



# Synthesis and characterization of platinum nanoparticles on *in situ* grown carbon nanotubes based carbon paper for proton exchange membrane fuel cell cathode

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## ABSTRACT

Multi-walled carbon nanotubes (MWCNTs) based micro-porous layer on the carbon paper substrates was prepared by *in situ* growth in a chemical vapor deposition setup. Platinum nanoparticles were deposited on *in situ* grown MWCNTs/carbon paper by a wet chemistry route at <100 °C. The *in situ* MWCNTs/carbon paper was initially surface modified by silane derivative to incorporate sulfonic acid–silicate intermediate groups which act as anchors for metal ions. Platinum nanoparticles deposition on the *in situ* MWCNTs/carbon paper was carried out by reducing platinum (II) acetylacetonate precursor using glacial acetic acid. High resolution TEM images showed that the platinum particles are homogeneously distributed on the outer surface of MWCNTs with a size range of 1–2 nm. The Pt/MWCNTs/carbon paper electrode with a loading of 0.3 and 0.5 mg Pt cm<sup>-2</sup> was evaluated in proton exchange membrane single cell fuel cell using H<sub>2</sub>/O<sub>2</sub>. The single cells exhibited a peak power density of 600 and 800 mW cm<sup>-2</sup> with catalyst loadings of 0.3 and 0.5 mg Pt cm<sup>-2</sup>, respectively with H<sub>2</sub>/O<sub>2</sub> at 80 °C, using Nafion-212 electrolyte. In order to understand the intrinsically higher fuel cell performance, the electrochemically active surface area was estimated by the cyclic voltammetry of the Pt/MWCNTs/carbon paper.

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## 1. Introduction

Renewable energy technologies are highly relevant for the current fuel crisis scenario faced by the entire world. Among various fuel cell types, proton exchange membrane fuel cells (PEMFCs) attract the foremost attention for light duty transportation, small-scale power generation for stationary/residential and portable power applications due to their lower operating temperature and (below 100 °C) [1]. However, the economical factors of the PEMFC components are the primary limitations for their early commercialization. Tremendous efforts are being taken by the US Department of Energy to advance the research and developmental activities addressing the performance, cost and durability aspects of PEMFCs [2]. Improving the utilization of expensive noble metal catalysts and development of high power membrane electrodes assemblies (MEAs) are the focus areas in many R&D laboratories through more accessible nanosize catalyst sites on durable catalyst support materials like CNTs and thinner active catalyst layers [3].

Platinum supported on high surface area carbon have been the most effective catalyst for PEMFCs due to their activity towards

oxygen reduction reaction at lower temperatures [4]. Various high-surface area carbon materials such as Vulcan XC-72R, single-walled CNT, multi-walled CNT, carbon fibers and aerogels have been investigated for support materials. It has been reported that the CNTs based supports have shown significant improvement in performance by providing high surface area for catalyst and also effective micro-porous layer for improved gas distribution [5,6]. Several studies have successfully used CNTs grown directly on the carbon paper substrate as electrocatalyst supports [7,8]. Another advantage of CNTs as catalyst support is improved oxidation resistance. However, their durability and stability in fuel cell operating conditions are not very clearly understood in the literature. More, recently Kannan et al. have developed a proprietary method for *in situ* growth of MWCNTs based micro-porous layer as well as catalyst support without using any hydrophobic agent that showed significant improvement in mechanical robustness as well as fuel cell performance at elevated temperature and low RH conditions [9].

Nanosized catalyst particles show unique properties such as high specific surface area and superior catalytic activity yielding higher performance with lower catalyst loadings. Numerous methods such as impregnation, electrodeposition, thin-film method, and sputter deposition have been reported for fabricating electrodes with low Pt-loading electrocatalyst for PEMFCs [10–14]. Low temperature route by a polyol process has been widely studied as it

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provides control over the Pt particles size and distribution without any stabilizers [15]. For example, Liu et al. have reported 2–6 nm Pt particles supported on Vulcan XC-72R as well as CNTs using microwave assisted polyol process [16]. Several reports on functionalizing CNTs with Pt nanoparticles have utilized both organic [17,18] and inorganic [19] reducing agents. However, high dispersion of Pt nanoparticles with controlled catalyst loading is still a challenge due to the inert surfaces of CNTs. Essentially, different non-covalent and covalent modifications methods have been investigated to functionalize CNTs for improved catalyst dispersion and molecular interaction [20]. Oxidation in strong acids or acid mixtures ( $\text{HNO}_3$ ,  $\text{HNO}_3 + \text{H}_2\text{SO}_4$ ) has been the chief route to incorporate carboxyl, carbonyl and hydroxyl groups on CNTs. However, citric acid treatment was reported to be a mild, more effective and facile method to functionalize CNTs [21]. Sun et al. have reported homogenous dispersion of platinum particles on MWCNT grown on carbon paper using silane surface modifying reagent [22].

