



Development of carbon nanotubes based gas diffusion layers by *in situ* chemical vapor deposition process for proton exchange membrane fuel cells

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ABSTRACT

A proprietary *in situ* chemical vapor deposition (CVD) process was developed for gas diffusion layer (GDL) by growing a micro-porous layer on the macro-porous, non-woven fibrous carbon paper. The characteristics of the GDL samples such as, surface morphology, wetting characteristics, and cross-section were characterized using electron microscopes, goniometer and focused ion beam, respectively. Fuel cell performance of the GDLs was evaluated using single cell with hydrogen/oxygen as well as hydrogen/air at ambient pressure, at elevated temperature and various RH conditions using Nafion-212 as an electrolyte. The GDLs with *in situ* growth of micro-porous layers containing carbon nanotubes (CNTs) without any hydrophobic agent showed significant improvement in mechanical robustness as well as fuel cell performance at elevated temperature at lower RH conditions. The micro-porous layer of the GDLs as seen under scanning electron microscope showed excellent surface morphology with surface homogeneity through reinforcement by the multi-walled CNTs.

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1. Introduction

Proton exchange membrane fuel cells (PEMFCs) are attracting considerable interest as alternative power sources for automotive, stationary and portable applications due to their higher power densities and environmental benefits. However, when air is used as oxidant the power density values are reduced due to mass transport limitations primarily at the cathode. GDL is one of the critical components of a fuel cell that has the ability to influence the H₂/air system performance at high current density region. The essential functions of gas diffusion layers in a fuel cell are: distribution of reactants to the active site of electrode (pore size as well as its distribution), management of water supplied and/or generated and enhancement of electrical contact between the electrode and the bipolar plates.

Hence, the ideal GDLs should have good gas diffusion properties with suitable permeability for allowing the reactant gases into the reaction zones, good water transport characteristics for water removal from the reaction sites avoiding flooding on the catalyst particles, good electronic conductivity for electron transfer between catalyst layer and the flow field plate, good thermal conductivity for temperature uniformity within the cell as well as for thermal management, crack-free surface morphology and

high mechanical integrity to sustain erosion resistance from the gas forces to avoid any particle shedding and enhanced oxidative stability for enhanced durability [1]. The water retaining (hydrophilicity) and water expelling (hydrophobicity) properties of the GDLs have to be carefully balanced to achieve optimal performance of the fuel cell without flooding when it operates under the condition of 100% RH. The hydrophilicity of the gas diffusion layers can also avoid drying of electrolyte at lower RH conditions. The motivation and the objective for the present study are to develop GDLs which will have superior fuel cell performance for automotive operating conditions. This will involve wider operating conditions like all current densities and lower and higher RH conditions.

There are many publications in the literature discussing various types of carbon for micro-porous layers for improved gas diffusion characteristics of GDLs in PEMFCs [2–4]. Recently, Kannan et al. have reported the use of CNTs as a better material for the fabrication of micro-porous layer for gas diffusion layer compared to Vulcan-XC72R [5]. Several GDL fabrication techniques have been investigated in the past, which include low-shear coating, rod coating, screen-printing, doctor blade, impregnation, etc. [6–11]. It is worth mentioning that the dry fabrication involves dry deposition of a mixture of carbon black and polytetrafluoroethylene powders onto water-proofed carbon paper in combination with a subsequent rolling process [12]. Three types of carbon materials, including Ketjenblack EC-600JD, Vulcan-XC72 and Denka, were investigated in the dry method. However, most of these methods including the

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dry powder method of GDL fabrication appear to be slow and are difficult to adopt for mass production.

Even though there are many publications addressing the direct growth of CNTs on carbon substrates, the application of the *in situ* grown CNTs as a possible gas diffusion layer has not yet been fully exploited for fuel cell applications [13–21]. The directly grown CNTs on carbon paper substrates have also been used for depositing Pt catalyst, but the fuel cell performance is rather low [16–21]. The present study focuses on the design and development of *in situ* grown CNTs as micro-porous layer by a proprietary CVD process, without any binding agent on the macro-porous carbon paper, as GDLs for efficient operation of PEMFCs at various RH conditions using Nafion-212 membranes at 80 °C at ambient pressure. The single cell fuel cell performance with CNT-based gas diffusion cathode exhibited a current density value of $>2 \text{ A cm}^{-2}$ at 0.4 V at 80 °C, 70% RH of H_2 and O_2 at ambient pressure.

