MS111.O05

Geometry limitation-free HT device for in situ/operando SCXRD

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Over fifteen years of experience in studying crystal behaviour and phase transitions at high temperature by single-crystal X-ray diffraction has brought to two fundamental issues about the use of in situ techniques, namely (1) the limitations imposed by the furnace itself to the excursions of goniometer circles, which impede to collect data to high resolution, and (2) the difficulties in controlling the physico-chemical and atmospheric conditions of the experiment.

Our new design, which is based on the Bruker FR559 heater, makes use of a vertical air or gas flow to heat the sample. It is mounted externally, just above the crystal at a fixed angle, and therefore it allows full liberty to the rotation of all goniometer circles. A thermocouple is placed at the exit of the gas flow. The blower can make use of air, N2 or other gases to heat the sample, thus allowing studying the high-temperature behaviour of crystals under different atmospheric conditions. Standard mounting in quartz vials is also compatible with this set-up and methods will be described for mounting by making use of quartz wool to keep the crystal in position and avoid the use of glues or cements and for sealing the vial under vacuum or other conditions.
Preliminary tests conducted so far allowed to reach the maximum temperature of ca 900 °C, however other tests are in hand to allow the HT device to reach 1100 °C. Calibration has been done by known melting points of several pure compounds and by the transition temperature of quartz. Temperature precision is ±2 K in the whole temperature range.

Examples will be shown about the study of phase transitions associated with the "jumping" effect in thermosalient crystals (project in collaboration with Zeljko Skoko, U. Zagreb, Croatia) and α-β phase transition in quartz-type ZIFs (project in collaboration with Ian D. Williams, HKUST, Hong Kong).
Keywords: high temperature, phase transitions, single crystal to single crystal transformations