In the current investigation, we have developed a MWCNTs based micro-porous layer without using any binding/hydrophobic agent by *in situ* growth on a carbon paper substrate through CVD process. A simple process to obtain homogenous dispersion of Pt nanoparticles with narrow distribution of particle size, on the *in situ* MWCNTs/carbon paper substrates using glacial acetic acid, was also developed. The organo-silane surface modification reagent was used for homogeneous dispersion of Pt particles on MWCNTs for high performance PEM fuel cell electrodes. The MEAs fabricated with Pt/MWCNTs/carbon paper show performance up to  $800 \text{ mW cm}^{-2}$ . The superior performance could be explained by the electrochemically active surface of about  $50 \text{ m}^2 \text{ g}^{-1}$ , as measured from the cyclic voltammetric data.

## 2. Experimental

### 2.1. *In situ* CNT growth on carbon paper

The fabrication of gas diffusion layer (GDL) is reported elsewhere in detail [9]. In brief, the carbon paper substrate (Hollingsworth and Vose Company, West Groton, MA) was surface modified on one side by a proprietary process in order to nucleate the MWCNTs growth, prior to the *in situ* chemical vapor deposition (CVD) process. In the CVD process the furnace was allowed to stabilize at  $800^\circ\text{C}$  and the carbon source/catalyst (xylenes/ferrocene) mixture was injected at a predetermined rate into the reaction zone, where the surface modified carbon paper substrate was placed (the surfaced modified side facing upwards). After the completion of the CNT growth, the furnace was allowed to cool in flowing Ar atmosphere to about  $200^\circ\text{C}$ . A dark black layer of CNTs was formed as micro-porous layer, on the top side of the carbon paper where surface modification was carried out. The surface morphology of the *in situ* MWCNTs/carbon paper was examined by SEM (JEOL JSM-5900LV). Wetting characteristics of the GDL samples was evaluated by NRL CA Goniometer (Ramehart 100). The contact angle of a droplet of water and ethanol mixture (1:1 ratio) on the micro-porous layer was measured after 5 min. The pore diameter and pore size distribution of the MWCNTs/carbon paper based GDL were measured by using PoreMaster-60 GT in both low and high pressure modes by Hg intrusion method in fixed speed mode.

### 2.2. Platinum nanoparticles deposition

Since MWCNTs are hydrophobic in nature, the *in situ* grown MWCNTs/carbon paper was treated in 0.1 M citric acid for 1 h and

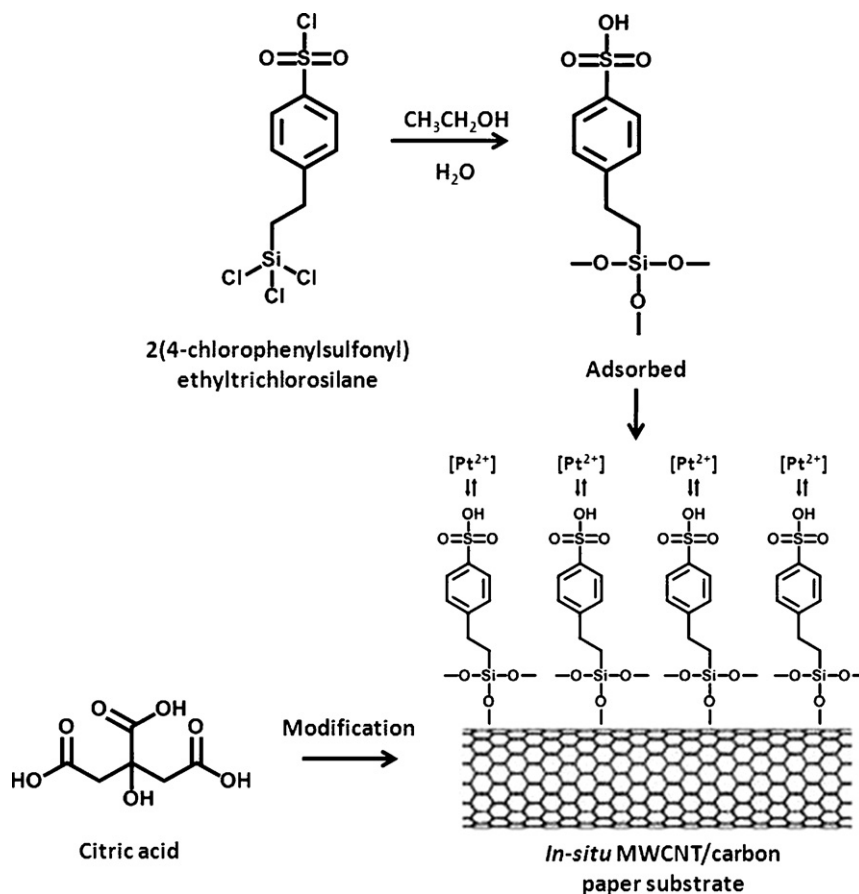


Fig. 1. Schematic representation of surface modification of *in situ* grown MWCNTs/carbon paper.