2. Experimental

2.1. Gas diffusion layer fabrication and characterization

In order to nucleate the growth of nanotubes, the carbon paper substrate provided by Hollingsworth and Vose company (West Gorton, MA) were surface modified by a proprietary process prior to *in situ* growth of carbon nanotubes by chemical vapor deposition (CVD). Once the CVD process temperature stabilizes at 800 °C, the carbon source along with catalyst (xylenes/ferrocene mixture) was injected into the pre-heater using syringe pump at a pre-determined rate. The xylenes/ferrocene vapor was carried by Ar carrier gas ($\sim 500 \text{ cm}^3 \text{ min}^{-1}$) into the reaction zone, where the carbon paper substrate was placed. The CVD process temperature was maintained for 30 min at 800 °C necessary for the *in situ* growth of CNTs. The furnace was allowed to cool in flowing Ar atmosphere to about 200 °C and the sample was removed. A dark black layer of CNTs was found on the top side of the carbon paper. It was observed that pristine carbon paper samples without any surface modification did not lead to any CNT growth at 800 °C.

For comparison purposes, GDL samples were also fabricated by wire-rod coating method using carbon slurry (75 wt.% Pure-black carbon and 25 wt.% nano-fibrous carbon (VGCF) with PTFE dispersion (25 wt.%) as described in our earlier publication [22]. The surface morphology of the GDL samples was examined by JEOL JSM-5900LV Scanning Electron Microscope. The nature of the *in situ* grown micro-porous layer was analyzed by dispersing the harvested sample in methanol for applying it on a lacey carbon grid to examine by TEM using Philips CM200-FEG. The wetting characteristics (contact angle) of *in situ* grown CNT-based micro-porous layer on the carbon paper GDL as well as the catalyst coated GDL samples were analyzed by a Ramehart Advanced Automated Goniometer using water drop. The surface profile (including the summit and valley) of the micro-porous layer was examined by a MicroXAM Laser Interferometric 3D Surface Profiler (Model #STD EX100-ENC, ADE Phaseshift (KLA-Tencor), San Jose, CA) by non-contact method. The bulk characteristics of the multi-walled carbon nanotubes (MWCNTs) based micro-porous layer of the GDLs were examined using Focused Ion Beam (Nova200 NanoLab) with integrated 3D-characterization and nano-machining by a gallium ion gun. The pore diameter and pore size distribution of the GDL samples were measured by using PoreMaster-60 GT in both low and high pressure modes by Hg intrusion method in a fixed speed mode.

2.2. Catalyst deposition on membrane and GDL

Catalyst coated membrane and the gas diffusion electrode (GDE) were fabricated as follows. Catalyst ink was prepared by

adding isopropanol (20 ml for 1 gm of electrocatalyst) after purging the Pt/C catalyst powder (TKK, Japan) in flowing nitrogen gas for about 30 min to avoid any flame/ignition. In order to extend the reaction zone of the catalyst layer, 5% Nafion® (Ion Power Inc., New Castle, DE, USA) solution (10 ml Nafion solution for 1 gm of electrocatalyst) was added to the catalyst slurry. Catalyst coatings on Nafion membrane (NRE 212, Ion Power Inc., New Castle, DE, USA) as well as on GDL with 5 cm² active area were fabricated as anode and cathode, respectively by spraying Pt/C catalyst ink using the micro-spray method. The catalyst loadings on the anode and cathode catalyst layers were about 0.5 and 1 mg Pt cm⁻², respectively. The catalyst coated Nafion-212 membrane (0.5 mg Pt cm⁻²) and the gas diffusion electrodes (1 mg Pt cm⁻²) were vacuum dried at about 70 °C for an hour before assembling them in the fuel cell. The particle size and the distribution of Pt catalyst particles deposited on the *in situ* grown micro-porous layer were harvested from the carbon paper substrate and examined by the TEM (Philips CM200-FEG) by dispersing the harvested sample in methanol for applying it on a lacey carbon grid.

2.3. Membrane electrode assembly and fuel cell performance

The GDE cathode and the catalyst coated membrane (CCM) anode were assembled by just sandwiching inside the fuel cell (Fuel Cell Technologies Inc., Albuquerque, NM, USA) along with a GDL at the anode side. Gas sealing was achieved using silicone coated fabric materials (Product #CF1007, Saint-Gobain Performance Plastics, USA) and with a uniform torque of 0.45 kg m. The single cell fuel cell performance for various GDLs was evaluated at 80 °C at various RH conditions (100, 90, 80 and 70%) using Greenlight Test Station (G50 Fuel cell system, Hydrogenics, Vancouver, Canada). The flow rates of gases were fixed at 200 and 400 SCCM for H_2 and O_2 (or air), respectively. The steady state voltage values were collected by holding the cell at each current density value for 60 s.

