

FORM 1
THE PATENTS ACT 1970
(39 of 1970) &
The Patent Rules, 2003
APPLICATION FOR GRANT OF
PATENT

(See section 7, 54 & 135, and rule 20(1))

(FOR OFFICE USE ONLY)

Application No:

Filing Date:

Amount of fee paid:

CBR No:

Signature:



700234006

Application No.	201841040380
Filing date:	26/10/2018
Amount of Fee paid:	8800/-
CBR No:	30510
Signature	

1. APPLICANT'S REFERENCE /
IDENTITY NUMBER. (AS ALLOTTED BY
OFFICE)

2. TYPE OF APPLICATION [Please tick (✓) at the appropriate category]

Ordinary (✓)		Convention ()		PCT-NP ()	
Divisional ()	Patent of Addition ()	Divisional ()	Patent of Addition ()	Divisional ()	Patent of Addition ()

3A. APPLICANTS

Name in Full	Nationality	Country of Residence	Address of Applicant
ALVA'S EDUCATION FOUNDATION	Indian	India	ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA

3B. CATEGORY OF APPLICANT [Please tick (✓) at the appropriate category]

Natural Person (✓)		Others ()
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4. INVENTOR(S) [Please tick (✓) at the appropriate category]

Are all the inventor(s) same as the applicant(s) named above?	Yes ()	No (✓)
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If NO furnish the details of the inventors

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ACHANTA VENUGOPAL	Indian	India	TATA INSTITUTE OF FUNDAMENTAL RESEARCH, MUMBAI - 400005 MAHARASHTRA, INDIA

5. TITLE OF THE INVENTION					
"ENHANCEMENT OF MICRO DIRECT METHANOL FUEL CELL (μ -DMFC) PERFORMANCE USING MICRO CHANNELS FABRICATED FROM <100> SILICON WAFER ORIENTATION AND P(VDF-TrFE) COATED NAFION MEMBRANE AS PROTON EXCHANGE MEMBRANE"					
6. AUTHORISED REGISTERED PATENT AGENT(S)					
		Name			
		Mobile no.			
7. ADDRESS FOR SERVICE OF APPLICANT IN INDIA		Name		ALVA'S EDUCATION FOUNDATION	
		Postal Address		ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA	
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		E-Mail ID		rpinto1942@gmail.com	
8. IN CASE OF APPLICATION CLAIMING PRIORITY OF APPLICATION FILLED IN CONVENTION COUNTRY, PARTICULARS OF CONVENTION APPLICATION					
Country	Application number	Filing date	Name of applicant	Title of invention	IPC (as classified in the convention country)
Not Applicable					
9. IN CASE OF PCT NATIONAL PHASE APPLICATION, PARTICULARS OF INTERNATIONAL APPLICATION FILED UNDER PATENT CO-OPERATIVE TREATY (PCT)					
International application number:			Not Applicable		
International filing date:			Not Applicable		
PATE OFFICE CHENNAI 29/10/2018 11:36					
10. IN CASE OF DIVISIONAL APPLICATION FILED UNDER SECTION 16, PARTICULARS OF ORIGINAL (FIRST) APPLICATION					

Original (first) application number: Not Applicable	Date of filing of Original (first) application: Not Applicable
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11. IN CASE OF PATENT OF ADDITION FILED UNDER SECTION 54, PARTICULARS OF MAIN APPLICANT OR PATENT

Main application/ Patent Number: Not Applicable	Date of Filing of main application: Not Applicable
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12. DECLARATION


i) Declaration by the inventors

(In case the applicant is an assignee: the inventors may sign herein below or the applicant may upload the assignment or enclose the assignment with this application for patent or send the assignment by post/ electronic transmission duly authenticated within the prescribed period)

We, the above named inventors are the true and first inventors for this invention and declare that the applicants herein are our assignee or legal representative.

(i)


(a) Date:

(b) Signature of the inventors: 

(c) Name: **ARJUN SUNIL RAO**

(ii)

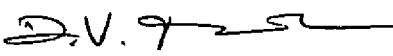
(a) Date: 12.10.2018

(b) Signature of the inventors: 

(c) Name: **JAYARAMA ARASALIKE**

(iii)

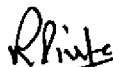
(a) Date: 11.10.18

(b) Signature of the inventors: 

(c) Name: **MANJUNATHA DODDABALLAPURA VEERABHADRAIAH**

(iv)

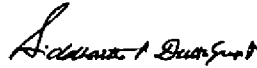
(a) Date:

(b) Signature of the inventors: 

(c) Name: **RICHARD PINTO**

(v)


(a) Date:

(b) Signature of the inventors: 

(c) Name: **SIDDHARTHA PRAKASH DUTTAGUPTA**

(vi)

(a) Date: 04/10/2018

(b) Signature of the inventors: 

(c) Name: **ACHANTA VENUGOPAL**

ii) Declaration by the applicant:

I, the applicant hereby declare that:-

- ☐ I am in possession of the above mentioned invention.
- ☐ The provisional/complete specification relating to the invention is filed with this application.
- ☐ The invention as disclosed in the specification uses the biological material from India and the necessary permission from the competent authority shall be submitted by us before the grant of the patent to us.
- ☐ There is no lawful ground of objection to the grant of the patent to us.
- ☐ I am the assignee or legal representative of true and first inventors. 6
- ☐ The application or each of the application, particulars of which are given in Para 5 was the first application in convention country/countries in respect of our invention.

- ☐ I claim the priority from the above mentioned applications filed in convention country and state that no application for protection in respect of the invention had been made in a convention country before that date by me/us or by any person from which we derive the title.
- ☐ My application in India is based on international application under Patent Cooperation Treaty (PCT) as mentioned in Para - 9.
- ☐ The application is divided out of our application particulars of which are given in Para-10 and pray that this application may be treated as deemed to have been filed on.....under section 16 of the Act.
- ☐ The said invention is an improvement in or modification of the invention particulars of which are given in Para-11

13. FOLLOWING ARE THE ATTACHMENTS WITH THE APPLICATION

(a) Form 2

Item	Details	Fee	Remarks
Complete Specification #	No. of Pages : 16		
No. of Claims	No. of claims : 1 No. of Pages : 2	₹ 8800/-	Addl. Pages - N/A
Abstract	No. of Pages : 1		Addl. Claims - N/A
No. of Drawings	No. of drawings : 18 No. of pages : 10		

In case of a complete specification, if the applicant desires to adopt the drawings filed with his provisional specification as the drawings or part of drawings for the complete specification under rule 13(4), the number of such pages filed with the provisional specification are required to mentioned here.

(b) Complete specification (in confirmation with the international application)/ as amended before the International preliminary Examination Authority (IPEA), as applicable (2 copies).

(c) Sequence listing in electronic format.

(d) Drawing (in confirmation with the international application)/ as amended before the International preliminary Examination Authority (IPEA), as applicable (2 copies).

(e) Priority document (s) or a request to retrieve the priority document(s) from DAS (Digital Access Service) if the applicant had already requested the office of first filing to make the priority document(s) available to DAS.

(f) Translation of priority document/ Specification/ International Search Report/ International Preliminary Report on Patentability.

(g) Statement and Undertaking on Form 3.

(h) Declaration of Inventor ship on Form 5.

(i) Power of Attorney.

(j) Total Fee ₹----- in Cheque bearing no: _____ dated this _____ drawn from _____ bank,

We hereby declare that to the best of our knowledge, information, and belief that the fact and matters stated herein are correct and we request that a patent may be granted to us for the said invention.

Dated this 10th day of October, 2018.

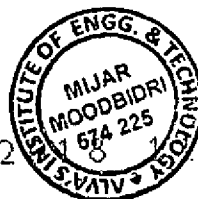
For ALVA'S EDUCATION FOUNDATION,

Authorized Signatory:

Name in full:

Designation:

Seal:



Mohan Alva.
chairman.

To,
The Controller of Patents,
Patent Office Chennai,
Chennai - 600 032.

