# Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF**.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

***Reports supporting requests for additional beam time***

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

## Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

## Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

| | **Experiment title:** Monitoring Laser-Induced Ice Nucleation II | **Experiment number**: SC-4798 (Proposal: P78988) |
|---|---|---|
| **Beamline**: ID09 | **Date of experiment**: from: May 31, 2018 to: June 06, 2018 | **Date of report**: March 02, 2020 |
| **Shifts:** 18 | **Local contact(s)**: Matteo Levantino | *Received at ESRF:* March 02, 2020 |

**Names and affiliations of applicants** (* indicates experimentalists):
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**Report:** *Experimental setup* – Details in the previous report (SC4531).

*Results* – We succeeded to induce for the first time ***homogeneous*** nucleation of ice within the bulk water drop in the volume element defined by the spatial intersection of two orthogonal laser beams with polarizations that are orthogonal and parallel within the horizontal plane and observed its diffraction signal with *Pink* X-ray beam as shown in Fig. 1. In these X-ray images sets of symmetry related diffraction peaks were identified. This allowed us (i) to determine the absolute orientation of the nuclei in this early stage of growth; (ii) to identify an unambiguous correlation between the directions and symmetry of the laser fields and that of the H-bonding arrays of the induced ice crystals, as shown in Fig. 2; (iii) to identify from the evolution of the symmetry-related diffraction peaks a particle attachment mchanism during the crystal growth – the broad peaks became narrower and more intense, a process that was accompanied by peak disappearance (not shown due to space restriction). Finally, the freezing temperatures under the pulsed laser illumination were significantly higher than in dark conditions, upto 12.4 ºC, depending on the laser beam configuration and intensity. A detailed description of the experiment, results, analysis and discussion appears in the manuscript we recently submitted to *J. Chem. Phys*.

We also probed the laser-induced ice nucleation with the multi-layer (ML) pulsed X-ray beam. However, the X-ray images (not shown) reaveled diffraction peaks that were much intense and spread over a smaller area on the detctor image relative to the images acquired with the Pink X-ray beam - indicating that frezing was rather detected at a later stage. Moreover, by contrast to the pink-beam images, here we could not determine whether the diffraction signal emerged from the volume element defined by the intersecting laser beams.

We faced several technical problems that effectively reduced our beamtime: adjusting the mirrors within the laser box; small range (12 mm) of motorized stages; and beamtime had fallen.

