ESRF	**Experiment title:** A Double-scale Approach for 3D Characterization of Ice Particles in Low-temperature Aviation Fuels	**Experiment number**: MA 4610
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Shifts: 12	**Local contact(s)**: Marta MAJKUT	

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Report:

The objective of the experiment was to take advantage of the ID19 beamline to significantly improve the resolution and quality of ice particle images previously obtained in 3SR. In particular, the plan was to use a double-scale approach: observing the ice particles at 6.5 µm and then zooming at 0.65 µm at specific locations where additional details are required.

Thus, two Hasselblad optics were mounted with x1 magnification for the Low Resolution (LR) and 10x magnification for High Resolution (HR). They were both equipped with a PCO EDGE camera. The LR optic was mounted on the fixed bench at the back of the hutch and the HR optic was mounted on the MR bench. The cold stage (CellStat) was installed on the MR rotation stage, leading to a sample to detector distance of 2.2 m for LR and down to about 5 cm for HR.

Due to the X-ray beam size, the LR field of view was: (width x height) 16.6x8.5 mm^2 (2560x1300 px^2) while the full field of view was used for the HR scans: 1.7x1.4 mm^2 (2560x2160 px^2).

Three macros were written for an easy switch between the LR and the HR, the central point of the imaged volumes was followed when (un)zooming and moving within the samples directly using the pixel coordinates. It was then possible to switch between the resolutions in less than 2 minutes.

The u13a and u176c insertion devices were used for X-ray beam energy of 26 and 19 keV. In both cases, the X-ray absorption of the sample was very low and projections showed almost only interference fringes.

The parameters for LR and HR scans were the same with 4000 projections on a full turn with no half acquisition (centered sample). The main difference was the gap of the insertion device that was decreased for the HR scans to keep a comparable beam transmission through the sample (ex: u13a with gap 24 mm and exposure time (ct) 0.04 s for LR, and gap 14 mm with ct 0.05 s for HR). The total scan time was about 4 minutes for LR and 5 minutes for HR.

The complete setup of the beamline and the CellStat installation took about two shifts.

Figures 1 and 2 show the middle slices of the reconstructed scans in LR and HR with optimised X-ray beam energy (19 keV) and distance for HR (about 5 cm). The sample is a suspension of ice particles in paraffin oil kept at -35ºC during the acquisitions (1 to 2 hours). The particles didn't "move" during the acquisitions, contrary to some of the air bubbles (not shown here).

Figure 1 (a) shows that the remaining phase field in the reconstructed images is quite important at the interface between:

- the sample holder (PMMA) and the paraffin oil,
- the ice particles and their pores.

Figure 1 (b) and (c) show that using delta/beta = 25 for the phase contrast filtering gives rather good results for the segmentation of ice particles in paraffin oil in LR scans.