3. Results and discussion

Fig. 1 shows the scanning electron micrographs of *in situ* grown MWCNTs on carbon paper. Each fiber of the macro-porous carbon paper is homogeneously covered by the micro-porous layer of CNT as shown in Fig. 1a. Fig. 1b shows a higher magnification SEM image of a single fiber of the carbon paper. As evident from this image, the micro-porous layer comprises nanotubes along with spherical (more oblong) carbon—a composite layer. It is interesting to see that the nanotubes are not ordered and they are entangled both in and through the plane to provide structural integrity of the micro-porous layer to the GDL substrate. The structural robustness and the effectiveness of adhesion of the composite nanotubes based micro-porous layer to the macro-porous layer were attained by subjecting the GDLs to ultrasonication.

In order to identify the nature of the *in situ* growth, the samples were also examined in TEM. As seen in Fig. 2a, the *in situ* growth on the carbon paper in the CVD process is MWCNT with an OD of $\sim 40 \text{ nm}$ and an ID of 10 nm. And the high resolution/high magnification TEM image ascertained that the CNT is highly crystalline (Fig. 2b). Fig. 3a and b shows the TEM images of the catalyst coated CNTs from the micro-porous layer of the GDLs. As seen from the images, the catalyst distribution is highly homogenous with particle sizes between 3 and 5 nm.

In order to view the *in situ* grown micro-porous layer to a depth of about 5 μm , nano-machining was carried out on the GDL surface by gallium ion gun, *in situ*. The possible damage to the GDL surface during the nano-machining was avoided by plating a thin

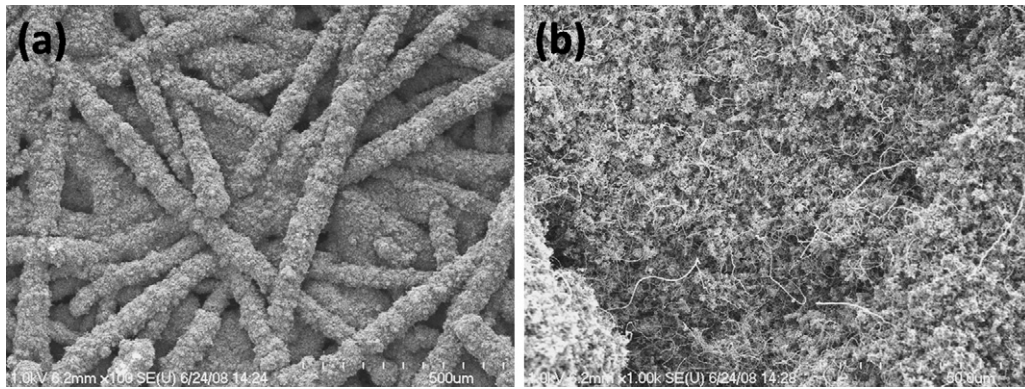


Fig. 1. SEM images of GDL surface fabricated by using *in situ* CVD on carbon paper at: (a) low (100 \times) and (b) high (1000 \times) magnifications.

