Nafion[®]—Titania Nanocomposite Proton Exchange Membranes

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ABSTRACT: Proton exchange membranes consisting of Nafion[®] and crystallized titania nanoparticles have been developed to improve water-retention and proton conductivity at elevated temperature and low relative humidity. The anatase-type titania nanoparticles were synthesized *in situ* in Nafion solution through sol–gel process and the size of the formed titiania nanoparticles is in the range of 3–6 nm. The formed nanoparticles are well-dispersed in Nafion solution at the titania concentration of 5 wt %. The glass transition temperature of the formed

Nafion-titania composite membrane is about 20°C higher than that of plain Nafion membrane. At elevated temperature (above 100°C), the Nafion-titania nanocomposite membrane shows higher water uptake ability and improved proton conductivity compared to pure Nafion membrane. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 120: 1186–1192, 2011

Key words: proton exchange membrane; titania; sol–gel process; water retention; ionic conductivity

INTRODUCTION

Proton exchange membrane fuel cells operating at elevated temperature and low relative humidity are being considered as the next generation of fuel cells because they could benefit from enhanced tolerance to impurity of the fuel gas, simplified water and heat management, and increased reaction rates at both cathode and anode compared with fuel cells operating below 80°C.1-4 Despite the advantages of this technology, one of the great technical barriers restricting their further widespread applications is the poor proton conductivity of membrane at elevated temperature and low relative humidity. The often used, commercially available proton exchange membrane for current fuel cell technology is perfluoronated Nafion[®] membrane produced by DuPont because of its robust structure and excellent proton conductivity in the hydrated state.^{5,6} However, shortcomings of Nafion membrane including the drastic decrease in proton conductivity at low relative humidity and the low glass transition temperature limit its utility at elevated temperature. If Nafion membrane is kept in hydrate state during the operation of fuel cells at elevated temperature, the increased system pressure could offset those benefits. Thus, development of membranes working at elevated temperature and low relative humidity becomes an active research area of fuel cells.

One interesting approach to improve proton conductivity of membranes at elevated temperature is to impregnate hygroscopic metal oxide nanoparticles inside Nafion membrane, as described in recent reviews.^{2,7} It has been observed that the metal oxides (SiO₂, ZrO₂, and TiO₂) doped Nafion membrane can bear the operating temperature of fuel cell up to 130°C, whereas the commercial Nafion115 membrane was damaged at 120°C within 1 h.8 Among the studied metal oxide fillers, nanosized titania doped Nafion membranes have shown rather promising results based on accomplished work on elevated temperature membranes.⁹⁻¹² Commercially available titania particles with diameters of 1 µm have been applied in Nafion-titania composite membrane,9 the decrease in cell resistance has been observed at 130 $^{\circ}\mathrm{C}$ and reduced relative humidity (77%). Sacca et al. 10 synthesized composite Nafion membranes by addition of preformed titania nanoparticles (~ 20 nm) with different percentages and

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tested cell performance using such membranes in the temperature range of 80–130°C using pure hydrogen and air. It was confirmed that the presence of titania nanoparticles is able to decrease the cell resistance above 100°C. The composite membrane with 3 wt % titania reached a power density of about 0.254 W cm⁻² at 0.5 V with 100% relative humidity under absolute system pressure of 4.0 bar. Chen et al.¹¹ fabricated composite membrane of Nafion doped with porous anatase titania using a recasting process. The titania dopants with diameter of 95 nm were presynthesized via sol-gel process using ionic liquids as template. Under 50% relative humidity at 120°C, the fuel cells using composite membrane with 3% fillers in weight delivered the power density of about 0.669 W cm⁻² at a voltage of 0.4 V using hydrogen and pure oxygen as fuels. Using in situ sol-gel technique, nanosized titania nanoparticles can be directly formed inside the commercially available Nafion membrane.¹² The formed composite membranes showed improved water uptake and thermomechanical properties compared to pure Nafion membrane. Although the role the titania particles played is not fully understood, titania doped Nafion membranes have shown the potential applications in elevated temperature fuel cell systems.