29/10/2018

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FORM 2
THE PATENT ACT 1970
(39 of 1970)



&
The Patents Rules, 2003
COMPLETE SPECIFICATION
(See section 10 and rule 13)

TITLE OF THE INVENTION

**"ENHANCEMENT OF MICRO DIRECT METHANOL FUEL CELL (μ -DMFC)
PERFORMANCE USING MICRO CHANNELS FABRICATED FROM <100>
SILICON WAFER ORIENTATION AND P(VDF-TrFE) COATED NAFION
MEMBRANE AS PROTON EXCHANGE MEMBRANE"**

APPLICANT'S NAME AND ADDRESS

ALVA'S EDUCATION FOUNDATION

ADDRESS

**ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY, as Indian having its
address at SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA -
574225, KARNATAKA, INDIA**

The following specification describes and ascertains the nature of this invention and
the manner in which it is to be performed:

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FORM 3
THE PATENTS ACT, 1970
(38 OF 1970)



&
The Patents Rules, 2003
STATEMENT AND UNDERTAKING UNDER SECTION 8
(See section 8 rule 12)

1. Name of the Applicant		We, ALVA'S EDUCATION FOUNDATION, addressed at ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY, an Indian having its address at SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA, do hereby declare that we have not made any application for the same/substantially the same invention outside India			
2. Name, address and nationality of the joint applicant. - N.A.					
Name of the country	Date of application	Application No.	Status of the application	Date of publication	Date of grant
NA	NA	NA	NA	NA	NA
3. Name and address of the assignee		Not Applicable			
4. To be signed by the applicant or his authorized registered patent agent.		<div style="text-align: right;"> Authorized Signatory: Name in full: Mohan Alva Designation: Chairman Seal: </div>			
5. Name of the natural person who has signed					
N/A					

To The Controller of Patents,
The Patent Office,
at Chennai

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26-Oct-2018/77527/201841040380/Form 3

ABSTRACT

According to the basic aspect of the present invention there is provided an enhanced μ -DMFC device performance by using micro-channels fabricated in <100> silicon wafer orientation for transport of fuel (methanol with water at anode and air/oxygen at cathode) and P(VDF-TrFE) coated nafion membrane as proton exchange membrane. The process comprises: cleaning of <100> silicon wafers, growing of silicon dioxide (SiO_2) by wet oxidation, spinning positive photo resist on both sides and front side UV exposure using level 1 mask (to feed methanol with water at anode and air at cathode), development of positive photo resist, etching front side SiO_2 using buffered hydrofluoric acid, stripping front side and back side positive photo resist using acetone, etching silicon by means of tetra methyl ammonium hydroxide for through holes, spinning positive photo resist on both sides and front side UV exposure using level 2 mask (micro channel mask), development of positive photo resist, etching front side SiO_2 using buffered hydrofluoric acid, stripping front side and back side positive photo resist using acetone, etching silicon by means of tetra methyl ammonium hydroxide for micro-channels, sequential sputtering of Chrome-Gold to form electrical contacts and electrodes on micro-channels. Further, Pt nano particles (for anode) and Pt-Ru nano particles (for cathode) are suspended in Iso Propyl Alcohol to form the catalyst solutions which are then coated on gas diffusion layers by brushing method. Further, nafion membrane is coated with P(VDF-TrFE) prepared by dissolving P(VDF-TrFE) powder in DMA (Dimethyl Acetamide). μ -DMFC device is assembled by sandwiching membrane electrode assembly (consisting of P(VDF-TrFE) coated nafion membrane sandwiched between two GDLs) between two silicon micro channels and the assembly is placed between two identical aluminum reservoirs both at anode and cathode of μ -DMFC for enhanced power output.

Ref: Fig 6.

FIELD OF INVENTION

The present invention is for the enhancement of micro direct methanol fuel cell performance using micro-channels fabricated from <100> silicon wafer orientation and P(VDF-TrFE) coated nafion membrane as proton exchange membrane. Particularly, the present invention contains processes like cleaning, etching and sputtering. The present invention uses micro-channels fabricated from <100> silicon wafer orientation and P(VDF-TrFE) coated nafion membrane as proton conducting membrane.

BACKGROUND OF THE INVENTION

Micro direct methanol fuel cells (μ -DMFCs) are among the leading contenders for energy generation for variety of applications and especially for wearable and portable devices. The advantage of these cells is that it uses methanol as fuel, practically at room temperature as opposed to hydrogen fuel cell which requires large hydrogen storage vessel and hence, not suitable for wearable and portable devices. The technology of μ -DMFC devices is maturing and these devices are increasingly finding applications in many types of electronic devices.

The new and renewable sources of energy are constantly evolving to decrease long-term dependence on oil and other fossil fuels. Fuel cells are potential candidates for this purpose. Proton exchange membrane (PEM) fuel cells such as μ -DMFCs have the potential to be a renewable energy source, owing to their high efficiency, high energy density and quiet operation. The PEM is the key component of μ -DMFCs. The chief function of the membrane is to allow the transport of protons generated at the anode and prevent the direct contact with the fuel and oxidant. Another key component of these fuel cells is flow field plate through which hydrogen fuel reaches the anode and oxygen reaches the cathode. The second function of the flow field plate is the electron collection for delivering power to the outside world.

Various direct methanol fuel cells have been devised in prior art some of the measures are as follows:

US6387559B1 relates to a fuel cell system and the method of forming the fuel cell system including a base portion, formed of a singular body, and having a major surface. At least one fuel cell membrane electrode assembly is formed on the major surface of the base portion. A fluid supply channel including a mixing chamber is defined in the base portion and communicating with the fuel cell membrane electrode assembly for supplying a fuel-bearing fluid to the membrane electrode assembly. An exhaust channel including a water recovery and recirculation system is defined in the base portion and communicating with the membrane electrode assembly. The membrane electrode assembly and the cooperating fluid supply channel and cooperating exhaust channel forming a single fuel cell assembly.

US6660423 relates to a fuel cell device and the method of forming the fuel cell device including a base portion, formed of a singular body, and having a major surface. At least one fuel cell membrane electrode assembly including a plurality of hydrophilic threads for the wicking of reaction water is formed on the major surface of the base portion. A fluid supply channel including a mixing chamber is defined in the base portion and communicating with the fuel cell membrane electrode assembly for supplying a fuel-bearing fluid to the membrane electrode assembly. An exhaust channel including a water recovery and recirculation channel is defined in the base portion and communicating with the membrane electrode assembly and the plurality of hydrophilic threads. The membrane electrode assembly and the cooperating fluid supply channel and cooperating exhaust channel forming a single fuel cell assembly.

US20060183015 relates to an efficient and passive micro fuel cell includes an anode plate, a reaction plate, a cathode plate and a condensation plate. The anode plate draws a dilute solution of methanol from a fuel tank to delivery to a series of upper oxidation reaction room through micro-channels by thermal capillarity. The

condensation plate separates carbon dioxide and vapor from each other. Meanwhile, the methanol solution is delivered to a plurality of lower oxidation reaction rooms. Protons pass through the inner walls of the reaction holes and a porous membrane layer and arrive in the lower reduction reaction rooms. The lower reduction reaction rooms and the lower oxidation reaction rooms have reaction holes whose inner walls have carbon nanotubes and catalysts. A plurality of upper reduction reaction rooms delivers oxygen for the reduction reaction and drains the reduced water at the same time.

CN101579632 relates to the field of fuel-cell catalyst, in particular discloses a nickel palladium/ silicon microchannel catalyst. The preparation thereof is as follows: the step of electroless plating deposit of a nickel palladium membrane is carried out on a silicon microchannel frame, and a nickel palladium/ silicon microchannel composite material is processed by rapid annealing for 6 to 10 minutes at the temperature from 300 to 500 DEG C under the argon atmosphere. The nickel palladium/ silicon microchannel catalyst can be used for preparing the electrode of the integratable direct methanol fuel cell.

CA 2400027 relates to a membrane, in particular, a membrane for use in a methanol fuel cell. The inventive membrane comprises complexing agents for cations and, therefore, functions like an anion exchanger. In a particular embodiment, the membrane comprises complexing agents selected from the group of crown ethers, cryptates, or of cryptate-like compounds based on carbon cyclic compounds or silicon cyclic compounds.

EP 1804326 relates to a flow connector for a microfluidic system through which a solution of at least an oxidable compound is fed to a feed manifold of an energy converting electrochemical device having a flat coupling area with the flow connector is made as a distinct article of manufacture applicable onto the electrochemical device. The flow connector comprises a monocrystalline silicon platform having at

least two distinct channels defined on the bottom side of the platform, on the top side of which a plurality of functional modules of said microfluidic system are fixed in correspondence of respective through holes communicating with a respective channel; a first channel connecting two or more of said through holes; the second channel connecting two or more other through holes; through holes first and second, respectively connected to said channels first and second, coinciding with a suction port and with a delivery port, respectively, of a micropump module fixed onto the silicon platform; at least the through hole coinciding with the suction port of the micro pump being connected through said first channel to a through hole coinciding with the outlet port of a first solvent release micro valve module from a first supply cartridge and to a through hole coinciding with the outlet port of a second oxidable compound release micro valve module from a second supply cartridge; upon coupling the channeled bottom side of said silicon platform to said flat coupling area of the device, an inlet of said solution feed manifold coinciding with said second channel. The depleted solution discharge manifold of the device has an outlet connecting to the first channel of the silicon flow connector and through a fifth through hole coinciding with the inlet of a third solution discharge micro valve module, to a third cartridge into which bleeding depleted waste solution.

