

Manufacturing of membrane electrode assemblies for fuel cells

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1 Abstract

Research in proton-exchange membrane fuel cells (PEMFC) and direct methanol fuel cells (DMFC) has matured to a point where, in addition to fundamental research in new catalysts, membranes and other materials, manufacturing processes used to make the various components in a fuel cell are drawing much needed attention. One of the key components in a fuel cell is the membrane electrode assembly (MEA) where the actual chemical reactions occur. Small-scale MEA manufacturing in academia and industry is done entirely by manual processes, which besides being time consuming and labor intensive, offer opportunities for inconsistency and defects. If the fuel cell technology is to evolve to a point where it can be commercially viable, the manufacturing costs of the MEA must be driven down, and continuous, automated production technologies developed and optimized, so that volume production can be undertaken.

In this paper, we present the design of a modular test bed, which is being used to study different coating methods for depositing the electrode material onto the polymer electrolyte, the key step in MEA fabrication. The flexible design of the test bed has allowed for the study of various coating technologies and process parameters, with a high degree of repeatability. Initial results of PEMFC MEA's produced on the test bed are also presented, which show much promise for the overall continuous manufacturing process being developed.

2 Introduction

As will be discussed in elaborate detail in the proceeding sections, the membrane electrode assembly is a key component of a PEMFC and DMFC fuel cell. Its components, its design and its fabrication directly contribute to the overall cost of the system and its performance.

The porous electrode in the MEA is formed by depositing 'catalyst ink' onto a substrate, which upon drying forms a three dimensional porous structure, which allows for the transport of the fuel and oxidant to the electrode and for the reaction products from the reaction sites on the catalyst surface. Additionally the electrode must also be electronically conductive to allow for the movement of the electrons produced in the reaction to the external circuit, and allow for the protons to move through the ionically conductive membrane to the other electrode to complete the overall reaction, as shown in Figure 1. Limiting the mobility and transport of any of these species reduces the catalyst utilization and the overall performance that can be extracted from a given amount of catalyst.

So it is essential to 'engineer' the electrode for maximum performance and minimal consumption of the expensive catalyst material. In this research effort it is hypothesized that the design and fabrication of the electrode is influenced by the composition of the 'catalyst ink' itself, the method of deposition and the rate at which it is solidified. In order to study the said variables it is essential to have a flexible platform where in a controlled and repeatable fashion the interactions of these process variables can be studied and optimized.

Additionally, it is important to develop a continuous manufacturing process for the fabrication of fuel cell MEA's to harness cost benefits and allow for volume production. A comparison based on performance, as well as economic considerations of the various ink deposition/ coating technologies available will allow for the design and construction of a cost effective MEA fabrication process. Such an effort again requires the availability of a platform where coating processes can be compared.

Having identified these two needs, we have developed a test bed which will allow us to: engineer the catalyst layer so as to optimize the transport of the reaction species through the MEA, as well test, consider and model different coating technologies which will lead to the development of a continuous manufacturing process for fabricating PEMFC and DMFC membrane electrode assemblies.

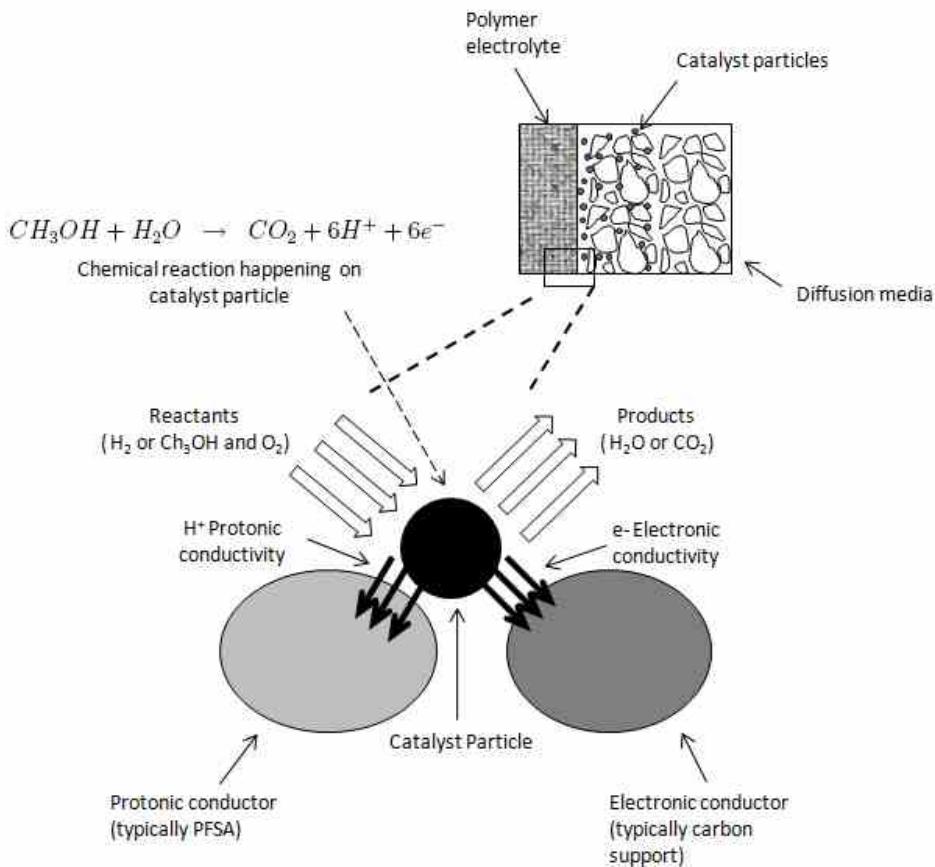


Figure 1: Reaction at a catalyst particle showing Mass, electronic and protonic transport

