ESRF	**Experiment title:** Electronic structure and bonding changes in siderite (FeCO3) melts at high pressure	**Experiment number**: ES-599
Beamline: ID20	**Date of experiment**: from: May 3rd 2017 to: May 9th 2017	**Date of report**: August 31st 2017 *Received at ESRF:*
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Report:

Background:

Carbonate melts at high pressure are of fundamental interest for understanding the Earth's deep carbon cycle and Fe containing magnesite ($MgCO_3$) has been proposed as a major carbon-containing phase in the lower mantle. Despite this, there is almost no information about the melt structure. Recent experimental and theoretical studies on $(Mg,Fe)CO_3$ have found evidence or a new crystalline phase above ~80 GPa consisting of corner sharing CO_4 tetrahedra [2,3]. If a similar transition to a tetrahedrally coordinated CO_4 phase were to occur in the melts, it would drastically change the melt properties, since unlike CO_3, CO_4 can form polymerisable networks [1].

The goal of these experiments was therefore to use non-resonant inelastic x-ray scattering (NRIXS) to investigate the pressure induced electronic structure and bonding changes of carbon and iron in siderite ($FeCO_3$) melts up to lower mantle pressures.

Experiments:

$FeCO_3$ glasses were synthesized in our home institution by laser heating crystalline siderite in diamond-anvil cells above 60 GPa, using either LiCl or KCl as pressure transmitting medium. This resulted in the formation of $FeCO_3$ glass spheres that were recovered back to ambient conditions and transferred into Be gaskets prepared at the ESRF (Jeroen Jacobs). The gaskets were then inserted in a panoramic DAC provided by ID20, in order to collect data in transmission mode, with the incoming X-ray beam going through the Be gasket to minimize absorption by the diamond anvils.

Although *in situ* high pressure data could not be collected at the carbon K-edge due to too large contribution from the diamond anvils to the sample signal, data were successfully recorded up to 50 GPa at oxygen K-edge and iron L-edge. All C, O and Fe edges spectra could be collected on free standing samples synthesized at high P-T conditions.

Results:

The C-edge spectra show no presence of sp3 carbon but show instead the broad contribution caracteristic of tetrahedrally coordinated carbon. The completion of these experiments thus proves that the transition from 3-fold to 4-fold carbon theoretically predicted in high pressure crystalline carbonates also takes place in the molten state. Besides, the results demonstrate that this transition that occurs above 40 GPa in the molten state is quenchable.
Data collected at the Fe-edge are being processed to check for any effect of the Fe spin transition.

[1] A.P. Jones et al., 2013, Rev. in Mineral. and Geochem., 75:289-322.
[2] A.R. Oganov et al., 2008, Earth Planet. Sci. Lett., 273:38-47.
[3] E. Boulard et al., 2015, Nat. Commun., 6:6311.