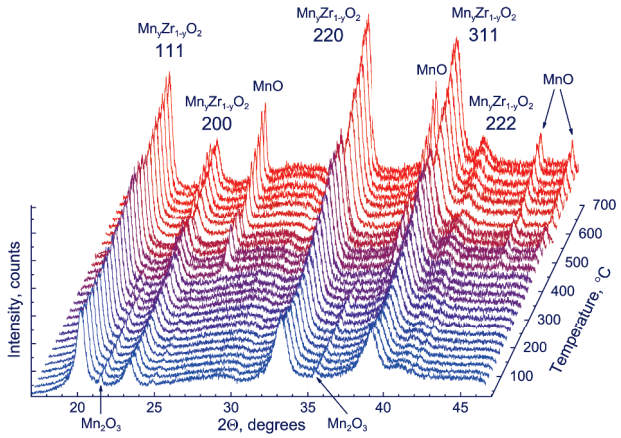


**Figure 1.** Series of diffraction patterns (λ = 1.0157 Å) recorded in situ during reduction with hydrogen in the temperature ranges from 30 to 700 °C

**Keywords:** in situ XRD, solid solution, reduction

## MS17-P11 A new micro-furnace for "in situ" high-temperature single crystal X-ray diffraction measurements

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Elastic properties reflect the nature of atomic bonding and allow the retrieval of crucial information about physical, chemical and mechanical behavior of materials. This explains the strong and increasing interest in quantifying elastic properties of materials in several scientific fields. To this aim several devices and methods have been developed so far. In particular, for high temperature devices for "in-situ" measurements, even if the small isothermal volume required for single-crystal X-ray diffraction experiments, 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.

Here we present a newly-developed H-shaped Pt-Pt/Rh resistance micro-furnace 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°. 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 diffractometer 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 measurements. The software has been modified to reduce chimney effects in the furnace and thus improve the stability by (i) restricting the χ circle movements to between -90° and +90°; (ii) optimizing the order of measurements to minimize χ circle movements (iii) imposing a waiting time after large angular movements on χ.

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. quartz and pure silicon).

**References**

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**Keywords:** X-Ray diffaraction, single crystal, high temeprature, thermal expansion