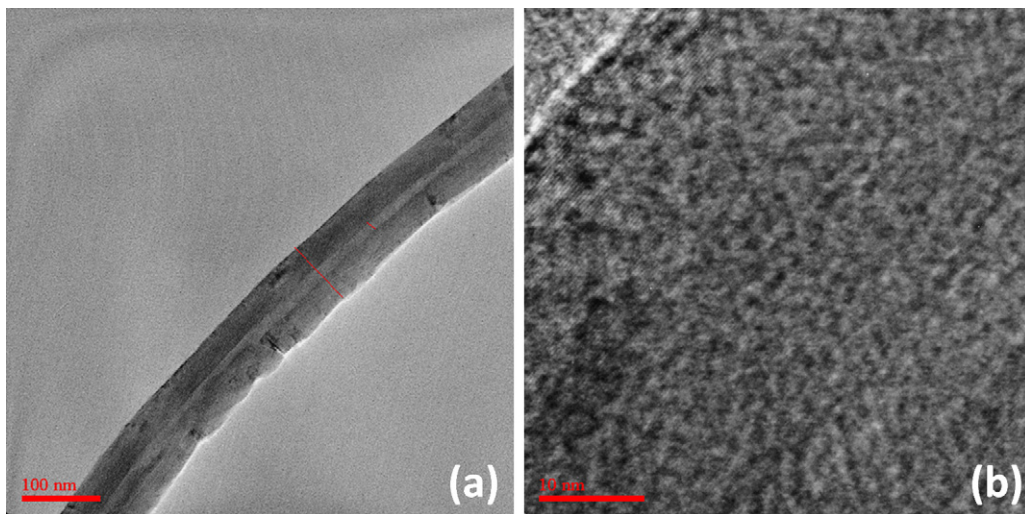


Fig. 2. Transmission electron micrographs of a multi-walled carbon nanotube: (a) low magnification showing the chain structure and (b) high magnification showing well defined crystallinity of the nanotube.

layer of platinum on the surface. The presence of porous nature within the bulk of the micro-porous layer is evident from the FIB images in Fig. 4. The pores within the micro-porous layer could contribute significantly to the low RH operating conditions in keeping the electrolyte wet. Fig. 4 shows the bulk nature of the micro-porous layer.

In order to understand the hydrophilic/hydrophobic characteristics, the *in situ* grown CNT-based micro-porous layer on the carbon paper GDL samples were analyzed by measuring wetting angle in a Ramehart goniometer. The wetting angle is the angle that the tangent makes with the point of contact of the drop with the surface under study; the surface characteristics and its

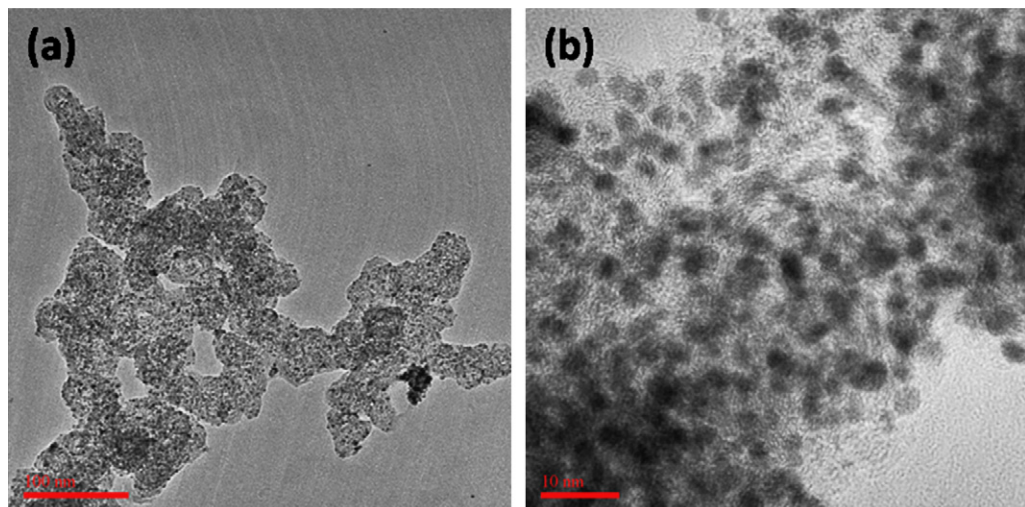


Fig. 3. Transmission electron micrographs of Pt deposited multi-walled carbon nanotubes: (a) low magnification and (b) high magnification.

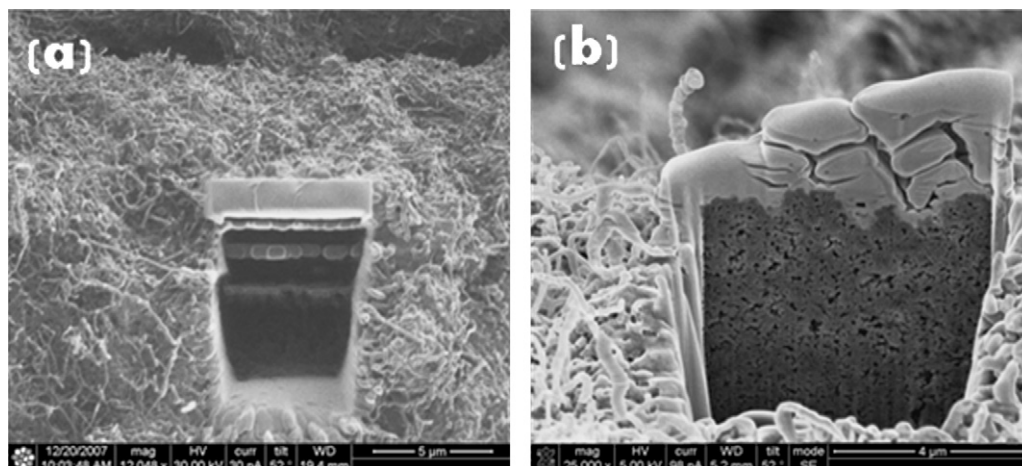


Fig. 4. FIB image for the cross-section of micro-porous layer of the *in situ* GDL showing the presence of porous nature.

