ESRF	**Experiment title:** Pressure induced polyamorphism and elastic properties of amorphous carbonates studied by High-Pressure Pair Distribution Function and Reverse Monte-Carlo analyses	**Experiment number**: HC-841
Beamline: ID11	**Date of experiment**: from: 21-06-2013 to: 24-06-2013; 26-07-2013 29-07-2013	**Date of report**: 11th August 2013
Shifts: 18	**Local contact(s)**: Jonathan Wright	*Received at ESRF:*

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Report: The experiment was divided in two visits (3 days each, in June and July). A symmetric diamond anvil cell was used for all the experiments. Stainless steel gaskets were loaded with amorphous and crystalline carbonate samples to study (1) the compressibility of the crystalline standards monohydrocalcite and hydromagnesite and (2) polyamorphism in the $Mg_xCa_{1-x}CO_3 \cdot H_2O$ system.

The study of the crystalline compounds was successful, and the equation of state has been determined for the first time for monohydrocalcite and hydromagnesite, two reference compounds of relevance for the study of amorphous calcium-magnesium carbonates.

An incident radiation of 35 KeV was used to study the amorphous compounds, with a focused beam of ~3×3 micron size, and a sample-detector distance of ~70 mm. With these settings, powder patterns with a q maximum of 90 nm^{-1} were obtained. The experiments were performed in the absence of any technical problem. The only problem was associated with the low Z elements present in the amorphous samples, and their low density. One of the samples (pure amorphous magnesium carbonate; $MgCO_3 \cdot H_2O$) could not be studied due to the low scattering intensity coming out in comparison with the high background due to Compton radiation from the diamonds. The scattered radiation from amorphous samples with mixed Ca and Mg compositions ($Mg_xCa_{1-x}CO_3 \cdot H_2O$) was also low, but patterns could be obtained to perform semi-quantitative analysis (see Figure 2; full quantitative analysis was

not possible due to the high background). To this end, pellets of compressed samples were prepared in a hydraulic press, grounded and loaded into the gasket. This procedure allowed obtaining better statistics in rasonable acquisition times (20 minutes per pressure point). Two amorphous samples with different Mg content were studied and a polyamorphic transition was observed in one of them, as it had been hypothesized. This result open new lines of research and further hypotheses to be tested.

We plan to perform new scattering experiments in these amorphous samples, with the goal of getting better statistics and be able to obtain powder patterns usable for Reverse Monte-Carlo structural analyses. To this end, we plan to use the Multi-Channel Collimator available at ID27. Using ID27 will also allow going to lower energies and increasing the coherent scattering cross section (higher flux is available at ID27 at lower energies, ~25 KeV. The lowest energy with a focused beam at ID11 is ~35 KeV).

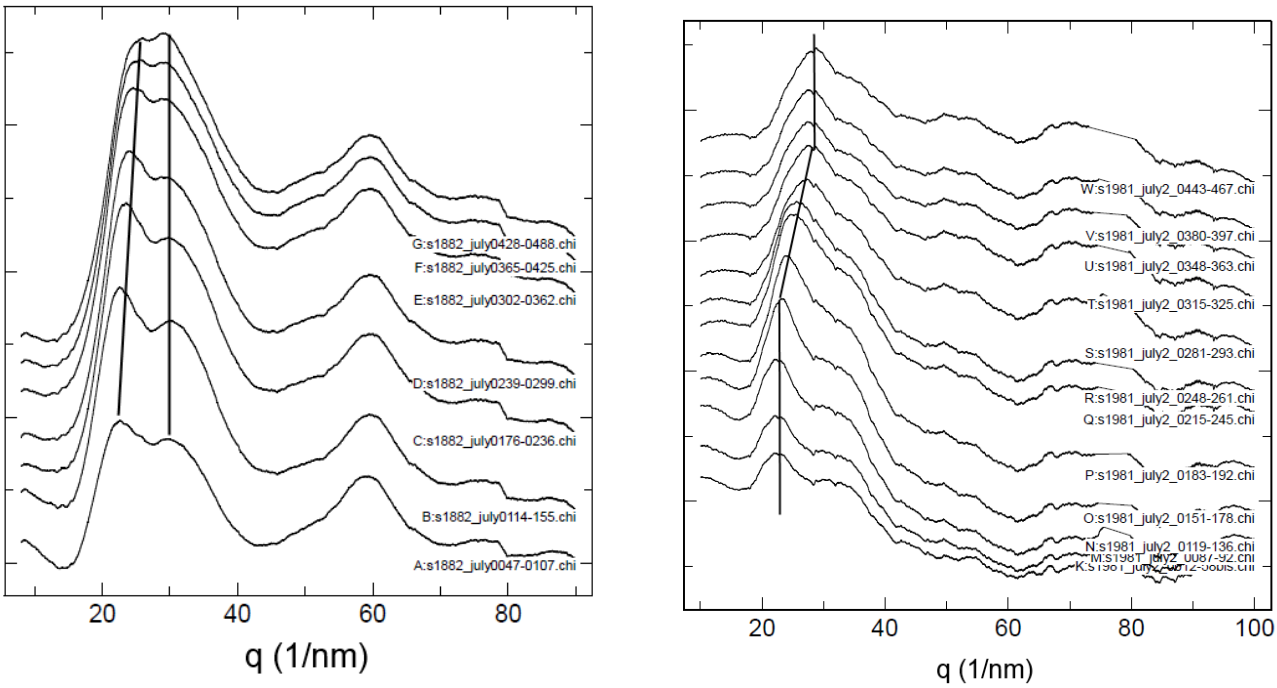

Figure 1. *Lines are eye guides to follow the position of the first two diffraction peaks.* ***Left****: Sample 1882. No phase transition is observed.* ***Right****: Sample 1981. An amorphous-amorphous phase transition is observed. The Mg content of the samples seems to be controlling polyamorphism, exerting 'chemical pressure'.*