| | | |
|---|---|---|
| ESRF | **Experiment title:** P/T phase diagram of BaCO3 and related structures and their bearing on the Earth's mantle stability of carbonate polymorphs | **Experiment number**: **ES- 382** |
| **Beamline**: | **Date of experiment**: from: 19 feb 2016 to: 22 feb 2016 | **Date of report**: |
| **Shifts:** | **Local contact(s)**: Wilson Crichton | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

Marco Merlini*
Davide Comboni*
Diego Gatta

## Report:

The experiments focussed on a time-resolved monochromatic X-ray powder diffraction investigation of phase transition in the BaCO3 system, a model system for carbonate behaviour at inner Earth's condition.
Temperature ramps have been performed at variable pressure, in the pressure interval 0-10 GPa.
We first addressed the question about a possible polymorphs existing above 7 GPa for BaCO3, for which a rhombohedral symmetry was proposed. On compression of BaCO3 in aragonite form, we detected a broadening of diffraction peaks and a partial amorphization from 7 GPa. On heating at 8 GPa, the sample crystallised in the post-aragonite form at temperatures as low as 300 °C, suggesting that if a different polymorph exist, it is a metastable or a very low temperature phase. Post aragonite is the stable polymorph even at moderate temperature above 7-8 GPa. Post aragonite tranformed back on decompression into aragonite. The temperature ramps at low pressure revealed the phase transition to disordered calcite and NaCl-type structures (figure 1)

.

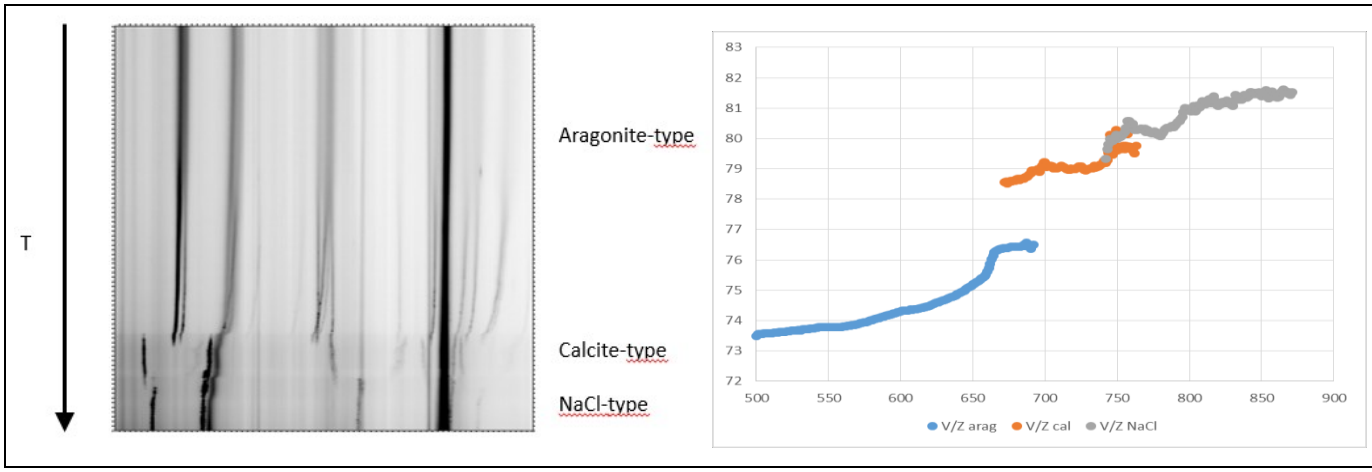


Figure 1 – Time resolved X-ray powder diffraction pattern of $BaCO_3$ during heating ramps. The phase transition are evident. The figure on the right reports the volume data obtained by Rietveld fit of the powder pattern and clarify the first order transition for aragonite to calcite-type structure (with volume discontinuity) and a secon order character for calcite to NaCl type transition. The cohexistance of calcite and NaCl-type structure is interpreted as a pressure oscillation during the phase transition. The x-axis reports the progressive number of diffraction scan.

The data analysis is in progress for all the P/T ramps together with a recalibration of exact P and T conditions.

Similar experiments have been performed on pure calcite system, to bracked accurately the phase bundary of calcite/aragonite polymorph as well as alkali carbonates, which, evidenced a richer polymorphism than reported in literature (figure 2)

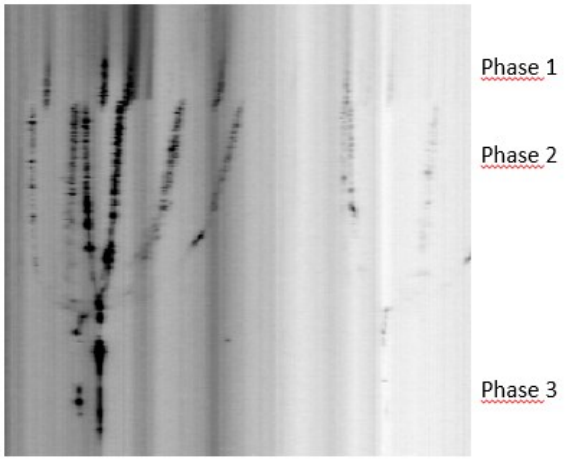


Figure 2- High pressure and temperature polymorphism of $K_2CO_3$ at 2 GPa and variable temperature.