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High-Performance Alkaline Direct Methanol Fuel Cell using a Nitrogen-Postdoped Anode

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A commercial PtRu/C catalyst postdoped with nitrogen demonstrates a significantly higher performance (~10–20% improvement) in the anode of an alkaline direct methanol fuel cell than an unmodified commercial PtRu/C catalyst control. The enhanced performance shown herein is attributed at least partially to the increased electrochemical surface area of the PtRu/C after postdoping with nitrogen.

In recent years, there has been growing interest to develop alkaline direct methanol fuel cells (ADMFCs) because of their advantages over acid-based direct methanol fuel cells (DMFCs). Benefits include enhanced electrokinetics for both methanol oxidation and oxygen reduction reactions,[1–3] which enable the consideration of non-noble-metal catalysts,[4] and lower methanol crossover from anode to cathode.[5,6] Despite these advantages, however, ADMFC performance demonstrated to date has not reached that of the best-performing acid-based DMFCs.[5–8] Besides the well-known issue with carbonate formation in ADMFC membrane electrode assemblies (MEAs), nonoptimized MEA structures also contribute to this underperformance. Recently, Surya Prakash et al.[9] demonstrated a catalyst-coated substrate (CCS) method for ADMFC MEA fabrication that yielded a record-breaking performance (~160 mW cm^{-2} at 90 °C) by using a Tokuyama-006 membrane. Although the CCS method was postulated to improve the membrane–catalyst interfacial structure, and hence performance, the study employed very high catalyst loadings for both the anode (PtRu black 8 mg cm^{-2}) and cathode (Pt black 8 mg cm^{-2}). Such catalyst loadings are likely prohibitive for ADMFC commercialization, even in high-value applications.

In this communication, we demonstrate a nearly comparable ADMFC performance (140 mW cm^{-2} at 80 °C) using a 3 mg cm^{-2} loading of commercial PtRu/C catalyst (JM Hi-spec 10000) that has been postdoped with nitrogen. Postdoping is accomplished by nitrogen-ion-implantation of the commercial carbon supported PtRu/C catalyst after it has already been synthesized and supplied.[10–12] The modification of carbon materials with nitrogen is a fast-growing field of research with many energy and sustainability applications.[13,14] The motivation to use postdoping comes from our earlier studies, which have shown that PtRu/C catalyst stability is enhanced greatly by a high surface nitrogen content.[15] Recent investigation of a series of commercial catalyst materials postmodified with nitrogen at several dosages revealed that in acid media materials postmodified at higher dosages not only show increased electrochemical surface areas (ECSAs) but also demonstrate an improved retention of the ECSA.[10]

This communication shows the benefits of N-modification in alkaline media, which demonstrate that the performance of the N-doped catalyst is significantly higher than that of an otherwise identical MEA made from the same commercial catalyst without N-doping. Electrochemical and microstructural evidence suggests that the nitrogen postdoping process improves performance in part by increasing the ECSA of the catalyst, likely by catalyst resputtering and refinement caused by the doping process.[10] Although the performance of the MEA fabricated from the N-doped PtRu/C is considerably better than that of the undoped control, both MEAs yield maximum power densities that are among the highest reported for ADMFCs.

The MEAs were fabricated by coating the PtRu/C (3 mg cm^{-2}), N-doped PtRu/C (3 mg cm^{-2}), and Pt/C (2 mg cm^{-2}) catalyst inks directly onto the alkaline membrane (Tokuyama A-201) using the same direct-membrane coating protocols optimized previously for high-performance Nafion-based acid DMFCs.[16] The PtRu/C was used directly as supplied by the manufacturer, and the N-doped PtRu/C was first doped with nitrogen using an ion implantation procedure as detailed elsewhere.[10] X-ray photoelectron spectroscopy (XPS) of the N-doped PtRu/C sample reveals that nitrogen was successfully incorporated into the materials with concentration 2.8 ± 0.7 at %. (The elemental compositions of undoped PtRu/C and N-doped PtRu/C in at % are provided in Supporting Information Table S1).