3 Background

3.1 Working Principles of PEMFC and DMFC Fuel Cells

Fuel cells convert the free energy of a chemical reaction directly into electrical energy. A typical DMFC using a polymer electrolyte is shown in Figure 2. On the anode methanol and water react to produce carbon dioxide, protons and electrons. On the anode, the protons produced migrate through the polymer electrolyte membrane to the cathode side where they react with oxygen to produce water. The electrons are free to travel through the external electrical circuit where they can drive an electrical load [1].

A PEMFC works in very similar fashion: on the anode side, humidified hydrogen gas is supplied through the flow fields in the bipolar plate, which then permeates through the porous diffusion media, into the anode electrode, where it breaks down into protons and electrons. The electrons travel through the external circuit driving the load, whereas the protons permeate through the polymer electrolyte, and combine with oxygen on the anode side to form water.

3.2 Key Components of a Typical PEMFC and DMFC

As shown in Figure 3, the key components of a fuel cell are;

- **Membrane Electrode Assembly**

The membrane electrode assembly (MEA) provides sites for chemical reactions occurring inside a fuel cell to take place, to convert the fuel into usable electrical power. Different research groups and manufacturers use different terminology. Some refer to the MEA as just the membrane and electrodes ; whereas some refer to the five-layer assembly (membrane, catalyst layers, and diffusion media) as the MEA.

- **Bipolar Plates**

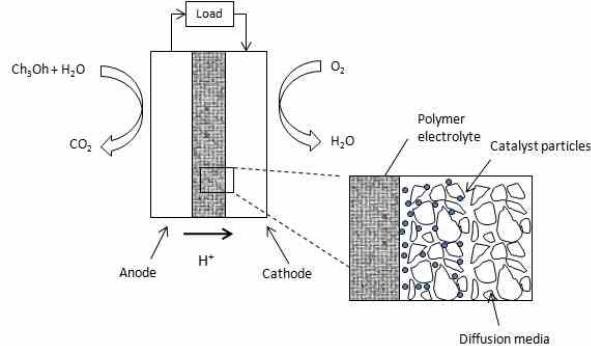


Figure 2: DMFC using a polymer electrolyte membrane and supported platinum catalyst

Bipolar plates perform a few key functions in a fuel cell. They provide paths for the fuel and oxidant to reach the membrane electrode assembly and for the reaction products to be removed, function as a current collector and are connected to the external circuit, and also provide mechanical stability to the entire assembly.

- **Sealing Media**

Sealing media is placed in between the MEA and the bipolar plates so as to prevent any exchange of fuel or oxidant between the cell and the environment.

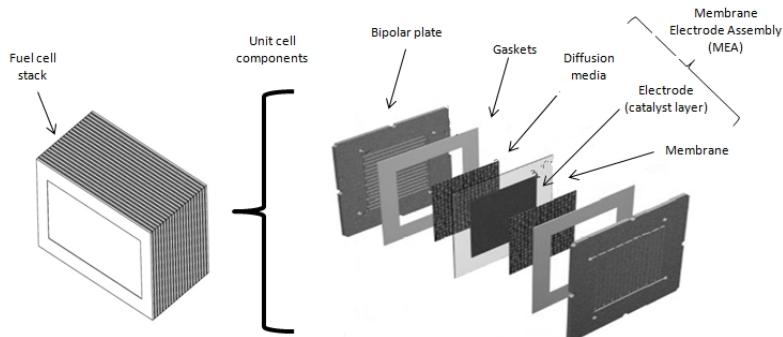


Figure 3: Anatomy of a typical direct methanol fuel cell

3.3 MEA Components

The membrane electrode assembly (MEA), shown in Figure 4, is essentially the heart of a DMFC. As the name implies an MEA consists of a proton-conducting membrane sandwiched between anode and cathode electrode layers, with a gas diffusion layer (GDL) on either side. A brief discussion of each component of a MEA follows.

3.3.1 Membrane

The membrane has two major functions in a fuel cell; the first is to separate the fuel and the oxidant, and the second is the transport of protons across from the anode to the cathode to complete the redox reaction. It also must provide strong mechanical, chemical, and electrochemical stability in a harsh chemical rich environment over a range of operating conditions, while at the same time offer a long life, low reactant permeability, high proton conductivity and serve as an effective electrical insulator.