EP 1191621 relates to an inorganically modified membrane consists of an organic sulfonated polymer and an inorganic phase made of oxides of silicon, organically modified silicon, titanium and/or zirconium. The oxides can be partially replaced by phosphates.

US6833167 relates to a methanol fuel cell comprising a membrane which conducts metallic cations, in which the metallic cations induce the transport of the charge inside the membrane and are advantageously guided in a circuit in the form of a base from the cathode chamber to the anode chamber. The inventive methanol fuel cell prevents the methanol drag associated with proton-conductive membranes, thus

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producing higher power outputs on a regular basis. A separate transport of the water produced by the reaction is not necessary.

CN 103490081 provides a modified nafion proton exchange membrane and a preparation method thereof. The method comprises the steps as follows: a nafion proton exchange membrane is soaked in a polybenzimidazole phosphoric acid dissolved liquid at vacuum; the soaked nafion proton exchange membrane is dried; and the modified nafion proton exchange membrane is obtained. The invention further provides a direct methanol fuel cell membrane electrode and a preparation method thereof. The method comprises the steps as follows: a cathode catalyst layer and an anode catalyst layer are loaded on a cathode gas diffusion layer and an anode gas diffusion layer respectively; a cathode electrode and an anode electrode are obtained; the anode electrode, the modified nafion proton exchange membrane and the cathode electrode are stacked sequentially and subjected to hot pressing; the membrane electrode is obtained; and the modified nafion proton exchange membrane is adjacent to the anode catalyst layer and the cathode catalyst layer respectively. The nafion proton exchange membrane modified by phosphoric acid dissolved polybenzimidazole to reduce a transfer space of a hydrotropic substance, so that methanol solution penetration can be reduced; and the properties of the membrane, such as a proton conduction rate and the like, are better, so that the property of a cell can be improved.

WO2017084377 relates to the technical field of fuel cells, and specifically, to a novel proton exchange membrane for a methanol fuel cell. The proton exchange membrane comprises a porous anode aluminum oxide template. A nafion membrane layer is disposed on the surface of the porous anode aluminum oxide template. An inert metal layer is disposed on the surface of the nafion membrane layer. In the present invention, the structure is compact and does not have a crackle, which effectively restrains the infiltration of methanol in the methanol fuel cell, and improves the utilization rate of the methanol of the methanol fuel cell and the service

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life of the methanol fuel cell; the proton exchange membrane has a simple structure and low production cost.

WO2016205972 discloses a poly(oxadiazole aryl ether-co-bisphenol fluorine) proton exchange membrane and a preparation method thereof. The proton exchange membrane is obtained by one step; specifically: mixing and reacting a base polymer obtained from a decafluorobenzadiazole and bisphenol fluorine copolymer with a functionalized reagent and forming a membrane to obtain the proton exchange membrane. The reaction with the functionalized reagent is primarily realized via nucleophilic aromatic substitution. The proton exchange membrane obtained by the present invention has a high ion conductivity; at 30°C the conductivity is $58 \text{ mS}\cdot\text{cm}^{-1}$, and at 70°C it reaches $137 \text{ mS}\cdot\text{cm}^{-1}$. Further, the proton exchange membrane has strong mechanical properties and a low methanol permeability, the methanol permeability being less than half that of a nafion® 117 membrane. Upon assembling the membrane in a direct methanol fuel cell and testing, results showed that at 90°C, the maximum power of the cell was $75 \text{ mW}\cdot\text{cm}^{-2}$, and at 100°C, the maximum power reached $85 \text{ mW}\cdot\text{cm}^{-2}$.

CN105680077 belongs to the technical field of a fuel cell, in particular to a proton exchange membrane. The proton exchange membrane is prepared from a combination comprising a sulfonated poly ether ether ketone solution, sulfonated polyphenylene oxide sppo, a polyaniline filter liquid, heteropoly acid and a titanium dioxide fluid. The proton exchange membrane has the advantages of favorable mobility and processability; with the combination of the titanium dioxide fluid, the sulfonated poly ether ether ketone solution, the sulfonated polyphenylene oxide sppo and the polyaniline filter liquid, the reduction of proton conductivity can be reduced, the methanol permeability of the composite proton exchange membrane is effectively reduced, the comprehensive performance of the proton exchange membrane is improved, the methanol permeation problem when an nafion membrane is used for a direct methanol fuel cell (DMFC) is solved, the proton conductivity is also not

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reduced, and the requirement for PEM used by the DMFC is met; and the proton exchange membrane can be used for the DMFC and also can be used for an alcohol fuel cell such as a direct ethanol fuel cell.

The present invention overcomes the inadequacies of the prior art by providing μ -DMFC devices by using micro-channels fabricated with <100> silicon wafer orientation for the flow of reactants and P(VDF-TrFE) coated nafion membrane as an efficient proton exchange membrane.

OBJECT OF THE PRESENT INVENTION

One or more of the problems of the conventional prior art may be overcome by various embodiments of the present invention.

Accordingly, the primary object of the present invention is to provide a fuel cell device (μ -DMFC) with higher efficiency by using silicon micro-channels etched in <100> silicon wafer orientation for the flow of the reactants and P(VDF-TrFE) coated nafion membrane as PEM..

It is another object of the present invention, wherein the said fuel cell device uses processes of silicon wafer cleaning, ultra violet lithography, silicon etching and Cr-Au deposition methods for preparation of silicon micro-channels.

It is even another object of the present invention, wherein the said fuel cell device uses Pt nano particles (at anode) and Pt-Ru nano particles (at cathode) as catalysts.

It is even another object of the present invention, wherein the two catalyst nano particles are suspended in Iso Propyl Alcohol and are coated on GDL by brushing technique.

It is one aspect of the present invention, wherein the said P(VDF-TrFE) is prepared by dissolving P(VDF-TrFE) powder in DMA (Dimethyl Acetamide) as a solvent.

It is one aspect of the present invention, wherein the said P(VDF-TrFE) is coated on nafion membrane by dip coating method.

It is one aspect of the present invention, wherein the said fuel cell device uses aluminium reservoirs for storage and supply of methanol and water at anode and oxygen/air at cathode.

It is one aspect of the present invention, wherein the said fuel cell device is assembled by sandwiching P(VDF-TrFE) coated nafion membrane in between two GDLs which is placed between two silicon micro channels and further sandwiched between two aluminium reservoirs.

SUMMARY OF THE PRESENT INVENTION

According to the basic aspect of the present invention there is provided an enhanced μ -DMFC performance by using micro-channels fabricated in <100> silicon wafer and P(VDF-TrFE) coated nafion membrane as PEM. The process for fabrication of micro-channels comprises: cleaning of <100> silicon wafers, growing of Silicon dioxide (SiO_2) by wet oxidation, spinning positive photo resist on both sides and front side UV exposure using level 1 mask (methanol feed mask), development of positive photo resist, etching front side SiO_2 using buffered hydrofluoric acid (BHF), stripping front side and back side positive photo resist using acetone, etching silicon by means of tetra methyl ammonium hydroxide for through holes, spinning positive photo resist on both sides and front side UV exposure using level 2 mask (micro channel mask), development of positive photo resist, etching front side SiO_2 using buffered hydrofluoric acid, stripping front side and back side positive photo resist using acetone, etching silicon by means of tetra methyl ammonium hydroxide for micro-channels, sequential sputtering of Chrome-Gold (Cr-Au) to form electrical

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contacts on micro-channels. Pt nano particles (for anode) and Pt-Ru nano particles (for cathode) are suspended in Iso Propyl Alcohol and are coated on GDL by brushing technique. P(VDF-TrFE) is prepared by dissolving P(VDF-TrFE) powder in DMA (Dimethyl Acetamide) as a solvent and is coated on nafion membrane by dip coating method. Aluminium reservoirs are used for storage and supply of methanol and water at anode and oxygen/air at cathode. Fuel cell device is assembled by sandwiching P(VDF-TrFE) coated nafion membrane in between two GDLs which is placed between two silicon micro channels and further sandwiched between two aluminium reservoirs.