heat treated in muffle furnace at 300 °C for 30 min to improve the wetting characteristics. Citric acid treatment is a well-accepted process for functionalizing CNTs for incorporating carboxyl and hydroxide groups [21]. The *in situ* grown MWCNTs/carbon paper was further surface modified by immersing in a mixture of 1 cm<sup>3</sup> of 2(4-chlorosulfonylphenyl) ethyltrichlorosilane (50 wt.% in Toluene, Gelest Inc.) and 50 cm<sup>3</sup> of ethanol (70 vol.% in water) for 2 h to incorporate sulfonic acid-silicate groups on the exterior surface of MWCNTs. Fig. 1 gives schematically the silane modification process applied prior to Pt deposition step. The sulfonic acid-silicate intermediates are formed during hydrolysis in ethanol and are adsorbed on to CNT surface by coupling reaction of silanol group, whereas the non-silyl functionality has the ability to form hydrogen bond with polar groups in the solution phase [23].

The wet MWCNTs/carbon paper was then immersed in a solution of 10 wt.% platinum (II) acetylacetonate (Pt(II)acac) in acetone. Two different Pt loadings (0.3 and 0.5 mg cm<sup>-2</sup>) were obtained by varying the amount of Pt(II)acac in acetone. After about 2 h, 10 cm<sup>3</sup> of glacial acetic acid was slowly added to the above solution to reduce the Pt<sup>2+</sup> ions to Pt nanoparticles directly on the MWCNTs/carbon paper. The solution with the MWCNTs/carbon paper was heated on a hot plate which was maintained at <100 °C for 4 h under the vacuum hood in order to evaporate the solvent, during this period the pale yellow solution changed slowly to dark brown and finally to black upon complete evaporation of the solvent. The MWCNTs/carbon paper was thoroughly rinsed with methanol and DI water, successively to completely wash of the remaining acetic acid. The MWCNTs/carbon paper was dried in a vacuum oven overnight

to completely remove the solvent and further heat treated in a muffle furnace at 350 °C for 3 h in air. In order to determine the Pt particle size and their distribution on the *in situ* grown MWCNTs based micro-porous layer, a portion of the layer was harvested from the carbon paper substrate and examined in TEM (Philips CM200-FEG). TEM samples were prepared by dispersing the Pt/MWCNTs in methanol and applied on a lacey carbon grid.

### 2.3. Membrane electrodes assembly and fuel cell testing

The MEA consisting of the catalyst coated membrane (anode side) and the gas diffusion electrode (GDE) as cathode were fabricated as follows. Anode catalyst ink was prepared by adding isopropanol (20 cm<sup>3</sup> for 1 g 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 wt.% Nafion<sup>®</sup> (Ion Power Inc., New Castle, DE, USA) dispersion (10 cm<sup>3</sup> for 1 g of electrocatalyst) was added to the catalyst slurry. Catalyst layer on Nafion-212 (NRE 212, Ion Power Inc., New Castle, DE, USA) with 5 cm<sup>2</sup> active area was fabricated as anode by spraying Pt/C catalyst ink using the micro-spray method. The cathode consisted of the Pt deposited *in situ* MWCNTs/carbon paper sprayed with Nafion<sup>®</sup> dispersion (0.5 cm<sup>3</sup> of 15 wt.%). The catalyst loading on the anode was about 0.5 mg Pt cm<sup>-2</sup>. Catalyst loading on Pt deposited *in situ* MWCNTs/carbon paper was estimated from TGA studies. The catalyst coated Nafion-212 membrane and the GDE were vacuum dried at about 70 °C for an hour before assembling them in the fuel cell test cell. For