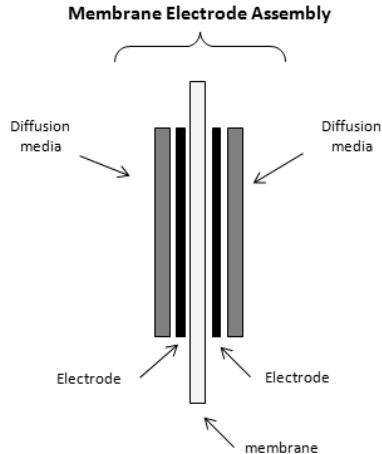


Figure 4: Cross section of a typical membrane electrode assembly

3.3.2 Gas Diffusion Layer

The diffusion layer, is porous media which allows for the transport of reactant from the flow field and evenly disperses it over the catalyst sites. Besides this key role, it also serves as a conducting path to the external circuit via the bipolar plates, therefore it should have low electronic resistivity. Carbon cloth or paper being conductive and stable in the fuel cell environment is the obvious choice, and has been widely employed as the diffusion media.

3.3.3 Electrodes

The electrodes are typically porous structures of nanometer-sized catalyst particles dispersed in a suitable binder medium. Additionally, catalysts can be supported on carbon media to promote dispersion and lower catalyst loading. It is important to allow adequate pathways for diffusion of the reactant media to the catalyst site, removal of reaction products, as well as good electrical and protonic conductivity. The electrons generated during the reaction have to be transported to the external circuit, whereas the protons have to cross the electrolyte to complete the reaction at the counter electrode. Electrical conductivity in a electrode is either provided by the carbon support, in case of supported catalyst, or the catalyst particles themselves in case of unsupported ones. The interfaces between the MEA components and the three phase boundary between the reactant, the catalyst and the ionomer dictate the performance of the fuel cell.

Previously PTFE was used as a binder in the electrode and subsequently very high catalyst loading were required for adequate performance. Wilson et al.[2, 3] proposed a new electrode structure where the catalyst and ionomer are blended together to form an “ink” and cast to form the catalyst layer, with no PTFE content. Such electrodes, where an ionomer is used as a binder (typically Nafion) are called the “thin film” electrodes.

4 MEA Types

There are essentially two types of MEA's, and they are differentiated on the basis on where the catalyst ink is deposited to form the electrode, as shown in Figure 5. ”Catalyst Coated Membranes” (CCM) are those where catalyst ink is coated directly onto the membrane. If the catalyst ink is coated onto the diffusion media to form an electrode and then sandwiched onto a polymer membrane, such MEA's are called ”Catalyst Coated Substrate” (CCS). Additionally an intermediate step can be introduced in making CCM type MEA's, by casting the electrodes on a temporary substrate such as Teflon film, which are then hot pressed on to a membrane and then the Teflon film is peeled off. This method is called the “decal transfer method”(DTM).

DTM method helps solve the problem of Nafion swelling, which causes considerable problems in coating and drying of the electrode directly onto the membrane. Results reported in the literature [4]

show that CCM type MEA's prepared with direct application of ink on the membrane performed the best, followed by MEA's made by the decal transfer method, and the lowest performance is by the CCS type MEA's. This is attributed to the fact that the direct application of ink on the membrane produces a more intimate contact between the catalyst layer and the membrane extending the three phase region.

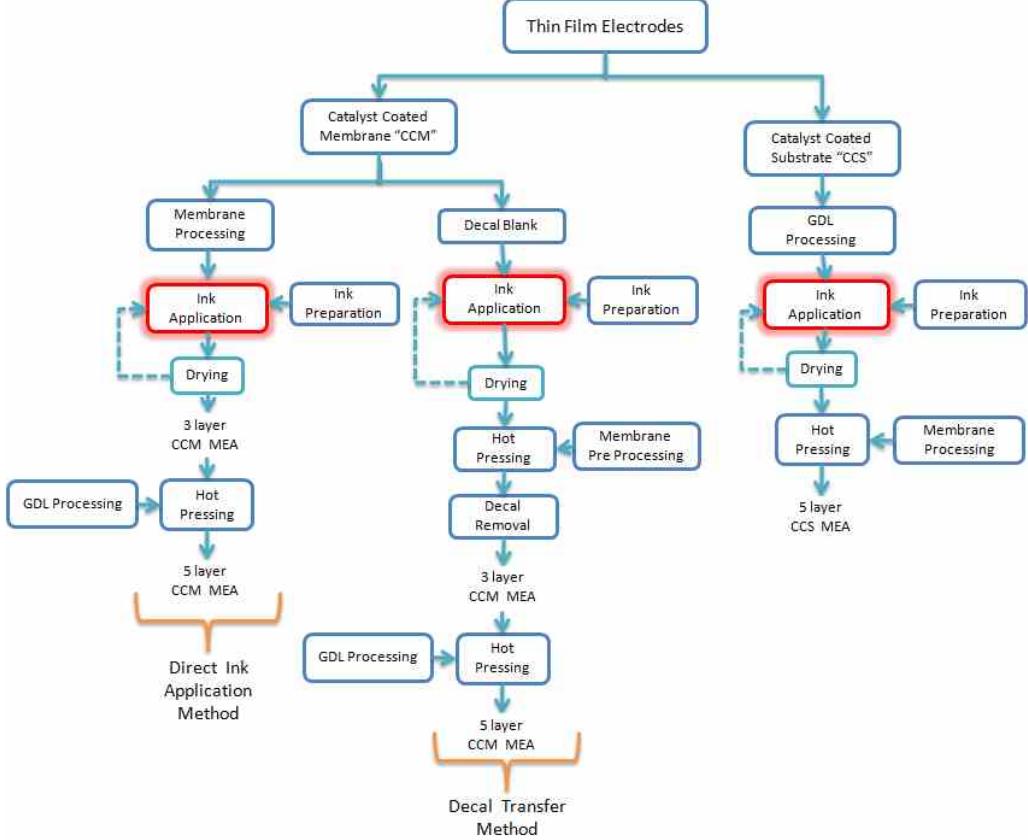


Figure 5: Types of Membrane Electrode Assembly (MEA)

5 MEA Manufacturing

This section discusses the steps involved in processing each of the three MEA components. The processing required for the diffusion media and the membrane is relatively simple and is discussed briefly, whereas the electrode fabrication process is discussed in elaborate detail.