It is generally believed that the improved proton conductivity of composite membranes is attributed to the surface hydroxide groups of additives which retain water and increase surface acidity at elevated high temperature.^{13,14} The well developed hybrid membranes require the additives uniformly and well distributed in the membrane to minimize the interfacial resistance of resin and dopants. However, Nafion/titania composite membranes are usually prepared either by recasting of mixture of preformed titania nanoparticles and Nafion ionomer,^{9–11} or by impregnation of Nafion membranes with titania precursor solution followed by in situ sol-gel reaction.¹² In the first approach, the doped titania nanoparticles make the particle/Nafion interface unsatisfactory as the mechanical properties of the membrane could be affected. In the second approach, hydrolysis and condensation reactions of precursor may drive the distribution of titania nanoparticles inside the membrane unevenly.

In the present work, Nafion/titania composite membranes were formed by *in situ* hydrolysis of precursors in Nafion solution through sol–gel technology, followed by a recast process. Nafion molecules were expected to be adsorbed on the surface of formed titania nanoparticles through electrostatic interactions and to stabilize the initial formed titania nanoparticles. The motivation of this work is to improve thermal property and proton conductivity of Nafion membrane by doping nanosized titania particles inside the membrane.

EXPERIMENTAL

Nafion solution (DE-520, EW1100) was purchased from DuPont Ind., which contains 5 wt % of perfluorosulfonate resin (H⁺ form) and 95 wt % of isopropanol/water mixture (10 : 9 weight ratios). Tetrabutyl titanate was received from Shanghai Reagent (China). Water was deionized through a Milli-Q system (Barnsted Nanopore, resistivity = $18.0 \text{ m}\Omega \text{ cm}^{-1}$). *N*-methyl-2-pyrrolidone (NMP) was dried over CaH₂ and distilled under vacuum. All the other solvents and chemicals were reagent grade and were used as received. The referenced titania particles for zeta potential measurements were formed by hydrolysis of tetrabutyl titanate precursors in NMP/water solution at different pH values.

Commercial Nafion solutions were evaporated at 120°C and redissolved in NMP. The desired quantity of precursor solutions was added dropwise to the Nafion solution under vigorous stirring in an inert nitrogen atmosphere at 120°C and allowed to cool down to 40°C. After adjusting the pH of the dispersion to ~ 2.0 using 2*M* HCl solution, the mixture was continuously stirred for 8 h at 40°C and a clear sol containing hybrid Nafion-titania nanoparticles was obtained. Unless otherwise stated, the final concentration of Nafion is about 2 wt % based on our previous results.^{15,16}

Hybrid membranes were prepared using a recasting process: the hybrid sol was first placed in a Petri dish, followed by the solvent evaporation at 100°C for 8 h and then posttreated at 150°C under vacuum for 3 h. The formed membranes were than cleaned using a standard procedure at 80°C for 30 min in 5% H_2O_2 solution, in deionized water, in 0.5M H_2SO_4 solution, and finally in deionized water again.

Zeta potential measurements were carried out on Zetasizer Nano-ZS (Malvern, UK) using laser Doppler velocimetry and phase analysis light scattering. The temperature of the scattering cell was 25°C and the data were analyzed with the software from supplier. Dynamic light scattering measurements were carried out with Malvern HPPS Laser Particle Size Analyzer (Malvern, UK) with scattering angle of 90° at 25°C using He-Ne laser (633 nm). The size of formed nanoparticles was examined using high resolution transmission electron microscopy (TEM, JEM-2010FEF equipped with Energy Dispersive Spectrum Analyzer). Samples for TEM measurements were prepared by directly placing a drop of the solution on a thin carbon film supported by a copper grid. Surface homogeneity of composite membranes was examined using scanning electron microscopy (SEM, JEOL JSM-5610LV). Fourier transform infrared spectra (FTIR, Bio-Rad FTS 300) were recorded with a resolution of 4 cm^{-1} .

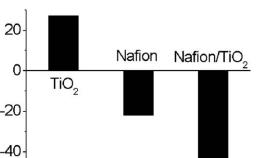
The water-uptake of membranes at different relative humidity was calculated as the ratio of the difference between the swollen and the dry weight of the membrane. The weight of swollen membrane was measured rapidly after keeping the membrane at 100°C under desired relative humidity for 10 h in a temperature and relative humidity-controllable oven. The weight of dry membrane was determined directly after drying the sample at 100°C for 2 h. The weight of the completely hydrated membrane was measured after boiling the membrane in deionized water for 24 h and wiping out the surface adsorbed water.

