X-Ray Tomographic Microscopy of Phosphoric Acid in High Temperature PEFC Components

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In high-temperature polymer electrolyte fuel cells (HT-PEFC) the quantity and concentration of phosphoric acid (PA) in the catalyst layer and, if wetted, in the gas diffusion layer (GDL) depends on operating conditions. The distribution, concentration and mobility of the PA relates to performance and durability of the HT-PEFC [1]. X-ray radiography (providing 2D information) has been developed for in-situ investigation of PA in HT-PEFC [2, 3]. However, while 2D imaging has advantages with time resolution, the determination of the PA concentration is tedious due to expansion of the acid with dilution [3]. Therefore, in order to better understand this correlation, X-ray tomographic microscopy workflows are developed to determine the PA properties in the GDL. As a basis the X-ray properties of PA are measured ex-situ in impregnated GDLs.

When light passes through a medium, part of it will be absorbed. This can be described by the complex index of refraction

 $n=1-\delta+i\beta$

the imaginary part β relates to the absorption when the electromagnetic wave propagates through the material, while the real part δ relates to the phase velocity. Figure 1 shows the complex index of refraction for phosphoric acid of two different concentrations as a function of photon energy.

In X-ray imaging real and imaginary part of the refractive index may be exploited. In particular, when using coherent synchrotron radiation, also the phase information may be utilized. The two methods are termed absorption and phase contrast and are giving complementary information on the sample.

Ex-situ experiments were performed at the TOMCAT beamline of the Swiss Light Source (SLS). In phase contrast the different concentrations lead to well separated peaks in the histogram. This can be exploited to determine the PA concentration in GDLs and full cells.

When GDLs (assembled in a membrane electrode assembly) are saturated ex-situ with PA (Figure 2), in phase contrast imaging the grey scale is indicative for the PA concentration. Figure 3 shows the correlation of grey scale with PA concentration filled in the GDLs. The results show that PA concentration can be determined in the GDL using X-ray tomographic microscopy. Future data will include results from in-situ imaging.



Figure 1: Complex refraction index of phosphoric acid with concentrations of 85 wt% and 40 wt%.



Figure 2: Orthogonal slice of a GDL filled with phosphoric acid (40 wt%) imaged in phase contrast mode at 20 keV.



Figure 3: Relationship of PA concentration and grey scale in X-ray tomographic imaging obtained in phase contrast mode at 20 keV.

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References

- Schmidt, T.J., *High Temperature Polymer Electrolyte Fuel Cells: Durability Insights*, in *Polymer Electrolyte Fuel Cell Durability*, F.N. Büchi, M. Inaba, and T.J. Schmidt, Editors. 2009, Springer: New York. p. 199-221.
- Maier, W., T. Arlt, K. Wippermann, C. Wannek, et al., Correlation of Synchrotron X-ray Radiography and Electrochemical Impedance Spectroscopy for the Investigation of HT-PEFCs. J. Electrochem. Soc., 2012. 159, F398-F404.
- Kuhn, R., J. Scholta, P. Krüger, C. Hartnig, et al., Measuring device for synchrotron X-ray imaging and first results of high temperature polymer electrolyte membrane fuel cells. J. Power Sources, 2011. 196, 5231-5239.