5.1 Membrane Processing

The polymer electrolyte membrane is usually procured in roll form as an end product from its manufacturer, and the only processing steps required on it is to boil it in hydrogen peroxide, dilute sulphuric acid and water. These steps serve to clean the membrane, and to ensure that it is protonated[5].

5.2 Diffusion Layer Processing

The gas diffusion layer is made up of two layers, the Macroporous layer and the Microporous layer. The Macroporous layer is essentially a region where the diffusion media has been treated with a hydrophobic agent(PTFE). Hydrophobicity is a key requirement to prevent flooding of the channels in the diffusion media with water. The precursor for the diffusion media is generally carbon cloth or carbon paper, and the macroporous layer is formed by applying teflon solution onto the substrate. The microporous layer which assists in managing the water content of the MEA [6], is a much thinner layer made by coating the macroporous layer with a mixture of carbon black and PTFE. PTFE content in both layers is also very important, it directly affects the porosity and hydrophobicity of the diffusion media which in turn

affects the mass transport of reactant media to the catalyst layers [7]. The manner in which this mixture is deposited is very similar to how the electrode layers are formed, so the same processes can be used to fabricate the microporous layer. The processing steps in the manufacturing of the diffusion layer are summarized in Figure 6.

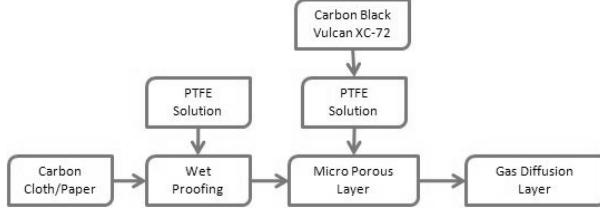


Figure 6: Gas diffusion layer fabrication steps

5.3 Electrode Fabrication

As mentioned in the previous paragraphs, there are two types of MEA's, the catalyst coated substrate (CCS) and the catalyst coated membrane (CCM), and the difference between them is basically the substrate on which the electrode is cast. The following paragraphs discuss the key steps in thin film electrode fabrication.

5.3.1 Ink formulation

Thin film electrodes, follow the same general recipe as developed by Wilson [2, 3] at LANL. The catalyst ink typically consists of 4 components

- Solvent (DI water, IPA ,Glycerol, etc)
- Binder (mainly PFSA , or other ionomer in protonated form)
- Catalyst (supported / unsupported platinum , platinum-ruthenium)
- Additives (gelling agents, release agents, pore formers etc..)

Various ink formulations have been developed and are available in literature[2, 5, 8], but all contain a binder which is usually the same ionomer as used in the membrane. The viscosity of the ink can be tuned to suit the application process. The solid content of an ink is typically defined as the percentage of the mass of solid in an ink, divided by the total mass of the ink. This gives an indication as to how 'thick' the ink is. When it comes to coating processes this factor plays an important role, because it essentially dictates the wet layer thickness that has to be deposited on the substrate to form the electrode.

Typically DMFC utilize a supported platinum-ruthenium catalyst, and the catalyst loading is around 4 gm/cm², whereas for a PEMFC, a supported platinum catalyst is used, with a loading of about an order of magnitude lower, as low as 0.1 mg/cm². A wet layer thickness model was developed to understand the requirement for a coating process. Figure 7 shows how the required wet layer thickness varies with respect to the ink solid content and the catalyst loading. These results are for a particular ink composition, but depict the general trend.

5.3.2 Ink Application

The catalyst ink application onto the surface of a substrate is a common step for electrode fabrication (Figure 5), irrespective of the type of MEA being made. In the ink application step, the catalyst ink is uniformly deposited onto the surface of the substrate. It is of extreme importance that catalyst loading is kept uniform across the width and length of the electrode. In academia as well as by some commercial entities, ink application is done manually, on discrete units of substrate. Such manual, low volume procedures result in high cost and variation in MEA quality, and increased defects. Figure 8, shows typical ink application processes used to make MEA's [2, 3, 4, 9, 10, 11, 12]. The microstructure of the electrode formed during the ink application and drying stage is critical, it is important to avoid agglomeration of the catalyst particles, or of the binder, so as to ensure a electrode where the catalyst

