Nano scale chemical analyses of ultra-high pressure melting experiments

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Modelling the deepest episodes of the Earth's formation and evolution through time requires constraints on the partitioning of elements between solid and molten silicate. Such data provide insights into the early development and composition of different reservoirs of the deep Earth. To elucidate the elemental partitioning at high pressure and high temperature conditions requires state of the art experiments that combine laser heating in a diamond anvil cell with focused ion beam sample recovery and precise and calibrated chemical analyses. Using an in-house laser heating system we have investigated the partitioning of elements during the melting of a basaltic glass at different pressures. The recovered samples were cut using the focused ion beam (FIB) method into a 4 micron thick lamellae. The composition of the molten and solid area were analysed by electron microprobe, an EDX-ray spectrometer mounted in the FIB and using a transmission electron microscope (TEM), after further polishing down to 100 nm.

FIB and TEM analyses were calibrated using natural and synthetic mineral standards that were calibrated using the electron microprobe. The sample of interest was then thinned to an electron transparent lamella using the FIB and an identical location was then measured with EDS systems on both the TEM and FIB.

Sample characterization was performed at the Bayerisches Geoinstitut using a Titan™ G2 80-200 TEM operating at an acceleration voltage of 200 kV and equipped with ChemiSTEM™ technology. This allows high quality X-ray spectra to be recorded with short acquisition times, due to the use of SDDs that are symmetrically mounted in the microscope column around the optical axis with a 0.7 srad solid angle. The EDS system works with a much reduced solid angle, but the large beam-sample interaction volume results in a higher X-ray generation. The data measured with both EDS systems are comparable and show the potential for obtaining full quantitative results from recovered laser heated DAC samples.