# Experimental report

01/11/2022

**Proposal:** 5-24-639 **Council:** 10/2019

**Title:** Improved sample environment for insitu neutron powder diffraction using sapphire single-crystal cells

**Research area:** Chemistry

**This proposal is a new proposal**


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**Samples:** Si
Pd
Na2Ca3Al2F14

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D20 | 3 | 2 | 25/09/2020 | 26/09/2020 |
| | | | 15/02/2021 | 16/02/2021 |


**Abstract:**

The present proposal aims for several improvements concerning the sample alignment in order to optimize beamtime usage, a new heating system for in situ powder diffraction at D20 and a validation of a newly developed gas pressure cell.
A x/y/z/omega-stage has been developed and will be tested. Further, a new heating system will be tested in order to reach higher temperature and allowing simultaneous raman spectroscopy and neutron diffraction. Additionally, Six new and untested sapphire single crystals need to be characterized by omega scans on the neutron diffractometer D20 to test their suitability for in situ neutron powder experiments. Finally, the validation of a newly developed gas pressure cell needs to be redone due to technical issues (see experimental report Test 2994).

# Improved sample environment for in situ neutron powder diffraction using sapphire single-crystal cells: sample alignment, sample heating and validation of a new gas pressure cell (experiment 5-24-639)

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## Objectives

The first aim of this study was to test an xyzω-stage sample alignment system for gas-pressure cells for *in situ* neutron powder diffraction investigations on D20. The second aim was to optimize a hot air heating system for investigations using sapphire single-crystal based gas-pressure cells [1] and validation by deuteration of palladium powder.

## Experimental Details

All experiments were conducted at D20 ($\lambda$ = 187 pm) by Dr. Thomas C. Hansen (ILL). Due to COVID-19 regulation user access was restricted. Because of this, the originally planned testing of an xyzω-stage sample alignment system was not performed and the first aim (see above) was abandoned. For the second part the deuteration part was replaced by heating of palladium powder in air due to reasons of safety and to account for the reduced experimental team.

## Results and Discussion

Palladium powder was filled into the sapphire single-crystal crucible inside a quartz vessel (Fig. 1), which was mounted on the neutron diffractometer D20. Neutron diffraction patterns were collected for 300 s each after reaching thermal equilibrium at 473 K, 773 K and 1073 K (set temperature at hot air blower). The neutron diffraction data were evaluated using the Rietveld technique. From the refined lattice parameters, the temperature could be determined to be 895 K for the highest tested heater power. Refined thermal displacement parameters were in accordance with literature values.

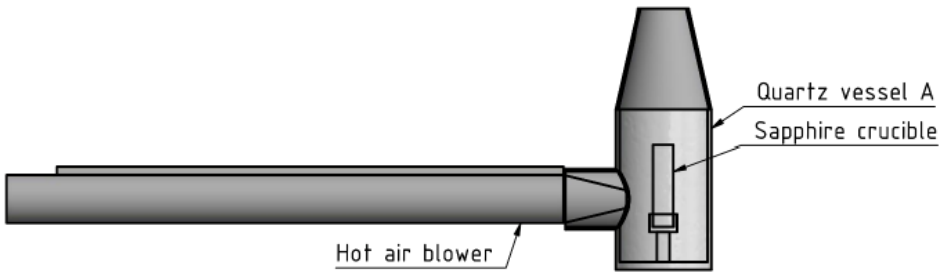


Fig. 1: Setup for heating of a sapphire crucible with hot air for *in situ* measurements on D20.

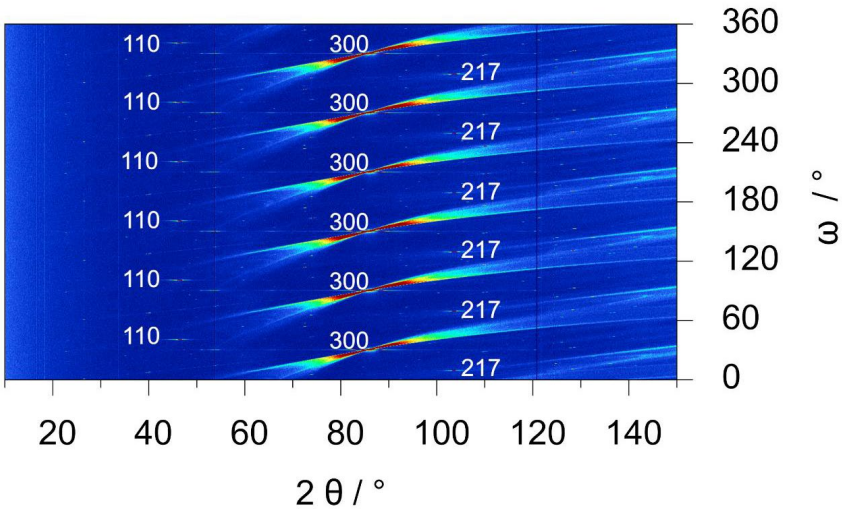


Fig. 2: ω scan of a sapphire single-crystal crucible with 1 mm wall thickness with 30 s data collection time per step and 0.5° steps.

The ω scan of a sapphire single-crystal crucible with 1 mm wall thickness (Fig. 2) clearly shows the single-crystal reflections of sapphire. By proper orientation (turning around vertical axis in Fig. 1) single crystal reflections can be avoided and a clean background is achieved.

**Literature**

[1] R. Finger, N. Kurtzemann, T. C. Hansen, H. Kohlmann, *J. Appl. Crystallogr.* **2021**, *54*, 839–846.