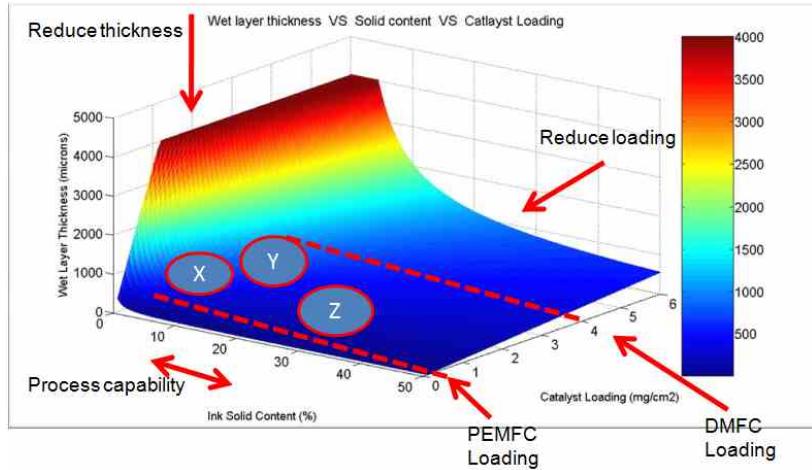


Figure 7: Wet layer thickness model

is thoroughly dispersed, and the triple phase boundary is maximized. This will ensure high catalyst utilization.

As PEMFC and DMFC technology nears commercialization, it is important to identify processes that can be scaled up for mass production of MEA's. It must be pointed out that for every ink application process, suitable ink has to be formulated, where essentially the ink viscosity is modified to match the application.

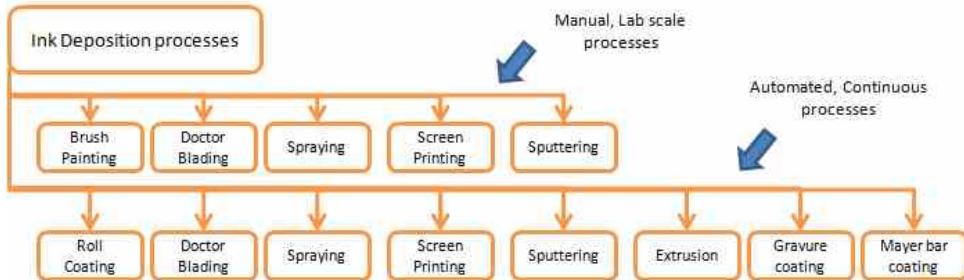


Figure 8: Common ink application processes

Additionally processes that would readily lend themselves to be automated, and perform coating on a continuous web of substrate are also identified in Figure 8. Continuous MEA fabrication processes aim to scale and improve on the above mentioned thin-film electrode fabrication processes, the foremost goal being to reduce the costs associated with manufacturing a MEA, maintain uniform quality, reduce and eliminate defects, while maintaining the actual fuel cell performance levels.

A few of the bigger players in industry have developed continuous coating lines, but obviously due to the proprietary nature of the information, no one is willing to talk about the particular coating method they have employed. A patent search was carried out to survey what processes have been patented by which company, because at the very least that might identify the processes that are being actively considered in the industry. Table 1, provides a snapshot of some of the processes along with the year, and company name.

There is minimal available in literature on how the performance of a fuel cell varies across these different ink application methods. From purely a coating process standpoint, quite a few of the technologies can deposit the required wet layer thickness of material on a substrate, but there is no good answer as to how the choice of the process and the drying parameters effect the actual microstructure, if at all, and what is the best process, and optimal processing parameters. Furthermore from an economic standpoint cost comparison of these processes are also necessary so that an economically feasible process is selected and the over all MEA fabrication process is designed around it.

Table 1: Commercial MEA Fabrication Processes

Component Type	MEA Fabrication Process	Company	Year	US Patent Reference
CCM	spray coating	Samsung SDI	2007	7169500
CCM	Flexographic printing	Dupont	2007	7316794
MEGA, CCS	Spraying	Hyundai	2007	7056612
CCM	Magnetron DC sputtering	Samsung	2004	6749892
CCS,CCM	Electron beam PVD	Gore	2007	7303834
GDL, CCM	Extrusion,Calendering	3M	2002	6432571,5338430
CCM	tri layer co-extrusion	Ballard	2001	6291091
CM+CCS	Screen printing	Japan gore-tex	2004	6723464
CCM	Screen printing	Honda	2007	7306876
CCM	Bar coater	Asahi glass	2007	7157176
CCM	Die coater	Matsushita	2005	6855178
CCM+CCS	screen printing	Umicore	2006	7141270

5.3.3 Drying

The microstructure of the electrode is formed during the drying stage, and the rate of drying will have a significant effect on the quality of the electrode. The drying time is intimately related to the solvent used in the ink formulation. Drying too fast causes the surface of the electrode film to boil and peel and is highly undesirable. In a continuous process typically a combination of infrared and convective heating is used in the dryer, whereas in laboratory scale processes, MEA's are typically vacuum dried in an oven.

5.3.4 Substrate Management

An added step in continuous manufacturing which would be common to all the ink application processes is the substrate management step. The substrate can be either the polymer electrolyte membrane, which is typically Nafion, or alternatively it can be an intermediate substrate such as teflon film for decal transfer, or if CCS type MEA's are being made, it can even be the diffusion media, which is typically carbon cloth or carbon paper. Each of these substrates will have to be properly unwound, and rewound and proper tension applied during the entire process. Nafion poses a significant challenge in this regard, because it can absorb significant amounts of water/ solvent and swells up. Upon ink application and drying, different areas of the material have different levels of hydration, which cause the Nafion substrate to wrinkle up. So if a Nafion substrate is used, web management becomes a significant problem, that is why generally the DTM method is preferred, as it simplifies the web management.

