STUDIES ON CARBON/CARBON COMPOSITE BIPOLAR PLATE FOR PEM FUEL CELL IN AEROSPACE APPLICATION

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DEPARTMENT OF CHEMICAL ENGINEERING

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by

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submitted

in partial fulfillment of the requirement of the degree of Doctor of Philosophy

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Dedicated to

"My late grandmother Mrs. Shaanti Devi and my kidney failure younger sister Ms. Seema Rani who were the real propeller in the completion of this piece of work"

Statement

The thesis 'Studies on Carbon/Carbon Composite Bipolar Plate for PEM Fuel Cell in Aerospace Application' is my original work carried out by me at Vikram Sarabhai Space Centre, Indian Space Research Organisation, Thiruvananthapuram, Kerala, India and Indian Institute of Technology Delhi, Hauz Khas, New Delhi, India under the supervision of Dr. S.C. Sharma, Deputy Director, Vikram Sarabhai Space Centre and Dr. Anil Verma, Associate Professor, Department of Chemical Engineering, Indian Institute of Technology Delhi. I certify that no conflict of interest in any form is associated with the present work. Further, it is not presented in any R&D lab and institute for claiming any degree or diploma. I also certify that the details and information taken from the works of other investigators have been acknowledged in the thesis.

Thakur Sudesh Kumar Raunija

Certificate

This is certified that the work contained in the thesis entitled **'Studies on Carbon/Carbon Composite Bipolar Plate for PEM Fuel Cell in Aerospace Application'** by **Thakur Sudesh Kumar Raunija** for the award of the degree of **Doctor of Philosophy** has been carried out under our supervision.

The results contained in this thesis have not been submitted, in part or in full, to any other university or institute for the award of any degree or diploma.

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Preface

Proton exchange membrane (PEM) fuel cell is best suitable in aerospace application due to quick start-up, and low temperature and pressure operations. The bipolar plate is the key constituent of PEM fuel cell, which accounts for around 80% weight and almost all of the volume of a fuel cell. The present thesis describes the development of a novel method to rapidly make carbon/carbon (C/C) composite, and fabrication of bipolar plate therefrom followed by functional testing of bipolar plate. The entire work was divided into seven chapters.

Chapter 1 gives an introduction about fuel cell, emergence of fuel cell, usage of fuel cell, types of fuel cell, working principle of PEM fuel cell, constituents of PEM fuel cell, functions of main constituent i.e. bipolar plate of PEM fuel cell, materials used for the fabrication of bipolar plate, and comparative analysis of advantages and disadvantages of different materials.

Chapter 2 presents comprehensive literature survey to establish the problem. As part of literature survey, the fundamental aspects of carbon is presented initially, and thereafter a brief review on the carbon reinforcement and carbon matrix precursor is presented. Further, the types of carbon composite, its processing methodologies and properties are briefed. The same is followed by the detailed review of carbon/carbon (C/C) composite. In later part of the chapter, the usage of C/C composite for the fabrication of bipolar plate and its present scenario is discussed. At the end of the chapter, objectives of the present research work are defined.

Chapter 3 describes about the raw materials used in the processing of C/C composite at the beginning. The details of pre-processing of reinforcement by chopping and exfoliation, and synthesis and stabilization of primary matrix precursor (PMP) are presented thereafter. Further, the characterization techniques to check the suitability of pre-processed raw materials are briefed. The same is followed by a brief description about the processing of C/C composite and detailed description of the characterization techniques along with standard test methods. Further, brief description of bipolar plate fabrication and detailed methodology for the system level testing of C/C composite bipolar plate through composite stability test, corrosion current test and fuel cell testing are presented at the end of the chapter.

Chapter 4 deals with detailed experimental work carried out to rapidly develop C/C composite by novel way of two-steps processing. In the first step, C/C composite is made by high pressure hot-pressing (HP) method comprising of mixing, moulding, drying, hot-pressing and carbonization. During HP method, various key parameters like hot-pressing pressure and heating rate, reinforcement loading, temperature and time shifts, and matrix precursor modification are studied in detail through visual and SEM analysis, density, mechanical properties, yield, yield rate and yield impact. In second step, the densification of C/C composite obtained from HP method is carried out using secondary matrix precursor (SMP) through low pressure impregnation-thermosetting-carbonization (ITC) method. The impact of HP method parameters on the densification is studied in detail through visual and SEM analysis, density, porosity, mechanical properties, and permeability analysis.

Chapter 5 describes the processing of C/C composite of size 125 mm × 125 mm × 20 mm based upon the optimum processing parameters obtained in Chapter 4. Later, description about the fabrication of C/C composite bipolar plate of size 95 mm × 95 mm × 3.5 mm using conventional grinding, wire electron discharge machining, and milling is presented. Further, post fabrication inspection of bipolar plate flow field channels through SEM analysis is discussed. The results of system level testing of C/C composite bipolar plate obtained through composite stability test, corrosion current measurement, and fuel cell testing are presented and discussed. At the end of the chapter, the comparative analysis of fuel cell results of C/C composite bipolar plate with graphite bipolar plate is made.

