ESRF	**Experiment title:** Disordering and melting of dolomite-ankerite: implications for the long term C-cycle	**Experiment number**: **HS- 4712**
Beamline:	**Date of experiment**: from: 31 oct 2012 to: 6 nov 2012	**Date of report**:
Shifts:	**Local contact(s)**: Wilson Crichton	*Received at ESRF:*

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Report:

The experiments focussed on a high-resolution monochromatic X-ray powder diffraction investigation of dolomite-ankerite solid solution at accurately controlled pressure, temperature and oxygen fugacity conditions, employing the newly developed Large Volume Press (LVD) at ID06 beamline.

The experiments have been performed with a 25/10 octahedra assembly, with sample contained into a graphite capsule, surrounded by BN sleeve and graphite furnace. A Pt/Rh thermocouple have been inserted into the assembly, close to the sample, to accurately monitor and control temperature, throug an improved feedback control. Pressure was determined against well established thermal equation of state of standard materials (BN, MgO, Au), measured by X-ray diffraction. Two samples have been investigated, a pure dolomite, $CaMg(CO_3)_2$, and an intermediate composition of the dolomite-ankerite compositional join, $Ca(Mg_{0.6}Fe_{0.4})(CO_3)_2$. X-ray diffraction data have been collected with a single point detector, equipped with slits for rejection of X-rays scattered from gaskets and furnace materials. The full data analysis is underway. Preliminary data indicate two main results: the detection of order/disorder process with extreme accuracy in pressure and temperature, monitored both by superstructure peak intensity variation and lattice parameter axial ratio evolution, and the determination of the melting processes, indicated by significant deviation form linearity of cell volume, a variation of intensity of the main diffraction peaks, related to significant recrystallisation, and an increase in background intensity. The in-situ results, coupled with ex-situ

examination of the run product (figure 3), will therefore help in reconstruction of accurate phase diagram and melting properties of carbonates, which to date are only restricted to Fe-free compositions.

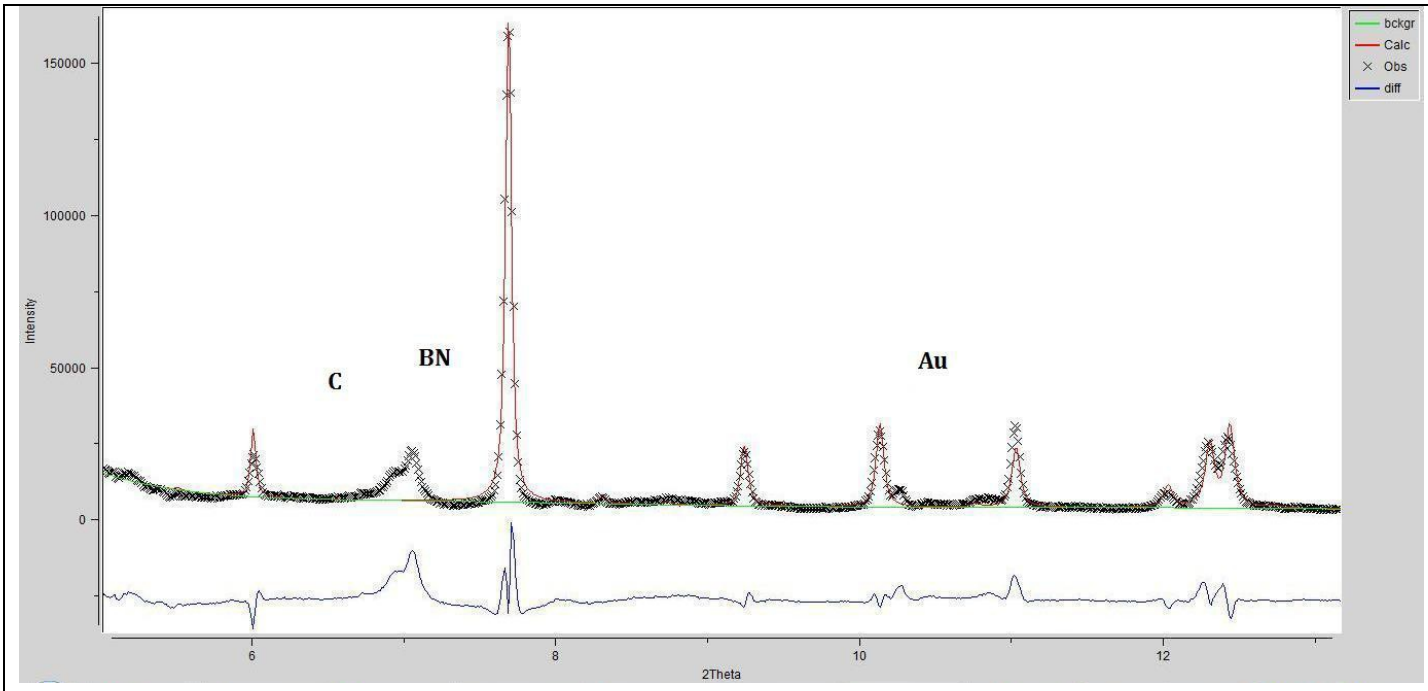
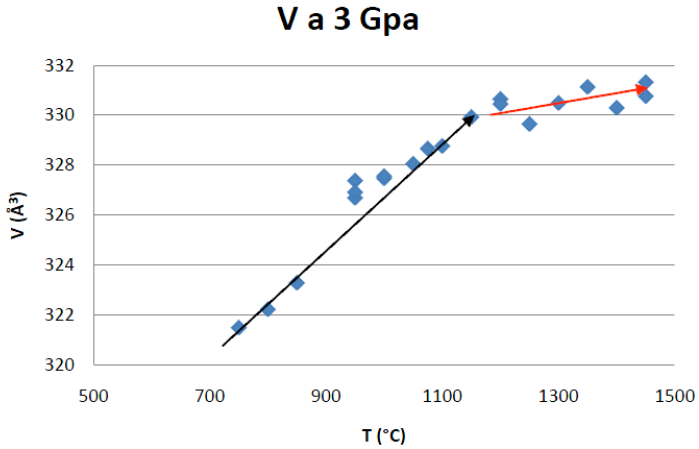


