ESRF

Experiment title:
HIGH-PRESSURE AND HIGH-TEMPERATURE X-RAY DIFFRACTION STUDY OF $MgSiO_3$ PEROVSKITE

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Local contact(s):
D. HÄUSERMANN / M. HANFLAND

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Names and affiliations of applicants (*indicates experimentalists):

G. FIQUET, P. GILLET and I. MARTINEZ — Ecole Normale Superieure Lyon
D. ANDRAULT, P. RICHET — Dpt Géomatériaux IPG Paris
F. GUYOT — IPG / Paris VII
B. REYNARD — Géosciences Rennes
JP ITIÉ — Laboratoire de Physique de la Matière Condensée PARIS VI

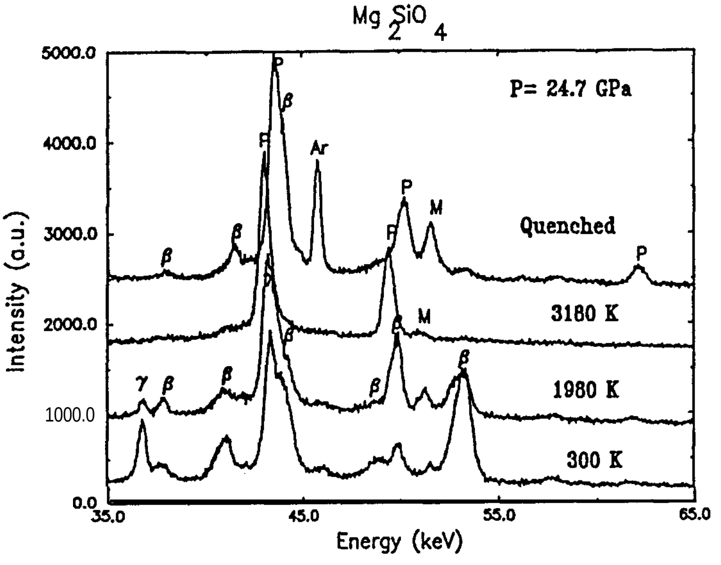
Report:

X-ray diffraction measurements have been carried out at simultaneous high-pressure and high-temperature in a laser-heated diamond anvil cell. X-ray diffraction patterns have been recorded up to 28 GPa and temperature in excess of 2500 K in an energy dispersive configuration. These experiments were conducted on polycrystalline discs or polished thin sections (10-15 μm thick), loaded cryogenically in dry argon acting as pressure transmitting medium, and heated by a $C0_2$laser (continuous mode, 120W, TEM 00).

We used an optical set-up designed for the on-line pressure and temperature measurements, where ruby fluorescence and thermal emission of the sample where collected during X-ray diffraction acquisition and directed with optical fibers to a spectrometer placed outside the hutch. The diffraction patterns were collected between 7 and 140 keV at $2\theta=6.8°$, after collimation of the incident X-ray beam to a 20x20 μm sized spot. Exposure times of the order of 300 s were long enough to collect diffraction patterns with usable intensities up to 100 keV.

A first set of data was obtained on periclase (MgO), to complete a preliminary series of measurements realized at LURE (Orsay) and check the influence of the sample size and thickness. This was possible with short acquisition times at the ESRF.

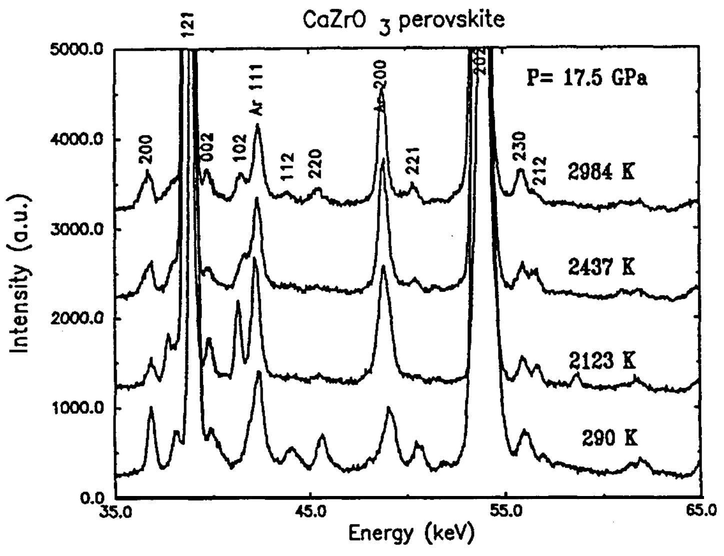
During a second set of experiments, the $Si0_2$ quartz-coesite-stishovite phase boundaries were examined in the temperature range 2400-3000 K. We also investigated the Mg2Si04 phase diagram (see Figure l). Forsterite Mg2Si04 is subjected to three main phase transitions with increasing pressure and temperature. At high temperature, 13-phase is formed at around 14 GPa and transforms to γ-phasc Mg2Si04 with increasing pressure, before it breaks down to perovskite ($MgSi0_3$) and periclase (MgO) at around 24 GPa.



The study of this phase diagram is of primary importance for the understanding of the Earth's transition zone located between 400 and 660 km depth.

Figure 1: Mg_2SiO_4 diffraction patterns at 24.7 GPa and various temperature conditions. P: perovskite, M: magnesiowustite, Ar: argon, β: beta-phase, γ: gamma-phase.

Some runs have been also dedicated to the study of the symmetry changes of **CaZr03** and MgSi03 perovskites at high pressure and temperature. Many solids with perovskite structure are known to undergo phase changes toward **less** distorted structures (higher symmetry) with increasing temperature, which highly affect their physical properties.



This behaviour was checked first on $CaZrO_3$ (see Figure 2), which still displays an orthorhombic structure at 2980K at 17.5 GPa. Preliminary results obtained on MgSi03 at 26 GPa seem to indicate that the orthorhombic structure is also preserved for this compound at very high temperature.

Figure 2: CazrO3 diffraction patterns at 17.5 Gpa and different temperature conditions.

A few runs have been performed with a monochromatic X-ray beam and image plates. Images have been recorded on $CaZrO_3$ perovskite up to *21.5* GPa and temperatures in excess of 3000 K, with exposure times between 30 and 45 mn. Although these experiments were more difficult to achieve, this technique appears to be very promising for this kind of investigation.

These results have been obtained in June and their analysis has not been completed yet. Consequently, no application for beam time will be submitted to the 1st september 1995 proposal round.