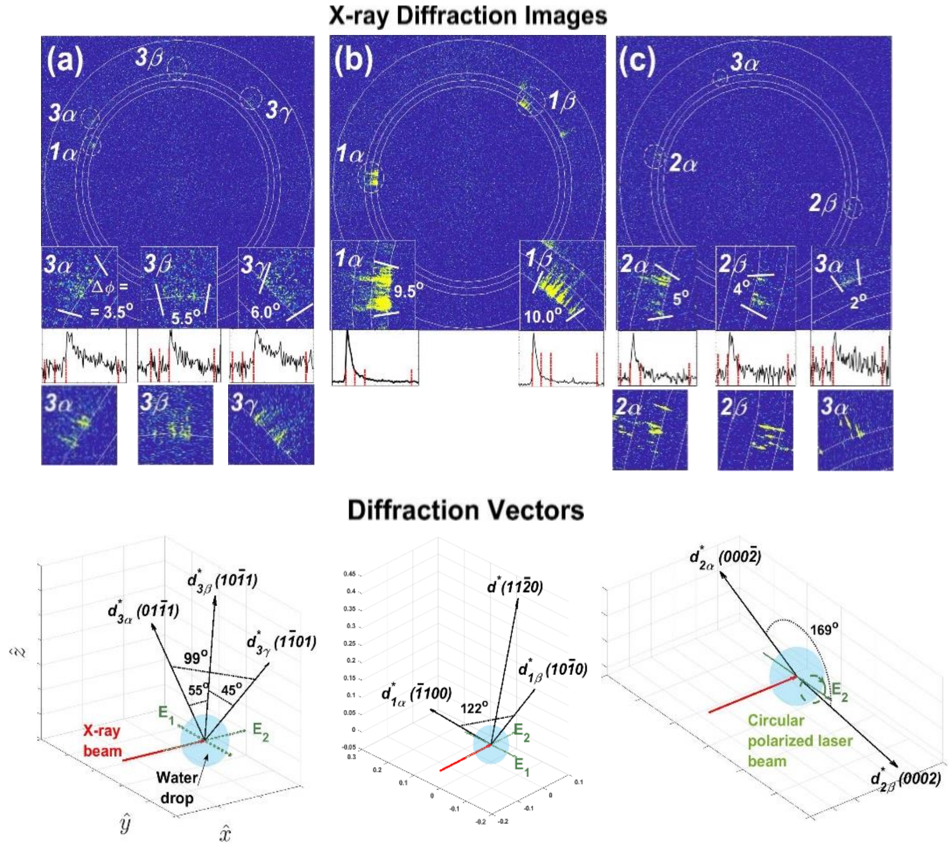


Figure 1. Three examples of first appearance of X-ray diffraction peaks of $I_H$ from a single 100 ps X-ray pulse impinging on a water drop, delayed 100 ns after laser illumination.

(**a**, **b**) Diffraction after illumination of the supercooled water drop by a linearly polarized double laser pulse (see Fig. 1a). (**c**) Diffraction after illumination by a circularly polarized pulse from the side of the water drop. To substantiate presence of diffraction peaks magnified images thereof are displayed in insets above their azimuth-integrated radial profiles; whereas below them, magnified diffraction peaks appear more intense being measured 700 ms after their first appearance. In the insets, the azimuthal angular spread values $\Delta\phi$ are indicated. The diffraction peaks *1* to 3 in each panel belong to an assemblage of similarly oriented ice crystals; symbols $\alpha$, $\beta$, $\gamma$ attached to peaks with the same number denote they are symmetry-related; the orientations of their diffraction vectors are displayed in the bottom row as well as the ***E***-laser fields and incident X-ray beam.

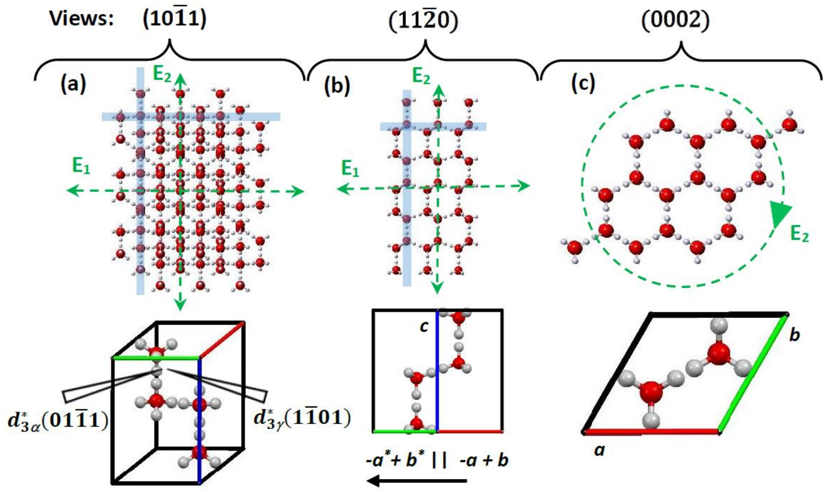


Figure 2. The oriented ice crystal structures and corresponding laser electric fields.
The super- and unit-cell structures of the oriented ice crystals, viewed perpendicular to the corresponding laser-induced *(hkil)* planes. These planes are approximately parallel to that defined by the linearly polarized $E_1$ and $E_2$ vectors (parallel to the glass slide) for panels (a) and (b), and perpendicular to the circularly polarized $E_2$ beam for panel (c). The water units appear as tetrahedra since the crystal structure of $I_H$ is proton-disordered[16] (see supplementary material Sec. S1). The pale-blue bars in (a) and (b) indicate directions of primary H-bonding arrays that are parallel to $E_1$ and $E_2$.