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| ESRF | **Experiment title:**<br>**Equations of state of hydrous minerals at high pressures: Consequences for their stability and for the water cycle within the deep Earth** | **Experiment number:**<br>**HS 132** |
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## Report:

Monochromatic angle-dispersive X-ray diffraction measurements have been carried out at high pressure (HP) at both room temperature using a membrane diamond anvil cell and high temperature (HT) with an externally heated Mao-Bell type diamond anvil cell. These experiments were conducted on powdered samples. Argon or Nitrogen, loaded cryogenically, served as pressure transmitting media for the room-temperature measurements; pressure was deduced from the shift of the $R_1$ fluorescence line of several ruby chips. During HT experiments, sodium chloride was used as pressure transmitting medium and pressure calibrant. The diffraction patterns have been collected on image plates, between 1 and $50° 2\theta$, with an X-ray spot of 50x50 $\mu m^2$ at the sample. Exposure times were ranging from 600 s to 1200 s.

*Three sets of data were obtained at high pressure and room temperature* on:

- lawsonite $CaAl_2Si_2O_7(OH)_2.H_2O$ up to 18 GPa (23 data points)
- phengite $KAl_3(Mg,Fe)Si_7AlO_{20}(OH,F)_4$ up to 15 GPa (21 data points)
- antigorite $Mg_3Si_2O_5(OH)_4$ up to 12.7 GPa (15 data points).

The proceeding of phengite and antigorite data is still under way, as we first need to refine the structure of these two minerals at ambient conditions. A fit of the lawsonite unit cell volume data to a Birch-Murnaghan equation of state, setting K'=4, leads to an isothermal bulk modulus $K_0$ of llO(3) GPa. This result is close to the 96(2) GPa value recently obtained from single crystal measurements by Comodi and Zanazzi (1996), but much lower than the 191(5) GPa determined by Holland et al. (1996). The slight difference between our results and those of Comodi and Zanazzi (1996) is likely to be due to our quasi-hydrostatic experimental conditions. This equation of state is only valid up to 10 GPa, since the X-ray patterns recorded clearly indicate that lawsonite undergoes a phase transition at 10 GPa (Fig. 1). The structure of this high-pressure new phase has not yet been determined.

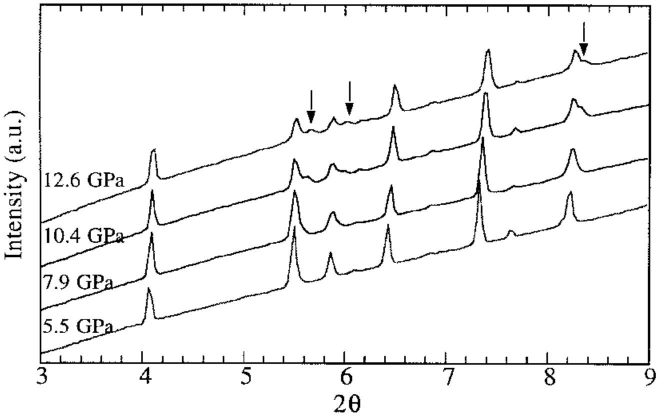

Figure I: selected diffraction pattern of lawsonite across the transition. The arrows show the new reflexions which appears in the new high-pressure phase

We have focussed our HP/HT measurements on lawsonite. *Three isothermal sets of data were recorded at 484, 660 and 759 K, up to pressures of* 10 *GPa.* The bulk moduli, calculated as the reciprocal of cell-volume compressibility are respectively 136(2) GPa, 151(2) GPa and 155(3) GPa. Compared to the 100 GPa value obtained at 300 K by Comodi and Zanazzi (1996), this clearly indicates a positive dK/dT value. This unsual behaviour of lawsonite has still to be explained.

At this point, it is important to further investigate the behaviour of hydrous minerals at simultaneous HP and HT. Hence, we ask for a continuation of this proposal in order to measure the equation of state at HP and HT of another important hydrous mineral: zoïsite $Ca_2Al_3O.SiO_7.SiO_4.OH$.

*Publications:* Daniel, I., G. Fiquet, Ph. Gillet, M. Schmidt, M. Hanfland, High-pressure high-temperature behaviour of lawsonite as viewed by *in situ* X-ray diffration and Raman spectroscopy. *Abstract accepted* for *oral communication* at *EUG 9.*