In situ synchrotron investigations in large volume presses at high pressure and high temperature

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Spinels (AB_2X_4) are compounds that stabilize in the cubic spinel structure at ambient conditions. Considerable interest has been attracted in current studies due to the large diversity and the practical usefulness of their physical and chemical properties, including humidity-sensing, oxygen-sensing, photoelectrical and super-para-magnetic as well as high temperature ceramic properties [1, 2].

The temperature and pressure dependent volume change were Spinels seem to be important constituents of the deep interior of the Earth while transition with spinel or pseudospinel structure strongly influence the dynamic of the mantle. On the other hand, Spinels are widely used as artificial material [3].

The Large Volume Presses were located at the Hamburger Synchrotron Laboratory (HASYLAB) at the DORIS III storage ring. The experiments were carried out using the high pressure multi anvil devices MAX80 (F2.1 beamline) and MAX200x (W2 beamline).

The F2.1 beamline was a bending magnet beamline with a critical energy of 16.6 keV and an energy range up to 75 keV. The W2 beamline was a hard-wiggler beamline with a critical energy of 26.4 keV and an energy range up to 175 keV. Energy-dispersive X-ray diffraction was used to determine the pressure and temperature induced volume change. Isothermal experiments were performed up to 15 GPa at ambient temperature. The pressure dependence of the thermal expansion coefficient is examined on two different ferrite, while systematically varying the iron contents. The two minerals, Magnetite ($Fe^{2+}Fe^{3+}{}_2O_4$) and Franklinite ($Zn^{2+}Fe^{3+}{}_2O_4$) adopt the normal spinel structure. Derived from compression experiments using MAX80 apparatus up to 5 GPa at temperatures of 298, 500, 700, 900 and 1100 K.

In MAX200x was measured at room temperature up to 15 GPa. To obtain the bulk moduli, the data points were fitted to a 3nd order Birch-Murnaghan equation of state yielding to K_T = 184(3) GPa and K' = 4.5(2) for Magnetite, K_T = 178(3) GPa and K' = 4.6(4) for Franklinite. The uncertainties in parentheses represent the 2 sigma uncertainties of the fit result.

[1] W.F.J. Fontijn, et al., J. Appl. Phys. 85 (1999) 5100 – 5105.

[2] S.A. Oliver, H.H. Hamdeh, Phys. Rev. B 60 (1999) 3400 – 3405.

[3] T. Irifune, et al., Phys. Chem. Miner., 29, (2002) 645-654.