

Tetragonal-cubic phase transition under high pressure in perovskite PbVO_3

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PbVO_3 is a perovskite prepared at high pressure (HP) of 6 GPa. It has a tetragonal structure at room temperature (RT) (space group $P4mm$; $a = 3.80391 \text{ \AA}$ and $c = 4.67680 \text{ \AA}$) and it is isotypic with PbTiO_3 . PbVO_3 is a candidate multiferroic material because it has the magnetic ion (V^{4+}) and its polarization was calculated to be $101 \mu\text{C}/\text{cm}^2$ (using the atomic displacement from the ideal positions (Figure 1)).

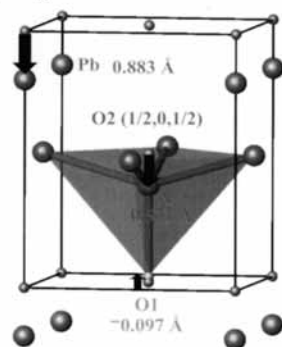


Figure 1. Structure of PbVO_3 refined from the synchrotron XRD data (BL02B2).

At ambient pressure (AP) in air, the Curie temperature is above the decomposition temperature of 300°C .

However the tetragonal(T)-cubic(C) phase transition was observed at HP. Powder XRD data were taken in cubic anvil-type HP apparatus at BL14B1.

Figure 2 shows the XRD patterns at RT. The C phase appeared at 2 GPa and the T phase almost disappeared at 5.9 GPa. After releasing the pressure, the C phase was still observed even at 0.3 GPa and completely disappeared only at AP.

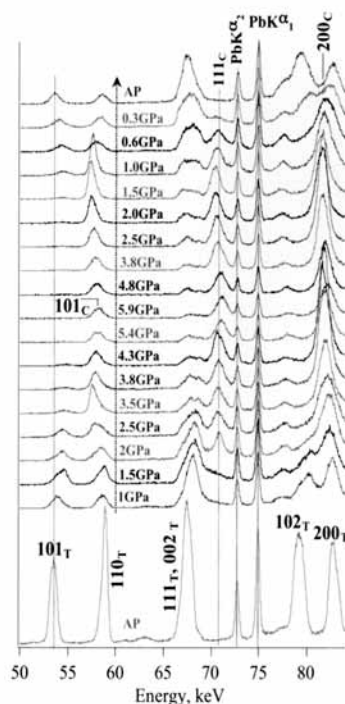


Figure 2. XRD patterns of PbVO_3 at RT from 0 to 5.9 GPa.

At 2 GPa, the T phase disappeared from 673 K. After cooling, the C phase was present at RT and 2 GPa and disappeared only at AP in the same manner as in Figure 2.

The HP behavior of PbVO_3 resembles that of BaTiO_3 rather than PbTiO_3 .

Particle size distribution analysis for nanometer particle using Ultra Small Angle X-ray Scattering

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Inorganic powder with nanometer size which called nano powder is beginning to be used on progress with integration of a semiconductor device. In case of the nano powder using Epoxy Molding Compound, it is necessary to know particle size distribution precisely. The laser diffraction and/or scattering technique has been applied in general. It is impossible to measure the particle diameter with a few decade nanometer or less because of the limitation for the range of 50nm or more. In this investigation, Small Angle X-ray Scattering (SAXS) technique has been used for particle size distribution analysis of the nano powder. SAXS measurement has been performed by BL15XU at SPring-8 which can measure at $2\theta = 0.1^\circ$ or less (Ultra-SAXS, U-SAXS).

Two kind of amorphous SiO_2 powders were measured. Energy of incident X-ray was 8keV and U-SAXS data were collected between $2\theta = 0.0^\circ$ and 1.0° . The Guinier Plots ($k^2\text{-Log(Int.)}$) were shown in Fig.1. In Sample B, the inclination of the Guinier Plot at near $k^2 = 0$ was more sharp than that of Sample A. The particle size distribution analyzed by Modified Fankchen technique[1] were shown in Fig. 2. It was confirmed that particle size distribution which normalized by specific diameter were obtained for the two kind of SiO_2 . It was able to measure the wide diameter range from 1nm to over 200nm using BL15XU with Modified Fankchen

technique. This technique is more useful for particle size distribution analysis of all nano powders.

Furthermore, particle size distribution into an epoxy resin also measured by U-SAXS. Nevertheless, the results of particle size distribution for only powder and that for its resin compound was not same. It is considered that the filling density of powder in resin is too low.

[1] H. Hashimoto *et al. Adv. X-ray Chem. Anal.* 35 in contribution.

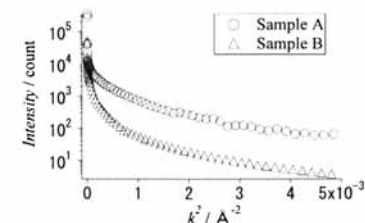


Fig. 1. Guinier Plots of the two kind of SiO_2 .

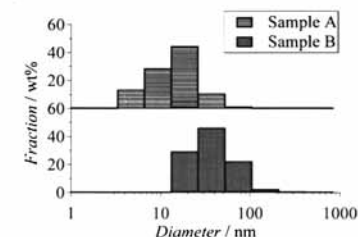


Fig. 2. Particle size distribution of the two kind of SiO_2 .