Figure 1 - Monochromatic diffraction pattern and Rietveld fit of ankerite at 900 K and 3 GPa



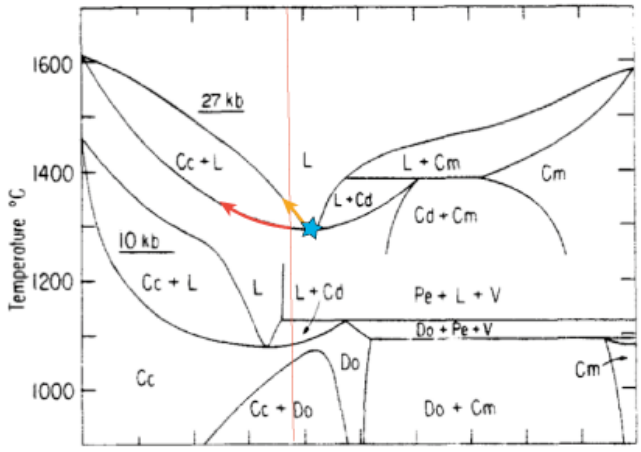


Figure 2- Volume variation of dolomite and preliminary qualitative interpretation of melting process

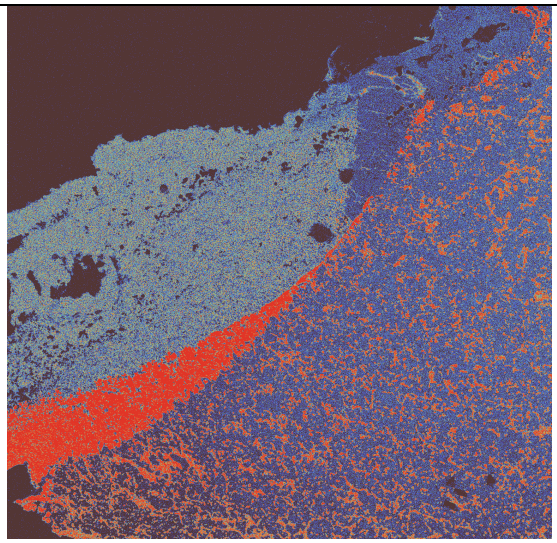


Figure 3 – ex-situ characterization of experimental run quenched from 1400 °C, 3 GPa. Fe X-ray map of Fe-periclase (red) formed upon incongruent melting of Fe-dolomite. Hour-glass shape os Fe-rich portions of the run charge provide microstructural evidence of melting and a high-temperature constraint for phase compositions at the end of the experiment.