ESRF	**Experiment title:** Time-resolved X-ray tomography of sea ice freezing and percolation	**Experiment number**: ES-1028
Beamline: ID19	**Date of experiment**: from: 07.11.2012 to: 10.11.2012	**Date of report**: 20.02.2015 *Received at ESRF:*
Shifts: 9	**Local contact(s)**: Alexander Rack	

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Report:

Sea ice is a porous medium that covers on average 5-7 percent of the earth's oceans. Its physical properties depend on its microstructure, characterised by the presence of liquid brine (concentrated seawater), solid salts and air in pore networks and inclusions. For non-destructive 3-d observations of sea ice microstructure, X-ray tomography (XRT) is the primary candidate. To date synchrotron XRT has been applied to image snow (e.g., 1) and atmospheric ice particles (2) . Tomographic imaging of brine-ice networks of sea ice is more challenging than air-snow: Due to the small absorption contrast of ice and water, conventional tomography based on transmission of X-rays cannot properly distinguish between brine and ice phases. To circumvent this problem, studies have been performed with ice grown from NaCl solutions doped by CsCl (3). However, to what degree such ice represents natural sea ice is unclear.

To perform observations of the internal freezing and melting process of pores in natural sea ice thus requires phase-sensitive imaging techniques, that are available at the ID19 beamline (4). However, as sea ice is a multi-phase medium that rapidly changes its morphology when removed from in situ conditions, sampling transport and storage are other challenging tasks. To obtain images of sea ice microstructure that resemble its in situ state, we thus have recently applied synchrotron-based XRT to image natural sea ice that has been centrifuged after sampling (5,6). This approach is suited to image pore networks from high contrast of ice and air that has replaced the brine by centrifugation.

The goal of the present experiment was to to evaluate the applicability of phase-sensitive 3-d in situ microtomographic imaging of sea ice and its internal freezing/melting during cooling/warming. Based on our previous results (5,6) we performed three kinds of experiments:

I. XRT imaging of „dry“ centrifuged natural sea ice samples from the Arctic as performed in previous work

II. Seawater brine/concentrated seawater was reinjected into the centrifuged samples (submerging the sample). Images are taken after submersion during cooling and warming and a time period 1 to 3 hours.

III. Upward freezing experiments with liquid seawater were performed to obtain 4-d time-lapse tomography series of the initial freezing process.

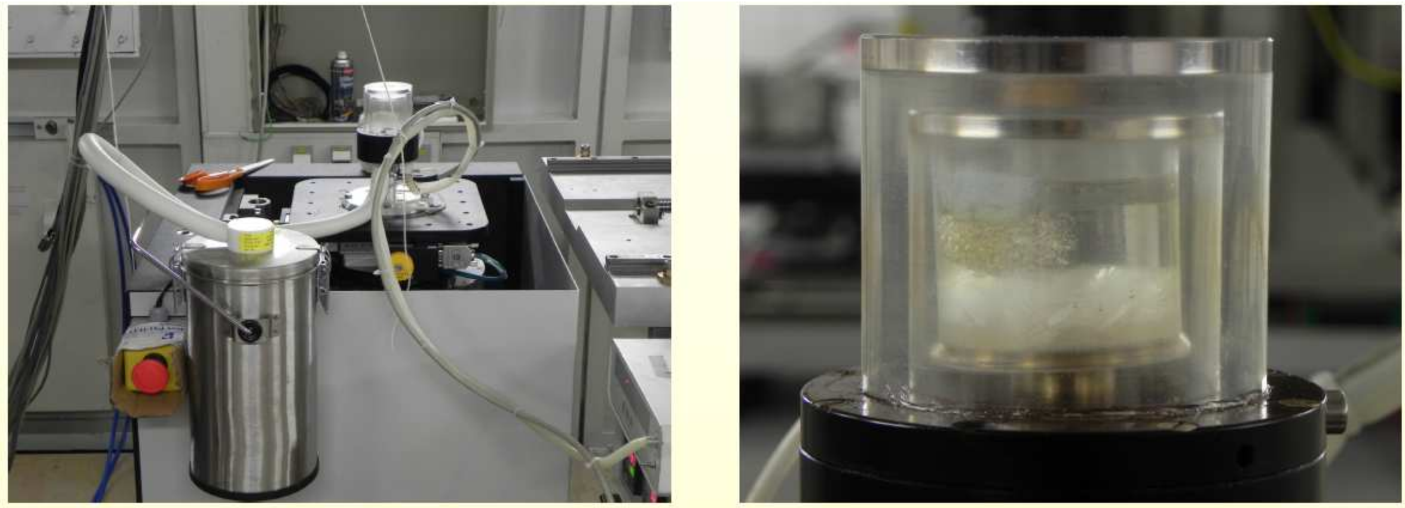

Fig.1: Nitrogene-based cooling setup with cooling from below. Right: Upward ice growth into the cell filled with natural seawater. The inner cell is 4.5 cm in diameter.

