| ESRF | **Experiment title:** Cubic-tetragonal phase transition in (Mg,Fe)SiO3 majorites and garnet symmetry in the Earth's transition zone | **Experiment number:** HS1080 |
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| **Beamline:** | **Date of experiment:** from: 16/11/1999 to: 21/11/1999 | **Date of report:** 29/02/2000 |
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**Names and affiliations of applicants** (* indicates experimentalists):

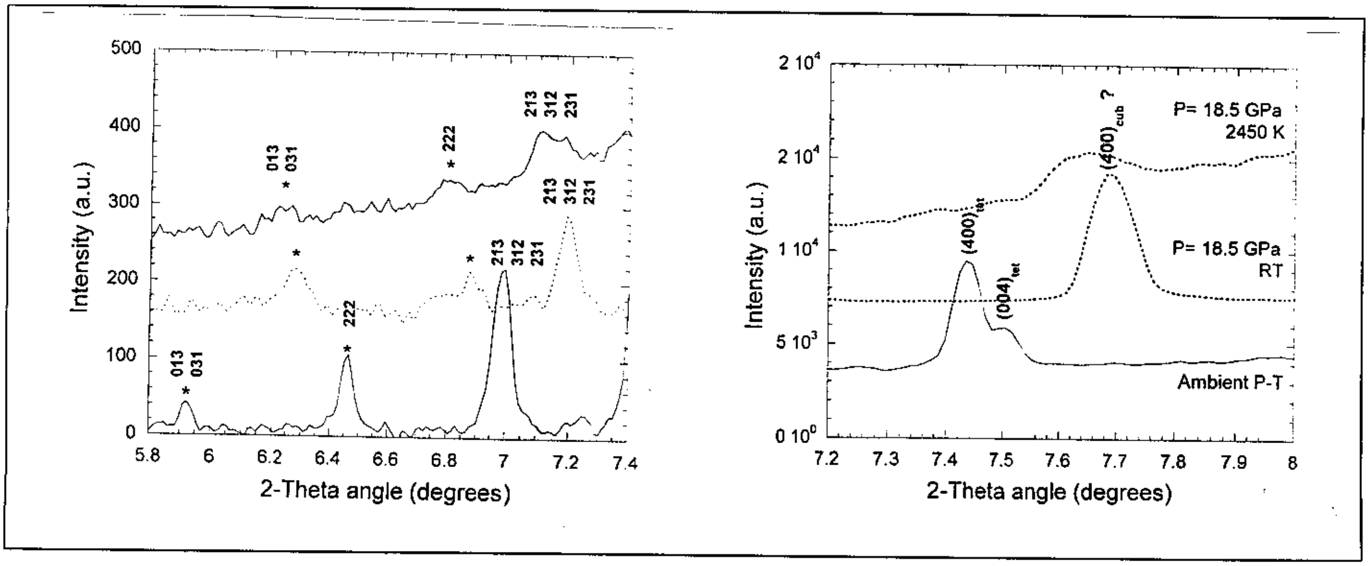
**Guillaume Fiquet** *(formerly at Lab. Sciences de la Terre - ENS Lyon) now at:
Laboratoire de Minéralogie et Cristallographie UMR 7590
Université Paris VI – 4 Place Jussieu
75252 Paris cedex 05

**Bruno Reynard * & Gilles Montagnac ***
Laboratoire de Sciences de la Terre - ENS Lyon
46 Allée d'Italie 69364 Lyon cedex 07

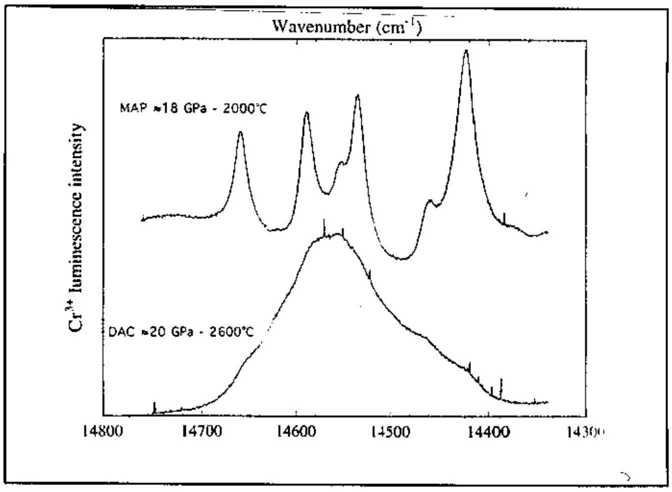
**Report:**

Garnet phases, rich in a $MgSiO_3$ component, probably account for a significant fraction of the Earth's upper mantle above the 670-km discontinuity. Although most garnets have cubic symmetry, some $MgSiO_3$-rich garnets have been reported to have tetragonal symmetry, which arise from cation ordering on the octahedrally coordinated sites in the structure. Angel et al. [American Mineralogist, 74, 1989] single-crystal X-ray diffraction study confirmed the tetragonal symmetry on a $MgSiO_3$ garnet synthesized at 17 GPa and 2100 K. This measurement, however, was achieved on a quenched sample. The question of the actual symmetry of $MgSiO_3$-garnet in the Earth's transition zone still remains unanswered. We therefore report here X-ray diffraction experiments that have been carried out between 18 and 22 GPa at high temperature (T > 2100 K) in a laser-heated diamond-anvil cell, where pure $MgSiO_3$ enstatite glass or crystal where loaded in argon. Angle dispersive X-ray diffraction experiments were achieved at the high-pressure beamline ID30 of the ESRF. X-ray beam at a wavelength of 0.3738 Å was used in association with imaging plates to collect data over a 2-theta interval from 4 to 25° while the samples were kept at high-pressure and high-temperature.

The following figures show detailed views of the X-ray diffraction pattern, recorded on samples at different P-T conditions in the diamond-anvil cell. From the bottom to the top, one can see the pattern of a majorite sample synthesized in a multi-anvil press which clearly shows a tetragonal distortion with the doublet (400) – (004) and supperlattice reflections denoted by stars. On the other hand, a sample synthesized and quenched at high-pressure during these laser-heating experiments shows a very weak distortion, with the absence of the doublet but almost a single (400) reflection instead. In addition, the supperlattice reflections are much weaker. At high-temperature (on the top), these reflections almost vanish, thus indicating a structure close to cubic.




These observations have been corroborated by a Raman spectroscopic study of the quenched samples, which also shows a large disorder of the Mg and Si cations among octahedral sites, as evidenced by the $Cr^{3+}$ luminescence spectroscopy shown on the right. Again one can observe a large difference between the sample synthesized in the multi-anvil cell on the top, where the tetragonal distortion is marked by the occurrence of two distinct 2E doublets corresponding to $Cr^{3+}$ replacing Si and Mg in the symmetrically distinct octahedral sites, whereas the sample synthesized with laser-heating shows almost a single large peak, indicating a large disorder in the "cubic" $MgSiO_3$ majorite sample.




We have here some experimental evidences for a tetragonal to cubic phase transition at high-temperature for $MgSiO_3$ majorite composition. Some detector problems and one anvil breakage prevented us to confirm this transition and to poursuie these in situ X-ray diffraction experiments on some slightly enriched iron compositions, relevant to the Earth's upper mantle. This leads us to ask for a HS1080 continuation of 12 shifts.