constituents influence this property. The wetting angle profiles using water droplet are compared for (a) plain carbon paper, (b) surface modified carbon paper, (c) *in situ* CNT grown micro-porous layer sample on the carbon paper and (d) Pt deposited CNT surface are compared in Fig. 5. As seen from Fig. 5(b), the wetting angle of the surface modified carbon paper is very low compared to the plain paper. However, once the CNTs are grown *in situ* by the CVD process, the wetting angle increased to $\sim 145^\circ$ due to the hydrophobic characteristics of the multi-walled carbon nanotubes (Fig. 5c). The hydrophobicity increases to about 150° on the Pt catalyst coated CNT-grown carbon paper (Fig. 5d). In general, the composite micro-porous layer grown *in situ* by the CVD process is more hydrophobic compared to the fibrous carbon-based macro-porous layer. Wetting characteristics of the micro-porous layer

will be useful as an *ex situ* property for designing GDLs for specific operating condition of the fuel cells. The contact angle image and the value for the wire-rod coated GDLs were given in our previous publication [22]. The contact angle value for the wire-rod coated GDLs with 1:1 water and ethanol mixture was about 120° . However, the contact angle value for the MWCNTs/carbon paper GDLs were measured with only water and hence could not be compared.

Fig. 6 compares the topography of MWCNT-based micro-porous layer of the GDL with a plain carbon paper substrate. The 3D image of the surface appears to be very non-uniform compared to the blank carbon paper as we can observe lot of crests and troughs. The white areas depict the highest points on the surface and the dark red regions are the lowest points on the surface. The yellow areas are the levels between the highest region and the lowest region.

