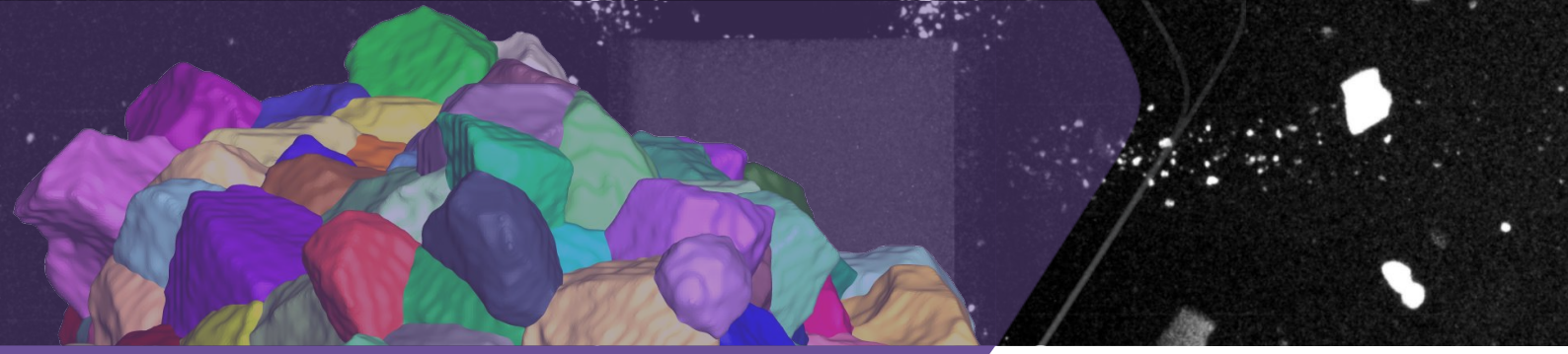

GrainMapper3D *Spotlight*

Ice

Sample Description

- Frozen tap water
- Crystal system: hexagonal (P 6 3 /mmc)
- Container: Carbon crucible, 0.1 mL volume
- Sample dimensions: 4 mm diameter, 6 mm height
- Sample conditions:

  Frozen is-situ and kept at -18 °C during experiment
- Cooling device:

  Deben CT160 in-situ Peltier-based cooling stage

3D microstructure of ice

Being able to characterize the 3D microstructure of ice is critical for understanding the physical properties and behavior of ice in both natural and engineered systems, advancing knowledge in fields ranging from glaciology to cryopreservation.
Here, an X-ray imaging experiment on a model system comprising frozen tap water with grain sizes ranging from 0.1 to 2 mm to show that lab-based DCT can be used to perform high-resolution 3D imaging of ice. Both crystal orientations, grain boundaries, porosities, and impurities are imaged in the ice, all of which are essential parameters for eventually examining dynamic processes like recrystallization, impurity migration and mechanical deformation.

In-situ cooling stage – CT160

The Deben CT160 in-situ cooling stage is a commercial product that allows for real-time analysis of material properties under controlled temperature and load conditions for X-ray microscopy. The CT160 system features a peltier-based cooling stage that quickly adjusts sample temperatures from +160°C to -20°C and supports 360° rotation for full imaging.