The ECSAs for both the N-doped PtRu/C anode and the undoped PtRu/C anode were measured using the methanol stripping voltammetry (MSV) technique.[17] Notably, CO stripping curves could not be obtained for either the N-doped PtRu/C or the undoped PtRu/C even after purging the CO gas (0.1% CO in Ar) for 30 min. It is believed that the non-Teflonized gas diffusion layer (GDL) used in the anode side of the MEAs hinders the CO gas permeation, which prevents the adsorption of CO

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gas on the PtRu/C catalysts. Thus, MSV analysis was used to provide an alternative assessment of the ECSA. The MSV results show that the N-doped PtRu/C anode exhibits a significantly higher methanol intermediate oxidation current (which is most likely a result of CO species[18]) than the unmodified PtRu/C anode (Figure 1). The resulting ECSA values for the N-doped PtRu/C and undoped PtRu/C anodes are 51.5 and 35.4 $m^2 g^{-1}$, respectively, which is a nearly 32% improvement in the ECSA for the former over the latter catalyst. For both catalysts, the CO oxidation onset takes place at the same potential (0.32 V vs. the dynamic hydrogen electrode; DHE), and the peak potential deviates by just 10 mV between the two anodes. These results suggest that the main difference between the two catalysts is metal surface area, a conclusion that is substantiated by the TEM comparison of the two samples (Figure 2). In addition, XPS[19] also indicates a change in the surface content. The total amount of Pt and Ru determined from XPS of the N-doped PtRu/C is 8.2 vs. 7.2 at% for the undoped PtRu/C. We hypothesize that the nitrogen-ion-implantation process causes resputtering and reorganization of the PtRu metal phase on the surface of the carbon support, which leads to a higher ECSA for the doped catalyst. Notably, the ECSA values for the Pt/C catalyst in cathodes of both the undoped and N-doped MEAs are almost identical (37.4 and 35.7 $m^2 g^{-1}$, respectively, for undoped and N-doped MEAs; Figure S1), and thus we eliminate the cathode as a possible source of difference between the performance of the two cells.

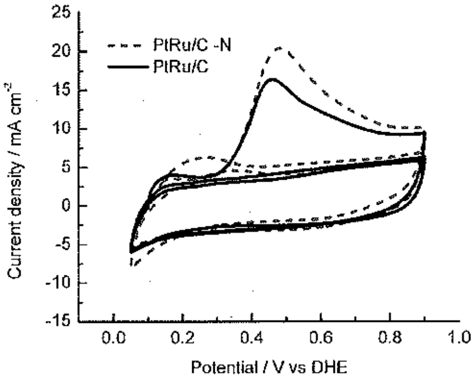

Figure 1. ECSA for N-doped PtRu/C (51.5 $m^2 g^{-1}$) and undoped PtRu/C (35.4 $m^2 g^{-1}$) catalysts measured by MSV at 25 °C (scan rate: 5 $mV s^{-1}$).

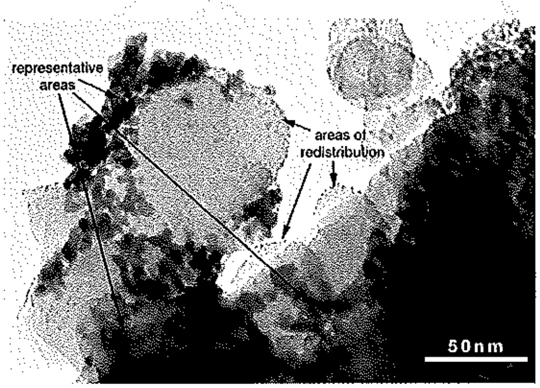

Figure 2. TEM image that shows representative areas (typically found in undoped PtRu/C) and unique areas of redistribution found only in the N-doped PtRu/C.

