A temperature-controlled sample stage for micro-X-ray diffraction of mirabilite-containing samples from Wolf Spring, Axel Heiberg Island, Nunavut, Canada.

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Introduction: Studies of mineral precipitates from a perennial saline cold spring at Wolf Spring, Axel Heiberg Island, Nunavut, returned samples which changed mechanical properties upon adjusting to ambient laboratory conditions [1]. The minerals involved were believed to be hydrated forms of sodium sulphate (Na₂SO₄). Thenardite (the non-hydrated form) was identified via bulk XRD [1], but in order to analyse the samples in an in situ environment a temperature-controlled (TC) stage was created for use with micro-X-ray diffraction (μXRD).

Temperature-Controlled Sample Stage:

The TC stage (Fig. 1) featured the following components:
- A single stage thermoelectric (TE) module with cooling face dimensions of 40 x 40 mm and a maximum ΔT of 66°C.
- A temperature controller with a power supply module that provided 12 V DC at 12 A output.
- A sample holder consisting of two parts; a 40 x 40 x 3 mm block of copper which contacted the TE module, and a copper plate containing a 1 mm depression 25 mm in diameter to contain the sample.
- A quick-disconnect thermocouple inserted into a 1.6 mm hole bored into the sample holder bottom block.
- A 51 x 51 x 25 mm copper block with a 12.7 mm hole with threaded-in copper hose couplings on each end serving as the heat sink.

Demonstration Experiment:

The performance of the stage was demonstrated through the analysis of a synthetic sample of mirabilite (Na₂SO₄·10H₂O) held at −25 °C and then incrementally warmed to ambient laboratory temperatures during the acquisition of in situ microdiffraction data [2]. No phase changes were seen in the mirabilite (XRD spots) during the ~2 hours held at −25 °C (compare Figs. 2a & 2b) despite the μXRD enclosure being at ambient temperature and humidity. However, the amount of secondary thenardite (polycrystalline Debye rings) increased over time (Figs. 2a & 2b).

Beginning at 0 °C and continuing until thermal equilibration at ambient laboratory temperatures, mirabilite was observed to undergo a phase transformation to thenardite upon dehydration. Trace amounts of water entered the sample holder when warming above 0 °C from the melting of condensation ice crystals around the edge of the holder.

The presence of water in contact with portions of the sample caused both thenardite and mirabilite to crystallize <5 µm crystals in the sample holder during the warming from 0 to 23 °C (Fig. 2c). At ambient conditions, mirabilite completely dehydrated; only polycrystalline thenardite remained (Fig. 2d, and 3b).

Ongoing Study:

Preliminary data collected from the Wolf Spring samples show mirabilite present in a number of samples at sub-zero temperatures, which dehydrates and is not detected once the sample equilibrates to ambient laboratory conditions (Fig. 4). Certain phases—halite, thenardite, and gypsum—are present throughout the data collection at all temperatures.

References:


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