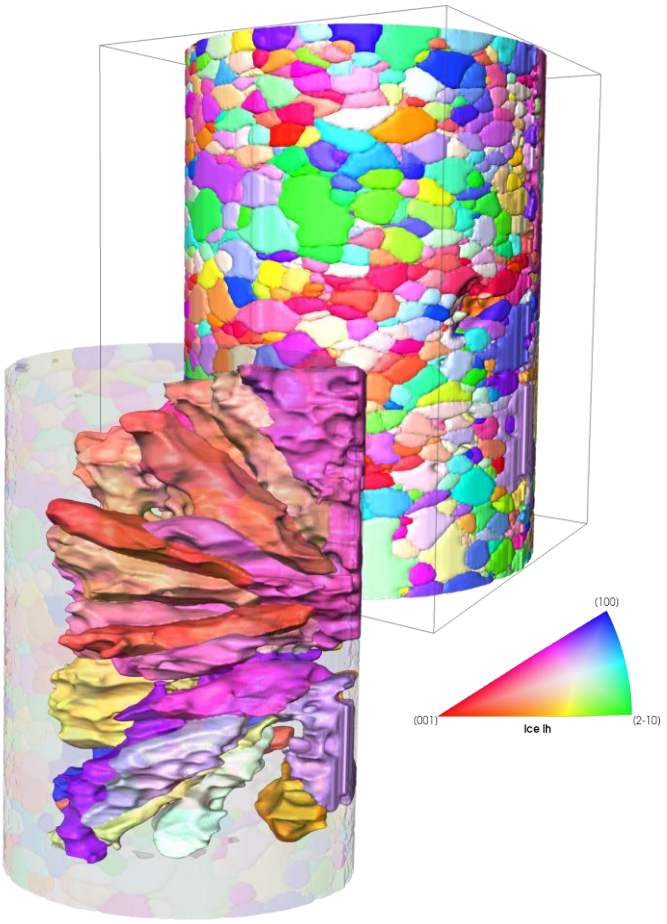

Figure: Microstructural analysis of ice revealing more than 3000 grains within a few hours. The image illustrates smaller, spherical grains on the periphery and a central region dominated by larger, elongated grains.

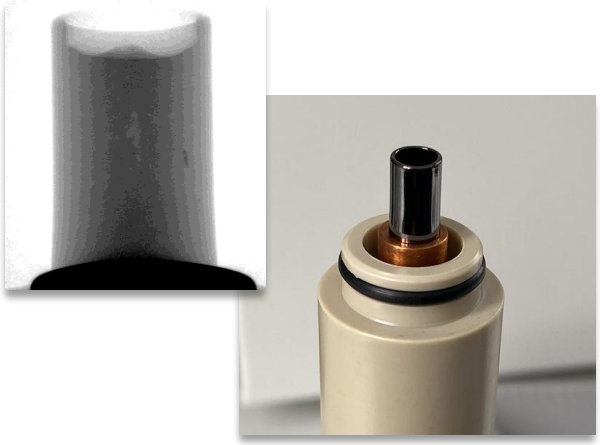

Figure Projection from absorption scan of frozen tap water with air bubbles and impurities in carbon crucible on the CT160 in-situ cooling stage (left) and photograph of experimental setup (right).

