ESRF	**Experiment title:** Kinetics of the transformations in the binary system Mg_2SiO_4 – Fe_2SiO_4 system: toward a detailed understanding of the srtucture of the planets'interior	**Experiment number**: HS3530
Beamline:	**Date of experiment**: from: 02/07/08 to: 08/07/08	**Date of report**:
Shifts:	**Local contact(s)**: J.-P. Perrillat	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Mélanie Chollet*, Isabelle Daniel*, Laboratoire de Sciences de la Terre, UMR 5570 CNRS-ENS Lyon – UCB Lyon1

Jean-Philippe Perrillat*, ESRF

Report:

Whithin the depth of the terrestrial planets, values of the mantle thermodynamic properties are deduced through seismological and geodynamical probes. Incompressibilities calculated from such observations are sensitive to the kinetics of phase transitions. Hence, the knowledge of transformation rates may bring tight constraints on mantle discontinuities, in particular in the transition zone (410 km in the Earth) where the $(Mg_{1-x},Fe_x)_2SiO_4$ α–β–γ transformations take place.

A pioneer time-resolved experiment was performed last year at ID27 (see experimental report HS 3260). Among significant results, **we observed in one run, the disappearance of the α-phase diffraction lines at the onset of the α-γ transition**. This observation was made possible thanks to the high time resolution achieved, *i.e.* 3s acquisition every ca. 30s. It suggests that the α-phase becomes highly disordered at the early stages of the transition, which may change our interpretation of the related seismic discontinuities. The purpose of run HS3530 was thus multiple; (*i*) first, to acquire kinetic data at high temperature (T>1000 K) relevant to martian and terrestrial mantle geotherm; (*ii*) second, to investigate transformation rates for various X_{Fe} content; and (*iii*) most importantly, to confirm and detail the above reaction mechanism.

During experiment HS3530, X-ray diffraction experiments were carried out on two synthetic samples, $(Mg_{0.57},Fe_{0.43})_2SiO_4$ X_{Fe} = 0.43 and $(Mg_{0.42},Fe_{0.58})_2SiO_4$ X_{Fe} = 0.58. They were prepared at ambient pressure and high temperature, under controlled oxygen fugacity at the CRPG in Nancy (coll. G. Libourel). The starting materials, in the α-form (space group Pbnm), were pressurized in the Paris-Edinburgh press and heated to enter the α-γ transformation loop (Fig. 1). Sintered diamond anvils were used to reach 11 GPa (the maximum pressure in this experiment), while heating was achieved using either graphite or $LaCrO_3$-Re heaters. Au and NaCl powders were mixed with the sample as (P,T) gauges. Pressure and temperature were calculated using the EoS cross-calibration method. As the α–γ loop was scanned and the transformation proceeded, angle dispersive diffraction data were recorded at an incident wavelength of 0.6199 Å (Molybdenum K-edge).

Seven runs were performed between 5.5-11 GPa and 850-1180 K. Unfortunately, we were unsuccessfull to achive temperatures higher than 1200 K. Either increasing in power supply was no longer effective to raise the sample temperature, or the experimental assemblage in the Paris-Edinburgh press was no longer stable at high temperature. We actually had 3 blow out during the experiment. This is likely to be due to the very high thermal conductivity of the sintered diamond anvil. Indeed, the power to be delivered for a given temperature was very high and increased with time. This is characteristic of a limited insulation. Moreover, the high power injected probably leads to heat the whole assemblage, which loses its mechanical

strength. **Further developments for a better insultation of the sample are required to reach T > 1200 K and to limit pressure relaxation during heating.**

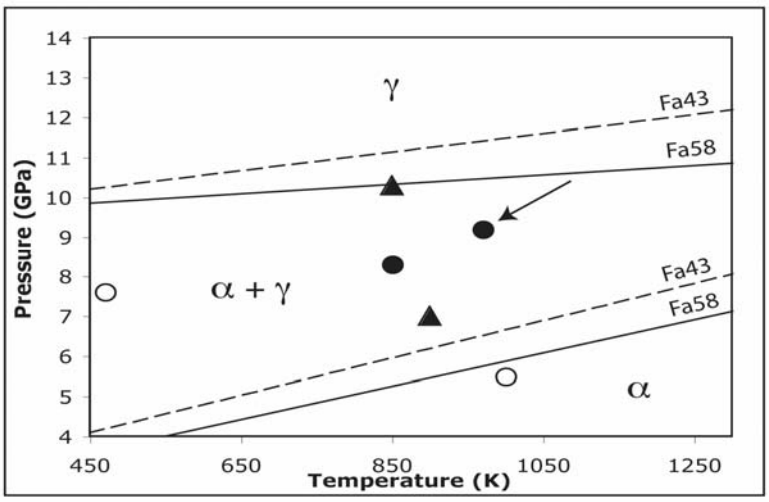

Fig. 1 : Experimental (P,T) conditions and α–γ loop for the studied compositions X_{Fe} = 0.43 (dashed line) and X_{Fe} = 0.58 (solid line).