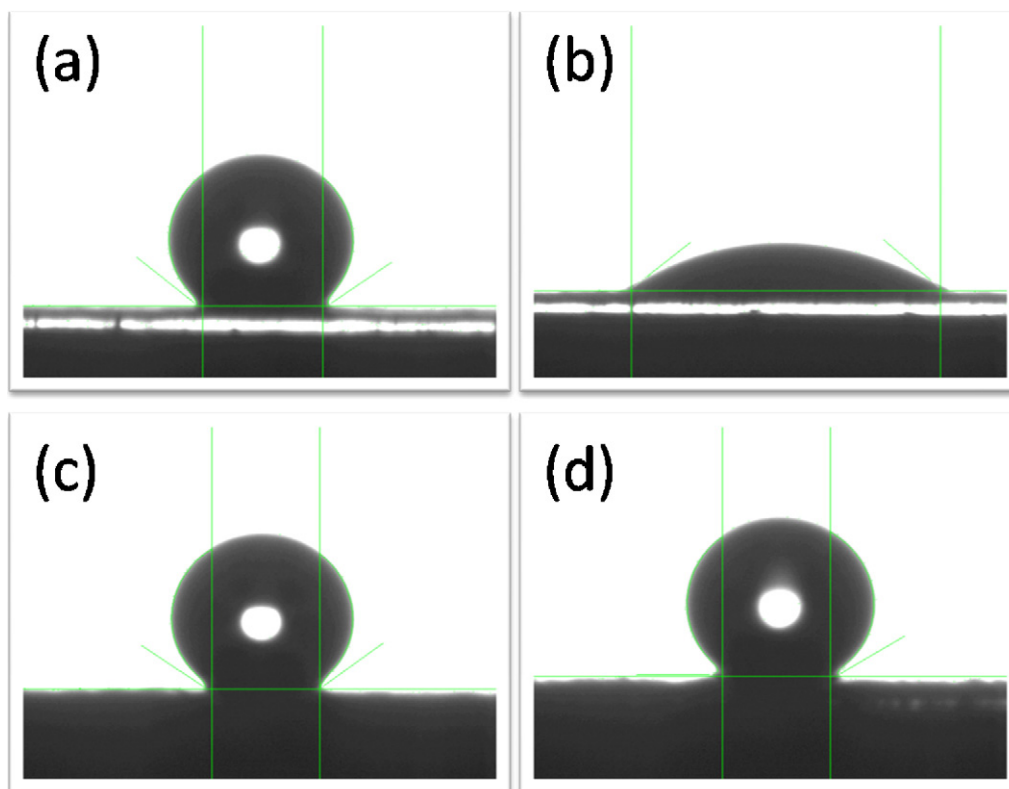


Fig. 5. Wetting angle images of (a) plain carbon paper, (b) surface modified carbon paper, (c) *in situ* CNT grown carbon paper and (d) Pt deposited surface of the *in situ* CNT grown carbon paper. The wetting angle values are 143° , 35° , 145° and 150° , respectively.

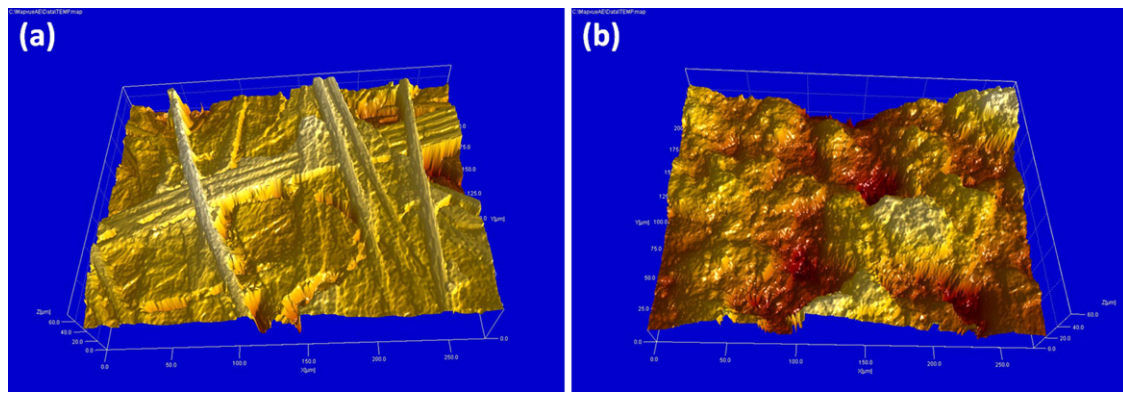


Fig. 6. Laser interferometry images: (a) plain carbon paper and (b) *in situ* MWCNTs grown on carbon paper.

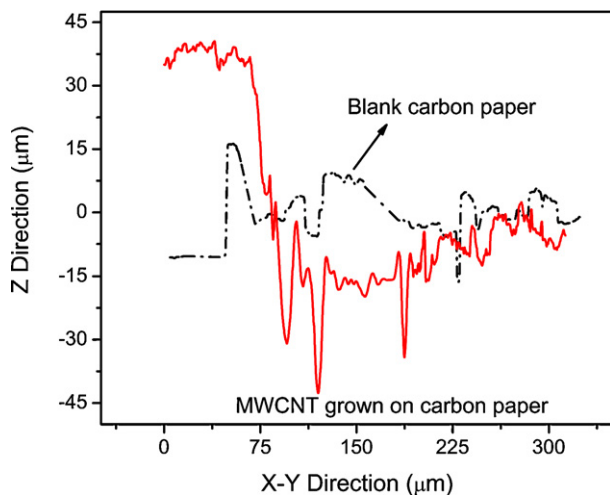


Fig. 7. Laser interferometry profile data for CNT/carbon paper along with that for plain carbon paper.

The Z-range depth profile of carbon paper along with that for the CNT-based GDL is shown in Fig. 7. It can be observed that the carbon paper has got relatively uniform surface compared to the CNT surface. But still we can observe some non-uniform surface of the carbon paper as it contains the non-woven fibers.

Fig. 8 compares the pore size distribution data measured by Hg porosimetry in low and high pressure modes for Pore size distribution.