BRIEF DESCRIPTION OF DRAWINGS

Figure 1 illustrates cross section of an anisotropic wet etch on a silicon wafer that creates a groove with a trapezoidal shape, wherein a represents $\langle 100 \rangle$ plane, b represents $\langle 111 \rangle$ plane, c represents etch mask (SiO_2), d represents silicon wafer, according to present invention.

Figure 2 illustrates schematic of μ -DMFC, wherein f represents inlet for methanol with water, g represents silicon micro-channels, h represents outlet for carbon dioxide, i represents GDL, j represents Pt catalyst, k represents nafion membrane (PEM), l represents Pt-Ru catalyst, m represents outlet for water, n represents inlet for air (oxygen), e^- represents electrons, H^+ represents hydrogen ions, according to present invention.

Figure 3(a-b) illustrates level 1 mask design for through-holes (methanol feed mask) and level 2 mask design for micro-channels (micro channel mask), according to present invention.

Figure 4(a-k) illustrates the diagrammatic representation of process flow for fabrication of silicon micro-channels, according to present invention.

Figure 5 illustrates the isometric view of Aluminium reservoir design for storage of fuel (methanol with water) and air with dimension 20mm × 17mm × 5mm, wherein o represents the inlet through which fuel is fed, p represents opening for outlet of fuel which then flows into the silicon micro-channels, according to present invention.

Figure 6 illustrates cross section of μ -DMFC along with two Aluminium reservoirs, wherein f represents inlet for methanol with water, g represents silicon micro-channels, h represents outlet for carbon dioxide, i represents GDL, j represents Pt catalyst, k represents nafion membrane (PEM), l represents Pt-Ru catalyst, m represents outlet for water, n represents inlet for air (oxygen), q represents aluminium reservoirs, r represents Cr-Au contacts, s represents P(VDF-TrFE) coating, according to present invention.

Figure 7 illustrates the flow chart of process flow for fabrication of silicon micro-channels, preparation of P(VDF-TrFE) polymer and assembling of μ -DMFC according to present invention.

DETAILED DESCRIPTION OF THE INVENTION WITH REFERENCE TO ACCOMPANYING DRAWINGS

The preferred embodiment of the present invention will now be explained with reference to the accompanying drawings. It should be understood however that the disclosed embodiments are merely exemplary of the invention, which may be embodied in various forms. The following description and drawings are not to be construed as limiting the invention and numerous specific details are described to provide a thorough understanding of the present invention, as the basis for the claims and as a basis for teaching one skilled in the art how to make and/or use the invention. However in certain instances, well-known or conventional details are not described in order not to unnecessarily obscure the present invention in detail.

With reference to Figure 1, the present invention of fabricating micro-channels from $\langle 100 \rangle$ silicon wafers for the flow of reactants in μ -DMFC. Silicon wafers come with various orientations. The orientations of silicon wafers are classified using Miller indices. These indices include such descriptions as $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$. Wet anisotropic etching of silicon wafers is done using Tetramethylammonium hydroxide (TMAH) solution to fabricate micro-channels. The etching angle resulting from above orientations depends on the orientation. A V-groove is formed as a result of wet anisotropic etching of Si $\langle 100 \rangle$ wafer making an angle 54.74° between the $\langle 100 \rangle$ and $\langle 111 \rangle$ planes.

The cross section of micro-channels fabricated from $\langle 100 \rangle$ silicon wafers is trapezoidal in shape. Trapezoidal channels have less volume when compared to that of rectangular channels. Methanol with water is fed into the channels through gravity flow and air is fed through natural breathing. The linear velocity of the fuel in trapezoidal channels is more than that of rectangular channels. As a result, the fuel consumption is more, consequently leading to improved cell efficiency. The micro channel design fabricated from $\langle 100 \rangle$ silicon wafer which results in better efficiency of μ -DMFC and it needs to be protected.

μ -DMFCs are among the leading contenders for clean energy generation for variety of applications. μ -DMFC consists of the following: A proton conducting membrane (Nafion) is sandwiched between two gas diffusion layers (GDLs) at anode and cathode (electrodes) of μ -DMFC; this Membrane Electrode Assembly (MEA) is the heart of the μ -DMFC. Methanol diffuses through the micro-porous GDL (which regulates the transport of methanol) to the catalyst which generates protons and electrons. The protons then diffuse through the nafion membrane to the cathode. The electrons move from anode to cathode through external circuit. Protons and electrons react with oxygen at the cathode to form water.

The equations for the process are as shown below:

Anode reaction: $\text{CH}_3\text{OH} + \text{H}_2\text{O} \rightarrow 6\text{H}^+ + 6\text{e}^- + \text{CO}_2$ (Oxidation)

Cathode reaction: $\frac{3}{2}\text{O}_2 + 6\text{H}^+ + 6\text{e}^- \rightarrow 3\text{H}_2\text{O}$ (Reduction)

Overall reaction: $\text{CH}_3\text{OH} + \frac{3}{2}\text{O}_2 \rightarrow 2\text{H}_2\text{O} + \text{CO}_2$ (Redox reaction)

A schematic of μ -DMFC is shown in Figure 2.

The entire MEA is sandwiched between two silicon chips with micro-channels which regulate the flow of methanol at the anode and air at cathode as shown in Figure 2. The negative charge (electrons) collected by the metallic electrode moves into the external circuit from anode to cathode, thus balancing the charge transfer process.

The heart of the μ -DMFC is MEA which consists of micro-porous layer (Toray carbon paper) which regulates the flow of methanol to the catalyst at the anode, a high efficiency catalyst layer (Pt) for the generation of protons (H^+) from methanol, a high proton conductance membrane layer (nafion) for the transfer of protons and a high efficiency catalyst (Pt-Ru) at the cathode for the conversion of oxygen, protons and electrons into water. Micro-channels etched in silicon wafer for the flow of methanol at the anode and air/oxygen at the cathode serve as flow channels. A metallic layer (Cr-Au) on the micro-channels of silicon chips (both at anode and cathode) is used for the exit of electrons from anode through the external circuit and entry into the cathode for charge balance.

Figure 3(a-b) shows the mask design for lithography. The lithography process is carried out in two levels. The first level of lithography is for etching through-holes (which connect reservoirs of methanol/air and micro-channels), while the second level of lithography is for etching micro-channels in silicon wafers.

The detailed process flow for fabrication of silicon micro-channels is shown below, which is only illustrative and should not be construed as limitation. With reference to Figure 4(a-k) and Figure 7 the said process flow comprises:

- 1) RCA cleaning of silicon wafers with following features: 2 inch, <100>, Single Sided Polished (SSP) and resistivity: 4-7ohm-cm.
- 2) Wet oxidation of wafers for growing oxide (1000nm thickness).
- 3) Positive Photo Resist (PPR) spinning at the rate of 300rpm for 10sec and 3000rpm for 30sec on both sides of the wafers.
- 4) UV exposure for through-holes using methanol feed mask (level 1). Development of the PPR for 25 sec using photoresist developer MF319.
- 5) Oxide removal using 5:1 BHF for 10min.
- 6) Strip PPR on both sides of silicon wafers using acetone. TMAH etching of Silicon for 4 hours to etch 150microns for methanol feed holes.
- 7) PPR spinning at the rate of 300rpm for 10sec and 3000rpm for 30sec on both sides of the wafers.
- 8) UV exposure for micro-channels using micro channel mask (level 2). Development of the PPR for 25 sec using photoresist developer MF319.
- 9) Oxide removal using 5:1 BHF for 10min.
- 10) Strip PPR on both sides of silicon wafers using acetone. TMAH etching of Si for 3 hours to obtain micro-channels of depth 140microns.
- 11) While etching the micro-channels, the through holes (etched earlier in step 6) will also be etched along with the micro-channels. As a result through holes will be formed.

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- 12) Chrome-gold sputtering to deposit Cr (10nm) and Au (100nm) for conduction of electrons (that accumulate on the GDL surface facing the micro-channels) towards external electrical circuit.

Preparation of catalyst:

- 13) Platinum (Pt) nano particles is used as catalyst at the anode of the μ -DMFC while Platinum-Ruthenium (Ru) nano particles is used as catalyst at the cathode of μ -DMFC. These nano particles are taken in two separate glass bottles (i.e. for anode and cathode) and the solvent (Iso Propyl Alcohol (IPA)) is added. The mixture is sonicated for 1 hour for the catalyst nano particles to be suspended in the solvent.

- 14) The catalysts are coated on GDLs by brushing technique.