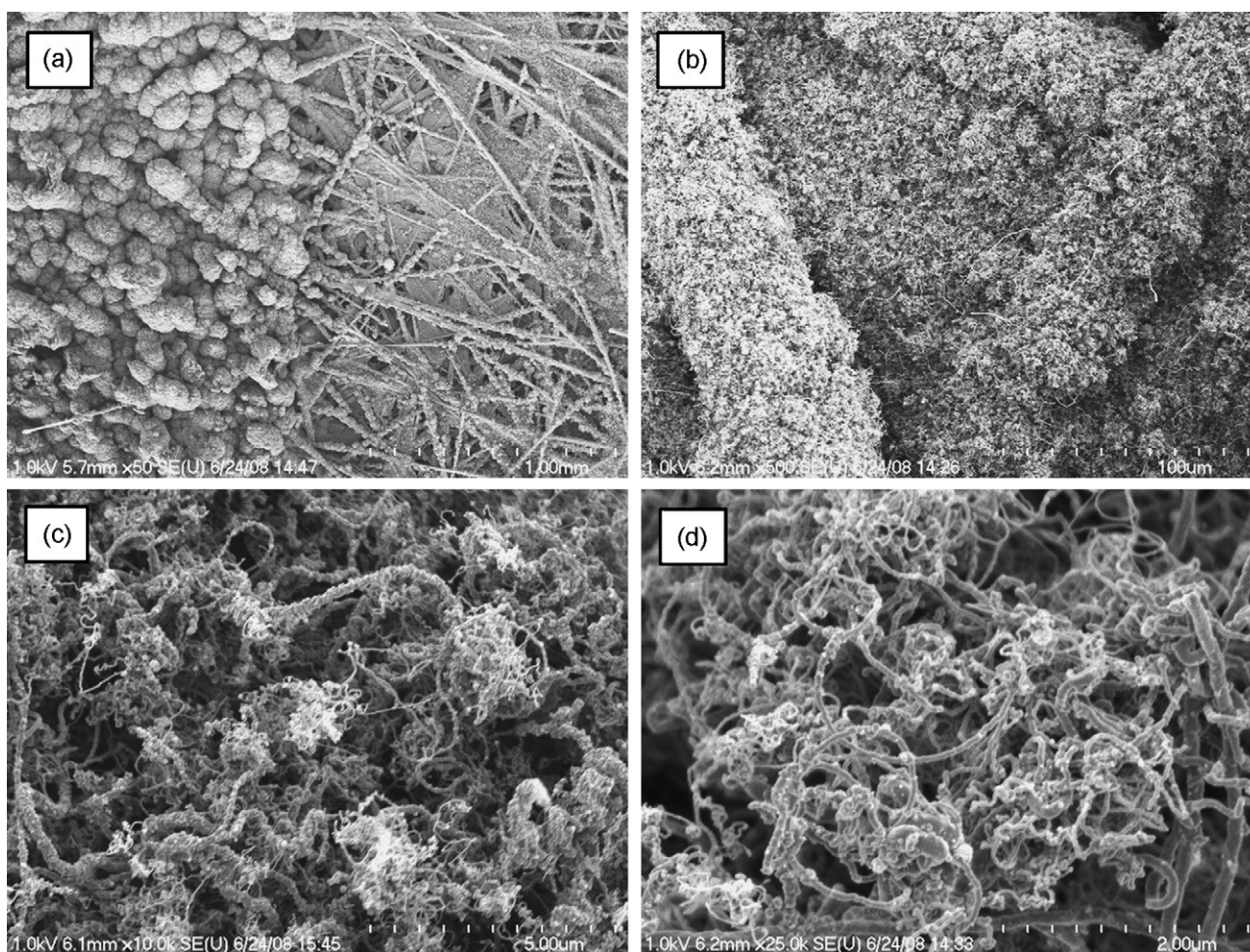


Fig. 2. SEM images of the *in situ* grown MWCNTs/carbon paper at various magnifications.



comparison purposes, the MWCNTs/carbon based GDLs were also evaluated by using catalyst coated membrane with Pt loading of  $0.5 \text{ mg cm}^{-2}$  (TKK, Japan), both on anode and cathode, respectively. Cyclic Voltammetry was carried out using PARSTAT for measuring Electrochemically Active surface area (ECA) of the MEAs. The test cell was equilibrated for about 2 h with humidified hydrogen and nitrogen gases at  $25^\circ\text{C}$  before the voltage scanning for CV measurement.

The Pt/MWCNTs/carbon paper based GDE cathode and catalyst coated membrane (CCM) anode were assembled by sandwiching inside the single cell test cell (Fuel Cell Technologies Inc, Albuquerque, NM, USA) along with a GDL (fabricated by a wire rod coating method) at the anode side. Gas sealing was achieved using silicone coated fabric gasket (Product # CF1007, Saint-Gobain Performance Plastics, USA) and with a uniform torque of  $0.45 \text{ kg m}$ . The single cell fuel cell performance was evaluated at  $80^\circ\text{C}$  at 100% RH using Greenlight Test Station (G50 Fuel cell system, Hydrogenics, Vancouver, Canada). The flow rates were fixed at 400 and 400 sccm for  $\text{H}_2$  and  $\text{O}_2$ , respectively. The steady state voltage values were collected by holding the cell at each current density values for 60 s.

