ESRF	**Experiment title:** Trace element distribution between Earth mantle phases and carbonates at in-situ conditions	**Experiment number**: ES-567
Beamline: ID27	**Date of experiment**: from: 5/7/2017 to: 11/7/2017	**Date of report**: 09/02/2018
Shifts:	**Local contact(s)**: Gaston Garbarino	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Karen Appel*, Nicole Biedermann*, European XFEL, Schenefeld, Germany

Max Wilke*, Georg Spiekermann*, University of Potsdam, Germany

Anna Pakhomova*, DESY, Hamburg, Germany

Report:

The aim of this project is to better understand element distribution processes between carbonates and silicates at mantle conditions and to elucidate the importance of carbonates as possible trace element carriers in the mantle. To achieve this goal, in this experiment, we performed in-situ laser-heating experiments with combined XRD and XRF at beamline ID27 at ESRF of selected carbonate-silicate reactions. We used the following samples and experimental conditions:

a. Samples: Natural enstatite with Fe contents of 30 wt.%, natural olivine (San Carlos type) and synthetic enstatite with more mantle-related Fe contents of only 10 wt.% were used as silicate starting materials. Carbonate starting materials in the different experiments were strontianite, Sr-doped calcite and calcite.
b. Sample geometry: To account for the difficulty of XRF analysis of single phases when dealing with two phases, in some experiments we placed adjacent platelets, cut with a FIB, in form of half circles and with known dimensions into the sample chamber (see Fig.1). The remaining experiments were performed with silicate crystals or glass and carbonate powders.
c. We used Ar as a pressure transmitting medium. Results from a previous beamtime with carbonates have shown that K-carbonate likely formed by reaction of $SrCO_3$ with the pressure medium KCl.

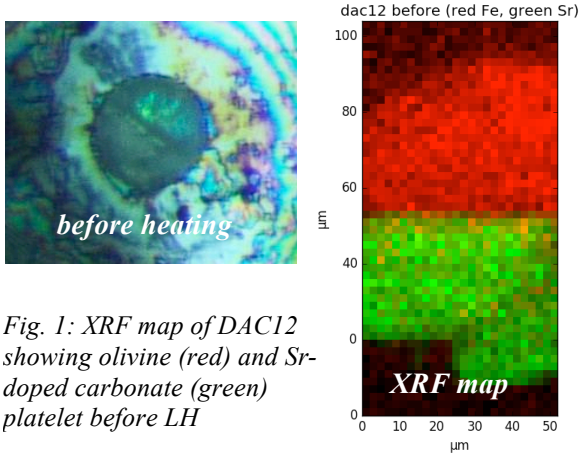

Fig. 1: XRF map of DAC12 showing olivine (red) and Sr-doped carbonate (green) platelet before LH

A CO_2-laser (off-axis) was used for heating the sample material with low Fe contents. X-ray diffraction images were collected at an energy of 33.17 keV before, during and after the heating process. In addition, XRF maps were recorded before and after the heating to monitor migration of Fe and Sr. The XRF measurements were performed using a confocal setup as described by Wilke et al. (2010) and Petitgirard et al. (2012) and XRF signal was collected in 20° from the incoming beam. As an example, XRF maps and XRD patterns of the starting assemblage olivine + $SrCO_3$ (DAC13) are shown in Fig. 2.