The ADMFC performance of the N-doped PtRu/C and undoped PtRu/C catalysts using 2 M NaOH + 2 M CH_3OH at an operating temperature of 80 °C are compared in Figure 3. The open circuit voltage (OCV) values of both ADMFCs are comparable (0.950 and 0.930 V for oxygen and air, respectively) and are significantly higher than the OCV values typically obtained for acid-based DMFCs that employ Nafion-type membranes (~ 0.8 V).[20] This can be attributed to the lower methanol crossover associated with the alkaline-type Tokuyama (A-201) membrane. Although the OCV values of both MEAs are similar, the MEA that contains the N-doped PtRu/C anode catalyst exhibits an improved polarization performance, particularly in the Ohmic and mass-transfer regions. This improvement can be attributed in part to the higher ECSA of the N-doped PtRu/C anode catalyst. The N-doped anode also demonstrates a higher performance than the undoped anode for other NaOH and methanol feed concentrations, which include higher and lower concentrations (Figure S2). For the conditions of the results shown in Figure 3, the N-doped PtRu/C MEA delivers maximum power densities of 140 (oxygen) and 110 $mW cm^{-2}$ (air), whereas the undoped PtRu/C MEA delivers maximum power densities of 117 (oxygen) and 100 $mW cm^{-2}$ (air). This represents a 10–17% improvement in performance for the N-doped PtRu/C MEA compared to the undoped PtRu/C MEA. To the best of our knowledge, the ADMFC performance of the N-doped PtRu/C MEA is among the best reported under similar catalyst loading and measurement conditions (comparisons are provided in Table 1).

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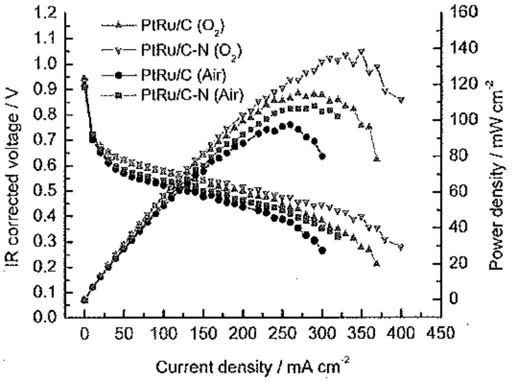

Figure 3. ADMFC performance curves for the N-doped PtRu/C and undoped PtRu/C catalysts in 2 M NaOH + 2 M CH_3OH at 80 °C.

To further investigate the reasons for the improved ADMFC performance, the methanol oxidation reaction (MOR) activities of both anodes were probed by linear sweep voltammetry

Table 1. Comparison of our ADMFC performance with some literature data.

Ref.	Anode catalyst	Catalyst loading [mg cm^{-2}]	Fuel	T [°C]	Maximum power density [mW cm^{-2}]
our data	PtRu/C	3	2 M NaOH/2 M CH_3OH	80	100 (air) 117 (O_2)
our data	PtRu/C-N	3	2 M NaOH/2 M CH_3OH	80	110 (air) 140 (O_2)
[24]	PtRu	1	1 M CH_3OH	80	2.6 (O_2)
> ~~[25]~~ [4]	PtRu	4	1 M KOH/1 M CH_3OH	50	6.0 (air)
[9]	PtRu	8	2 M KOH/1 M CH_3OH	90	160 (O_2)
> ~~[26]~~ [25]	PtRu	1	0.5 M KOH/1 M CH_3OH	80	60 (O_2)

(LSV) under the same operating conditions used for the ADMFC performance tests in 2 M NaOH + 2 M CH_3OH solution (Figure 4). Higher MOR current density is obtained at 0.4 V vs. DHE for the N-doped PtRu/C anode (390 mA cm^{-2}) ^ compared

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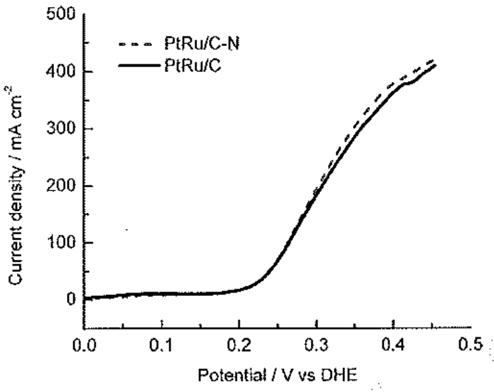

Figure 4. MOR polarization curves for the N-doped PtRu/C and undoped PtRu/C catalysts in 2 M NaOH + 2 M CH_3OH at 80 °C (scan rate: 5 mV s^{-1}).