### 3. Results and discussion

Pt(II)acac is one of the organo-metallic precursors commonly used for depositing Pt thin films via Metal-Organic CVD. The acetylacetonate anions form complexes by bonding each oxygen atom

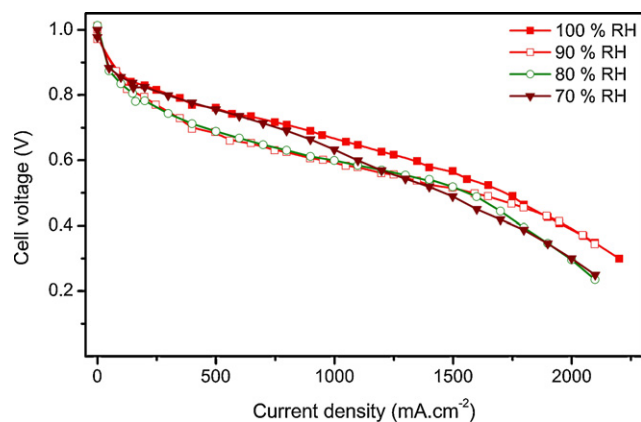


Fig. 3. Fuel cell performance of an MEA with MWCNTs/carbon paper electrode using catalyst coated ( $0.5 \text{ mg cm}^{-2}$ ) Nafion-212 membrane at  $80^\circ\text{C}$  and  $\text{H}_2/\text{O}_2$  at various RH conditions at ambient pressure.

to the metal cation to form a six-member chelate ring, due to this property these complexes are used as catalytic precursors and reagents. In the current study Pt(II)acac is reduced via wet-chemistry reduction method to deposit Pt nanoparticles on *in situ* grown MWCNTs/carbon paper. Pt(II)acac is highly soluble in organic solvents such as acetone making it convenient for reduction using

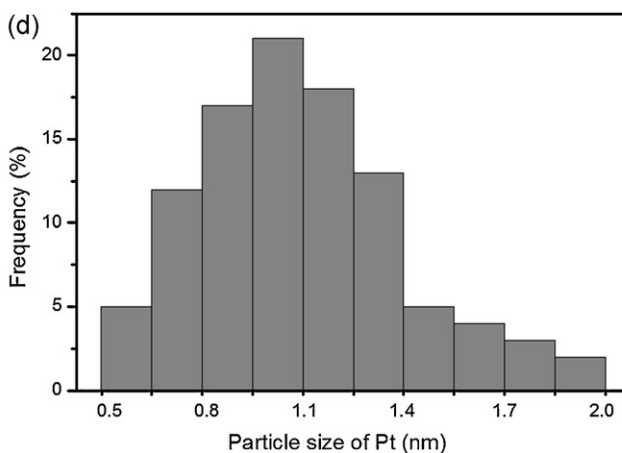
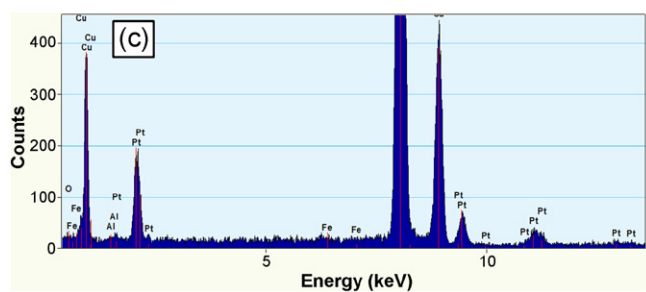
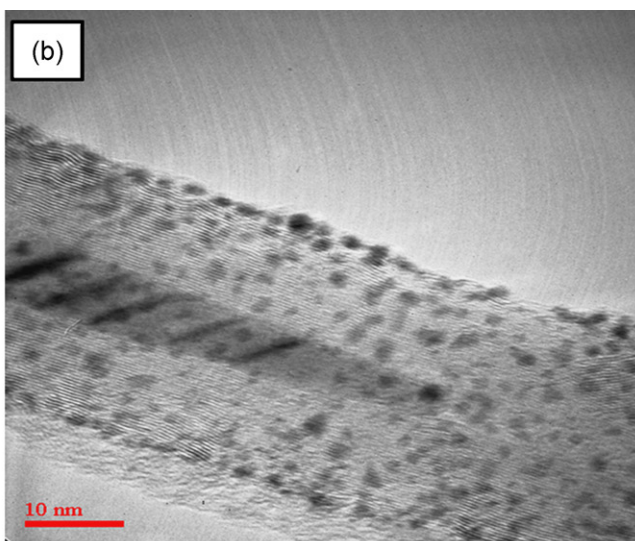
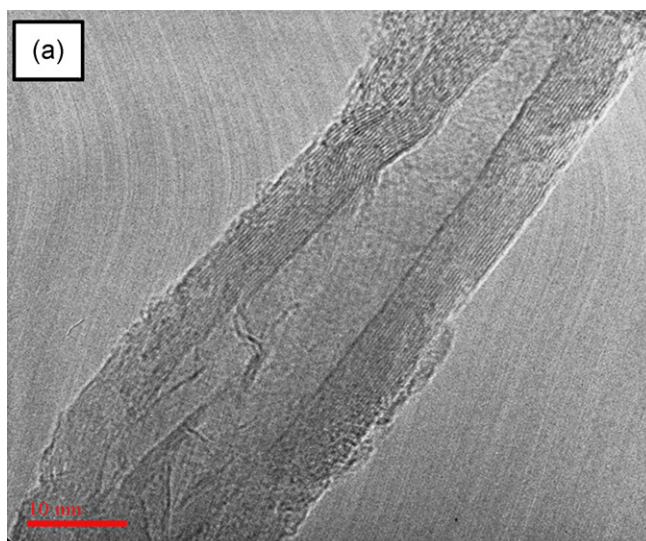


