Report on MA-3234 experiment “3D real-time and in situ characterisation of the forming of cellulose nanofibril foams by ice-templating”

Allocated shifts: 9 shifts. This report refers to the session from 16 Nov. 2016 to 18 Nov. 2016 (6 shifts).

Objective of the proposal

Cellulose nanofibril (NFC) foams can be fabricated from aqueous colloidal NFC suspensions by ice templating followed by freeze drying, i.e., by freezing NFC suspensions and sublimating the formed ice crystals. In this project, we aim (i) to establish the influence of cooling rates and sublimation conditions on the architecture and the deformation of cellulose nanofibril (NFC) foam cells. Current understanding of ice templating is based on 2D visualization of NFC foam structures. The water solidification of NFC suspensions, and the resulting phase separation that occur during ice crystal growth, has never been studied using 3D in situ and real time imaging techniques. Furthermore, the inherited structure of foams after freeze drying has also never been studied using 3D images. Thus, the proposal consists of acquiring 3D images of (i) the NFC suspensions during in situ quenching at several temperatures, and (ii) the foam cellular structures during in situ ice-sublimation experiments.

Summary of the work done during the session from 16 Nov. 2016 to 18 Nov. 2016

During this session, several in situ solidification experiments were performed using various NFC suspensions and processing conditions. For the first time for this kind of materials, we could follow in real time the solidification front, the NFC cell formation and consolidation during freezing experiments. Several types of cellulose nanofibrils (NFC) were used, allowing the effect of various aspect ratios, various surface charge density to be studied. We also studied the effect of NFC content on the solidification of suspensions (Fig. 1).

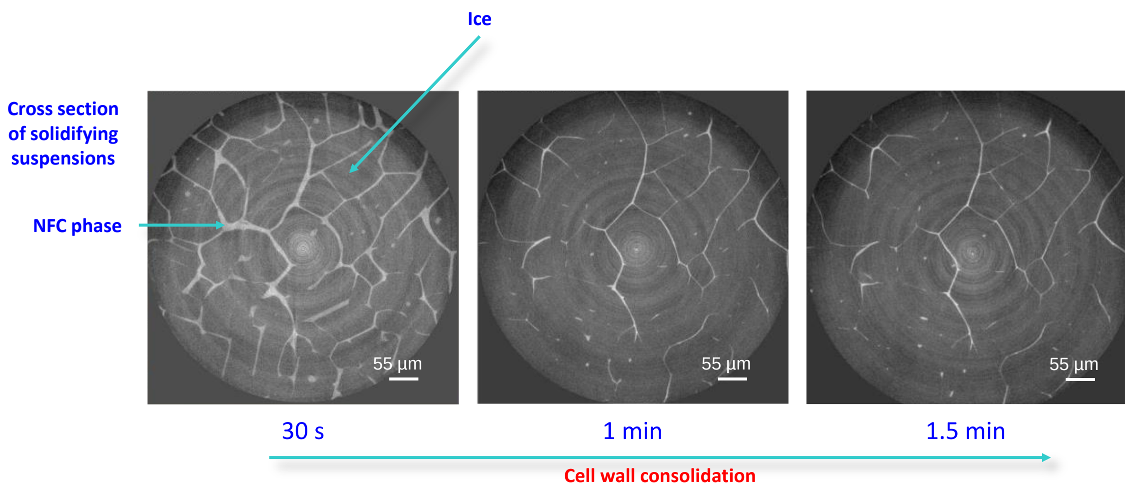

Fig. 1 – Horizontal cross sections of a solidifying NFC suspension at various times, showing the consolidation of the cell wall of the forming NFC foam.

To perform these experiments, we designed a new experimental setup which enabled a directional control of the freezing of the suspensions and the monitoring of freezing temperature (Fig. 2). These experiments corresponded to the schedule that was described in the proposal. We consider that these experiments are extremely successful with an exceptional quality of the acquired images. Currently, 3D images are being reconstructed and analysed to extract several microstructural descriptors to characterize the growth of ice crystals and the associated mechanisms and kinetics of cell wall formation. During the second shift, we plan to study the sublimation of ice crystals and the resulting architecture of NFC foams.

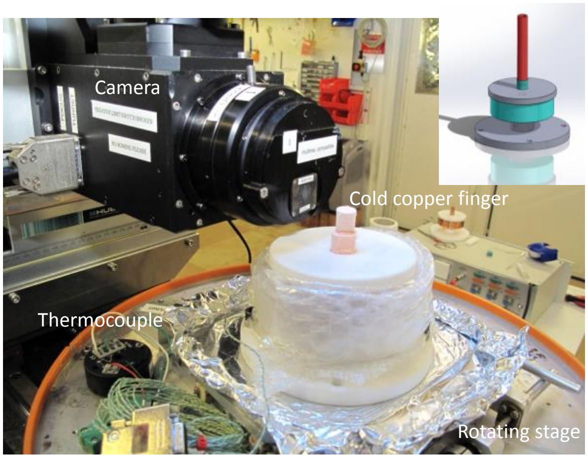

Fig. 2 – View of the solidification device installed on the rotation stage of the high resolution of the ID19 ESRF beamline. Inset: schematic view of the device with the tube (red) containing the NFC suspension.

Communication

Florian Martoïa, Thibaud Cochereau, Pierre Dumont, Laurent Orgéas, Sabine Rolland du Roscoat, Elodie Boller, Maxime Terrien, Naceur Belgacem, Links between ice-templating conditions, microstructures and mechanical properties of cellulose nanofibril foams, 5th International Conference on Multifunctional, Hybrid and Nanomaterials, Lisbon, Portugal, 6-10 March 2017.