Preparation of PEM:

- 15) Nafion is cleaned with IPA and acetone. It is post-baked at 100°C for 1 hour to remove any moisture or water molecules present. Further, nafion membrane is coated with P(VDF-TrFE) prepared by dissolving P(VDF-TrFE) powder in DMA (Dimethyl Acetamide) by dip-coating method.

Preparation of MEA:

- 16) The P(VDF-TrFE) coated nafion membrane is sandwiched between two GDLs at anode and cathode to form the MEA of μ -DMFC.

Aluminium reservoirs

- 17) With reference to Figure 5, the said design for Aluminium reservoirs comprises: a reservoir to contain methanol with water at the anode which is supplied continuously to GDL through micro-channels, and another reservoir at the cathode for air which is supplied to GDL through micro-channels via natural breathing. Two identical aluminium reservoirs sandwich the μ -DMFC, thereby

serving also as an external protection for the μ -DMFC besides serving as reservoirs for methanol with water at anode and air at cathode.

Assembly of μ -DMFC

18) With reference to Figure 6, the said process for assembling the μ -DMFC, starting from anode to cathode, comprises: silicon micro-channels, GDL, a coat of Pt catalyst, P(VDF-TrFE) coated nafion membrane, a coat of Pt-Ru catalyst, GDL and silicon micro-channels. This assembly is sandwiched between two identical aluminium reservoirs both at anode and cathode. The assembling of μ -DMFC is secured by using 3M tape.

Although the invention has been described with reference to specific embodiments, this description is not meant to be construed in a limiting sense. Various modifications of the disclosed embodiments, as well as alternative embodiments, will be apparent to persons skilled in the art. It is therefore, contemplated that the appended claims will cover all modifications that fall within the true scope of the invention.

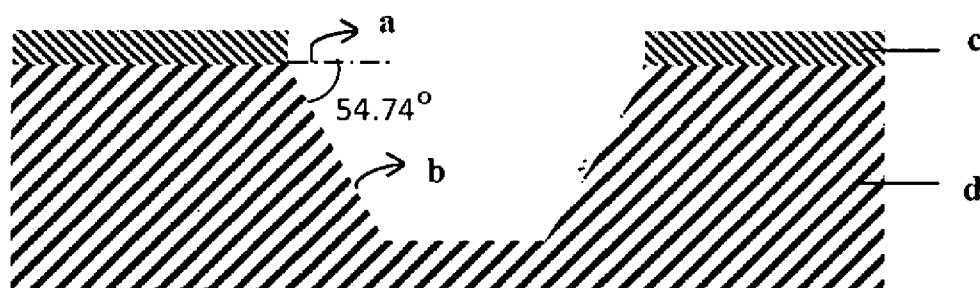
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FIGURE 1

Total number of sheets: 10

Number of sheet: 1/10



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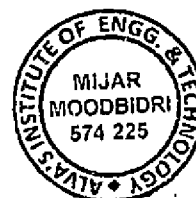
For ALVA'S EDUCATION FOUNDATION,

Authorized Signatory:

Name in full: Mohan Alva

Designation: Chairman.

Seal:



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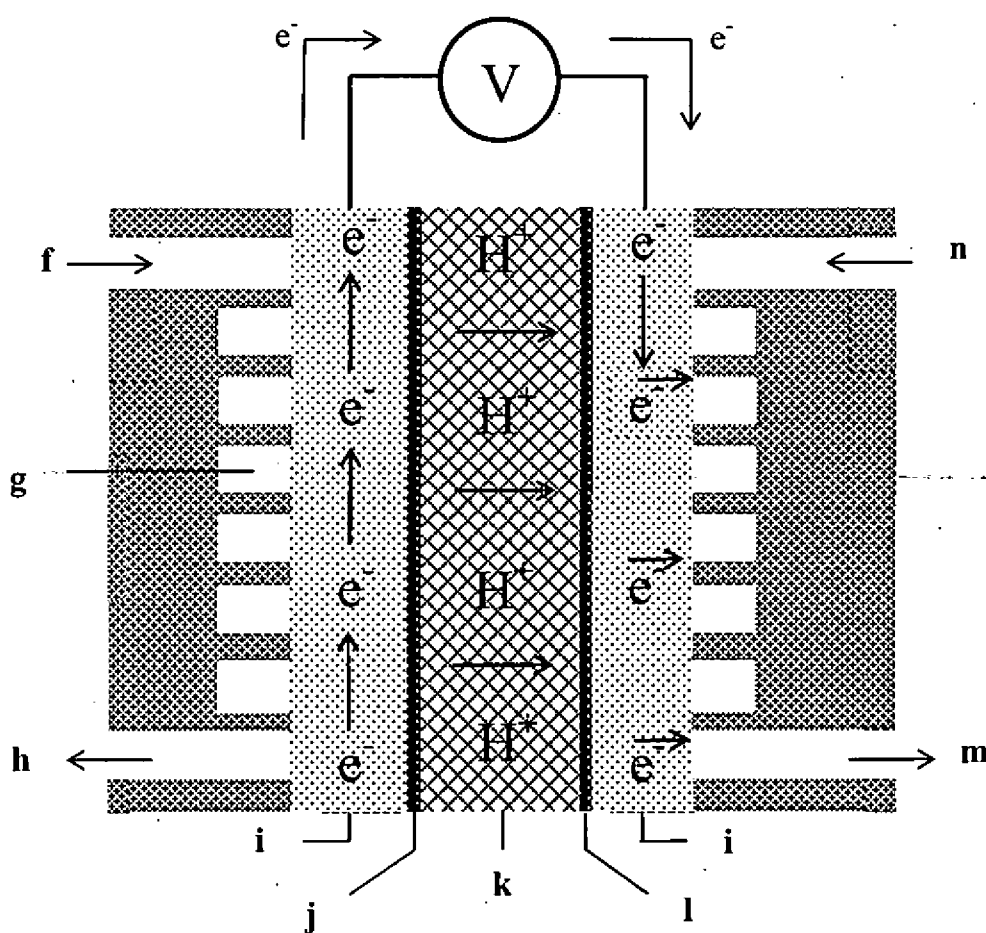
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FIGURE 2

Total number of sheets: 10

Number of sheet: 2/10



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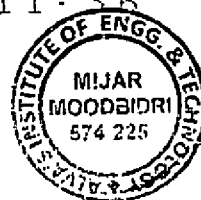
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Name in full: Moham Alva

Designation: Chairman

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APPLICATION NO:

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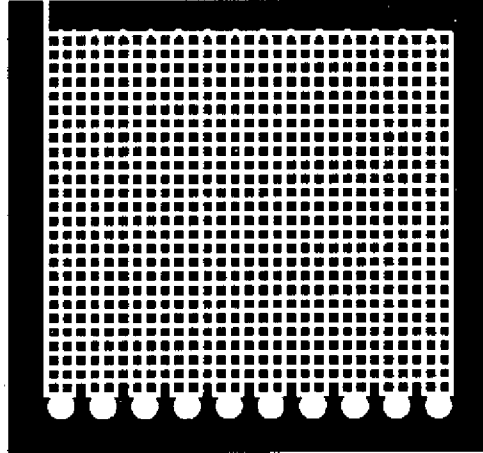
FIGURE 3(a)

Total number of sheets: 10

Number of sheet: 3/10



FIGURE 3(b)



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Designation: Chairman.



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APPLICATION No:

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FIGURE 4(a)

Total number of sheets: 10

Number of sheet: 4/10

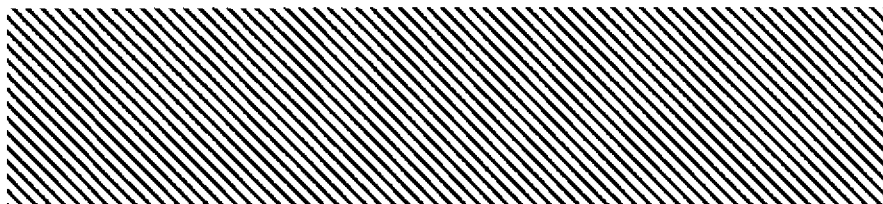


FIGURE 4(b)