Fig. 4. High resolution TEM images of (a) pristine *in situ* MWCNT harvested from carbon paper substrate, (b) Pt deposited on *in situ* MWCNTs/carbon paper ( $0.5 \text{ mg Pt cm}^{-2}$ ), (c) EDAX pattern of Pt deposited *in situ* MWCNTs (from the image shown in 4(b)) and (d) frequency distribution of Pt particles on MWCNTs (from the image shown in 4(b)).

glacial acetic acid. Recently, Saha et al. have reduced Pt(II)acac by dissolving in glacial acetic acid to deposit monodispersed Pt nanoparticles on MWCNTs [24]. As given schematically in Fig. 1, the  $\text{Pt}^{2+}$  ions in solution are anchored by the sulfonic acid–silicate groups adsorbed onto MWCNTs/carbon paper which yield high dispersion of Pt particles upon reduction with acetic acid.

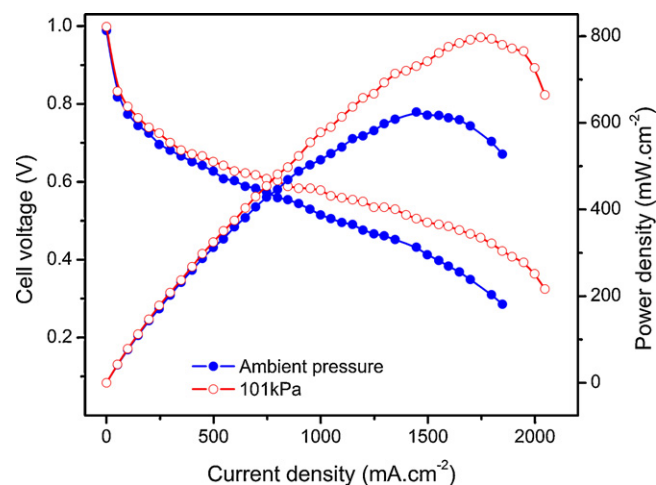
The SEM images in Fig. 2 shows the characteristics of the *in situ* grown MWCNTs on carbon paper using the CVD process at different magnifications. Fig. 2(a) compares the surface morphology of the CNT grown area with the pristine carbon paper. Evidently, the *in situ* growth of CNTs can be controlled to any specific area through surface modification process. High density of *in situ* grown MWCNTs all over the surface of the carbon paper substrate is seen from Fig. 2(b). Fig. 2(c) and (d) shows the evolution of MWCNTs on the surface of the fibers on the carbon paper at higher magnifications. It can be seen that MWCNTs are not ordered but are entangled in and through the plane to provide structural integrity of the micro-porous layer to the GDL substrate. High density carbon nanotubes network grown on the substrate is an ideal micro-porous layer for uniform gas distribution as well as active sites as support for electrocatalyst particles. The *in situ* grown MWCNTs/carbon paper fabricated without any binding agent is also hydrophobic in nature as confirmed by the contact angle measurements which yielded a wetting angle of  $\sim 145^\circ$  [9]. Hence surface treatment was necessary to improve the wettability, which was accomplished by citric acid treatment prior to Pt deposition by silane method (see Fig. 1).