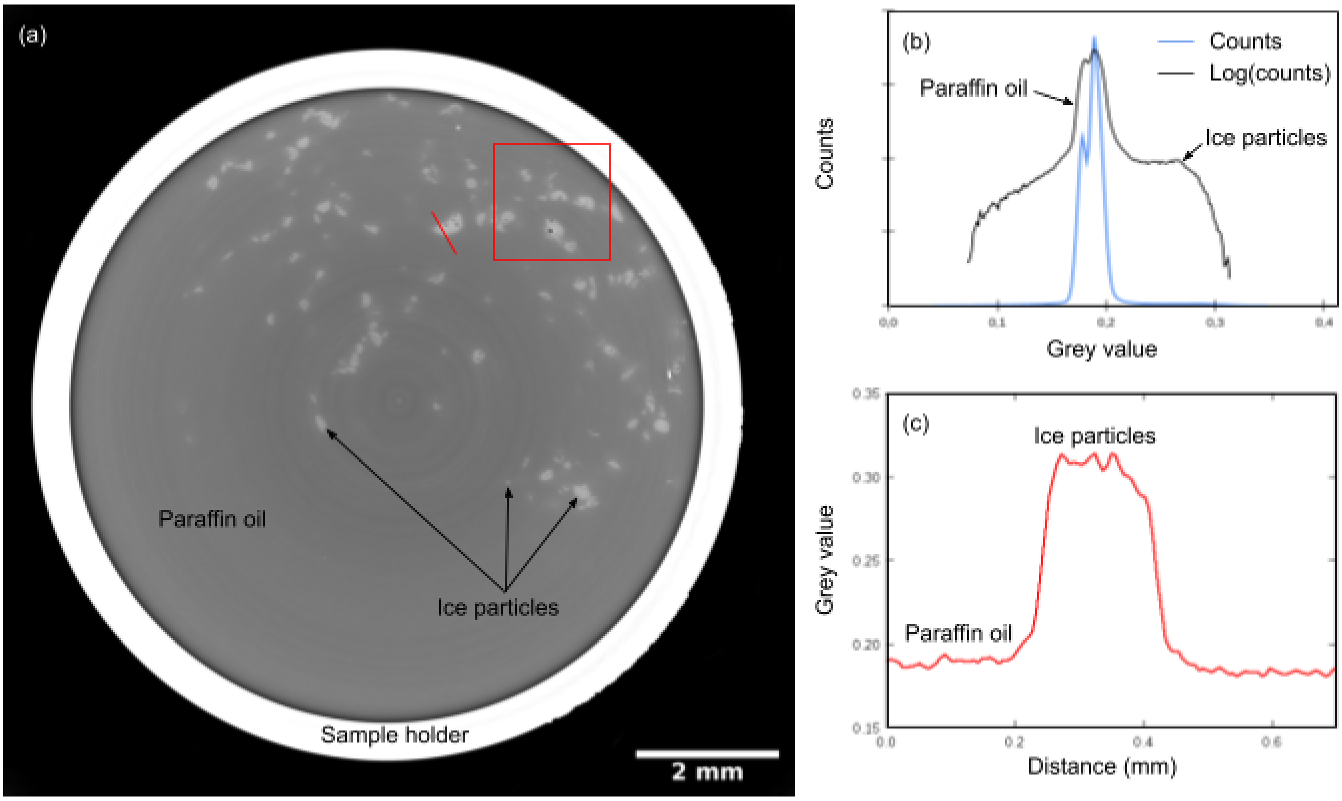

Figure 1: LR scans of ice particles in paraffin oil. (a) Reconstructed middle slice with delta/beta = 25, brightness and contrast are enhanced to fit the sample values. (b) Histogram of the grey levels inside the sample holder. (c) Grey level profile along the red line in (a).

Figures 2 (a) and (b) show the middle slice of the reconstructed scans in HR after zooming in the red ROI (see Figure 1 (a)). Here again, the remaining phase contrast at the mentioned interfaces is important with a delta/beta = 1500 optimised for the ice particles in the paraffin oil. However the local tomography and/or cupping artefact are limited.

The strong ring artefacts need to be corrected, the tests with Paul Tafforeau's scripts give rather good results. Further investigations are presently carried out to correct these artefacts (the double flatfield doesn't give good results, nabu ring correction gives good results, still need to try moving flatfield).

After image segmentation, the ice particle size distributions at LR and HR will be processed and compared.

