In situ X-ray diffraction (XRD) during ice formation on natural alkali feldspar

Johanna Seidel[1], Alexei Kiselev[1], Bärbel Krause[2], Michal Kaminski[2], David Heuser[3], Elena Petrishcheva[3], Rainer Abart[3]

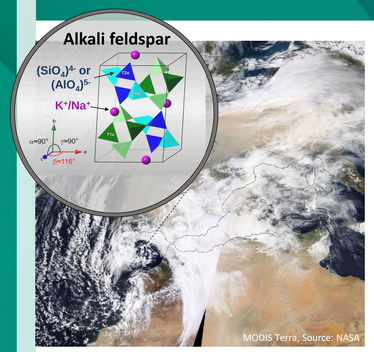
[1]Karlsruhe Institute of Technology, Institute of Meteorology and Climate Research Atmospheric Aerosol Research, Karlsruhe, Germany
[2]Karlsruhe Institute of Technology, Institute for Photon Science and Synchrotron Radiation, Karlsruhe, Germany
[3]Department of Lithospheric Research, University of Vienna, Vienna, Austria

Contact: johanna.seidel@kit.edu

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Motivation



- Alkali feldspar is a **highly ice-nucleation-active** constituent of **mineral dust aerosol** and can strongly influence precipitation formation and cloud optical properties. [1,2,3,4]
- The ice activity is attributed to its **complex perthitic microstructure**, consisting of Na- and K-rich **exsolution lamellae and defects** such as cracks, pores, and step edges. [5]
- In microscopic freezing experiments, **oriented ice growth** with an epitaxial relationship to the non-cleavable feldspar (100) plane has been observed on natural perthites, but the molecular mechanisms are still unclear.[6]

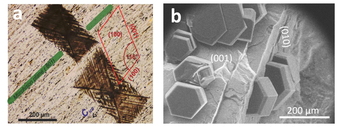
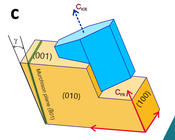
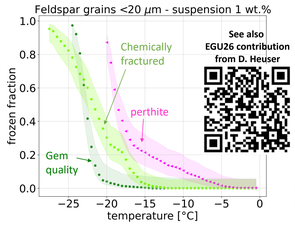


Figure 1: a) Ice crystals on a perthitic feldspar (010)-surface growing in supercooled sucrose solution with their c-axis oriented parallel to the non-cleavable feldspar (100) plane. [from 7] b) Secondary electron (SE) image of oriented ice crystals in ESEM on a naturally cleaved perthitic feldspar surface. c) Schematics of the epitaxial relation [from 7].



Better understanding of the fundamental ice nucleation process on the molecular level will help to clarify the importance of alkali feldspar for atmospheric processes, improve cloud models and might also help to identify other highly ice-active materials

Methods & Experiment



1) IR coldstage

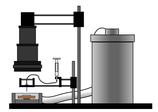


Figure 2: Comparison of the freezing activity of different alkali feldspar samples using an infrared coldstage setup

I. Sample pre-characterization

2) Lab XRD



Figure 3: Photograph of the laboratory X-ray diffractometer setup used to characterize the feldspar orientation

3) Environmental scanning electron microscopy (ESEM)

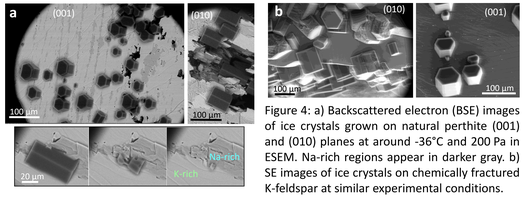
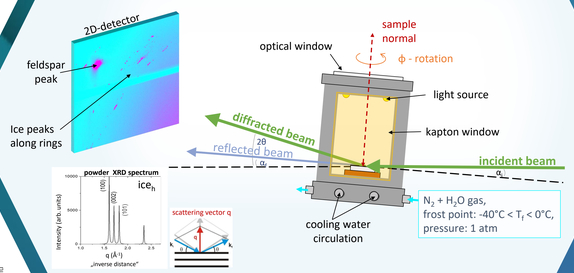


Figure 4: a) Backscattered electron (BSE) images of ice crystals grown on natural perthite (001) and (010) planes at around -36°C and 200 Pa in ESEM. Na-rich regions appear in darker gray. b) SE images of ice crystals on chemically fractured K-feldspar at similar experimental conditions.