Most experiments were performed with 360 degree offset mode to obtain a large FOV of 6 cm, and images with voxel size of 30 micron. Figure 1 show the in-house cooling stage used in the experiments. For experiments with submerged samples the sample was fixed to the wall with styrofoam, then a dry image was obtained, before the sample holder was flooded with brine. In the following we only show results from submerged sample runs II and III, yet note that the „dry“ runs gave images as in our earlier work (5,6).

(II) Centrifuged natural sea ice

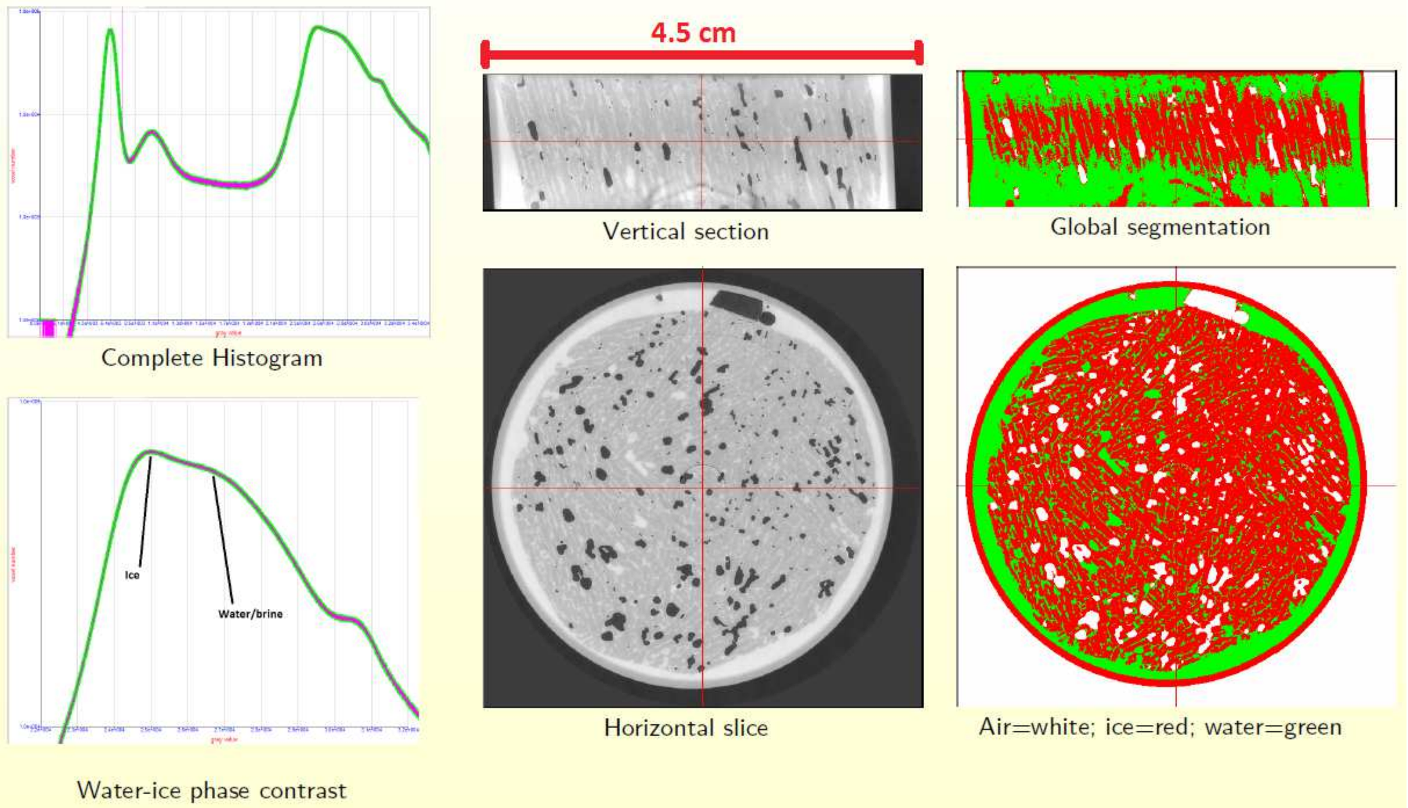

Fig.2: Histogram (left), raw image (middle) and segmented image (right) of centrifuged natural sea ice that had been centrifuged. Upper images are horizontal sections, lower are vertical slices. Raw – dark:air, grey: ice, bright: seawater brine. Semented - red: ice, green: seawater, white: air.

Figure 2 shown that we can clearly distinguish air (dark), ice(grey) and brine (bright). A minor problem was that (seen in the upper horizontal images) the sample was not illuminated vertically homogeneously (for some unkown reason we eventually see the vertical profile of the beam), which precludes global segmentation as we show in the images. The main problem with these experiments was that, when the brine displaces air upon submerging the sample, only a fraction (roughly half) of the centrifuged pore spaces refills with brine. We attempted to increase the brine absorption of the sample (slow and rapid pouring, etc.), yet without substantial changes. Hence, while phase-contrast imaging turned out to be successful, the refilling of the samples did not. We thus conclude that the centrifuging method cannot be applied to analyse the internal freezing and melting of natural sea ice, as we are not able to reinject the brine into the centrifuged samples. To make further progress here one needs to image uncentrifuged samples, for which the isothermal transport from the high lattitude field to the synchrotron is a non-trivial challenge.