The weight loss of membranes was measured using a high resolution thermogravimetric analyzer (TGA, Perkin-Elmer Instrument) at a rate of 10° min⁻¹ under nitrogen. The sample was immersed into deionized water for 1 h at room temperature and was further treated under vacuum at 80°C for 2 h before TGA measurements. Thermomechanical measurement (TMA) was carried out using a TMA-202 analyzer (NETZSCH, Germany) with 50 cm³ min⁻¹ of nitrogen purge gas. The sample size was 12.0 mm \times 4.0 mm. Experiments were run from 30 to 200°C with a temperature ramp rate of 20°C \min^{-1} .

Proton conductivity measurements were carried out in-plane using an impedance analyzer (Autolab PG30/FRA, Eco Chemie, Netherland) at different temperatures without external humidification using homemade conductivity testing cell. Two goldcoated copper sheets were placed onto the as-formed membranes (3.5 cm \times 2.2 cm) in the same face. The distance between two gold-coated copper electrodes was 3 cm. Membranes with two electrodes were sandwiched between two polytetrafluoroethylene sheets. Electrochemical impedance spectra were recorded in the frequency range of 1 Hz and 105 KHz and the signal amplitude of 10 mV. Membranes were first hydrated before assembling into the testing cell. The measurements were carried out from lower temperature to higher temperature. Membranes were stabilized under each testing condition for 15 min before data were recorded. It also should be noted that Nafion membranes are isotropic according to the literature¹⁷ and the throughplane conductivity of membrane is similar to in-plane conductivity.

RESULTS AND DISCUSSION

Titania nanoparticles were formed through in situ hydrolysis of tetrabutyl titanate precursors in Nafion solution. Since hydrolysis and polymerization of precursor occur rapidly in the presence of water, the used Nafion solution for the *in situ* growth of titania nanoparticles is required to be anhydrous before hydrolysis of precursors to have better control of size distribution and morphology of the formed tita-



Zeta potential / mV

-60-

Figure 1 Zeta potentials of TiO₂ particles, 2 wt % of Nafion, and Nafion/TiO₂ hybrids in \hat{NMP} /water (1 : 1, v/v) at pH value of 2.0.

nia nanoparticles. To minimize undesired water content in the system, Nafion solution was heated up to 120°C under nitrogen atmosphere and the addition of precursors were then performed under the same condition.

As Nafion molecules were expected to be adsorbed on the surface of formed titania nanoparticles through electrostatic interaction, zeta potentials of titania particles, Nafion and Nafion-titania hybrids were first investigated. Figure 1 shows zeta potentials of different systems at pH value of 2.0. It can be seen that zeta potential of titania particles is about 27 mV at pH value of 2.0, indicating that the formed titania particles are positively charged. For Nafion solution, the dissociation of sulfonic acid end-groups on side chains leads to negatively charged molecules, also indicated by zeta potential of -20 mV for Nafion dispersion with concentration of 2 wt % at pH value of 2.0. Once positively charged titania nanoparticles were formed in Nafion solution, the adsorption and coverage of ionomer molecules on the particle surface can occur through electrostatic interaction. Zeta potential of -53 mV for the Nafion-titania hybrid dispersion confirmed the hypothesis. The large difference in zeta potential for Nafion and Nafion-titania hybrid nanoparticles attributes to the adsorption of Nafion molecules at titania surfaces, leading to a higher negative charge (SO₃⁻ on Nafion side chains) density. Accordingly, higher zeta potential was observed.

