



	Experiment title: Crystal structure of magnesite-II at Mbar pressures	Experiment number: ES- 142
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Shifts:	Local contact(s):	<i>Received at ESRF:</i>
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Report:

We investigated crystal structure of carbonates up to Mbar pressures, with the main goal to stabilize tetrahedrally coordinated carbon in ultrahigh pressure structures.

Preliminary data indicate that magnesite and siderite maintain their rhombohedral structure up to 135 GPa at ambient pressure. This behaviour is contrasting with the polymorphism observed in Ca-rich carbonates. In particular calcite transforms into high pressure polymorph on compression at ambient temperature above 2 GPa. Dolomite, $\text{CaMg}(\text{CO}_3)_2$, transforms to dolomite-II above 16 GPa, and, we investigated during this session, $\text{CaMn}(\text{CO}_3)_2$, which transforms into a high pressure polymorph at 40 GPa.

Magnesite and siderite transform only at high temperature. We studied a low Fe-magnesite ($\text{Mg}_{0.9}\text{Fe}_{0.1}\text{CO}_3$), an intermediate magnesite-siderite sample ($\text{Fe}_{0.6}\text{Mg}_{0.4}\text{CO}_3$) and pure siderite. The iron content in magnesite sample was not enough for laser radiation absorption and high temperature. On the contrary, it was very easy to transform intermediate siderite-magnesite and pure siderite. We detected a red-ox reaction, resulting into a Fe^{3+} carbonate (figure 1) and Fe^{3+} oxide (figure 2), as revealed by structure determination. By chemical balance, diamond should have formed. Fe13O19 was determined both in the intermediate magnesite-siderite and in pure siderite sample. The high pressure structure determination of siderite is in progress.

High pressure experiments were performed at ID09A. We used a monochromatic beam ($\lambda=0.414314 \text{ \AA}$) with beam size $15 \times 15 \mu\text{m}^2$ on the sample. The detector used was a Mar555 flat panel. The crystals were placed in the high pressure chamber of a membrane type DAC,

equipped with Boheler-Almax designed diamonds, beveled with lower section 150 μm diameter. We used rhenium gasket, preindented to a thickness of 30 μm . The sample chamber was gas loaded with Ne, which acted as both pressure transmitting medium, pressure marker and insulating material for high temperature heating. We also monitored the pressure by measuring the fluorescence line of a Sm-doped Sr-borate. We performed single crystal data collections by omega-step scan rotation, with 1° step, in the angular range $-30/+30^\circ$. Normally, we performed two data collections at different chi angle positions: $\text{chi}=0$ and $\text{chi}=90^\circ$, in order to collect all the diffraction peaks in the accessible reciprocal space determined by the DAC opening cone. Diffraction data were handled using the CrysAlis software, with instrumental geometrical parameters (sample to detector distance, detector tilt) previously refined against standard quartz single crystal sample. The integrated single crystal data were processed with Jana2006 and Superflip software

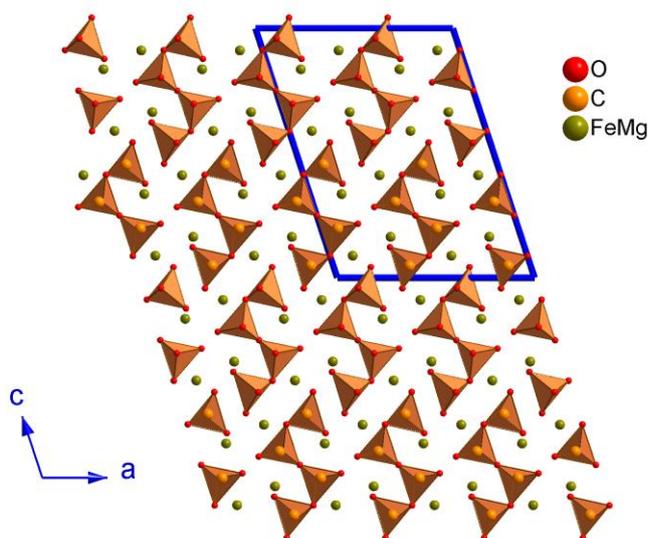


Fig. 1. Crystal structure of $\text{Mg}_2\text{Fe}_2(\text{C}_4\text{O}_{13})$, based on truncated tetrahedral carbonate chains.

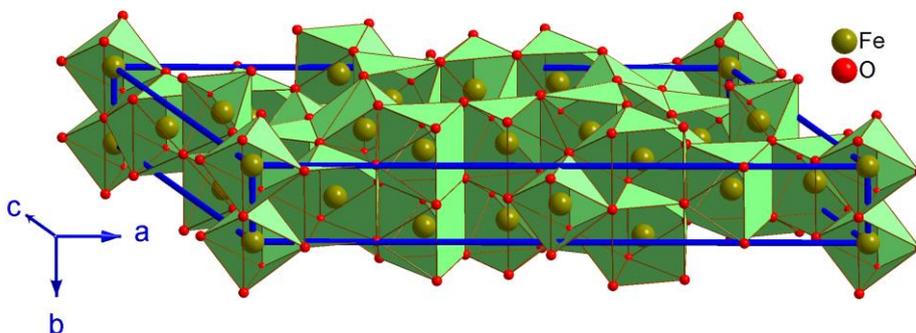


Fig. 2. Crystal structure of $\text{Fe}_{13}\text{O}_{19}$