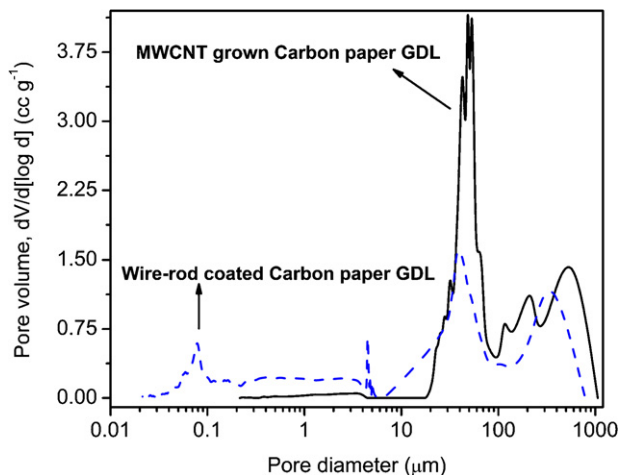


Fig. 8. Pore size distribution of *in situ* grown CNT-based GDL and wire-rod coated GDL.

of *in situ* grown CNT-based GDL and wire-rod coated GDL samples. As observed from the pore size distribution data in Fig. 8, the GDL with Pureblack and nano-fibrous carbons in the micro-porous layer fabricated by wire-rod coating show pores with 0.075, 5, 40 and 225 μm diameters. However, the *in situ* grown CNT-based GDL shows only larger pores with 30 μm diameters. The CNT-based GDLs without Teflon as binder is preferable to hold product water in the fuel cell working at reduced RH conditions, even though the pores are not very narrow compared to that with the wire-rod coated GDLs. The relatively larger pores in the MWCNTs based micro-porous layer-based GDLs could lead to better gas diffusion (less hindrance) as well as quicker removal of product water even at higher RH conditions.

Fuel cell performance data at 80 °C at different RH conditions using oxygen and air as oxidants using Nafion-212 membrane are given in Figs. 9 and 10, respectively. The *in situ* CNT/Carbon paper GDLs show excellent performance both using H_2/O_2 and H_2/air at all RH conditions. The cell voltage (or power density) values remain fairly stable at all RH conditions. The peak power density of the wire rod coated GDLs based MEA is higher than that of the MWCNT-based GDLs at 100 and 90% RH. However, the peak power density value (810 mW cm^{-2}) is identical for both wire-rod and MWCNT-based GDLs at 80% RH. But, the peak power density value of the MEA with wire-rod coated GDLs reduced to 655 mW cm^{-2} and that of the MWCNT-based GDLs showed >780 mW cm^{-2} at 70% RH. The reason for better fuel cell performance with MWCNTs/carbon paper GDLs at 100% RH without any electrode flooding is mainly due to the hydrophobic property of the MWCNTs which decorated homogeneously the fibers of the carbon paper, as seen from the SEM images in Fig. 1. In addition, the pore diameter of the micro-porous layer

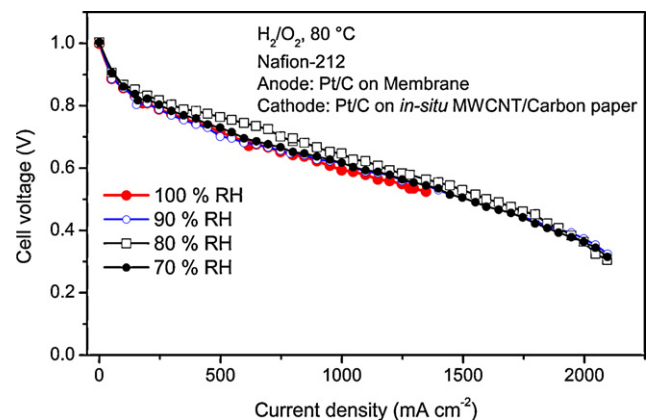


Fig. 9. Fuel cell polarization data for cathode using Pt deposited on the *in situ* MWCNT/carbon paper-based GDL at 80 °C using H_2/O_2 at various RH conditions.

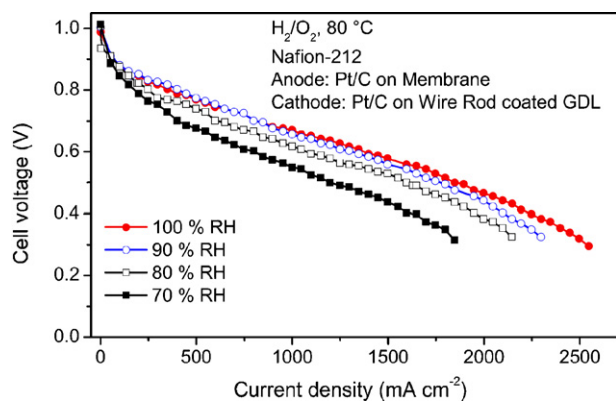


Fig. 10. Fuel cell polarization data for cathode using Pt deposited on the *in situ* MWCNT/carbon paper-based GDL at 80 °C using H₂/air at various RH conditions.