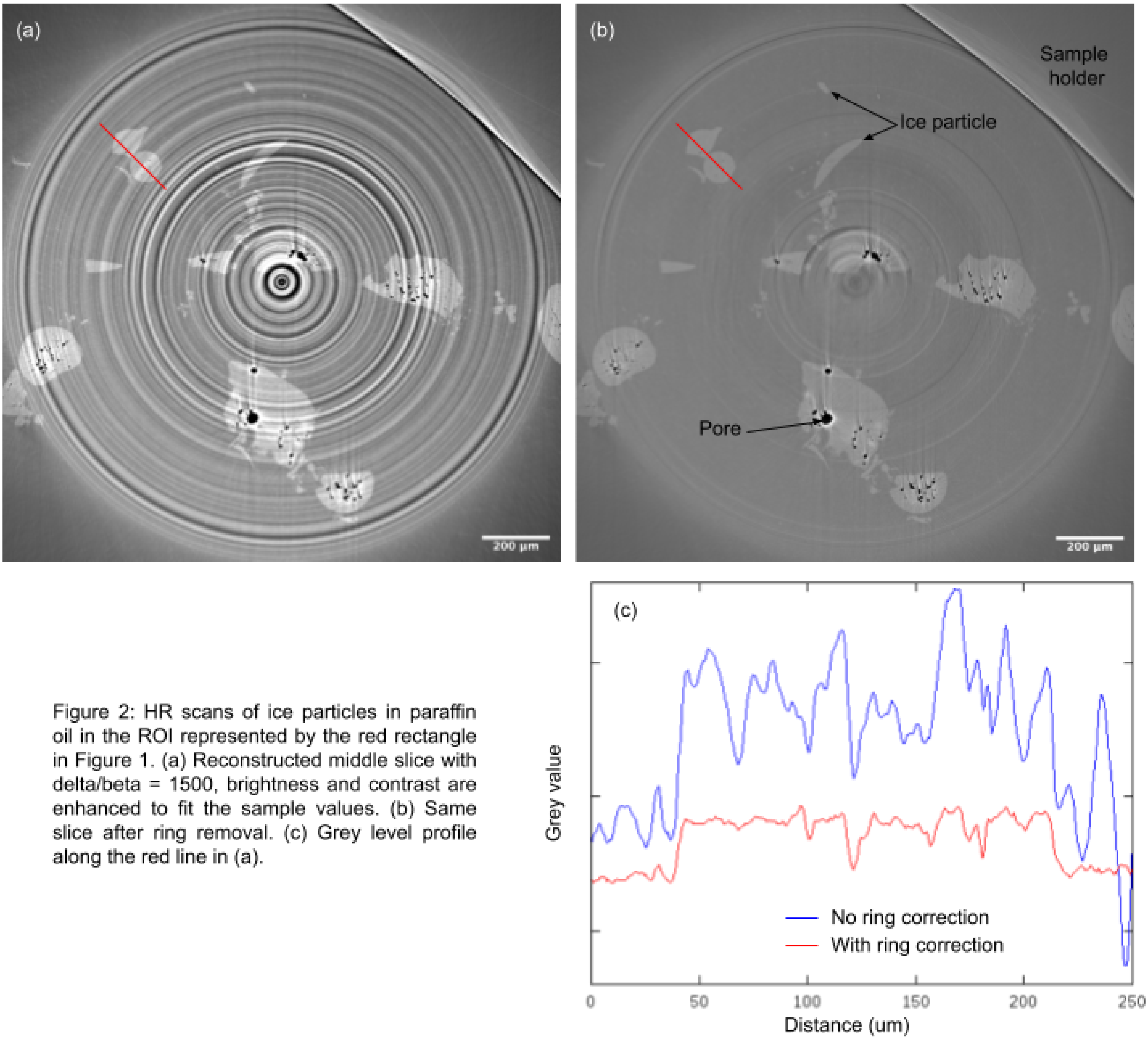

Figure 2: HR scans of ice particles in paraffin oil in the ROI represented by the red rectangle in Figure 1. (a) Reconstructed middle slice with delta/beta = 1500, brightness and contrast are enhanced to fit the sample values. (b) Same slice after ring removal. (c) Grey level profile along the red line in (a).

As a conclusion, the experimental setup at ID19 allowed us to scan samples of ice in paraffin oil at temperatures below -35°C at LR and to easily zoom in ROIs at HR. The principal ameliorations might be to stay at relatively low energy (19 keV) and:

1) to drastically decrease the LR sample to detector distance (while being able to change it during the experiment to easily optimise the propagation distances);
2) to decrease the HR sample to detector distance, which was limited by the MR rotation stage and bench configuration, up to the limits of CellStat.

Additional improvements might consist in decreasing the external diameter of the cryogenic cell (but to the detriment of sample size), allowing an even closer detector, offering thus optimised propagation distances.

Based on the difficulty to obtain very high quality images, only a limited number of samples of ice particles preserved in paraffin oil were finally scanned. Remaining beam time was devoted to test other impregnation products and sample types: snow in paraffin oil, snow in dodecane, snow in chloronaphthalene, snow in diethyl-ortho-phthalate, ice in silicone oil, snow with mineral dust particles. Some of these tests gave good quality images and interesting results. Images are now under process and may give rise to publications.