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## Distinction of Liquid Water and Ice using Dual Spectrum Neutron Imaging

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The use of fuel cells in automotive as well as in autonomous power supply applications requires them to be able to start and operate at sub freezing temperatures. At those temperatures, the water produced by the electrochemical reaction can freeze and block the access of gaseous reactants, leading to a failure of the cell. Previous experiments [1] indicated that liquid water in supercooled state plays an important role in the cell operation at sub-freezing conditions. In this context, a direct method from the distinction of liquid water and ice is highly desired. The measurement is based on the differences in microscopic cross-section between liquid and frozen water [2] –related to a different inelastic scattering behavior of the neutrons. To avoid time consuming energy-selective measurements, a “dual spectrum” method was developed and implemented at the ICON neutron imaging beamline (Paul Scherrer Institut, Switzerland). In this method, a Beryllium filter is used to remove the high energy part of the spectrum and focus on an energy range ( $< 5$  meV) where the differences of cross section are visible. Using the comparison of the sample attenuation with and without filter, a “liquid fraction parameter” calibrated to be 0 for ice and 1 for liquid water can be extracted [3]. As seen in Figure 1, the liquid fraction parameter for a test water column shows a clear step at  $t = 4$ h, where the state changes from ice to liquid water. At  $t = 8$ h, no transition is visible, as the water stays liquid in the supercooled state. Further experiments were performed using small scale fuel cells. Although the absolute detection of the aggregate state of water was not possible, the phase transitions between liquid water and ice (and inversely) were clearly identified.

[1] P. Oberholzer, P. Boillat, R. Siegrist, R. Perego, A. Kaestner, E. Lehmann, G.G. Scherer and A. Wokaun, Cold-Start of a PEFC Visualized with High Resolution Dynamic In-Plane Neutron Imaging, *J. Electrochem. Soc.*, 159(2), 2012.

[2] L. Torres, J.R. Granada, J.J. Blostein, Total cross sections of benzene at 90 K and light water ice at 115 K, *Nucl. Instr. Meth. B*, 251(1), 2006

[3] J. Biesdorf, P. Oberholzer, F. Bernauer, A. Kaestner, P. Vontobel, E. Lehmann, T.J. Schmidt and P. Boillat, Distinction of Liquid Water and Ice Based on Dual Spectrum Neutron Imaging, *ECS Transactions*, 58(1), 2013

### Summary

Dual Spectrum Neutron Imaging was established as a method for distinguishing liquid water from ice. Although an absolute detection remaining challenging, the identification of phase transitions in time series was demonstrated both in a test water column and in small scale fuel cells.

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