6 MEA Manufacturing Test Bed

Based on the discussion presented in the prior sections, the authors felt the need to develop a test apparatus where the various coating methods could be tested out and applied to the manufacturing of PEMFC and DMFC fuel cells. The goal is to be able to study the various ink application methods, different ink formulations and the effect of drying rates on the electrode performance. Such a study permits the direct comparison of the electrode fabrication process on the performance of a fuel cell a performance. Then the entire MEA process would be designed around this electrode fabrication step and further economic analysis can then also be done. Such a study would identify process which can then be further optimized and refined.

With this intent in mind an initial concept for a test bed for continuous electrode fabrication was developed, as shown in Figure 9. The concept as shown, has modules for web management: web unwinder, rewinder and the tensioning module. The catalyst ink is applied via the ink application or coating module and solidified the dryer. These key modules would be common to most of the coating processes that will be tried out on this platform. Such a flexible approach allows for the test bed to accept multiple coating heads, and the web parameters such as tension, feed can be modified to suit the particular web material and drying conditions.

The key design requirement are as follows

- Multiple coating heads/ Ink application modules
- Variable web feed rate
- Variable drying rate

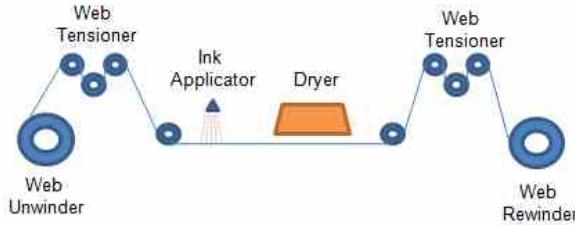


Figure 9: Coating test bed concept

- Web tension management
- Easy replacement of web cores
- Future expansion
- Modularity

6.1 Test Bed Design and Details

Keeping the requirement and constraints in mind, a design of a modular coating test bed has been developed. A CAD model of the machine showing the main parts and modules is shown in Figure 10, while the actual machine that has been built is shown in Figure 11. A six-inch-wide web can be mounted onto the machine using core holders, which allow for rapid mounting and removal of a new web. At this stage the design intent was to have the ability to very closely monitor the deposition process and the drying process, therefore the machine was designed to be able to operate at a web feed as low as 0.2 ft/min, and as high as 1.2ft/min. The entire drive train is very modular and if need be can be upgraded in future to allow for higher speeds and torques. A 3.5 KW combination infrared and convection dryer module has been designed and installed into the test bed. The dryer module is reconfigurable so as that individual heating elements can be repositioned and reoriented to better accommodate the drying requirement of the different ink formulations and application processes. Two zones have been built into the test bed where coating modules can be plugged in. One zone will provide for 'over web' access and the other for 'under web' access. This is required due to the nature of the specific coating modules used, for instance spraying needs 'over web' access, whereas a gravure coating head would require an 'under web' access. The coating modules are described in the subsequent sections. Web tension control is done via a disk brake arrangement which provides friction to the web unwind roller, whereas the drive unit is mounted onto the web rewind roller. As shown in Figure 10, at present all operations in the machine are manual but ample space has been left for further automation, which will be done when the application processes are further short listed.

6.2 Ink Application Modules

6.2.1 Spraying Module

The spraying module, allows for the movement of the spray head in the web feed direction. This in rapid traversal in combination with the web feed will allow for multiple spray passes on the substrate. A vacuum table is mounted under the web surface, so that in addition to continuous web handling, even discrete samples of substrate can be mounted in the test bed and coated. Two different spraying heads can be mounted, one is an ultrasonic spray head, which works without any pressure, which can potentially minimize catalyst loss, and the other spray nozzle is of a conventional type with a wide spray band. The height of the spray head is adjustable so as to allow for adjustment of the spray width.

6.2.2 Tape Casting/Doctor Blading Module

In Figure 13, the design of a tape casting head is shown. The doctor blade is held in place by micrometers, which can be used to adjust the gap. This controls the wet layer thickness of the ink being deposited onto the substrate. In the same figure the actual catalyst ink layer deposition is being shown.