- **Study the ice crystal formation on cleavage planes of natural and chemically modified gem quality and natural perthite alkali feldspar**
- **Gain high resolution information about crystallographic orientations and molecular phases using synchrotron radiation with a new in situ X-ray diffraction setup**

Real-time monitoring using grazing incidence XRD



II. Synchrotron experiments

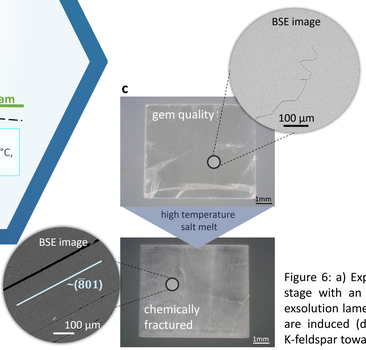
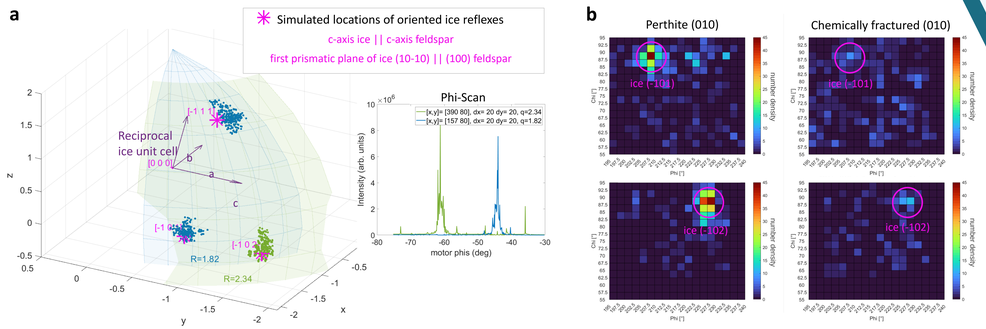


Figure 6: a) Experimental setup at DESY (Deutsches Elektronen-Synchrotron) [8]. b) Feldspar sample on our cooling stage with an example ESEM image of a naturally cleaved perthite surface used for the experiments. Na-rich exsolution lamellae (dark gray) extend along the K-rich feldspar framework (light gray). c) Cracks subparallel to (-801) are induced (direction sub-perpendicular to the direction of maximum tensile stress) by shifting a gem quality K-feldspar towards more Na-content by cation exchange in NaCl salt melt at 900°C for 4 days (method described in [9])

Preliminary results & conclusions



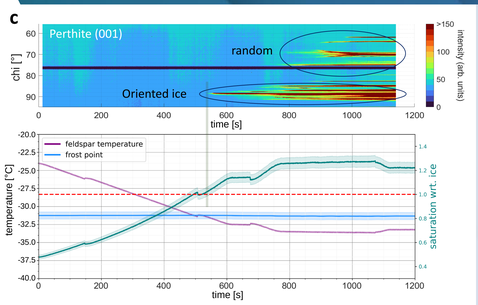


Figure 5: a) 3D reciprocal space map showing clusters of oriented ice crystal peaks during an experiment with a (010) perthite surface. A 2D-phi-scan of the integrated XRD signal within a 20x20 pixel^2 window on the detector is shown in the right panel. b) Comparison of the number density of ice crystal peaks for a natural perthite and a chemically fractured K-feldspar. (c) Time series of the feldspar temperature, frost point and saturation wrt. ice (lower panel) during an XRD-scan with fixed detector position. The upper panel shows the integrated XRD-signal along chi.

- **In situ XRD confirms the preferential orientation of vapor grown ice crystals on natural perthitic alkali feldspar under atmospheric pressure for the first time**
- **Chemical fracturing of gem quality K-feldspar leads to a small fraction of oriented ice but can so far not mimic the high degree of ice orientation on perthite**
- **After reaching the ice onset conditions, oriented ice crystals nucleate first**

Outlook

Final measurement campaign at DESY, PETRA III, beamline P08 planned this June 2026 to

- complement the dataset with experiments on gem quality reference samples
- increase the statistics on the average ice crystal orientation with respect to the crystallographic orientations of feldspar

Acknowledgements

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