In order to examine the GDLs fabricated by *in situ* growth of MWCNTs on carbon paper substrate, fuel cell performance of MEAs were evaluated at various operating conditions. Fig. 3 provides the fuel cell performance at  $80^\circ\text{C}$  at various RH conditions (100, 90, 80 and 70% RH of the reactant gases), using commercial catalysts ( $0.5\text{ mg Pt cm}^{-2}$ ) for anode and cathode, respectively. Since the micro-porous layers of the GDLs are without any hydrophobic agent (say, Teflon), the fuel cell performance does not degrade even at 70% RH. The physical characteristics of the MWCNTs/carbon paper based GDLs such as MWCNTs loading, micro-porous layer thickness, wetting angle and pore diameter values are consolidated in Table 1. Even though the pores in the MWCNTs based micro-porous layers are relatively larger (30, 45 and  $55\ \mu\text{m}$ ), the MWCNTs based GDLs without Teflon as binder is preferable to hold product water in the fuel cell working at reduced RH conditions.

The high resolution TEM image in Fig. 4(a) shows that the MWCNTs are highly crystalline and the outer diameter and inner diameter were estimated as  $\sim 80$  and  $\sim 10$  nm, respectively. Fig. 4(a) also shows the nature of MWCNTs along with the fringes depicting the multiple number of layers in a MWCNT. Fig. 4(b) illustrates the high resolution TEM image of Pt deposited *in situ* grown MWCNTs. It can be seen that the Pt particles are homogeneously distributed on the surface of MWCNTs. High loading of monodispersed Pt nanoparticles is facilitated by the sulfonic acid–silicate groups incorporated on to MWCNTs during silane functionalization step prior to Pt reduction. Moreover, Pt particle size in the range 1–2 nm is attributed to wet chemistry reduction of  $\text{Pt}^{2+}$  ions using glacial acetic acid. Fig. 4(c) shows the EDAX pattern of the Pt/MWCNTs which corresponds to TEM image shown in Fig. 4(b). The semi-quantitative EDAX pattern shows the presence of Pt and the high intensity peaks substantiates the high loading. Relatively small amount of Fe observed in the EDAX image is essentially from

**Table 1**  
Characteristics of MWCNT based micro-porous layer on the carbon paper substrate.

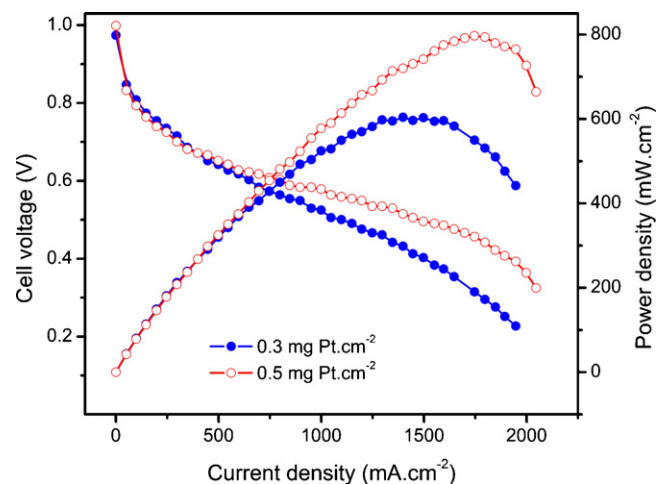
Loading of MWCNT ( $\text{mg cm}^{-2}$ )	1.5–1.75
Thickness ( $\mu\text{m}$ )	50–60
Wetting angle ( $^\circ$ )	135–140
Pore diameters ( $\mu\text{m}$ )	30, 45 and 55



**Fig. 5.** Fuel cell performance of MEA with MWCNTs/carbon paper electrode using Nafion-212 membrane at  $80^\circ\text{C}$  and  $\text{H}_2/\text{O}_2$  at 100% RH conditions with and without back pressure.

ferrocene catalyst used in the CVD process. The effect of Fe presence on the GDE was not studied in the present study. Fig. 4(d) shows the statistical distribution of Pt nanoparticles indicating a very narrow particle size distribution with a median of about 0.9–1.4 nm.