In strategic application of the fuel cell, the weight and volume requirements are more stringent along with electrical conductivity and mechanical strength. Hence, the bipolar plate, which accounts for 80 wt% and almost all of the fuel cell volume, should be very thin with lowest permissible density. Thus, new bipolar plate system of carbon/carbon (C/C) composite was studied in detail and presented in the major portion of this thesis. However, the ever sought demand of developing a bipolar plate system with very high specific strength for strategic sector made us to explore new system. Coincidently, the idea of ultra-thin (net thickness $\leq$ 0.25 mm) bipolar plate was evolved while working on the development of wire electron discharge machineable (EDM) carbon/silicon carbide (C/SiC) composite for air breathing propulsion engine of a launch vehicle. The need for the development of rugged and very thin bipolar plate made us to explore this system with ceramic matrix and carbon reinforcement for bipolar plate application. It is the first time when ceramic and carbon composite is being reported as bipolar plate material. The preliminary results on this system are presented in Chapter 6.

The studies carried out on C/C composite bipolar plate for PEM fuel cell in aerospace application and next generation C/SiC composite bipolar plate system are concluded in Chapter 7 with key results. The future scope of the work in the field is also briefed in this chapter. It will be of much help for the buddy researchers to formulate their objectives in the area of bipolar plate.

Thakur Sudesh Kumar Raunija

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In 2008, at Vikram Sarabhai Space Centre (VSSC) after completion of all joining formalities when I reported to Mr. S. Babu, Head (Retd.), CCL/VSSC, his second question was – When are you planning your higher studies? That was the real ignition of the fire for higher studies. But the real journey in this direction started when I got an opportunity to pursue PhD at IIT Guwahati as an external candidate of VSSC. Now, the wonderful journey is going to end with flying colors. Before, I conclude my journey full of knowledge and experience gained from various prominent personalities, I should give the due credit to the persons and the personalities who backed this journey in various capacities.

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I like teaching profession and consider it the effective tool for the transformation of the society. This was one of the reasons to pursue PhD so that I can become the part of this profession along with services to Indian Space Research Organisation. And the one and only one person, who made my reason right, grew a great interest towards this profession, taught me the real approach of a great teacher, is none other than Dr. Anil Verma. The lessons learnt from him and his experiences are unforgettable. I learnt many things from him, but his straightforwardness and supportive nature are the most influential qualities, I tried to learn from him. The all-time support received from Dr. Anil Verma is immense and incredible. To him, I owe my gratitude and deepest appreciation for nurturing this educational experience. Because of his sincere commitment to my educational and personal well-being both at IIT Guwahati and IIT Delhi, I can truly consider myself a better person and student.

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Thakur Sudesh Kumar Raunija

Abstract

Polymer electrolyte membrane (PEM) fuel cell is preferred over others for space applications because of quick start-up, high efficiency, high power density, and low temperature and pressure operations. Bipolar plate (BP) is one of the key constituents, which accounts for around 80% weight and almost all of the volume of a fuel cell stack. Therefore, the material used for its fabrication should be light in weight, strong enough to withstand natural and induced environment, and show excellence in performance. High specific energy (kW/kg) and durability are the mission requirement of spacecraft power system. Almost all of the conventional materials (graphite, metal, and carbon/polymer composite) used for its fabrication are associated with one or more problems, and the necessity to explore a material for the bipolar plate used in aerospace application arises. Carbon/carbon composite, which was first synthesised for thermo-structural applications, possesses all the necessary properties. However, the time consuming and high temperature processing of C/C composite are the major technical challenges, which are to be addressed. In the present work, a novel methodology for the rapid and effective fabrication of C/C composite bipolar plate is presented. The fabrication of C/C composite was carried out using exfoliated carbon fibers as reinforcement, wherein carbon matrix was derived from mesophase pitch and phenolic resin using high pressure hot-pressing (HP) method and low pressure impregnation thermosetting carbonization (ITC) method. The uniquely designed methodology explored for rapid fabrication could produce C/C composite in a very short time of 137 h as compared to 3-4 months in conventional processes while using a pressure not more than 25 MPa in any processing stage. The C/C composite thus obtained offered bulk density 1.75 g/cm^3, impact strength 4.8 kJ/m^2, tensile strength 45 MPa, flexural strength 98 MPa, compressive strength 205 MPa, electrical conductivity 190 (through-plane) & 595 S/cm (in-plane), and thermal conductivity 24 (through-plane) & 51 W/mK (in-plane). The bipolar plate of 95 mm × 95 mm × 3.5 mm with serpentine channels of 1 mm width and 1 mm depth fabricated out of the C/C composite made through rapid and effective methodology was tested in PEM fuel cell and compared with graphite bipolar plate. The performance of C/C composite bipolar plate cell in the ohmic polarization dominated region was found similar to that of graphite bipolar plate. However, voltage difference of around 0.05 V at maximum power density was obtained at 65^{o}C. Further, the cell with C/C composite bipolar plate at an operating temperature of 65^{o}C showed a voltage as high as 0.3 V for a current density of 1100 mA/cm^2 along with a power density as high as 370 mW/cm^2. The detailed characterization and evaluation of the C/C composite bipolar plate in fuel cell showed promising results for its utilization in the next generation PEM fuel cell used in aerospace applications. While working on the development of wire EDM machineable C/SiC composite for space application, the idea of making ultrathin (net thickness ≤ 0.25 mm) bipolar plate was evolved, which encouraged us to explore this system for bipolar plate application apart from C/C composite. It is the first time when carbon reinforced silicon carbide composite is being reported as bipolar plate material. Therefore, at the end of the thesis, the preliminary results carried on the development of C/SiC composite and fabrication of bipolar plate are presented.