Solid circles and triangles locate the observation of the transition for X_{Fe} = 0.58, ad 0.43, respectively.

Empty circles locate two experiments X_{Fe} = 0.58 where the transition was not observed. The arrow points to the data displayed in Fig. 2.

Kinetic data on the α-γ transformation were obtained in three runs. Improvement in the data acquisition software and binning of the MAR CCD images increased XRD acquisition rate to 4s exposure every 23s. Further changes in the motorization of the Soler's slits system should even improved this time resolution.

The α-γ transformation observed at 9.2 GPa – 970 K on sample Fa58 (X_{Fe}=0.58) reproduced with further details the disappearance of the diffraction signal signal of the α-phase at the onset of transformation into γ phase (Fig. 2). At t = 23s, diffracted intensity by the α-phase diffraction decreases rapidly until t = 69s, while the γ-phase grows and reaches a plateau at t = 69s. This suggests that the α-phase is disordered between 23-46 s. The latter timescale is comparable to that observed in HS3260.

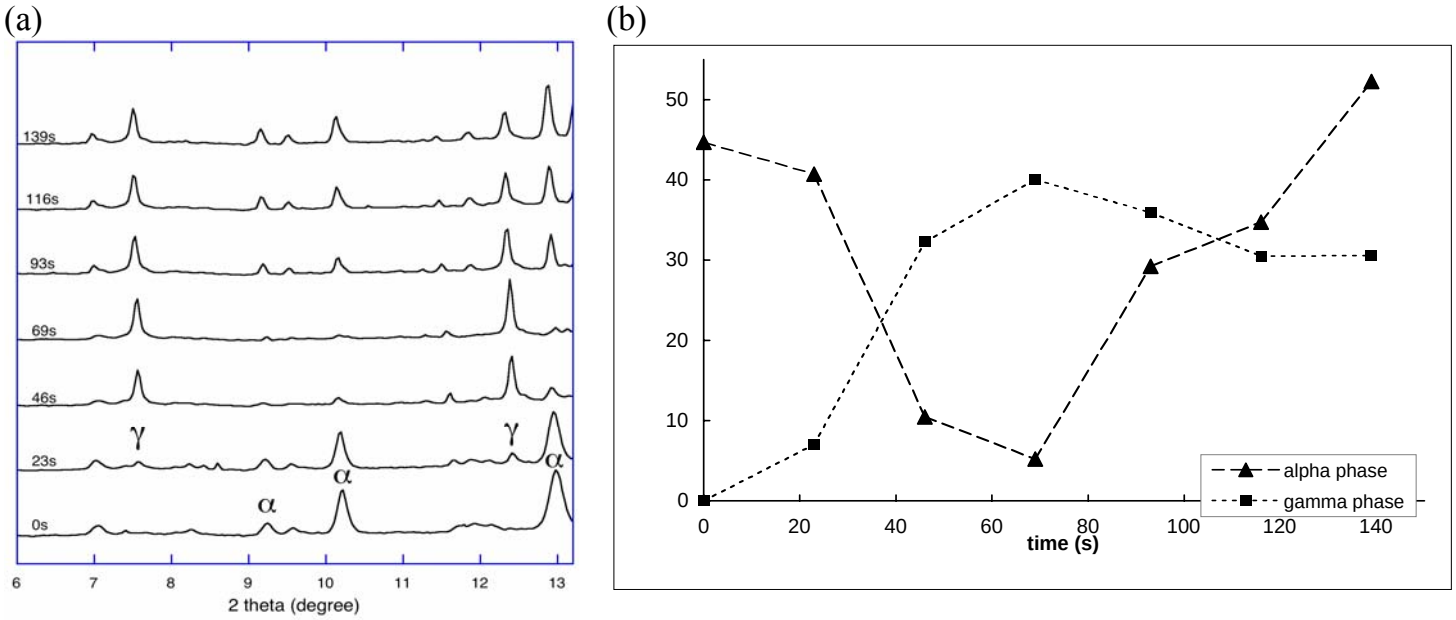

Fig. 2: (a) Time series XRD spectra of the α-γ phase transition on sample Fa58 between 9.2GPa – 970K and 6.1GPa – 1180K. (b) Evolution with time of the intensity of selected diffraction peaks of the α and γ phases. Both diagrams emphasize the fast decrease of the α-phase that almost disappears at 46s, while the γ-phase grows. Later (t = 93s), the α-phase re-appears, once the γ-phase is grown.

This phenomenon may have a great impact on propagation on the seismic waves through the terrestrial planets' mantle, and needs to be further constrained. Unfortunately, the Fe/Mg rearrangement could not be quantified during transformation, since the pressure dropped significantly during the transformation while temperature raised to 1180 K. Nevertheless, **the first goal of experiment HS3530 to confirm the disappearance of the α-phase at the onset of the transition was achieved**.