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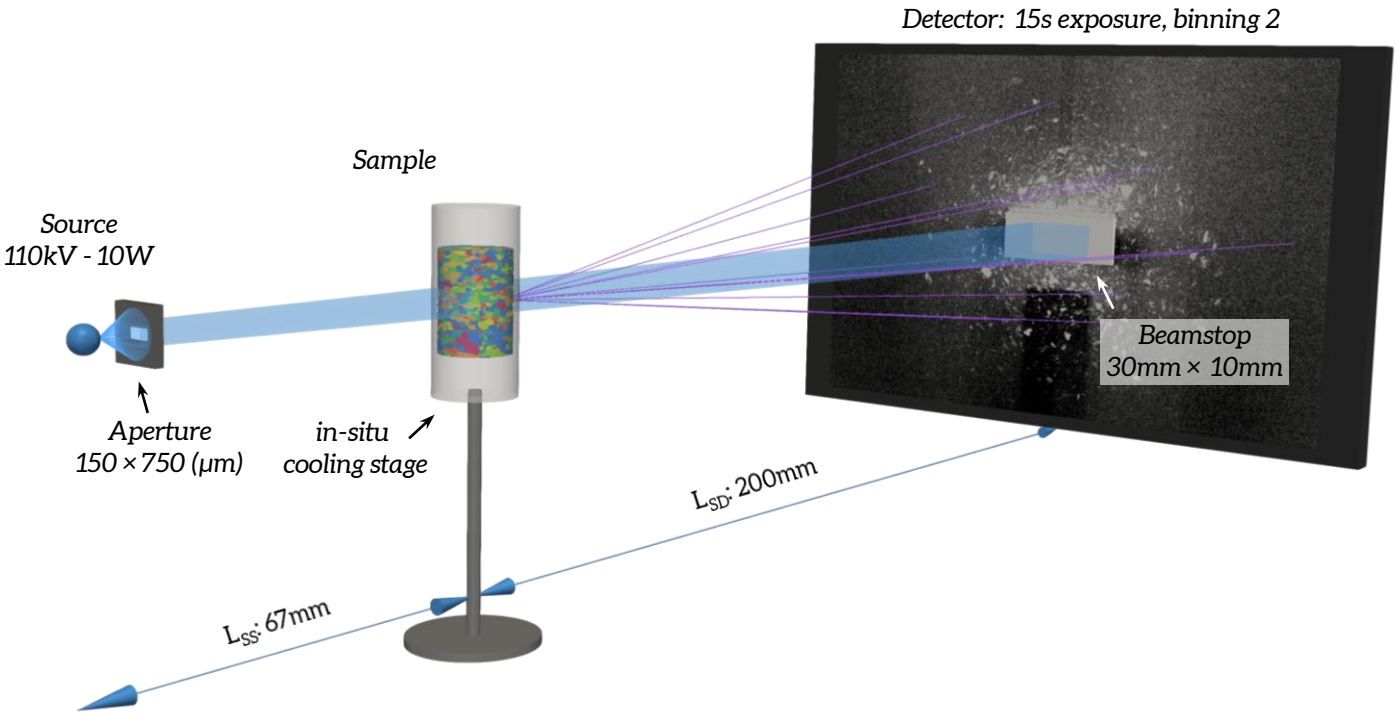

Figure: Schematic illustration of the setup for diffraction contrast tomography data acquisition of frozen tap water in M95 carbon crucible on 160CT in-situ cooling stage at -18°C. Key acquisition parameters are marked.

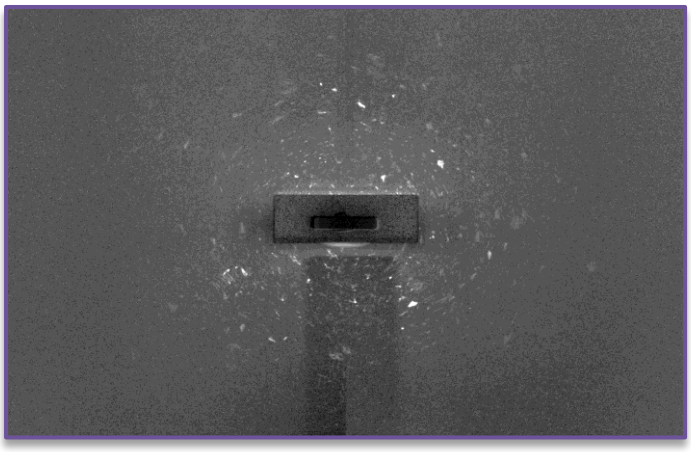

Figure (left): Example diffraction contrast projection at a certain rotation angle. The sample was scanned with projection geometry, with the shape of the diffraction spots representing the shape of the grains.

Data Acquisition Parameters

System: ZEISS Xradia 520 Versa with LabDCT Pro

Absorption Contrast Tomography

- *Voltage*: 50 kV
- *Power*: 4 W
- *Objective*: 0.4X
- *Source – Sample distance*: 22 mm
- *Sample – Detector distance*: 146 mm
- *Exposure*: 5s / binning 2
- *Number of projections*: 801
- *Voxel size*: 9.05 µm

Diffraction Contrast Tomography

- *Data acquisition mode:* Helical Phyllotaxis
- *Aperture*: DCT 150 × 750 (µm × µm)
- *Voltage*: 110 kV
- *Power*: 10 W
- *Objective*: Flat Panel Detector
- *Source – Sample distance*: 67 mm
- *Sample – Detector distance*: 200 mm
- *Exposure*: 15s / binning 2
- *Number of projections*: 978
- *3D Grain Map voxel size*: 18.10 µm

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