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Five runs were carried out in total during beamtime (9-15 april 2014), two days were dedicated to power-temperature calibration and cell loading. And five days were occupied with the experiments, one experiment per day. The most representative experiments among all are number 1, 3 and 4, at which samples were heated up till 500 °C, 500 °C and 800 °C respectively.

During these runs one diamond were replaced with the drilled diamond, which helped to reduce the thickness of diamonds and optimize X-ray transmission at Fe energy range (7 keV). Samples with 1.69 wt % and 0.6 wt % FeO were used in runs. High quality pre-edge and Fe K-edge were presented on XANES spectra with such low amount of iron using one drilled diamond.

The diamond and rhenium gaskets were used in different experiments in order to compare their influence on fO_2 . It is visible that XANES spectra has the same features and changes during experiments with different gaskets. Consequently there is no difference in using either diamond or rhenium gasket. However, for easy and fast cell loading it's better to use rhenium gasket.

The both kind of samples were used in experiments – single crystal and powder. As shown on fig.1 pre-edge feature of antigorite didn't change during heating. But white line of the main edge was shifted towards lower energies and height of the edge decreased, which can be an evidence of iron-speciation changes in the sample. The both types of samples (crystal and powder) show the same XANES spectra shifts.

All challenging parts of the experiment were checked and will be taken into account during future runs. The only limitation, which still must be improved is a heating system. Temperature were dropping down every run at high temperature, thus there was no chance to follow the behavior of the sample at high temperatures due to instability of heating system.

X-ray diffraction was carried out at each step in each run, what helped to trace phase transitions in the sample.

As a result many unclear questions were solved during beamtime such as thickness of diamonds, influence of gaskets on fO_2 , type of samples, amount of iron in a sample. And next experiment should give the information about behavior of iron at high temperatures at stable conditions including all improvements.

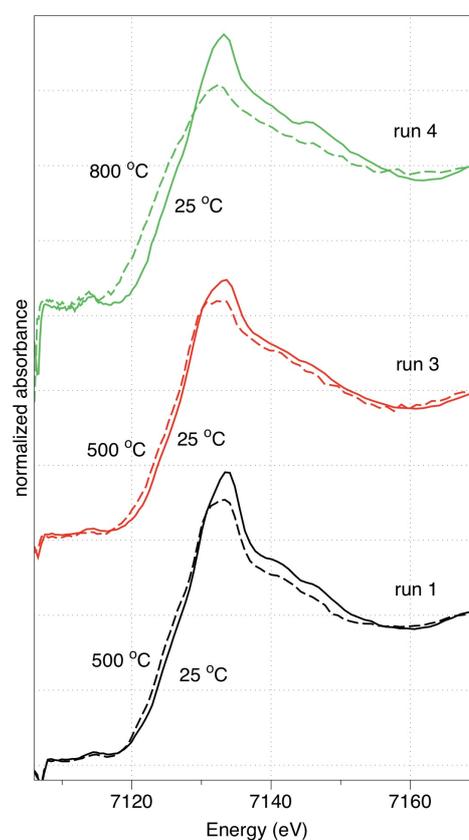


Fig. 1 Comparison of XANES spectra at 25 °C and highest temperature of runs 1, 3 and 4. Run 1 included diamond gasket and single crystal sample. Run 3 – Re gasket and powder sample. Run 4 – Re gasket, powder sample with low Fe content.