Hydrodynamic diameters of hybrid particles were investigated with different titania content using dynamic light scattering measurements, as plotted in Figure 2. When the titania content is about 5 wt % regarding to Nafion, only one type of particle was observed with hydrodynamic diameter of around 216 nm in size distribution curve. This result indicates that the formed titania nanoparticles (with adsorbed polymer layers) were evenly and well distributed in the dispersion, leading to the minimized

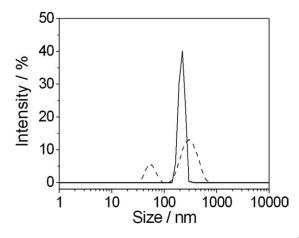


Figure 2 Distribution of scattering intensity of Nafion[®]/ TiO_2 dispersion with different weight ratios of TiO₂ to Nafion: 5% (solid line) and 15% (dash line).

interfacial interaction between Nafion molecules and titania nanoparticles. When the titania contents increases to 15 wt %, two types of particles with hydrodynamic diameters of 56 and 312 nm were observed, indicating the uneven distribution of particles in the dispersion. Particles with different hydrodynamic diameters correspond to different sizes of titania particles with self-assembled Nafion layers on surfaces.

Figure 3(a) shows transmission electron microscopy image of Nafion-titania hybrid dispersion formed in 2 wt % of Nafion solution in NMP. The final content of doped metal oxides in designed membrane is 5 wt %. It is evident that the diameter of the *in situ* formed nanoparticles was in the range from 3.0 to 6.0 nm [inset in Fig. 3(a)], indicating that the existence of Nafion molecules prevents further growth of the particles. Energy dispersive spectrometry was measured based on one nanoparticle in TEM image and the recorded spectrum was shown in Figure 3(b). The coexistence of the atom peaks for Ti, C, F, and S suggests the coverage of Nafion molecules on the formed metal oxide nanoparticles. In addition, clear lattice fringes of the formed titania nanoparticles were observed from the high resolution TEM image [Fig. 3(c)], indicating their high crystallinity. The lattice spacing in Figure 3(c) are found to be 0.35 nm, corresponding to the (101) planes of anatase phase.

The Nafion-titania hybrid membranes were formed using a recast process and were posttreated with a standard process, as described in the experimental section. To determine whether the doped titania nanoparticles affect the microstructure of Nafion membrane, X-ray diffraction (XRD) measurements of recast Nafion membrane and hybrid membranes were performed (data not shown). Compared to pure Nafion membrane, the diffraction maxima of Teflon-like domain¹⁸ for composite membrane

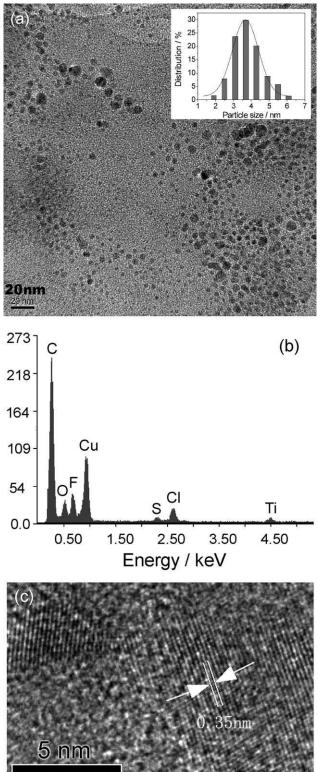


Figure 3 TEM micrographs of Nafion[®]/TiO₂ hybrid dispersions with 5% titania in weight regarding to Nafion[®] (a), energy dispersive spectra for one particle in TEM micrographs of Nafion[®]/TiO₂ (b), and high resolution TEM images of crystalline TiO₂ particles (c). The inset in (a) is the size distribution of TiO₂ nanoparticles.

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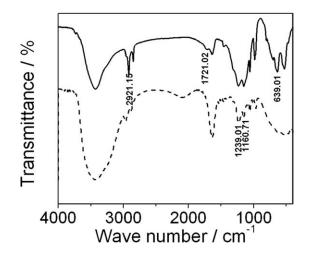


Figure 4 FTIR spectra of Nafion[®]/TiO₂ composite membrane with 5% titania in weight (solid line) and referenced recast Nafion[®] membrane (dash line).

appear at very similar scattering angles, indicating that the doped titania nanoparticles did not affect the crystallinity and structure of Nafion in the membrane significantly. Unfortunately, diffraction peaks related to crystallized titania particles could not be clearly observed, probably because of the low concentration of doped nanoparticles.