Electrocatalytic activity of the Pt/MWCNTs/carbon paper was evaluated by single cell PEMFC testing. Fig. 5 shows the polarization performance of Pt/MWCNTs/carbon paper ( $0.5\text{ mg Pt cm}^{-2}$ ) cathode and TKK catalyst ( $0.5\text{ mg Pt cm}^{-2}$ ) coated Nafion-212 membrane with GDL as anode at  $80^\circ\text{C}$  using  $\text{H}_2/\text{O}_2$  at 100% RH condition, with and without back pressure. As seen from the data, the performance at higher pressure conditions is much higher than that at ambient pressure. The current density obtained at 0.4 V is about 1.5 and  $1.9\text{ A cm}^{-2}$  at ambient pressure and 101 kPa, respectively. The peak power density values observed are about 600 and  $800\text{ mW cm}^{-2}$  at ambient pressure and 101 kPa back pressure, respectively. The polarization performance of Pt/MWCNTs/carbon paper cathode with lower Pt loading ( $0.3\text{ mg Pt cm}^{-2}$ ) was also investigated. Fig. 6 compares the single cell performance of cathodes with 0.3 and  $0.5\text{ mg Pt cm}^{-2}$  at  $80^\circ\text{C}$  using  $\text{H}_2/\text{O}_2$  at 100% RH with 101 kPa back pressure. The peak power density values are about 600 and  $800\text{ mW cm}^{-2}$  for 0.3 and  $0.5\text{ mg Pt cm}^{-2}$ , respectively. The specific power density at 0.4 V is about  $2\text{ W mg}^{-1}\text{ Pt}$  for  $0.3\text{ mg Pt cm}^{-2}$  load-



**Fig. 6.** Fuel cell performance of MEA with MWCNTs/carbon paper electrode ( $0.3$  and  $0.5\text{ mg Pt cm}^{-2}$ ) using Nafion-212 membrane at  $80^\circ\text{C}$  and  $\text{H}_2/\text{O}_2$  at 101 kPa back pressure and 100% RH condition.

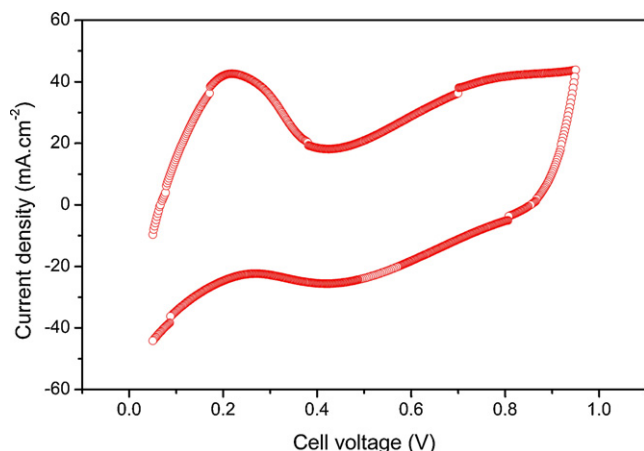


Fig. 7. Cyclic voltammogram of an MEA with Pt/MWCNTs/carbon paper ( $0.3 \text{ mg Pt cm}^{-2}$ ) at  $25^\circ\text{C}$  using humidified hydrogen and nitrogen gases.

ing as compared to  $1.6 \text{ W mg}^{-1} \text{ Pt}$  for the  $0.5 \text{ mg Pt cm}^{-2}$  loading. In the economic point of view, higher specific power density is definitely an advantage. In order to understand the intrinsically higher fuel cell performance, the electrochemically active surface area was estimated by the cyclic voltammetry of the Pt/MWCNTs/carbon paper. The cyclic voltammetry of the cathode fabricated with  $0.3 \text{ mg Pt cm}^{-2}$  on the MWCNT based micro-porous layer is now provided in Fig. 7 (see below) to highlight the electrochemically active surface area (ECA). The ECA is calculated to be about  $50 \text{ m}^2 \text{ g}^{-1}$  based on the hydrogen desorption.

#### 4. Conclusions

*In situ* grown MWCNTs on carbon paper were decorated with platinum nanoparticles in the size range 1–2 nm by reducing Pt(II)acac in glacial acetic acid at  $<100^\circ\text{C}$ . Homogeneous dispersion of Pt nanoparticles on *in situ* MWCNTs was observed from the high resolution electron micrograph. Fuel cell performance of the MEA with Pt ( $0.5 \text{ mg Pt cm}^{-2}$ ) supported on *in situ* MWCNTs/carbon paper exhibited a peak power density of about 600

and  $800 \text{ mW cm}^{-2}$  at ambient pressure and 101 kPa, respectively with  $\text{H}_2/\text{O}_2$  at  $80^\circ\text{C}$  using Nafion-212 electrolyte. The specific power density at  $0.3 \text{ mg Pt cm}^{-2}$  loading is much higher compared to that at  $0.5 \text{ mg Pt cm}^{-2}$ . The higher catalytic activity of the Pt/MWCNTs/carbon paper is evidenced by the ECA value of  $\sim 50 \text{ m}^2 \text{ g}^{-1} \text{ Pt}$ .

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