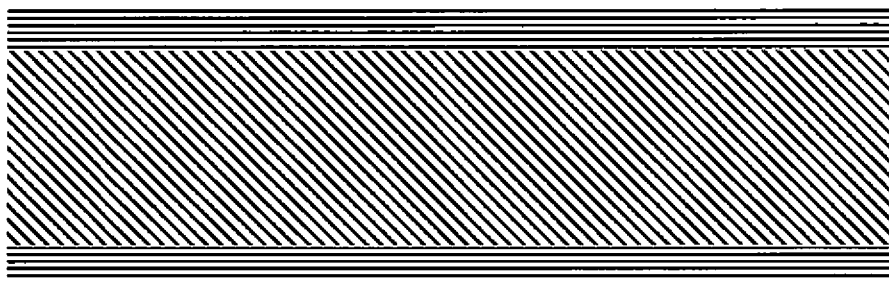
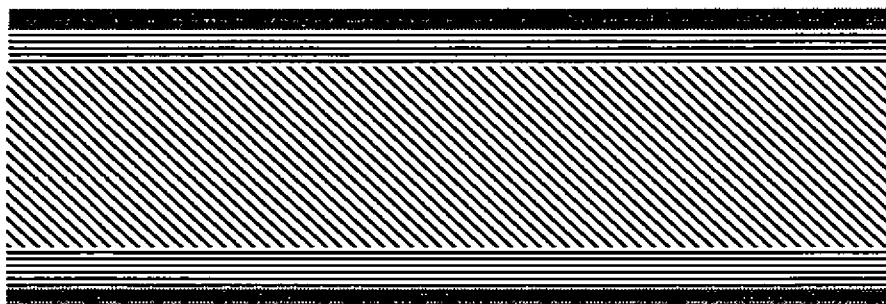


FIGURE 4(c)



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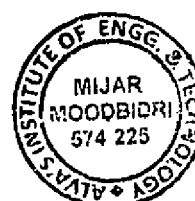
Authorized Signatory:

Name in full: Mohan Alva

Designation: Chairman

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FIGURE 4(d)

Total number of sheets: 10

Number of sheet: 5/10

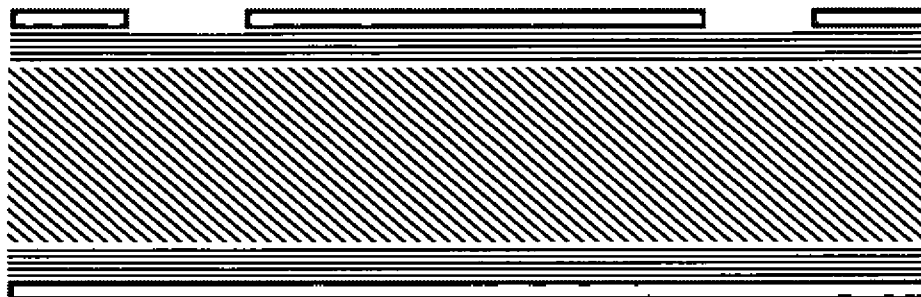


FIGURE 4(e)

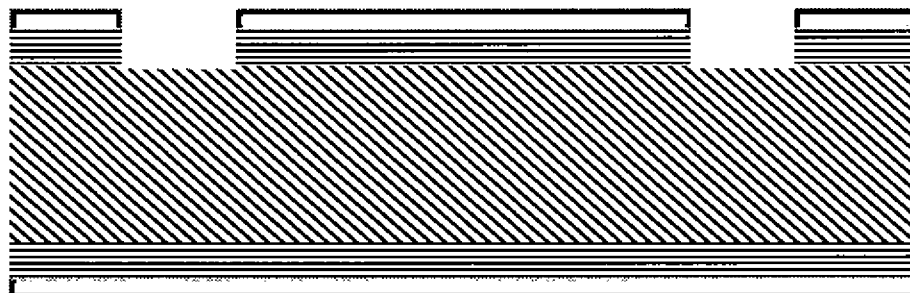
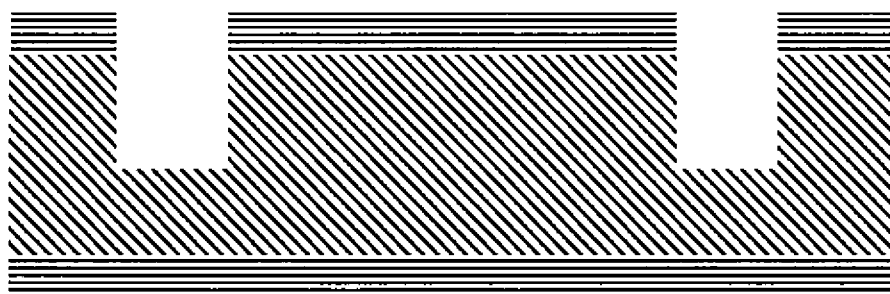


FIGURE 4(f)



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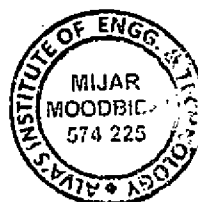
For ALVA'S EDUCATION FOUNDATION,

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Name in full: Mohan Alva

Designation: Chairman

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APPLICATION NO:

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FIGURE 4(g)

Total number of sheets: 10

Number of sheet: 6/10

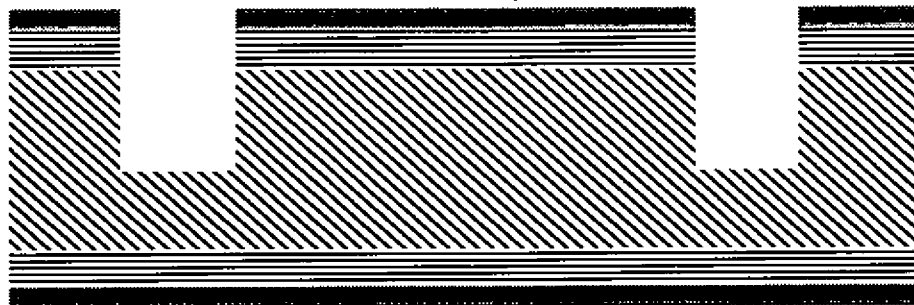


FIGURE 4(h)

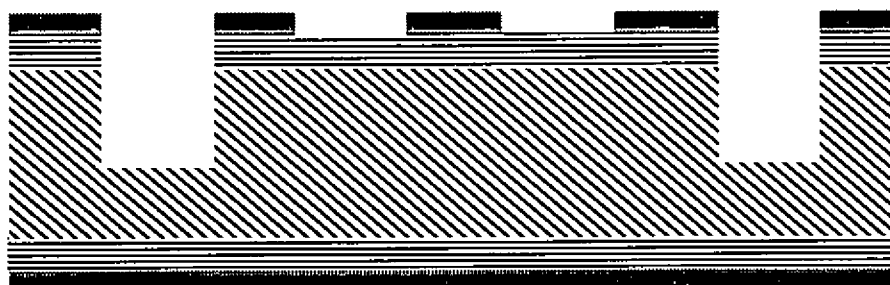
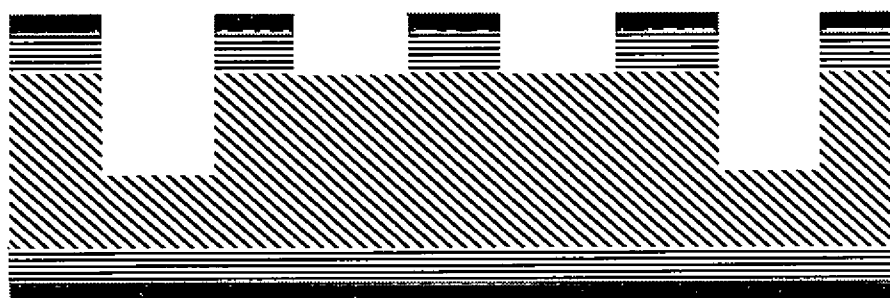


FIGURE 4(i)



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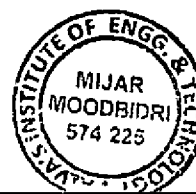
Authorized Signatory:

Name in full: Moham Alva

Designation: Chairman

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APPLICATION NO:

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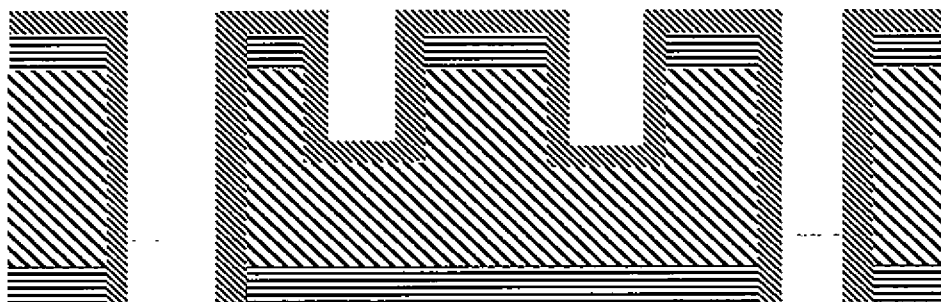
FIGURE 4(j)

