

Comparative Study of High Pressure Behavior in TiO₂ and Its Nanophase

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Beamline(s): X17B1

Introduction: Use of nanophase materials has shown a great advantage in producing high quality sintered industrial products^{1,2}. Nanophase of TiO₂ has been reported to have different behavior from its bulk sample³. In-situ x-ray diffraction indicated that the Rutile/ α -PbO₂-type phase boundary in nanophase TiO₂ is 2 GPa lower than that in TiO₂ bulk sample at high temperature. However, an increase in the transition pressure for wurtzite to rock salt transformations was observed in CdSe and CdS nanocrystals⁴. Therefore a detail study on the high pressure behavior of nanophase material is necessary to better understand the nature of nanocrystals.

Methods and Materials: We performed in-situ energy dispersive x-ray diffraction at high pressure and temperature using the DIA-type large-volume press at the superconductor wiggler Beamline X17B1. Nanocrystal anatase phase and bulk rutile sample are loaded into the high pressure cell, separated by a layer of mixture of NaCl and hBN. Sample pressure is determined by the equation of state of NaCl, and temperature is measure by a thermocouple.

Results: Diffraction patterns from both samples are recorded at 5 GPa and temperatures up to 1200oC. We found that with increase in temperature at this pressure the anatase nanocrystals transform into α -PbO₂-type phase and then rutile phase. Meanwhile the α -PbO₂-type phase also appears in the rutile sample at the temperature that the anatase nanocrystals transform into α -PbO₂-type phase. This indicates that the α -PbO₂-type phase is stable in both nanocrystal and bulk sample at this pressure and temperature condition. However a fraction of the α -PbO₂-type phase in the bulk sample is very small, and increase of the phase fraction as a function of time and temperature is very slow, which indicates the kinetics of the phase transformation at the pressure and temperature is slow.

Conclusions: Previous observation of change in the phase boundary for nanophase material might not reflect equilibrium phase boundary. Because nanocrystals have much higher surface energy with respect to the bulk sample, phase transformation rate in the nanocrystals may be much higher than that in the bulk material. The faster phase transition kinetics may have significant influence on hot press process.

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