



Development of a new micro-furnace for "in situ" high-temperature single crystal X-ray diffraction measurements

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Several experimental methods to reliably determine elastic properties of minerals at non-ambient conditions have been developed. In particular, different techniques for generating high-pressure and high-temperature have been successfully adopted for single-crystal and powder X-ray diffraction measurements. High temperature devices for "in-situ" measurements should provide the most controlled isothermal environment as possible across the entire sample. It is intuitive that in general, thermal gradients across the sample increase as the temperature increases. Even if the small isothermal volume required for single-crystal X-ray diffraction experiments makes such phenomena almost negligible, the design of a furnace should also aim to reduce thermal gradients by including a large thermal mass that encloses the sample. However this solution often leads to complex design that results in a restricted access to reciprocal space or attenuation of the incident or diffracted intensity (with consequent reduction of the accuracy and/or precision in lattice parameter determination).

Here we present a newly-developed H-shaped Pt-Pt/Rh resistance microfurnace for in-situ high-temperature single-crystal X-ray diffraction measurements. The compact design of the furnace together with the long collimator-sample-detector distance allows us to perform measurements up to $2\theta = 70^\circ$ with no further restrictions on any other angular movement. The microfurnace is equipped with a water cooling system that allows a constant thermal gradient to be maintained that in turn guarantees thermal stability with oscillations smaller than 5°C in the whole range of operating T of room-T to 1200°C . The furnace has been built for use with a conventional 4-circle Eulerian geometry equipped with point detector and automated with the SINGLE software (Angel and Finger 2011) that allows the effects of crystal offsets and diffractometer aberrations to be eliminated from the refined peak positions by the 8-position method (King and Finger 1979), and thus maximize precision in unit-cell parameter measurements. The software has been modified to restrict the χ circle movements to between -90° and $+90^\circ$ to reduce chimney effects in the furnace and thus improve stability. Moreover, the temperature stability during the measurements is further improved by optimizing the order of measurements to minimize χ circle movements, and then imposing a waiting time after large angular movements on χ to allow the temperature inside the furnace to re-equilibrate before each measurement. Temperature calibration has been performed iteratively by combining measurements with a standard small diameter thermocouple mounted in the same conditions as the sample together with the lattice parameter determination of materials with known thermal expansion behavior (i.e. silicon, quartz etc. . .). This procedure has the main advantage that the temperature calibration can be obtained with a large number of measurements over a large temperature interval (room-T to 1200°C) and allows the waiting time for the χ movements to be calibrated as a function of temperature.

References

- Angel RJ, Finger LW (2011) *JAppCryst*, 44:247-251.
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