Total number of sheets: 10

Number of sheet: 7/10



FIGURE 4(k)



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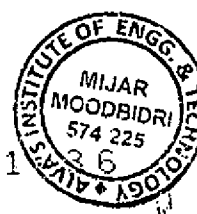
For ALVA'S EDUCATION FOUNDATION,

Authorized Signatory:

Name in full: Mohan Alva

Designation: chairman

Seal:



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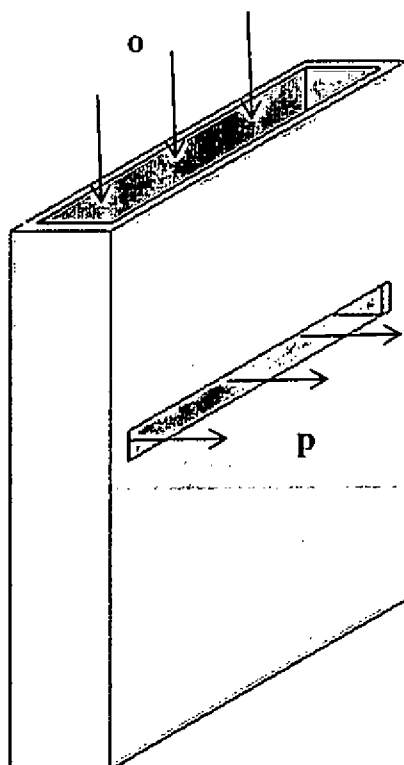
APPLICATION NO:

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FIGURE 5

Total number of sheets: 10

Number of sheet: 8/10



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Authorized Signatory:

Name in full: Mohan Alva

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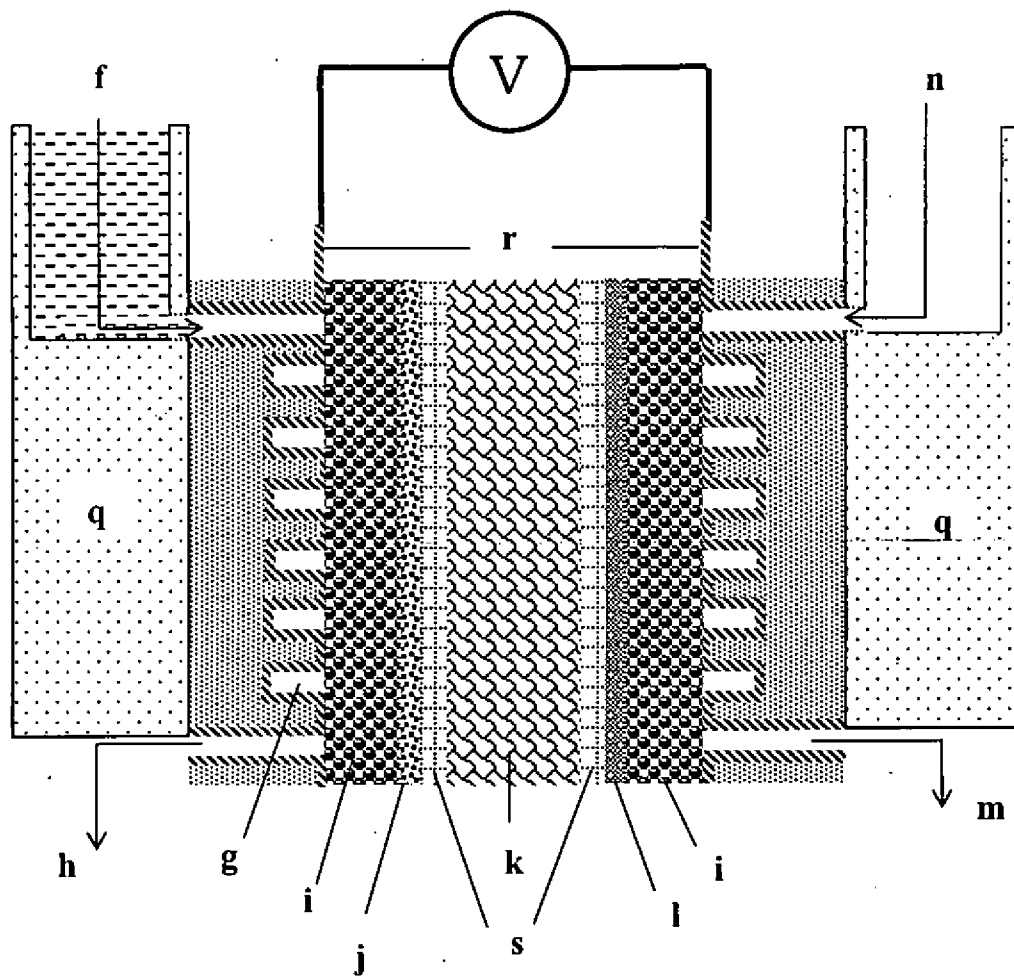
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FIGURE 6

Total number of sheets: 10

Number of sheet: 9/10



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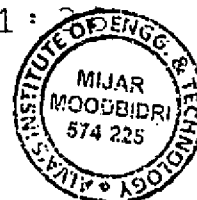
Name in full: Mohan Alva

Designation: Chairman

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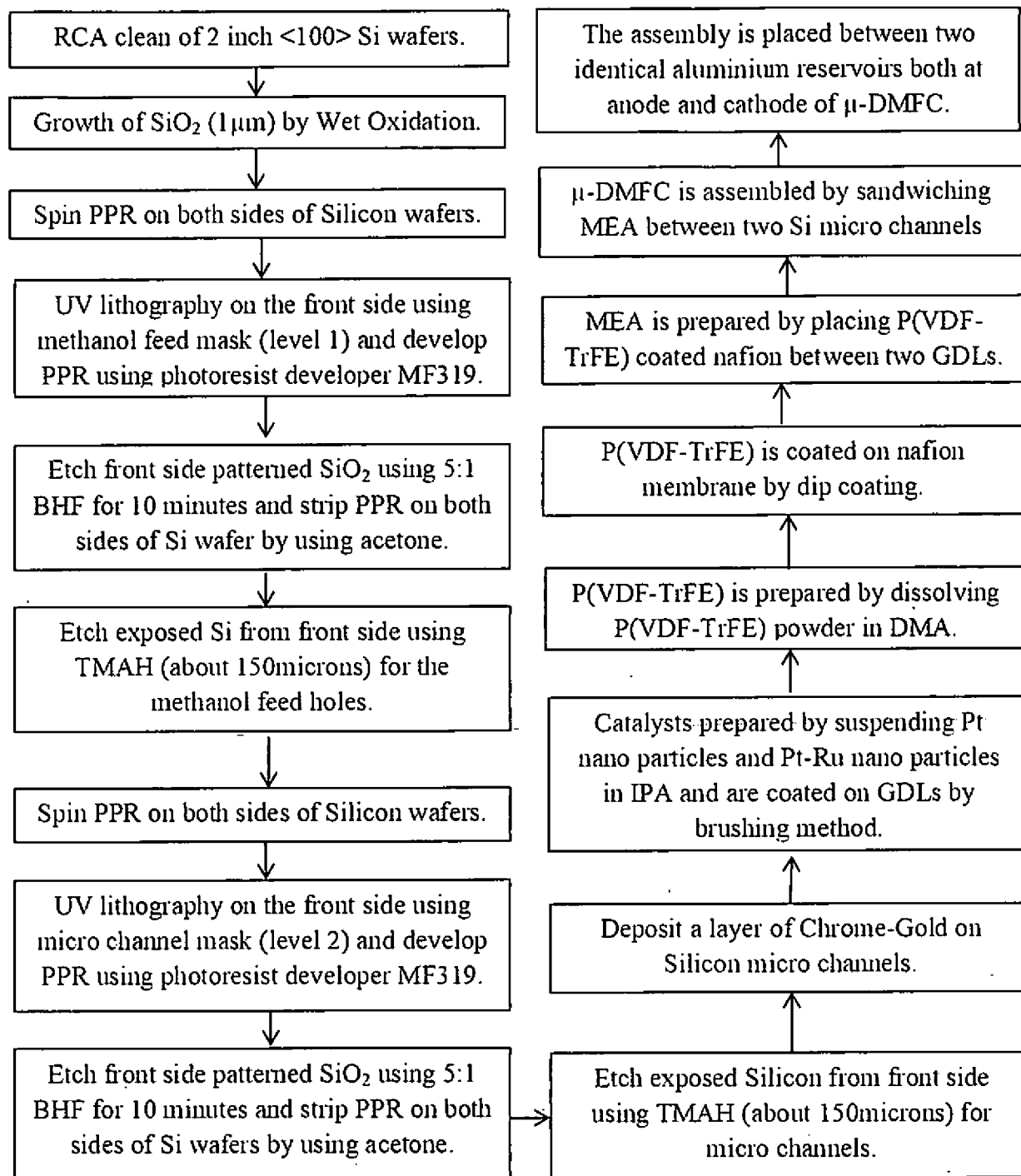
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FIGURE 7

