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Conductive AFM Study to Differentiate Between the Surface Ionic Conductivity of Nafion® and Electrospun Membranes

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Background

Our goal is to use multi-mode atomic force microscopy (AFM) to differentiate between Nafion® and electrospun nanofiber membranes. The results of this experiment will guide the design of new proton exchange membranes (PEM). Characterizing the electrospun nanofiber membranes will allow us to identify properties that are ideal for the next generation PEM. Engineering the proton conducting membrane for a PEM fuel cell that allows protons and inhibits other species to cross is vital to its long-term operation. When species other than protons are allowed to cross a PEM, issues arise in the form of electrode flooding, adverse chemical reactions, and reduced fuel cell efficiency.

One of the challenges with improving the efficiency of PEM fuel cells is to design a membrane that is both highly selective in which species are able to migrate across the membrane and resistant to negative hydration effects. The ionic selectivity has been shown to drastically change with the relative hydration of the PEM [2]. When the PEM is not hydrated, the transport of protons across the membrane effectively goes to zero due to the decrease in the disassociation of the ionic groups (-$SO_3^-H^+$) within the membrane and on the surface [1]. When a membrane is too hydrated, the polymer swells causing the ionic pathways through the membrane to open up and allow larger chemical species to migrate across the membrane. The purpose of making electrospun membranes is to increase the number of ionic pathways available for protons to move through the membrane, but also prevent the membrane from swelling at high relative humidity. The increased number of ionic pathways will raise the overall efficiency of the PEM fuel cell, while the prevention of membrane swelling will enable the PEM to be more selective.

The potential and limitations of AFM have been extensively studied by Aleksandrova et al [1]. AFM has the ability to operate in multiple modes; including static force, dynamic force, phase contrast (i.e. hardness), force modulation, spreading resistance (i.e. conductivity), and lateral force. While in static force mode, an AFM measures the surface topography by measuring the deflection of a cantilever as it moves across the sample. When the cantilever is deflected due to a change in surface topography, a laser detection system detects a change in the reflected laser beam. This information is used to produce an image of the surface topography. While operating AFM in spreading resistance mode, a voltage is applied to the surface of the sample through the use of a catalytic cantilever tip. The sample is hot-pressed onto a catalyst-coated electrode to establish a current pathway back to a current sensing assembly. The AFM setup for spreading resistance mode is shown in Figure 1.

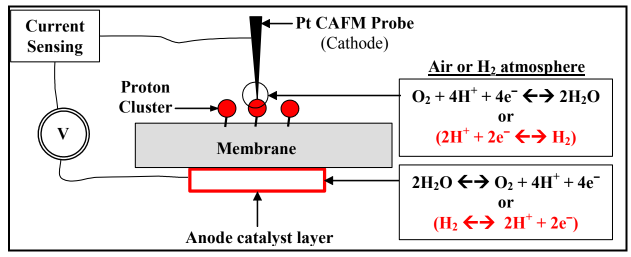

Figure 1. AFM Setup for Spreading Resistance Mode [3]

Experimental Procedure

A Nanosurf® FlexAFM was used to measure the surface topography, hardness, and conductivity of different proton conducting membranes. Nafion® 212 was compared to the electrospun nanofiber membranes made by Dr. Pintauro's research group at Vanderbilt University. In order to measure the conductivity (resistance) of the membrane, a platinum coated cantilever was used in coordination with a PEM hot pressed onto a platinum coated SGL35BC gas diffusion electrode. The Nanosurf® FlexAFM was operated inside a shielded cabinet to minimize noise while making measurements. Prior to and after each data collection period, an AFM calibration grid was used to ensure the accuracy of the FlexAFM.

Results and Discussion

The ability to characterize the surface of the electrospun membranes will be important for our group to validate whether these membranes have a sufficient number of ionic cluster sites for proton transport but also prevent the PEM from being over saturated with water which would allow non-proton species to cross the membrane. We hypothesize that there will be a high correlation between the phase contrast and spreading resistance modes. The ionic molecules on the membranes surface will be softer relative to the polymer's PTFE backbone. In addition, the setup for spreading resistance mode is more prone to errors than phase contrast mode due to a higher degree of complexity in the AFM setup. Statistical analysis of the AFM images will be used to characterize the surface of the PEMs. By correlating the two modes, we hope to use phase contrast mode in the future to predict the surface ionic activity. We expect electrospun membranes to have a better distribution of ionic pathways through the membrane compared to Nafion® 212.

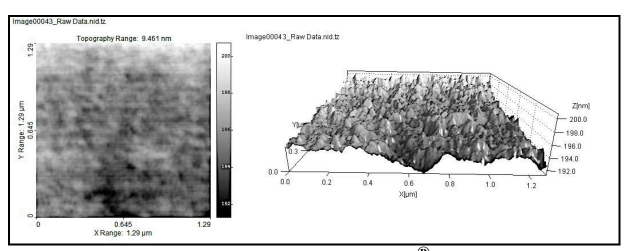

Figure 2. 2D/3D Images of Nafion® 212 in Dynamic Force Mode, Image Size 1.3 µm

References

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