ESRF

Experiment title:

High pressure equation of state of mantle pyroxenes by powder diffraction

Experiment number:

HC400

Beamline:

ID9-BL3

Date of Experiment:

from: 22-3-96 to: 27-3-96

Date of Report:

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Shifts:

4

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Report:

INTRODUCTION: High-pressure clinopyroxene with space group C2/c is a major mineral in the Upper Mantle of the Earth, but this phase inverts to low-clinopyroxene (space group P21/c) upon pressure release. The repeated cycling of pressure required to achieve higher pressures in the single-crystal diamond anvil cell resulted in the destruction of the single crystal as it passed repeatable through the phase transition. Furthermore, we have found it impossible to synthesise single-crystals of intermediate compositions that are of sufficiently good quality to allow high-pressure single-crystal diffraction measurements to be performed. We have therefore undertaken a high-pressure powder-diffraction study to measure the equations of state (EOS) of two intermediate compositions on the $MgSiO_3$-$FeSiO_3$ join, in order to determine whether the thermodynamic description of the join should include significant excess volumes at high pressures.

Studies at ESRF: Diffraction patterns from two clinopyroxenes of intermediate compositions ($Mg_{0.9}Fe_{0.1}SiO_3$ = En90 and $Mg_{0.3}Fe_{0.7}SiO_3$) were collected at high pressures using monochromatic radiation on ID9, our own design of diamond-anvil cell (Allan et al., 1996) and an image-plate as detector. Data from the image plate were reduced to a one dimensional diffraction pattern and cell parameters were obtained from these by full-pattern Rietveld analysis. Because of the high-background and low signal-to-noise only pattern parameters and cell parameters were refined; structural parameters were interpolated from the known structures of the end-members.

Results: The volume-pressure data from both samples, in separate experiments, exhibits a change in slope at pressures of about 10 GPa, which we attribute to the well-known freezing of the ethanol-methanol pressure media. However, our diamond-cell with the En90 composition pyroxene was loaded with sufficient ruby powder that we also obtained a refinable powder pattern of alumina. The refined cell volumes of the alumina deviate significantly from the well-constrained equation of state determined by Richet et al. (1988), and the c/a ratio deviates significantly from its hydrostatic value, indicating that non-hydrostatic stresses are present in our samples. Pressures determined from the unit-cell volume of the ruby and the ruby fluorescence shift deviate significantly above 10 GPa, and when the former are used to plot the unit-cell volumes of the En90 pyroxene, the change in slope with pressure at 10 GPa is removed. We therefore conclude that the largest effect of non-hydrostatic stresses in these experiments is upon the ruby fluorescence shift, and not upon the volume properties of either the ruby or our pyroxene samples. The pressure correction calculated from our ruby data is: P(corrected) = 0.67P(ruby fluor.) -3.3 GPa. This correction was also applied to the En30 data.

Equations of state fitted to both data sets yield bulk moduli in the range 115-130 GPa, depending upon the constraints imposed. These are slightly, but not significantly larger than the values previously determined for the end-members Enl00 and Fs100 by single-crystal diffraction and indicate that there is no significant excess volume effect at high pressures on the enstatite-ferrosilite join.

Allan et al., (1996) Rev. Sci. Instruments., in press,

Richet et al., (1988) Phys. Chem. Minerals., 16:207-211