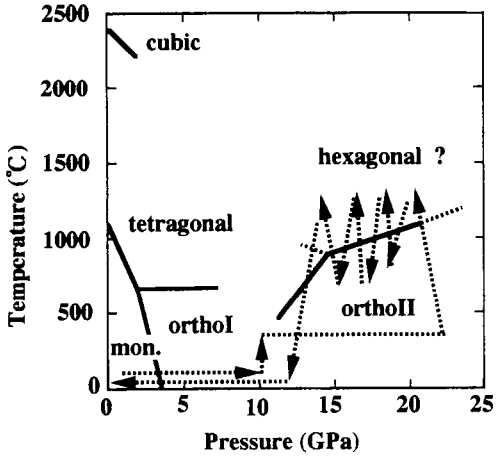

Fig. 1 P-T phase diagram of ZrO_2.

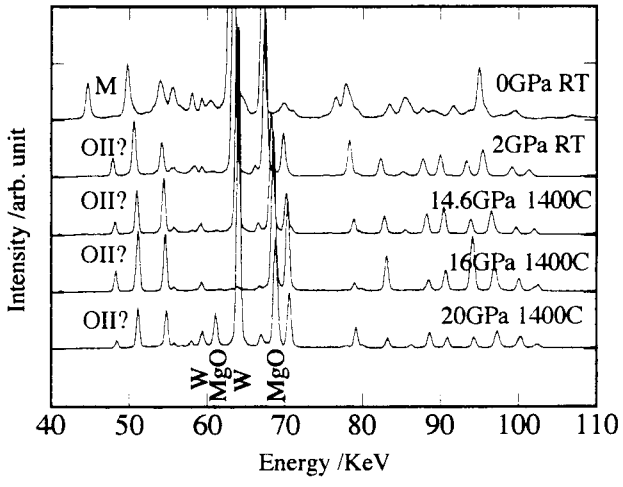

Fig. 2 X-ray diffraction patterns obtained at several P-T conditions during decompression

References

1 Ohtaka et al. Phys. Rev. B, 49, 9295 (1994)
2 Irifune et al. Science 279, 1698 (1998)