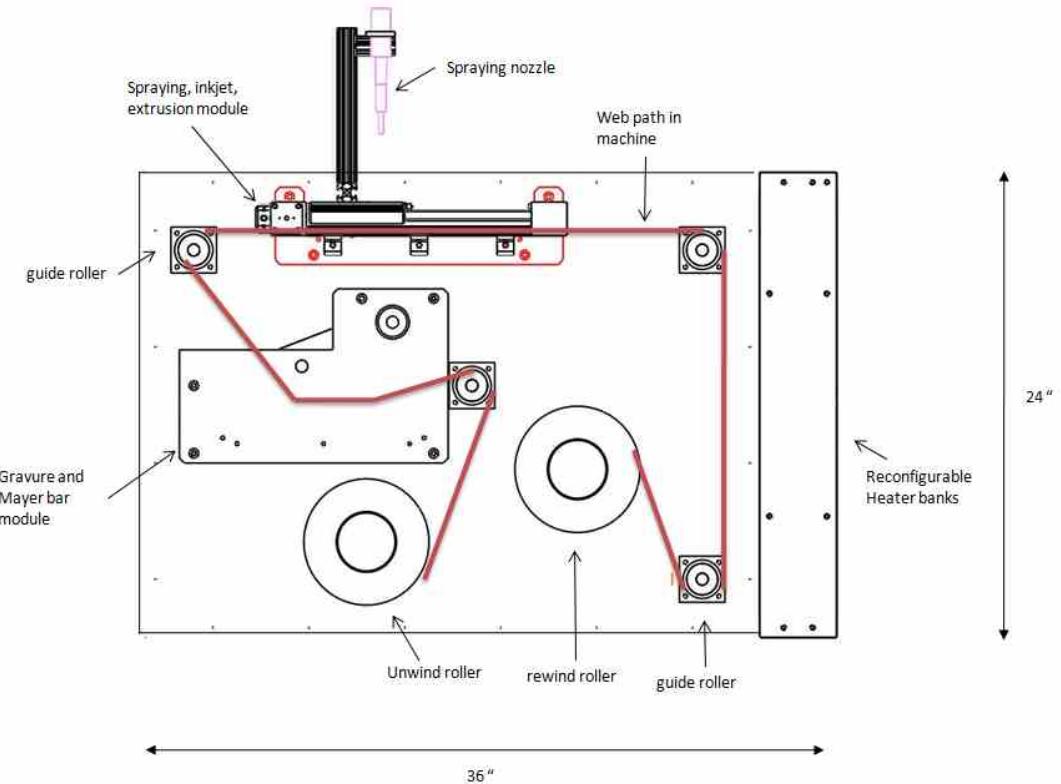


Figure 10: Different modules and parts of the coating test bed

6.2.3 MEA's manufactured by Tape casting

Figure 14, compares the results of MEA's manufactured by hand vs those manufactured by tape casting on the test bed. Manufacturing MEA's by hand is truly an art form, and the results shown represent the best efforts by us to date, in literature higher performances have been achieved. The results of the tape casting MEA compare favorably with typical results expected from the catalyst at the given loading and testing conditions. These are the first results and improvement are expected in the due course of this research.

Figure 15 show the electrodes as they are cast on to the substrate in a roll form. Individual electrodes are cut out from the roll and then hot pressed on to a membrane to form the overall MEA. The subsequent processing steps (die cutting and lamination) right now are done by hand but can be easily automated and incorporated into a continuous process.

6.2.4 Future Modules

Right now, two modules have been built and tested on the test bed. But the design has allowances for more modules and in the near future the following modules will be built and tested with the machine.

- Ink jet module
- Slot die extrusion module
- Gravure coating module
- Mayer bar coating module

7 Conclusion and Future work

This paper discusses the components and the manufacturing of the membrane electrode assembly for PEMFC and DMFC fuel cells. It further highlights the underlying need to engineer the porous electrode