Fourier transform infrared (FTIR) spectroscopy has been employed to further confirm the existence of titania nanoparticles inside the membrane and to investigate the interfacial properties of Nafion and titania nanoparticles (Fig. 4). As reference, FTIR spectrum of recast Nafion membrane is also included in the same figure. The IR absorption in the spectral range of 600-1000 cm⁻¹ was assigned to the surface vibrations of the Ti-O bonds.¹⁹ Absorption peaks at 2921 and 1721 cm⁻¹ were assigned to the C-H and C=O vibrations from the precursor of titania nanoparticles, indicating the incomplete hydrolysis of precursor. The slight shifting of the absorption band for antisymmetric stretching of SO₃groups from 1160 to 1152 cm⁻¹ was probably attributed to the hydrogen bond formation between sulfonic acid groups and existing hydroxyl groups on the surface of titania nanoparticles.

One of the shortcomings of Nafion membrane is relatively low glass transition temperature that limits its application at elevated temperature. TMA experiments were carried out to investigate glass transition temperatures of formed membranes and the TMA curves of Nafion and Nafion-titania composite membranes were plotted in Figure 5. The glass transition temperature of Nafion-titania composite membrane was found to be 135°C which is about 20°C higher than that of plain Nafion membrane. The increased glass transition temperature of composite membrane is attributed to the electrostatic interactions of sulfonic acid groups on side chain of Nafion and surface charges of titania nanoparticles. Jalani et al.¹² reported that the glass transition temperature of Nafion-titania composite membrane formed form *in situ* hydrolysis of titanate precursors inside the commercially available membrane is about 122°C whereas the glass transition temperature of commercial Nafion 112 membrane is about 110°C. Despite the similarity of the chemical composition, the distribution of the embedded titania nanoparticles reported here is more homogenous than that reported in the literature,¹² which resulted in the higher glass transition temperature in the current system.

The motivation of the addition of hygroscopic metal oxide nanoparticles into the Nafion membrane is to increase the water retention ability and accordingly to increase the ionic conductivity of the membrane at elevated temperature and low relative humidity since water is proton transporting medium for proton exchange membranes. At room temperature, the fully hydrated Nafion-titania hybrid membranes contained 25.5% of water in weight, which is higher than that of Nafion 211 membrane (18.7% in weight) and much higher than that of fully hydrated recast Nafion membrane (13.7% in weight). We further investigated the water uptake of hybrid membranes under different relative humidity at 100°C, as shown in Figure 6. As comparison, the water uptake of recast reference Nafion membrane as a function of relative humidity at 100°C was plotted in the same figure. It can be clearly seen that the formed composite membrane takes up more water than recast Nafion membrane does in the measured range of relative humidity at 100°C. The higher water uptake for the composite membrane can be attributed to the doped hygroscopic titania nanoparticles.

To investigate the water retention ability of the formed membranes, thermogravimetric analyses (TGA)

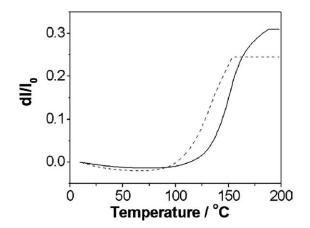


Figure 5 TMA curves of Nafion membrane (gray dash line) and Nafion-TiO₂ composite membrane (black solid line).

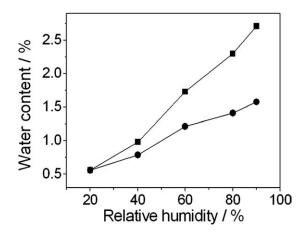


Figure 6 Water uptake of formed membranes as a function of relative humidity at 100° C: Nafion[®]-TiO₂ membrane with 5% titania in weight (squares), and referenced recast Nafion[®] membrane (circles). Solid lines are guide to eyes.

were performed for composite membrane and recast Nafion membrane, as shown in Figure 7. For both membranes, mass losses at about 200°C correspond to the water desorption from the membrane. The decomposition of sulfonic acid groups and the polymer molecules at about 280 and 400°C has been observed, respectively. The complete decomposition of the composite membrane seems to be accelerated compared to recast Nafion membrane, probably caused by the addition of titania nanoparticles. However, it is worthy to be noticed that the water desorption of composite membrane is much slower than that of recast Nafion membrane although composite membrane has higher water uptake, indicating an improved water retention ability of composite membrane.