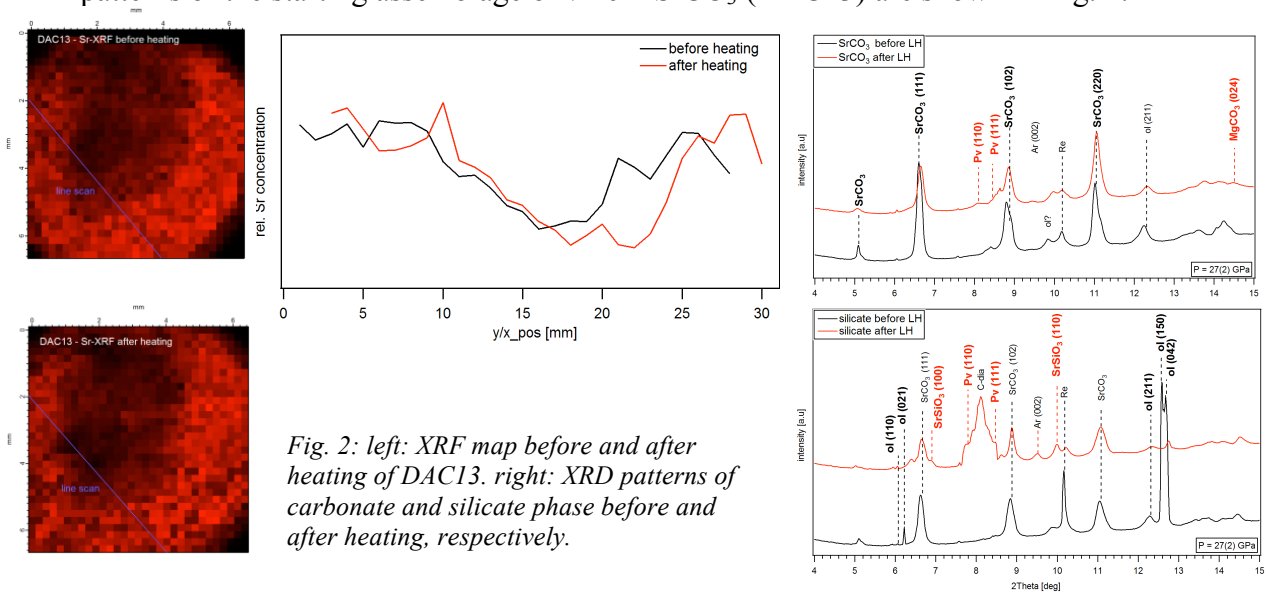

Fig. 2: left: XRF map before and after heating of DAC13. right: XRD patterns of carbonate and silicate phase before and after heating, respectively.

In total, 7 experiments were conducted during our beamtime. In most of these experiments we observed a phase transition from enstatite or olivine to bridgmanite (+MgO in case of olivine) at 25 – 38 GPa and during heating at a maximum temperature of 2400 K. The carbonate phase has mostly transformed into aragonite and decomposed into CaO during heating when the temperature was above 2400 K. In addition, cubic $CaSiO_3$ or $SrSiO_3$ perovskite phases as well as $MgCO_3$ were formed as a product of carbonate-silicate reactions and can be seen in Fig. 2 and 3. A comparison of the XRF intensity maps before and after heating indicates that changes in distribution of Sr across the heated area after annealing are negligible. Post-mortem analyses of the recovered sample material are necessary to clarify whether Sr is distributed into bridgmanite or not. Therefore, we cut TEM foils out from 3 samples for additional analyses which will give further insights in 3-dimensional space.

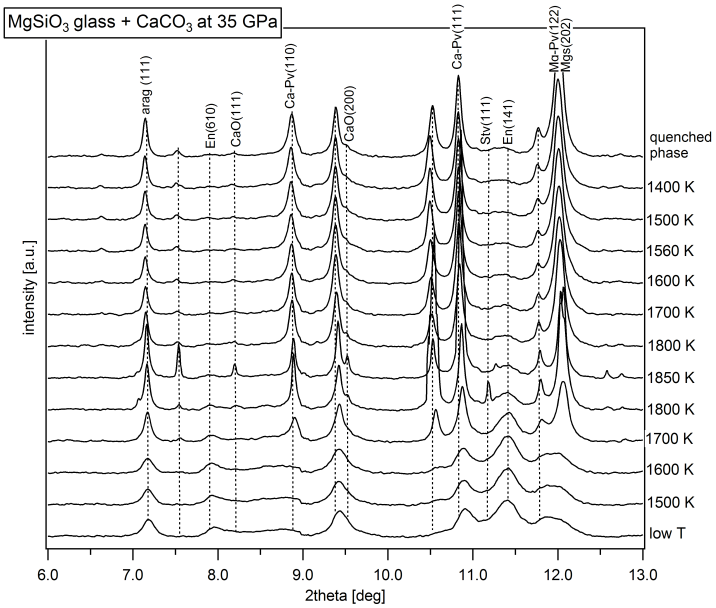

Fig. 3: XRD patterns of laser-heated $MgSiO_3$ glass and Sr:$CaCO_3$ starting materials. The sample was pressurized to 35 GPa and laser-heated to maximum temperatures of 1850 K. The following reaction could be obtained:
$MgSiO_3$ + Sr:$CaCO_3$ + $MgCO_3$ + $CaSiO_3$(Pv) + $MgSiO_3$(Pv)