Total number of sheets: 10

Number of sheet: 10/10



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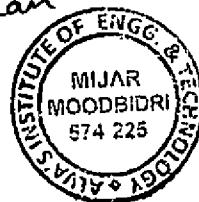
For ALVA'S EDUCATION FOUNDATION,

Authorized Signatory:

Name in full: Mohan Alva

Designation: Chairman

Seal:



WE CLAIM

1. A μ -DMFC device with enhanced performance with the use of <100> Silicon wafer orientation for fabrication of micro channels for the flow of reactants in μ -DMFC and with the use of P(VDF-TrFE) coated nafion membrane as proton exchange membrane in μ -DMFC, the process comprising of:
 - a) Growing of SiO_2 on <100> silicon wafers by wet oxidation.
 - b) Spinning of positive photo resist followed by UV lithography with level 1 mask (methanol feed mask) and development using photoresist developer MF319.
 - c) Etching front side SiO_2 using buffered hydrofluoric acid and stripping the positive photo resist on both sides using acetone and etching silicon by means of Tetra Methyl Ammonium Hydroxide.
 - d) Spinning of positive photo resist followed by UV lithography with level 2 mask (micro channel mask) and development using photoresist developer MF319.
 - e) Etching front side SiO_2 using buffered hydrofluoric acid and stripping the positive photo resist on both sides using acetone and etching silicon by means of Tetra Methyl Ammonium Hydroxide.
 - f) Sputtering Cr-Au on micro channels.
 - g) Preparing P(VDF-TrFE) polymer by dissolving P(VDF-TrFE) powder in DMA (Dimethyl Acetamide) and coating it on Nafion membrane by dip coating method.

- h) Preparing the catalysts by suspending Pt nano particles (as anode catalyst) and Pt-Ru nano particles (as cathode catalyst) in Iso Propyl Alcohol and coating it on GDLs by brushing technique.

Where μ -DMFC is assembled by sandwiching membrane electrode assembly (consisting of P(VDF-TrFE) coated nafion membrane sandwiched between two GDLs) between two silicon micro channels and the assembly is placed between two identical aluminum reservoirs both at anode and cathode of μ -DMFC for enhanced power output.

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For ALVA'S EDUCATION FOUNDATION,

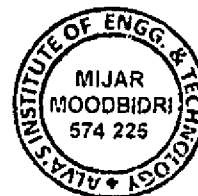
Authorized Signatory:



Name in full: Mohan Alva

Designation: Chairman

Seal:



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FORM 5
THE PATENT ACT, 1970
(39 of 1970)



&
The Patents Rules, 2003
DECLARATION AS TO INVENTORSHIP
[See section 10(6) and rule 13(6)]

1. NAME OF APPLICANTS

Name	Nationality	Address
ALVA'S EDUCATION FOUNDATION	India	SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA

I hereby declare that the true and first inventor(s) of the invention disclosed in the specification filed in pursuance of our application

Application no:

Title : ENHANCEMENT OF MICRO DIRECT METHANOL FUEL CELL (μ -DMFC) PERFORMANCE USING MICRO CHANNELS FABRICATED FROM <100> SILICON WAFER ORIENTATION AND P(VDF-TrFE) COATED NAFION MEMBRANE AS PROTON EXCHANGE MEMBRANE

Dated :

2. INVENTORS

Name	Nationality	Address
ARJUN SUNIL RAO	India	ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY, SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA
JAYARAMA ARASALIKE	India	ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY, SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA
MANJUNATHA DODDABALLAPURA VEERABHADRAIAH	India	ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY, SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA
RICHARD PINTO	India	ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY, SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA
SIDDHARTHA PRAKASH DUTTAGUPTA	India	INDIAN INSTITUTE OF TECHNOLOGY BOMBAY, POWAI, MUMBAI - 400076, MAHARASHTRA, INDIA
ACHANTA VENUGOPAL CHENNAI	India	TATA INSTITUTE OF FUNDAMENTAL RESEARCH, MUMBAI - 400005, MAHARASHTRA, INDIA

PATENT

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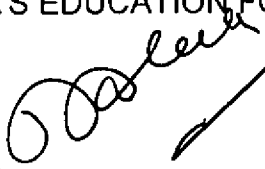
11/07/2018

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For ALVA'S EDUCATION FOUNDATION,

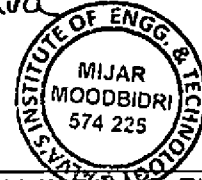
Authorized Signatory: 

Name in full:

Moham Alva

Designation: Chairman

Seal:



3. DECLARATION TO BE GIVEN WHEN THE APPLICATION IN INDIA IS FILED BY THE APPLICANT(S) IN THE CONVENTION COUNTRY:- **NA**

We the applicant(s) in the convention country hereby declare that our right to apply for a patent in India is by way of assignment from the true and first inventor(s) **NA**

4. STATEMENT **NA**

I assent to the invention referred to in the above declaration, being included in the complete specification filed in pursuance of the stated application.

Dated this 10th day of October 2018

Signature : **NA**

Name: **NA**

To
The Controller of Patents,
The Patent Office at Chennai.

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11/12/18



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CBR: 35519
DATE: 11/12/2018
Amt: 13750/-

11/12/18

FORM 9

**THE PATENTS ACT, 1970
(39 of 1970)**

**REQUEST FOR EARLY PUBLICATION
[See section 11A(2); rule 24A]**

We, **ALVA'S EDUCATION FOUNDATION**, addressed at **ALVA'S INSTITUTE OF ENGINEERING AND TECHNOLOGY SHOBHAVANA CAMPUS, MIJAR, MOODBIDRI, DAKSHINA KANNADA - 574225, KARNATAKA, INDIA** hereby request for early publication of my Patent application No **201841040380** dated **October 26, 2018**, for the invention **ENHANCEMENT OF MICRO DIRECT METHANOL FUEL CELL (μ -DMFC) PERFORMANCE USING MICRO CHANNELS FABRICATED FROM <100> SILICON WAFER ORIENTATION AND P(VDF-TrFE) COATED NAFION MEMBRANE AS PROTON EXCHANGE MEMBRANE** under section 11A(2) of the Act.

Dated this the 11th day of December 2018

For **ALVA'S EDUCATION FOUNDATION**,

Authorized Signatory:

Name in full: Mohan Alva

Designation: Chairman

Seal:

Chairman

Alva's Education Foundation (R)

MOODBIDRI-574227-D.K.

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11-Dec-2018/89435/201841040380/Form 9

From

RICHARD PINTO,
Alva's Education Foundation,
Alva's Institute Of Engineering And Technology
Shobhavana Campus, Mijar, Moodbidri,
Dakshina Kannada - 574225, Karnataka, India
Email: ariv@leintelligensia.com

October 26, 2018

D/71827



To

The Controller of Patents,
The Patents Office,
Guindy,
Chennai - 600032.

Sir,

Ref: Patent application for "ENHANCEMENT OF MICRO DIRECT METHANOL FUEL CELL (μ -DMFC) PERFORMANCE USING MICRO CHANNELS FABRICATED FROM <100> SILICON WAFER ORIENTATION AND P(VDF-TrFE) COATED NAFION MEMBRANE AS PROTON EXCHANGE MEMBRANE" in the name of **ALVA'S EDUCATION FOUNDATION**

Referring to the above, we are enclosing application for request for filing Complete Specification..

Form 1 original signed by applicant - 4 pages.

Form 3 original signed by applicant- 1 page.

Form 5 original signed by applicant -2 page.

Form2-Complete specification - 16 pages.

Total number of -Claims-1

Total number of pages for claims- 2 Page.

(additional claims -0)

Abstract-1 page.

Drawing Sheets- 10 pages.

Total No. of Pages- 29 pages.

We submit Fee of Rs.8800/- in cash.

We request you to take above on record.

Thanking You,

Yours faithfully,

R. Pinto
RICHARD PINTO.

Encl As above.

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26-Oct-2018/77527/201841040380/Other Patent Document