

rays which are insensitive to the scattering of light atoms and hence neutron diffraction data on a single crystal sample are essential in determining precisely the atomic co-ordinates during this possible transition. Previously only data up to 2.0 GPa have been collected using single crystal neutron diffraction. We present structures determined from a single crystal of 4 mm³ using time-of-flight Laue diffraction on the SXD instrument at the ISIS Facility at Rutherford Appleton Laboratory in the UK.

[1] Endo S. et al, *Nature*, **340**, 452, 1989.

Keywords: high pressure, single crystal diffraction, neutron diffraction

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Compression of Oxygen Vacancy Type Al-bearing MgSiO₃ Perovskite

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MgSiO₃ perovskite is believed to be a dominant constituent of the Earth's lower mantle. Experimental results on the effect of Al on the compressibility of MgSiO₃ perovskite have been pretty controversial. Two kinds of the Al substitution mechanisms are expected: 2Al³⁺ = Mg²⁺ + Si⁴⁺ and 2Al³⁺ = 2Si⁴⁺ + (as a vacancy site) O²⁻ [1]. Theory predicts that the latter mechanism significantly increases the compressibility [2].

Recently, Kojitani et al (2005) demonstrated the structural differences between these two types of Al-bearing MgSiO₃ perovskite on the basis of Rietveld analyses. In this work, the volume compression measurements were performed on the oxygen vacancy type Al-MgSiO₃ perovskite by using synchrotron radiation x-rays. Data were collected under hydrostatic conditions using helium pressure transmitting medium. Preliminary results show that the isothermal bulk modulus is reduced due to the incorporation of Al₂O₃ in perovskite with oxygen vacancy.

[1] Navrotsky A., Shoenitz M., Kojitani H., Xu H., Zhang J., Weidner D. J., Jeanloz R., *J. Geophys. Res.*, 2003, **108-B7**, ECV2-1. [2] Yamamoto T., Yuen D.A., Ebisuzaki T., *Earth Planet. Sci. Lett.*, 2003, **206**, 617.

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New High-pressure Phase of Calcite at Room Temperature

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A new high-pressure phase of calcite (CaCO₃) has been found by single-crystal X-ray analyses. Recently we determined the crystal data of calcite III (monoclinic C-centered lattice, Z=8) using single-crystal X-ray diffraction methods at 300K and pressures between 2.3 and 4.1 GPa [1]. The present studies have been carried out to determine the crystal data of calcite at room temperature and higher pressures.

The samples are single crystals of natural calcite from Creel, Chihuahua, Mexico. For high-pressure experiments, the Merrill-Bassett type diamond-anvil-cells were used. Pressures were estimated based on the ruby-fluorescence method. X-ray diffraction measurements were performed using an Enraf-Nonius CAD-4 diffractometer with MoK α radiation monochromatized by graphite. A film cassette for a flat imaging plate was attached on the diffractometer.

On the oscillation photographs taken at 2.5GPa, all the reflections could be assigned as calcite III being composed two twinned individuals. By increasing the sample pressure to 4.5GPa, several reflections disappeared. Then remained reflections were re-indexed and the cell parameters were determined with the angular data of 25 reflections. The triclinic unit-cell are $a=3.855(3)$, $b=4.815(2)$, $c=6.165(5)\text{\AA}$, $\alpha=84.10(4)$, $\beta=72.15(5)$, $\gamma=88.73(5)$, $Z=2$ at 300K and

4.5GPa. The cell parameters were determined at pressures between 4.3 and 5.6GPa. Thus the phase transition from calcite III to the new phase may occur at 4.1-4.3GPa.

[1] Hagiya K., Matsui M., Kimura Y., Akahama Y., *J. Mineralogical and Petrological Sciences*, 2005, **100**, 31.

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High-pressure Single-crystal Study of Chlorite

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Chlorite is a major constituent of hydrated oceanic crust, and may represent an important water carrier in subducting slabs. In this context, its stability under high pressure has relevant implications on rheology and melting behaviour of mantle rocks.

The structural effects of pressure on a natural chlorite from Val Malenco (Italy) [clinoclhor, polytype IIb-4, S.G. C-1, pseudomonoclinic metric, composition (Mg_{7.82}Al_{3.36}Fe²⁺_{0.52}Fe³⁺_{0.30})(Si_{7.70}Al_{0.30}) O₂₀(OH)₁₆] have been studied by X-ray diffraction on single-crystal mounted in a DAC. Pressure was calibrated through the EoS of α -quartz.

Structural refinements were performed at 0, 0.8, 1.8, 2.7, 3.5, 4.4, 5.1 GPa with intensity data collected on a CCD Xcalibur diffractometer (Oxford Instr.) equipped with monochromatized MoK α . Lattice parameters were measured with the point-detector mounted on the same instrument.

The compressibility data of chlorite (bulk modulus K= 83(1) GPa, K'= 4) are in fair agreement with data based on powder neutron [1] and synchrotron diffraction methods [2,3]. Axial moduli are 102(2), 97(3) and 63(1) GPa respectively for a , b and c axes. The main structural deformations affect the interlayer region where the hydrogen bonds are relevant to the structural properties of the phase. The OH-O distances decrease of about 4% in the 0-5 GPa range. Work is in progress and further details will be presented at the conference site.

[1] Welch M.D., Marshall W.G., *Am. Mineral.*, 2001, 1380. [2] Pawley A.R., Clark S.M., Chinnery N.J., *Am. Mineral.*, 2002, 1172. [3] Welch M.D., Crichton W.A., *Eur. J. Min.*, 2002, 561.

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Research of Nanostructured X-ray Amorphous Minerals by AFM

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Among products of geological processes the noncrystal substances are distributed. Before among geomaterials the basic attention was given to research of crystal mineral substance and microdisperse mineral phases. Progress of methods of research has allowed to find out and considerably to expand last decade ideas about nanodisperse mineral phases. Their study causes interest due to active influence of nanoconstitution on properties, to detection of some new unique forms in mineral substances.

In our work an attempt of search characteristics of nanoconstitution of various noncrystal mineral substances will be undertaken. Among them simple substances (ex., fullerene shungite Karelia), oxides (ex., natural photonic crystals – opals), complex substances (ex., solid hydrocarbons). Nanoconstitution visualized on a fractures surface off by atomic force and electronic microscopies. The quantitative superstructural data were received by a combination of microscopic researches with diffractational and statistical-geometrical analysis of morphological features.

As a result of the carried out researches local and global characteristics of supermolecular structures, in particular, a degree of orderliness of a relative positioning of elements nanostructures have been established. The multilevel fullerene-like structure of shungite