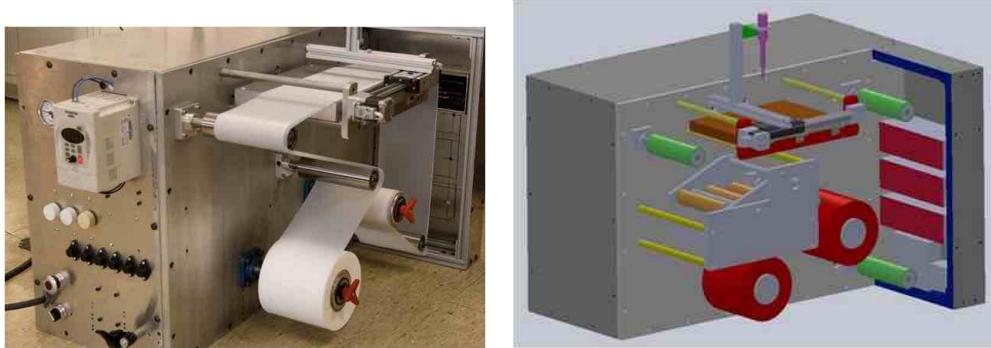


Figure 11: CAD model of modular coating test bed and actual machine

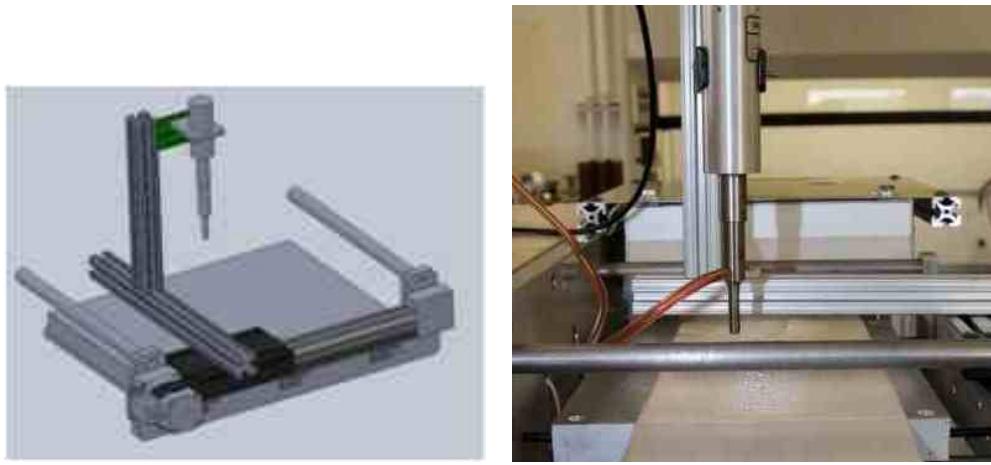


Figure 12: Spray head model and Ultrasonic spray nozzle

of a fuel cell for optimal mass transport; as well as the need for a continuous process for the manufacturing of MEA's. In lieu of these arguments, we have presented the design and the key features of a novel, modular coating test bed which will facilitate the study of various coating processes, drying parameters and ink formulations. Initial results of PEMFC MEA's manufactured using the test bed are also shown, which highlight the capability of the platform. Experiments conducted on this test bed will identify an optimal process for electrode fabrication, around which an overall MEA fabrication process can be designed.

Amongst the experiments that are planned, different ink compositions will be tested out, along with different substrates which can be used in the decal transfer method (DTM), and for direct catalyst coated membrane(CCM) processing, Nafion management schemes will be explored. The temperature distribution of the substrate in the dryer will be modeled analytically and measured as well, so that the influence of the drying rate on the performance can be studied. Once a thorough understanding of these interactions has been developed, process metrics will be identified to directly relate manufacturing variables to the performance of a fuel cell. Such schemes will allow for efficient control of the manufacturing process.

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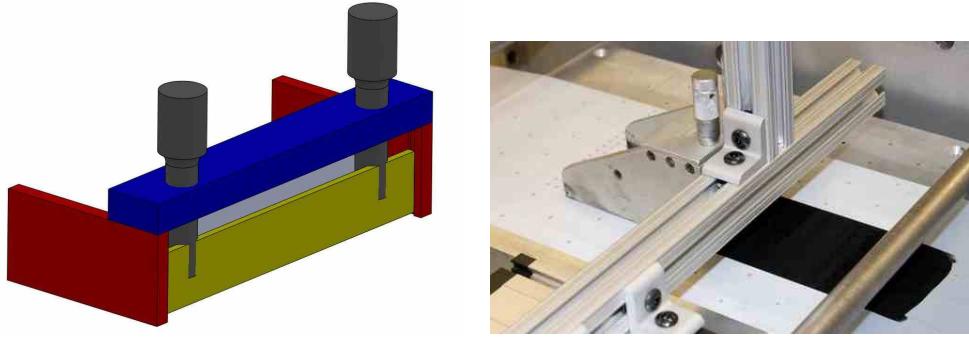


Figure 13: CAD model of tape casting module and actual tape casting results on machine

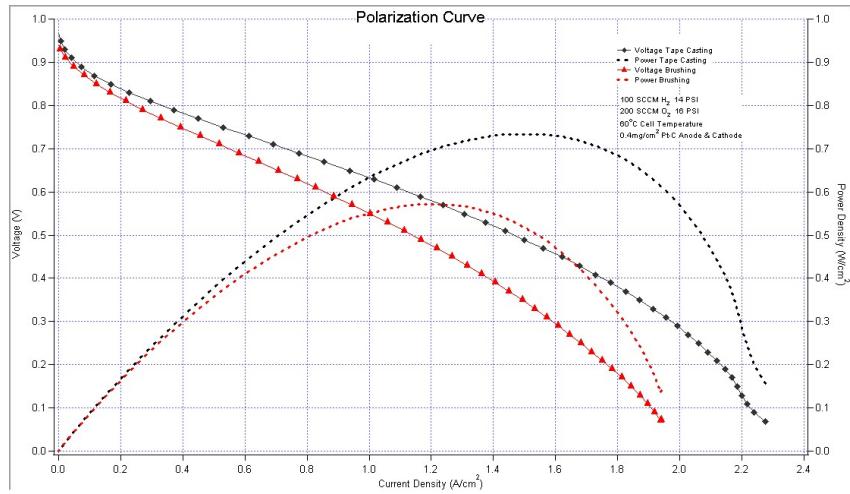


Figure 14: Results from MEA manufactured by Tape Casting on test bed and brushing done by hand. (both MEA's are 0.4 mg/cm^2 Pt-C, 100SCCM H₂ 200SCCM O₂, 14 psi H₂ 16psi O₂ back pressure, cell temperature = 60°C)

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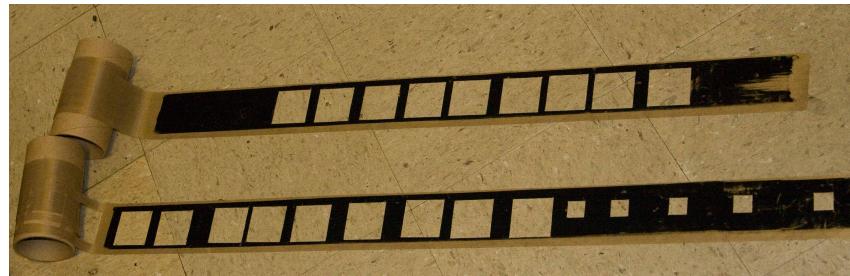


Figure 15: Tape Casting of electrodes on test bed

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