ESRF	**Experiment title:** High pressure - high temperature behaviour of Fe-bearing phase-X	**Experiment number**: HS4056
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Report:

Potassium in the Earth plays a fundamental role from being one of the major source of radiogenic heat of our planet, from the crust to the inner core, to being a leading alkalic component in metasomatic fluids and in diamond-forming silicate, carbonate-silicate and Cl-rich brine liquids as recorded by inclusions in kimberlitic diamonds worldwide [1]. Primary potassic minerals are very rare in mantle paragenesis due to their very restricted P-T-fluid conditions of formation. Some of them are hydrous phases (phlogopite, KK-richterite, phase-X and K-hollandite) that have been indicated, together with Dense Hydrous Magnesium Silicates, as possible candidates for carrying water deep into the Earth's mantle. In subduction slabs settings, experimental petrology suggested a sequence of phase-breakdown, by increasing pressure, from phlogopite to K-amphibole, to phase-X, a hydrous K-rich silicate, and to K-hollandite [i.e. 2].

Phase-X has a general formula $A_{2-x}M_2Si_2O_7H_x$ where A= K, Na, Ca, [], and M= Mg, Al, Cr^{3+} (with H_2O contents from 1.8 to 4.2 wt.%), and a stability in a Fe-free system from T=1150-1400ºC and P=9-17GPa [3-4]. This phase has the highest thermal stability of any yet investigated hydrous silicate, the only mineral to be stable along an average mantle adiabat.

We performed H*P* - H*T* diffraction experiments on a sample of Fe-bearing phase-X of composition $(K_{1.307}Na_{0.015})(Mg_{1.504}Fe^{3+}_{0.373}Al_{0.053}Ti^{4+}_{0.004})Si_2O_{7.00}H_{0.360}$ (hexagonal, sp. gr. $P6_3cm$, lattice constants a=5.005(1) Å, c=13.148(2) Å; V=285.23(9)Å^3) to define the structural stability of this phase, the EoS and the structural stability at high temperature.

<u>Single-crystal H*P* experiment</u>. We carried out single-crystal X-ray diffraction experiments collecting 28 points up to 40GPa. Single crystal was loaded in a DAC with a ruby chip as pressure calibrant, and He as pressure transmitting medium. At each point we carried out a data collection by rotating the DAC of 60° along the ω axis (from -32 to 32) with an angular step of 1° and time of 1.5sec per step.

No phase transition was detected up to 40GPa. The lattice parameters evolution was determined in the range 0.001 - 40 GPa by fitting the single-crystal data after a data reduction obtained by the CrysAlis software

(Oxford diffraction). The axial compressibilities (a/a_0 =1-0.00198(3)GPa and c/c_0=1-0.00156(1)GPa), show a clear anisotropy in the [001] direction that is stiffer than [100] direction (Fig.1). The cell volumes were fitted with a 3rd-order Birch-Murnaghan EoS in the range 0.001 - 34 GPa (Fig. 2), giving a bulk modulus K_0 = 144(1) GPa K'=2.4. Comparing this data with the H*P* behaviour of hydrous and anhydrous end-members of $K_2Mg_2Si_2O_7$ reported from a previous synchrotron X-ray diffraction study [5], (K_0 ~74GPa, K'=4), we found that our bulk modulus is twice those previously measured. In contrast, density functional based computations [6] found a bulk moduli of K_0=128GPa, K'=4.0 for the Na and K_0=132 GPa, K'=4.4 for the K anhydrous end-members of phase-X. Our data on the Fe^{3+}-bearing phase-X gives a bulk modulus more similar to the calculated than measured values for the potassium end-member. Structural refinements are in progress to define the structural evolution with pressure. Our data suggest that the Fe-bearing hydrous phase-X remains stable up to 35GPa and it may represent a possible storing phase for K and water under typical mantle geothermal conditions.

(*Comodi P, Nazzareni S, Bindi L, Borov A, IMA general meeting, Budapest 21-27 August 2010; Nazzareni S, Comodi P, Bindi L, Bobrov A, 89 Congresso SIMP Ferrara 13-15 Settembre 2010 (oral presentation)*)

<u>**H*P*-H*T* experiment**</u>: Powder of the same phase-X was loaded in a DAC with 3 chips of ruby and Ne as pressure transmitting medium. We started loading the sample up to 10 GPa before heating by using the YAG laser in the double side configuration. The sample was heated in a range between 2000 and 2800K at constant pressure of ca. 10 GPa, 13GPa and 23GPa for several cycles.

The sample showed a complex behaviour during heating with transformations into different crystalline phases . After the heating experiment we mapped the sample performing a data collections on a mesh grid to study the modifications that occurred after the experiment.

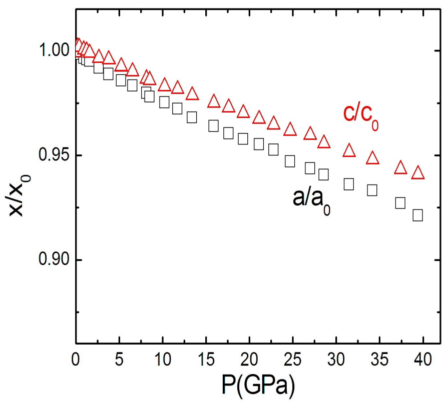

Fig. 1

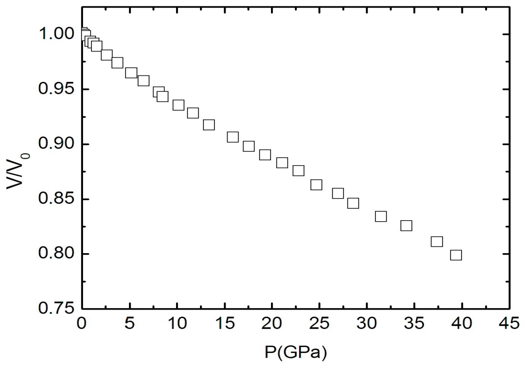

Fig. 2

References

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