Keywords: Bipolar plate, Carbon/carbon composite, Carbon fiber, Fuel cell testing, HP method, ITC method, Matrix precursor, PEM fuel cell.

पॉलीमर इलेक्ट्रोलाइट झिल्ली ईंधन सेल को त्वरित स्टार्ट-अप, उच्च दक्षता, उच्च शक्ति घनत्व और निम्न तापमान और दबाव संचालन के कारण अंतरिक्ष अनुप्रयोगों के लिए दूसरों की तुलना में पसंद किया जाता है। द्विध्रुवी प्लेट इसका एक मुख्य घटक है, जो ईंधन सेल स्टैक के कुल वजन और आयतन का लगभग 80% हिस्सा होती है। इसलिए, इसके निर्माण के लिए इस्तेमाल किया जाने वाला पदार्थ वजन में हल्का होना चाहिए, प्राकृतिक और प्रेरित पर्यावरण का सामना करने के लिए पर्याप्त मजबूत होना चाहिए, और प्रदर्शन में उत्कृष्ट होना चाहिए। उच्च विशिष्ट ऊर्जा (kW/kg) और स्थायित्व अंतरिक्ष यान की मिशन आवश्यकता होती हैं। इसके निर्माण के लिए उपयोग की जाने वाली लगभग सभी परंपरागत सामग्री (ग्रेफाइट, धातु और कार्बन/पॉलिमर मिश्रित) एक या एक से अधिक समस्याओं से जुड़ी होती हैं। इसलिए अंतरिक्ष अनुप्रयोग में इस्तेमाल योग्य द्विध्रुवी प्लेट के लिए एक नूतन सामग्री का पता लगाने की आवश्यकता है। कार्बन/कार्बन मिश्रित, जो पहले थर्मामीटरों-संरचनात्मक अनुप्रयोगों के लिए संश्लेषित था, सभी आवश्यक गुणों के पास है हालांकि, कार्बन/कार्बन मिश्रित बनाने में लगने वाला समय और उच्च तापमान प्रसंस्करण प्रमुख तकनीकी चुनौतियां हैं, जिन्हें संबोधित किया जाना है। वर्तमान कार्य में, कार्बन/कार्बन मिश्रित द्विध्रुवी प्लेट के तेज और प्रभावी निर्माण के लिए एक उपन्यास पद्धति प्रस्तुत की गई है। कार्बन/कार्बन मिश्रित का निर्माण सुदृढीकरण के रूप में एक्सफ़ोइएटेड कार्बन फाइबर का उपयोग करके किया गया है, जिसमें कार्बन मैट्रिक्स मैसॉफिस पिच और उच्च दबाव गर्म दबाव (एचपी) विधि और कम दबाव संसेचन थर्मोसेटिंग कार्बननाइजेशन (आईटीसी) पद्धति का उपयोग करके फेनोलिक राल से बनाई गई है। तेजी से फैब्रिकेशन के लिए खोज की गई विशिष्ट पद्धति से 137 घंटे के बहुत ही कम समय में और किसी भी प्रसंस्करण चरण में 25 एमपीए से अधिक दबाव का उपयोग किये बिना, कार्बन/कार्बन मिश्रित का उत्पादन किया गया है, जो पारंपरिक प्रक्रियाओं में लगने वाले 3-4 महीने के समय की तुलना में बहुत ही कम है। इस प्रकार प्राप्त कार्बन/कार्बन मिश्रित का थोक घनत्व 1.75 g/cm^3, प्रभाव शक्ति 4.8 kJ/m^2, तन्यता ताकत 45 MPa, फ्लेक्सुरल ताकत 98 MPa, संपीड़न ताकत 205 MPa, विद्युत

