

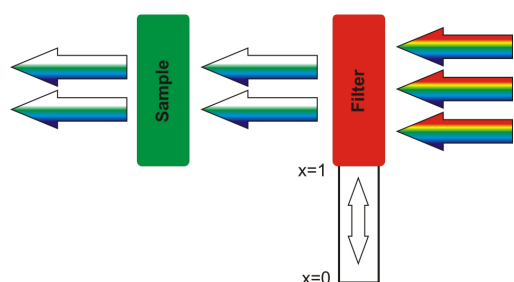
## Distinction of liquid water and ice based on dual spectrum neutron imaging

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Polymer electrolyte fuel cells (PEFCs) have attracted much attention during the last decade, because of extremely low emissions at very high power densities. However, before its commercialization, PEFC have to prove the durability during operation at ambient conditions, such as subfreezing temperatures. As the freezing mechanism is not fully understood up to now, imaging methods have to be improved to enhance the understanding. In this talk, a visualisation method to identify phase transitions in PEFC based on dual spectrum neutron imaging is going to be presented.

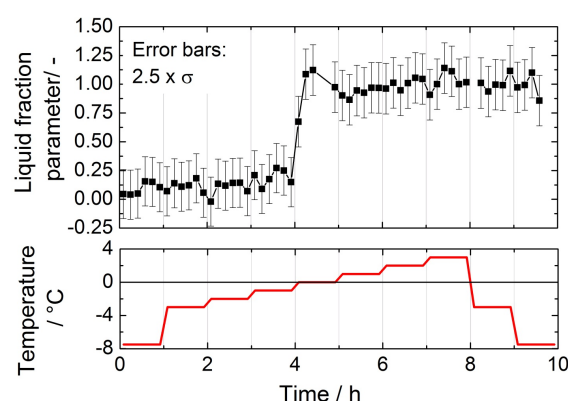


**Figure 1.** Illustration of the technical implementation of dual spectrum measurements using a motorized, polycrystalline filter.

The attenuation of neutrons passing through a specimen is strongly dependent on the incoming neutron energy. It has been shown [1,2] that increasing differences between the attenuations of frozen compared to liquid water appear with decreasing neutron-energy. At the ICON beamline [3] at the Paul Scherrer Institut, the energy spectrum has its average energy at 5 meV and no significant deviations between liquid and solid phase can be distinguished. Introducing a polycrystalline beryllium filter inside the neutron beam, the low energy part of the spectrum is emphasized and phase transitions between liquid water and ice are more distinctive. Since the spatial distribution of water inside a fuel cell is not known a priori, the analysis compares exposures with the filtered and unfiltered beam (cf. figure 1). Furthermore, this water distribution may change over time which implicates the capture of consecutive images with and without filter (dual spectrum).

In a first part, results based on a cylindrical water column ( $\text{\O}1.7 \times 10 \text{ mm}$ ) will be presented. Those measurements provide reference values for the ratio between the neutron attenuation by  $\text{H}_2\text{O}$  with and without filter for different thicknesses of water. As expected, due to the decreasing attenuation of  $\text{H}_2\text{O}$  with increasing energy [1,2], different aggregate states can be identified. However, the difference between the aggregate states is rather small and accounts only 1.8%. This slight deviation can be

attributed to the relatively small differences between water and ice in this energy range [1,2]. Referencing these ratios towards 0 in the frozen and 1 in the liquid state (liquid fraction parameter), phase transitions can be identified based on dual spectrum neutron imaging. Figure 2 shows the temporal evolution of the liquid fraction parameter (LFP) obtained during a temperature ramp (15 steps à 1h/image). As expected, the phase transition from frozen to liquid water can be identified at 0°C. During the last two hours, the water has been maintained in the supercooled state and the liquid fraction parameter maintains in the liquid state.



**Figure 2.** Identification of phase transitions based on dual spectrum neutron imaging.

Subsequently, the results obtained with a small scale differential PEFC will be analysed. At subfreezing temperatures, the product water is produced in the supercooled state [4,5]. In order to avoid temperature dependencies, a protocol has been developed, to compare liquid water (in supercooled state) and ice at constant temperature. Although the ratio of the attenuation between the filtered and non-filtered beam is not consistent in the fuel cell vs. the cylindrical scale measurement, phase transitions from liquid water to ice in the fuel cell have clearly been identified with this method.

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