ESRF	**Experiment title:** Phase relations of zircon ($ZrSiO_4$) at high pressure and high temperature.	**Experiment number**: 1-02-189
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Report:

This experiment represents a first test of the ID30 heatable diamond anvil cell in combination with the MAR-image-plate detector system on SNBL. The aim of the experiment was (i) to test the feasibility of a powder diffraction experiment at simultaneously high-pressure and high-temperature on BM01 and (ii) to explore the high-pressure/high-temperature behavior of the mineral zircone ($ZrSiO_4$). We performed the experiments by loading a powder of natural zircon together with NaCl, which served as P-T standard, and a 4:1 mixture of methanol:ethanol into the heatable DAC of ID30.

Although the analysis of the data is still in progress, we can already report some interesting results.

(i) The MAR system on SNBL is highly compatible with a high-pressure experiment using a heatable diamond anvil cell (DAC). Although we suffered the loss of one of the anvil diamonds towards the end of the run, we were able to collect powder diffraction patterns in a P-T range of 0 – 8 GPa and 300 – 570 K. The data collected proofed to be perfectly suitable for powder crystallography and can be analyzed with Rietveld refinement. This work is under way at the moment.

(ii) These data will allow us for the first time to determine a P-V-T equation of state of $ZrSiO_4$ and also to look at the structural evolution as a function of pressure and temperature. Clearly, our P-T range did not reach the values of any of the phase changes previously reported at 12 GPa or 1100 K, respectively. However, a preliminary analysis shows that the combination of P and T seems not to shift any of these phase changes into the P-T range of 600 K and 8 GPa as was suspected.

Apart from these preliminary results on the phase relations of zircone, we observed an unexpected behavior of the NaCl pressure standard. After the first heating cycle at 2.3 GPa, we increased pressure at room temperature to about 4 GPa. This led to the appearance of a strong new reflection, which, according to its similarity in 2-dim peak shape and preferred orientation, has to be related to NaCl. A first analysis seems to indicate that this represents a reaction between the methanole/ethanole pressure medium and the NaCl P-T-standard. Surprisingly, this has never been reported before. We suspect this observation to be linked to our extreme fine grain size of the NaCl powder we used. Further data analysis to clarify this intriguing observation is under way.

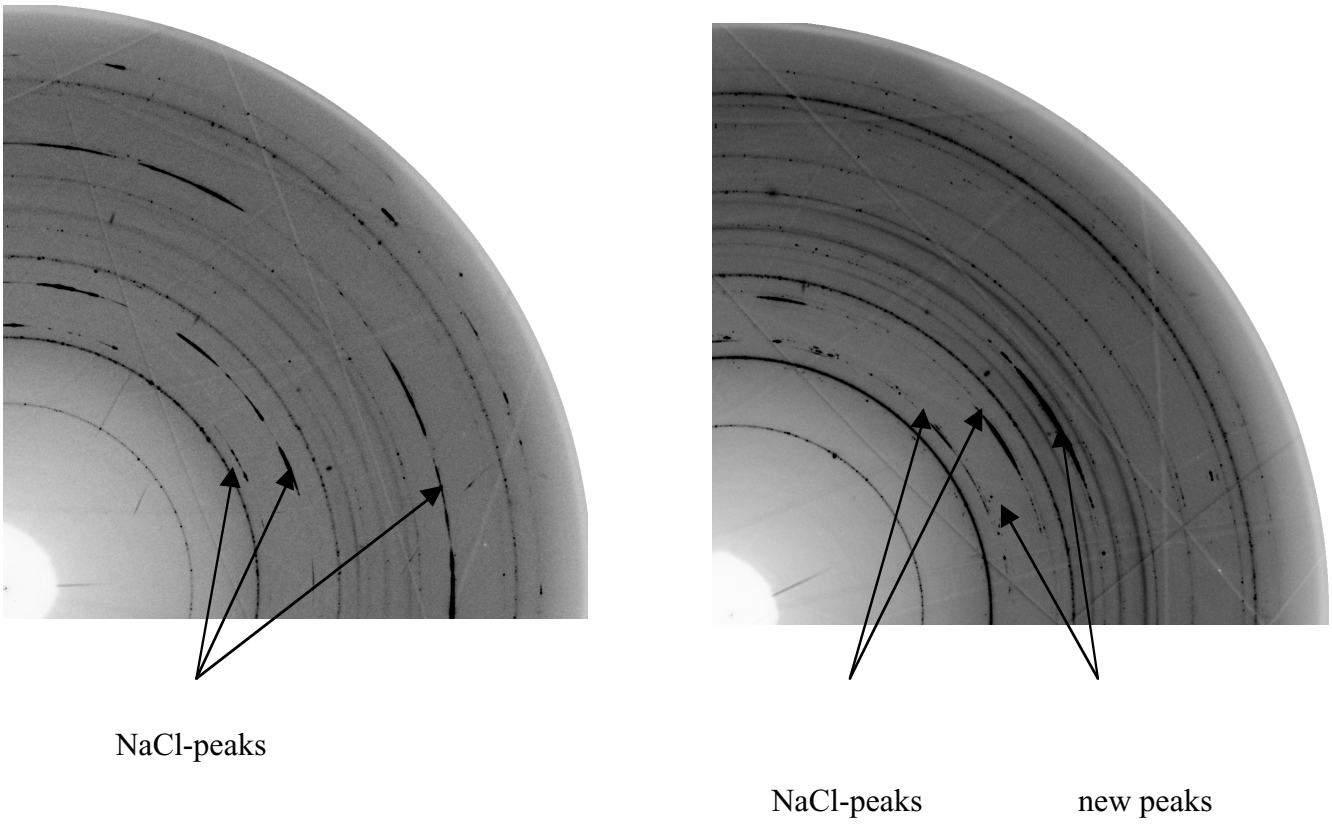

Fig. 1: Powder patterns of Zircone plus NaCl in 4:1 methanol:ethanol. Left after loading at ca 2.3 GPa; right after first pressure increase (4 GPa at room temperature). Note the change in the NaCl-type diffraction rings. A very strong new peak appears whereas a NaCl peak disappeared