to the undoped PtRu/C anode (365 mA cm^{-2}). Although only a minor improvement in the MOR current is observed for the N-doped PtRu/C compared to the undoped PtRu/C anode in 2 M NaOH + 2 M CH_3OH, ~~if~~ the MOR current is measured in other solution concentrations (e.g., 2 M NaOH + 1.5 M CH_3OH or 2 M NaOH + 2.5 M CH_3OH), larger improvements are observed that are consistent with the improvement in the fuel cell performance curves obtained in methanol with oxygen/air. The improvement in the MOR current density is observed only in the high anodic potential region for the N-doped PtRu/C anode, consistent with the DMFC polarization data, which showed significant performance improvement mostly in the >Ohmic and mass-transfer regions. In contrast, the MOR current density in the lower potential region (0.2–0.3 V) remains almost equivalent for the two anode catalysts, and the MOR onset potential does not change. We hypothesize that the higher ECSA of the N-doped PtRu/C anode does not impact the performance in the kinetic region significantly because in this region performance is determined mainly by the oxidation of methanol intermediates by the OH_{ads} species.[21] The concentration of these species is likely determined by the availability of excess OH^- ions supplied by the NaOH solution fed along with methanol as well as the migration of OH^- ions from the cathode to anode, rather than by the catalyst surface area. The higher ECSA of the N-doped PtRu/C anode contributes more significantly to the performance in the mass-transfer region, in which the additional active catalyst area may enhance the bifunctional mechanism and thereby facilitate the higher conversion of the CO intermediates into CO_2/CO_3^{2-}. This interpretation is also consistent with the results from the MSV (Figure 1).

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Replace 'O' by small 'o' in Ohmic

In conclusion, a commercial PtRu/C catalyst postdoped with nitrogen exhibits a significantly higher ADMFC performance compared to an unmodified version of the same catalyst and demonstrates a 32% increase in ECSA and a 10–17% increase in power density. The increased performance of PtRu/C after doping with nitrogen is at least partially attributed to its higher ECSA. In addition to the effects discussed in this work, based on our previous examinations in acid-based MEAs, N-doped PtRu/C might also improve the long-term durability of the ADMFC. The improved durability of the nitrogen-doped PtRu/C in acid-based DMFCs has been demonstrated in our previous studies[8,15,22,23] and investigation of the long-term durability of the ADMFC is currently underway.

Experimental Section

Preparation of the N-postdoped PtRu/C catalyst

The commercial 60 wt% PtRu/C (JM Hi-spec 10000) sample was implanted with nitrogen as reported elsewhere.[10] Briefly, approximately PtRu/C catalyst (500 mg) was placed in a rotating sample holder, and the chamber was evacuated to less than 5×10^{-6} Torr (1 Torr = 133.3 Pa).[11,12] Prior to nitrogen implantation, powders were outgassed by heating to above 180 °C for 15 min. Samples were then implanted by using a 3 cm DC ion source (Veeco) at a pressure of 1×10^{-3} Torr (N_2) and a beam current of 45 mA with a constant discharge voltage (55 V) and acceleration voltage (100 V) for 60 min.

Physiochemical characterization of the N-postdoped PtRu/C catalyst

XPS analysis was performed by using a Kratos Nova X-ray photoelectron spectrometer using energies of 160 eV for the survey spectra and 20 eV for the high-resolution spectra of C 1s/Ru 3d, N 1s, Ru 3p, and Pt 4f. Details of the processing can be found elsewhere.[16] TEM analysis of PtRu nanoparticles before and after postdoping were recorded by using a Philips CM200 transmission electron microscope.

MEA fabrication

The MEAs were 5 cm^2 in area and were fabricated using a spray-coating method described elsewhere.[13] The catalyst inks for the anode and the cathode were prepared by mixing appropriate amounts of 60 wt% PtRu/C (JM Hi-spec 10000; control as well as

N-postdoped catalysts) and 40 wt% Pt/C (JM Hi-spec 4000) with isopropyl alcohol and 30 wt% of alkaline ionomer solution. The prepared PtRu/C and Pt/C homogeneous catalyst inks were sprayed on either side of the Tokuyama A-201 membranes. The anode and cathode catalyst loadings were 3 and 2 mg cm^{-2}, respectively. Finally, the MEAs were hot pressed at 100 °C under a load of 150 kg for 3 min.

