# High-Pressure High-Temperature Synchrotron X-ray Diffraction Study of High Clinoenstatite $MgSiO_3$

Y. Zhao,[1] H. Xu,[1] J. Zhang,[1] C. Pantea,[1] K. Tait,[1] T. Uchida,[2] Y. Wang[2]

[1]Los Alamos National Laboratory, Los Alamos, NM, U.S.A.; [2]The University of Chicago, Chicago, IL, U.S.A.

## Introduction

Enstatite ($MgSiO_3$), a member of the pyroxene group, is composed of single chains of corner-sharing [$SiO_4$] tetrahedra that are cross-linked by parallel bands of [$MgO_6$] octahedra. Under different pressure and/or temperature conditions, enstatite exists in three polymorphs: low clinoenstatite, orthoenstatite, and high clinoenstatite. Structurally, these polymorphs differ in the conformations of the [$SiO_4$] chains and in the relative positions of successive layers of [$MgO_6$] [1]. This report focuses on high clinoenstatite, which, along with orthoenstatite, may be the major pyroxene components in the Earth's upper mantle. High P/T experiments in excess of 8 GPa and 900 °C have provided evidence for a phase transformation of orthoenstatite to high clinoenstatite [1-4]. Since high clinoenstatite cannot be quenched to ambient condition, it has been argued to be responsible for the "X discontinuity" near 300-km depth, occasionally observed in seismic profiles [5]. To fully address this question, however, accurate measurements of the P-V-T equations of state of high clinoenstatite and determination of its related thermoelastic parameters such as the temperature derivative of bulk modulus $\partial K/\partial T$, the pressure derivative of thermal expansion $\partial\alpha/\partial P$, and Anderson-Grüneisen parameter $\delta_T$, are essential. In this study, we have performed *in situ* synchrotron X-ray diffraction of high clinoenstatite in the pressure range from about 6 to 17 GPa and at temperatures up to 1200 °C. The obtained data are currently being processed using the Le Bail method with the General Structure Analysis System (GSAS) [6]. Our aim is to determine the equations of state of high clinoenstatite and the related thermoelastic parameters. Seismological implications of the obtained parameters will also be discussed.

## Experimental Methods

The *in situ* high P/T synchrotron XRD experiments were carried out at GeoSoilEnviroCARS beamline 13-BM, Advanced Photon Source, with powdered $MgSiO_3$ orthoenstatite as the starting material. Though not the stable phase of $MgSiO_3$ at room pressure and temperature (low clinoenstatite is the thermodynamically stable phase at ambient condition), orthoenstatite, which was synthesized at high P/T conditions, is quenchable. The XRD experiments were performed with the "T-cup" large-volume apparatus, a two-stage multi-anvil system designed for *in situ* X-ray diffraction, and using Co-doped MgO as the pressure medium [7]. An energy-dispersive method was employed using white radiation. The incident X-ray beam was collimated to dimensions of 50 × 100 μm, and diffracted X-rays were collected by a solid-state Ge detector at a fixed 2θ angle -5.6685°. The temperature was measured by a W/Re24%-W/Re6% thermocouple that was in direct contact with the sample and the internal pressure standard, CsCl. XRD patterns were collected in close proximity to the thermocouple junction; the errors in temperature measurements are therefore estimated to be less than 25 °C.

Seven heating-cooling cycles were applied in the pressure range 6-17 GPa and at temperatures up to 1200 °C. All XRD data were collected on cooling, at intervals of 200 °C. The counting time was 20-30 min for the sample and about 10 min for CsCl.

## Results

As shown in Fig. 1a, the starting material was orthoenstatite (space group *Pbca*). At the experimental temperatures and pressures (6-17 GPa, 25-1200 °C), it transformed to high clinoenstatite (space group *C*2/*c*) (see Fig. 1b for an example). Upon decompression and quenching to ambient condition, it converted to low clinoenstatite (Fig. 1c), the stable polymorph of $MgSiO_3$.

We are currently processing the high P/T data for high clinoenstatite using the Le Bail method (Fig. 2). Unit-cell volumes at different P/T conditions will be obtained, and the P-V-T equations of state determined. Moreover, related parameters such as isothermal bulk modulus, thermal expansitivity and their pressure and temperature dependences will be derived. The obtained results will also place additional constraints on the phase boundaries between high clinoenstatite and the low-pressure $MgSiO_3$ polymorphs.

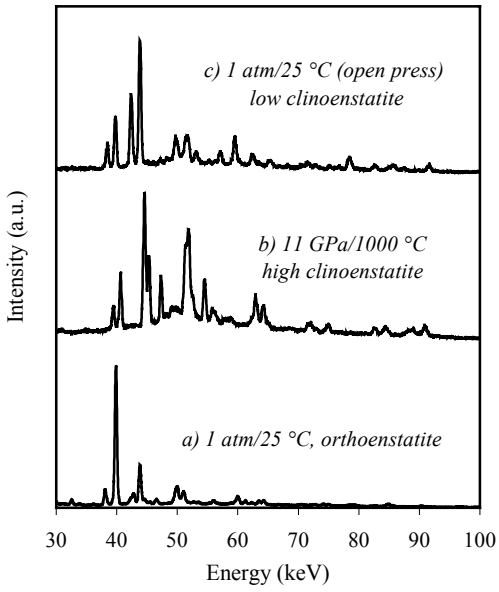


Fig. 1. Synchrotron XRD patterns of a) starting orthoenstatite, b) high clinoenstatite at 11 GPa and 1000 °C, and c) low clinoenstatite after decompression.

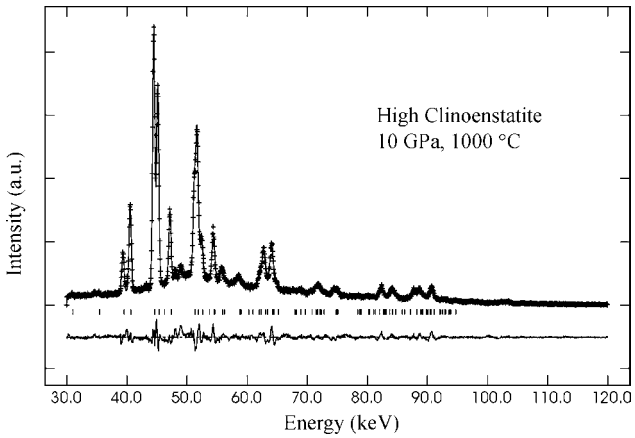


Fig. 2. Selected fitted synchrotron XRD pattern of high clinoenstatite at 10 GPa and 1000 °C.

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