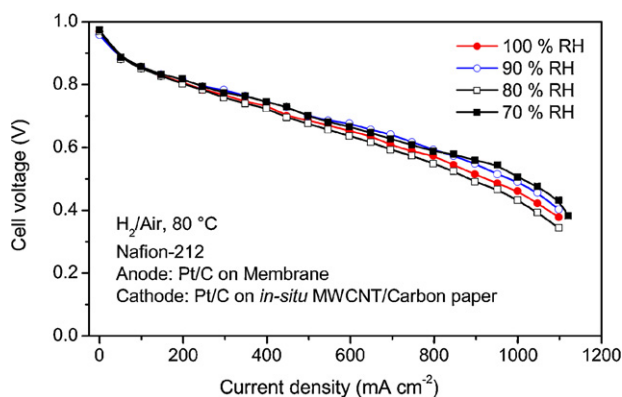


Fig. 11. Fuel cell polarization data for cathode using Pt deposited on the wire-rod coated GDL at 80 °C using H₂/O₂ at various RH conditions.

of the MWCNTs/carbon paper GDLs is also much bigger leading to quicker removal of the product water avoiding flooding.

The observed fuel cell performance with the MWCNTs grown on carbon paper could not be compared with the literature as the reported values are with back pressure [16–18]. In order to compare the fuel cell performance, wire-rod coated GDLs were also evaluated at 80 °C using H₂/O₂ and H₂/air at various RH conditions. As seen from Figs. 11 and 12, there is a significant reduction in cell voltage (or power density) values at lower RH values with electrodes using wire-rod fabricated GDLs. The operating RH inside the cell may be different than what is calculated and presented here based on the water bottle temperature and the cell temperature. However,

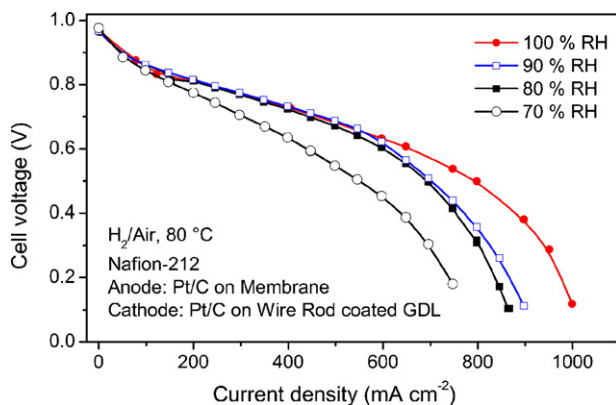


Fig. 12. Fuel cell polarization data for cathode using Pt deposited on the wire-rod coated GDL at 80 °C using H₂/air at various RH conditions.

fuel cell performance comparison in Figs. 9–12, the test conditions were kept identical. As the pores in the micro-porous layer of the *in situ* CNT-based gas diffusion electrodes are relatively larger, there is no water flooding when the RH of the reactant gases is 100%. The main reason for the superior performance even at reduced RH condition, of the CNT/carbon paper is due to the fact that it does not have Teflon leading to better hydrophilicity and higher electronically conducting network [14]. The presence of porous nature within the bulk of the CNT-based micro-porous layer, as observed in FIB was also responsible for the significant performance improvement at the low RH operating conditions in keeping the electrolyte wet. The micro-porous layers of the GDLs fabricated by the wire-rod method had 25 wt.% Teflon.

4. Conclusions

Highly efficient CNT-based GDLs were developed by a proprietary *in situ* CVD process using a commercial macro-porous, non-woven fibrous carbon paper without any binding agent/hydrophobic component. The surface morphology of the CNT-based micro-porous layer showed excellent surface homogeneity without any cracks. The composite layer also showed exceptional mechanical integrity through reinforcement by the carbon nanotubes. The bulk of the micro-porous layer, as examined by the FIB exhibited porous characteristics. The TEM images of the CNTs grown *in situ* were multi-walled and crystalline with an OD of ~40 nm and an ID of 10 nm. PEMFC data of the GDLs evaluated using single cell with hydrogen/oxygen as well as hydrogen/air at ambient pressure, at elevated temperature using Nafion-212 as an electrolyte showed very stable performance down to 70% RH conditions due to the fact that the micro-porous layers were without any hydrophobic agent like Teflon. The cost analysis and viable mass production methods of the MWCNTs/carbon paper GDLs which are important for commercialization are not considered in the present study.

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