III) In situ freezing

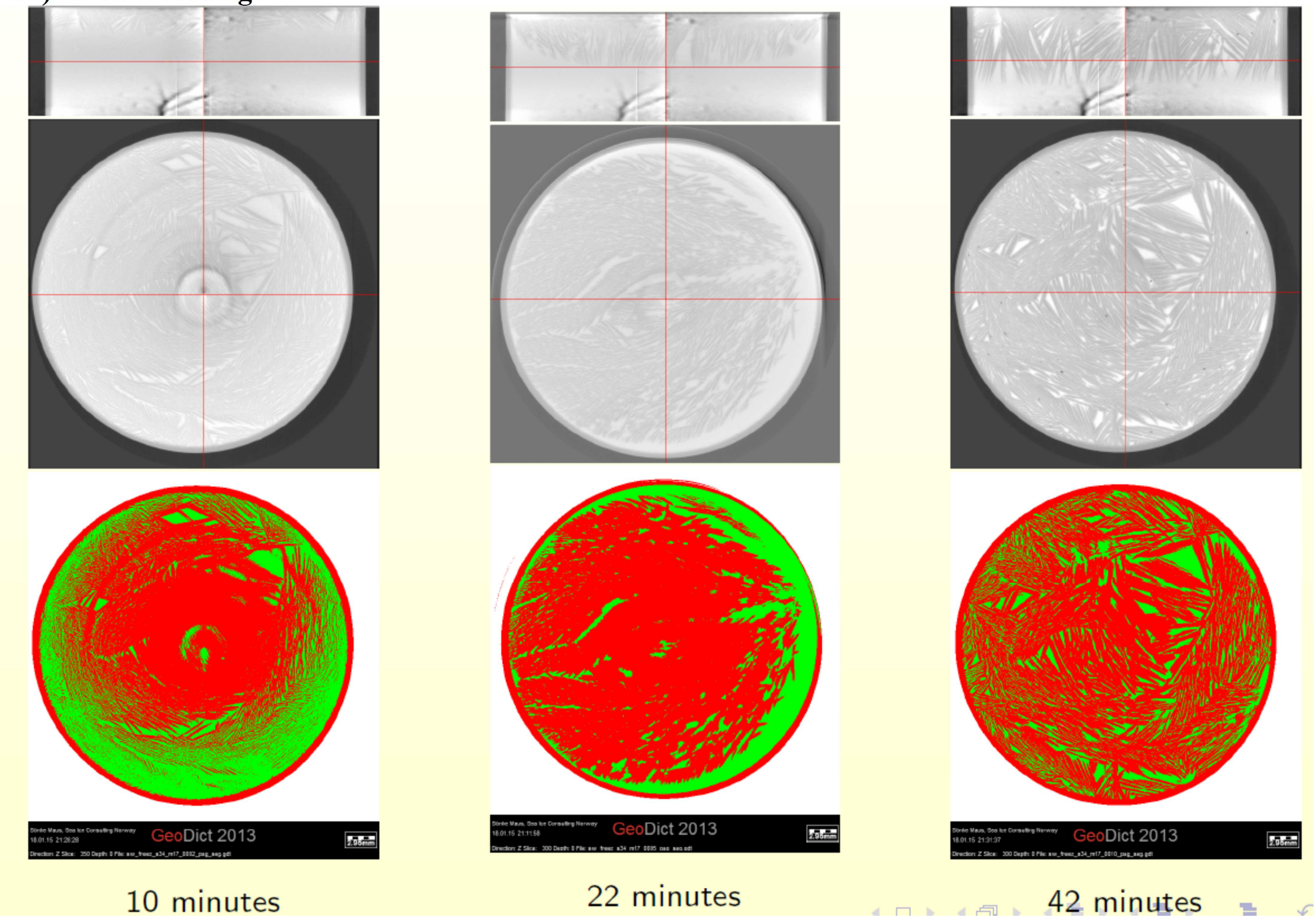

Fig. 3. Time lapse image series of upward freezing (note that up and down are inverted in the figure) of seawater. Upper images are raw horizontal sections, middle raw vertical slices, and lower images segmented vertical slices (green: seawater brine, red: ice).

The results of the upward freezing experiments were very promising and validate the applicability of phase-contrast imaging to study the freezing prcoess of seawater. The lammelar structure observed during all experiments resembles what is observed at the freezing interface of natural sea ice (5,7). However, natural sea ice is normally freezing downward under the presence of solutal convection, which is expected to influence the evolution of the pore structure, especially in the interior past the iterface where convective desalination creates brine channels. In future experiments one of the main goals will be to construct a cooling setup that allows for downward freezing.

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