ADMFC testing

To test ADMFC performance, the MEAs were assembled in single-cell test fixtures by placing non-Teflonized microporous carbon paper (Toray-060) and Teflonized microporous carbon paper (Toray-060-40%) that acted as GDLs on the anode and cathode side of the MEAs, respectively. Serpentine-type graphite separators with channel dimensions of 1 mm × 1 mm (depth × width) were employed. After assembling the MEAs, 1 M NaOH solution followed by deionized water were fed to the anode side of the MEA for 5 and 10 h, respectively, at 60 °C. Subsequently, preconditioning was performed using humidified hydrogen/air. Initially, the cell was preconditioned by discharging at 0.6 V for 2 h by feeding humidified H_2 (105 cm^3 min^{-1}) and air (350 cm^3 min^{-1}) to the anode and cathode, respectively, at 80 °C. The cell voltage was then maintained at 0.6 V for 10 min and 0.8 V for 5 min, and this discharging cycle was repeated for 10 h. After preconditioning, initial DMFC performance curves were obtained by feeding different concentrations of NaOH and methanol (2 mL min^{-1}) and humidified air/oxygen (300 cm^3 min^{-1}) to the anode and cathode, respectively, at ambient pressure and 80 °C. The ADMFC performance testing was performed galvanostatically by increasing the current in 15 min steps and the corresponding voltage values were recorded with a commercial test station (Teledyne Energy Systems, Inc., USA). The voltage values recorded for the last 5 min at each current step were averaged and used to plot the polarization curves.

Electrochemical characterization of MEAs

The ECSAs of the anodes were measured by MSV by first feeding 2 M NaOH + 1 M CH_3OH (2 mL min^{-1}) for 20 min and then deionized water (2 mL min^{-1}) for another 20 min to the anode by holding the potential at 0.1 V at 25 °C. The cathode was fed with humidified hydrogen (100 cm^3 min^{-1}) and acted as the DHE. Subsequently, the methanol stripping curves were collected in the potential region from 0.1–0.9 V at a scan rate of 5 mV s^{-1} at 25 °C. The ECSA of the cathode was measured by cyclic voltammetry (CV). The cathode of the MEA was fed with 2 M NaOH for 30 min at 25 °C before performing the CV measurement. The CV for the cathode was performed by purging H_2 (100 cm^3 min^{-1}) and N_2 (50 cm^3 min^{-1}) to the anode and cathode, respectively, in the potential region from 0–1.0 V at a scan rate of 20 mV s^{-1} at 25 °C. The ECSA was calculated from the charges associated with the hydrogen adsorption and desorption peaks formed in the potential region from 0–0.4 V vs. DHE during the CV. The MOR polarization curves were collected using LSV by feeding 2 M NaOH + 2 M CH_3OH (2 mL min^{-1}) and hydrogen (100 cm^3 min^{-1}) to the anode and cathode, respectively, in the potential region from 0–0.5 V at a scan rate of 5 mV s^{-1} at 80 °C.

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Keywords: electrochemistry · fuel cells · ion exchange · membranes · oxidation

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High performance: We demonstrate a high performance alkaline direct methanol fuel cell (ADMFC) that obtains high maximum power densities in oxygen as well as in air using a commercial PtRu/C catalyst. Upon doping the commercial catalyst with nitrogen, we are able to further improve the performance to the highest power densities demonstrated so far for an ADMFC. ■■ok? results should not be reported in the table of content to encourage the reader to read the original publication.■■

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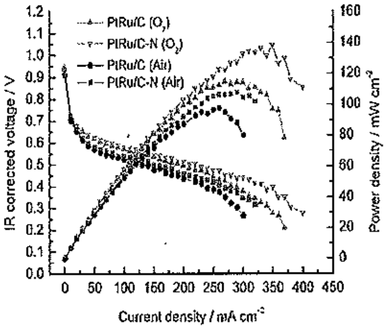

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High-Performance Alkaline Direct Methanol Fuel Cell using a Nitrogen-Postdoped Anode

Researchers @coschoolofmines describe a new N-doped #PtRu/C catalyst for #fuelcell anodes `SPACE RESERVED FOR IMAGE AND LINK`

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