One of the very important properties that a qualified fuel cell membrane should have is the proton

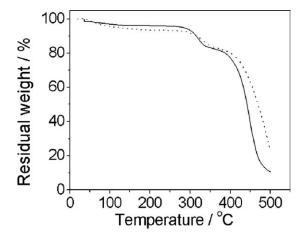


Figure 7 Thermogravimetric analyses on the Nafion[®]/ TiO_2 membrane with 5% titania in weight (solid line) compared to recast Nafion membrane (dash line).

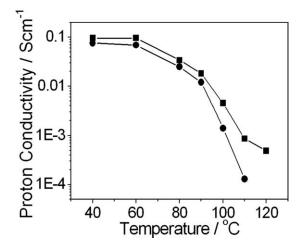


Figure 8 Proton conductivities of the formed membranes as a function of temperature without external humidification: Nafion[®]/TiO₂ membrane with 5% titania in weight (squares), and recast Nafion[®] membrane (circles). Solid lines are guide to eyes.

conductivity, which decides the performance of the assembled fuel cells. Figure 8 shows the proton conductivities of formed membranes at different temperature without external humidification, as described in the experimental section. It is evident, that the proton conductivity of Nafion-titania nanocomposite membranes is higher than that of recast Nafion membrane in the measured temperature range, attributed to the enhanced water retention property for titania nanoparticles doped membranes. With the increase in temperature (higher than 80°C), the proton conductivities for both the composite membrane and the recast Nafion membrane decrease dramatically. However, the titania doped membrane, still show better proton conductivity. At 100°C, the proton conductivity of composite membrane without external humidification is about 5 \times 10⁻³ S cm⁻¹ whereas the conductivity of the recast Nafion membrane is about five times less. This behavior can be attributed to the water entrapped into the membrane that dehydrates with the increase of temperature, indicating higher water retention ability of composite membranes. Yang et al.20 have observed that the addition of hygroscopic inorganic particles (zirconium phosphate) could lead to the decrease in conductivity of membranes, which is contrary to our observation. This difference may be probably attributed to the size and distribution of the doped nanoparticles. The particle size in literature²⁰ is about 11 nm. Such large particles may block the proton transporting channel in the membrane although the particles can improve the water retention ability of the membrane. Accordingly, the proton conductivity decreases. In our case, the diameter of formed particles is about 5 nm, which is similar to the diameter of hydrophilic domains inside Nafion membrane. The formed particles may therefore stay inside the hydrophilic cores to improve the proton transporting process, which leads to an enhanced proton conductivity of composite membrane.

It is worthy to be noted that water molecules always exist in the fuel cell when it is under operation as the only product of electrode reaction is water. Therefore, the membrane can be humidified to some extent in an operating fuel cell, leading to higher conductivity compared to the result from external experiments in this communication. Thus, the titania doped Nafion membranes formed in this study can be potentially used in the elevated temperature PEMFCs. The fuel cells performance using titania nanoparticles doped membranes is still under investigation.

CONCLUSIONS

Nafion-titania nanocomposite membranes were formed through *in situ* sol-gel process in this study. Crystallized anantase type titania nanoparticles with diameter in the range of 3-6 nm were in situ synthesized in Nafion solution by hydrolysis and condensation of precursors. The existing Nafion molecules can be adsorbed onto titania particles through electrostatic interactions and prevent the further growth of the initially formed nanoparticles. The higher glass transition temperature of composite membranes compared to plain membrane makes the application at elevated temperature possible. The formed Nafion-titania nanocomposite membranes also show enhanced water retention ability and higher proton conductivity compared to recast pure Nafion membrane at all measured temperature range, attributed to the small size and well distribution of the formed nanoparticles. Although the proton conductivity of the composite membrane decreases with increasing temperature, the proton conductivity without external humidification for

composite membrane is much higher than that for recast Nafion membrane at elevated temperature. The hybrid membrane developed here has the potential for PEMFC applications at elevated temperatures and low relative humidity.

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