चालकता 190 (विमान के माध्यम से) और 595 S/cm (विमान में), और थर्मल चालकता 24 (विमान के माध्यम से) और 51 W/mK (विमान में) पाई गईं। 95 mm × 95 mm × 3.5 mm की द्विध्रुवी प्लेट 1 mm चौड़ाई और 1 mm की गहराई के साँप चैनल के साथ तीव्र और प्रभावी पद्धति के माध्यम से बनाये गए कार्बन/कार्बन मिश्रित से बनाई गई और ईंधन सेल में इसका परीक्षण किया गया। ओमिक ध्रुवीकरण वाले क्षेत्र में, कार्बन/कार्बन मिश्रित की द्विध्रुवी प्लेट का प्रदर्शन ग्रेफाइट की द्विध्रुवी प्लेट के समान पाया गया। अधिकतम बिजली घनत्व पर 65°C के तापमान पर लगभग 0.05 V का वोल्टेज अंतर पाया गया किया गया। इसके अलावा, 65°C के ऑपरेटिंग तापमान पर ईंधन सेल ने कार्बन/कार्बन मिश्रित द्विध्रुवी प्लेट के साथ 1100 mA/cm^2 के करंट घनत्व के साथ साथ 370 mW/cm^2 के पावर घनत्व पर 0.3 V ज्यादा वोल्टेज दिखाई। ईंधन सेल में कार्बन/कार्बन मिश्रित द्विध्रुवी प्लेट का विस्तृत लक्षण वर्णन और मूल्यांकन करने के बाद अंतरिक्ष अनुप्रयोगों में उपयोग की जाने वाली अगली पीढ़ी की ईंधन सेल में इसके उपयोग के लिए अच्छे परिणाम दिखाई दिए। अंतरिक्ष अनुप्रयोगों के लिए तार ईडीएम मशीनक कार्बन/सिलिकॉन कार्बाइड मिश्रित का विकास करते हुए, अल्ट्राथिन (नेट मोटाई ≤ 0.25 मिमी) द्विध्रुवी प्लेट बनाने का विचार विकसित हुआ, जिसने हमें कार्बन/कार्बन के अलावा द्विध्रुवी प्लेट अनुप्रयोग के लिए इस प्रणाली का पता लगाने के लिए प्रोत्साहित किया। यह पहली बार है जब कार्बन प्रबलित सिलिकॉन कार्बाइड मिश्रित द्विध्रुवी प्लेट सामग्री के रूप में सूचित किया जा रहा है। इसलिए, थीसिस के अंत में, कार्बन/सिलिकॉन कार्बाइड मिश्रित के विकास और द्विध्रुवी प्लेट के निर्माण पर प्रारंभिक परिणाम प्रस्तुत किए गए हैं।

कीवर्ड्स: द्विध्रुवी प्लेट, कार्बन/कार्बन मिश्रित, कार्बन फाइबर, ईंधन सेल परीक्षण, एचपी विधि, आईटीसी विधि, मैट्रिक्स अग्रदूत, पीईएम ईंधन सेल।

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α	Thermal diffusivity (cm^2/s)
σ_c	Specific enhancement in compressive strength (MPa/h)
σ_f	Specific enhancement in flexural strength (MPa/h)
θ_{an}	Actual porosity of the compact after n densification cycles (%)
θ_h	Porosity of the compact after HP method (%)
θ_{tn}	Theoretical porosity of the compact after n densification cycles (%)
φ	Permeability for definite thickness and pressure at constant temperature (cm^3/cm^{2}s)
∂_{ch}	Specific compressive strength after HP method (MPa)
∂_{ci}	Specific compressive strength after ITC method (MPa)
∂_{fh}	Specific flexural strength after HP method (MPa)
∂_{fi}	Specific flexural strength after ITC method (MPa)
ρ	Bulk density (g/cm^3)
ρ_h	Bulk density of the compact after HP method (g/cm^3)
ρ_{m1}	Maximum possible density of PCM after HP method (g/cm^3)
ρ_{m2}	Density of SCM (g/cm^3)
ρ_n	Bulk density of the compact after n densification cycles (g/cm^3)
ρ_r	Density of reinforcement (g/cm^3)
ρ_s	Density of SMP (g/cm^3)
ρ_{th}	Theoretical density of the compact after HP method (g/cm^3)
a	Contact area (cm^2)
c_h	Compressive strength after HP method (MPa)

c_i	Compressive strength after ITC method (MPa)
c_p	Specific heat (J/gK)
E_v	Electrical conductivity (S/cm)
f_h	Flexural strength after HP method (MPa)
f_i	Flexural strength after ITC method (MPa)
k	Thermal conductivity (W/mK)
l	Length of the sample (cm)
n	Number of densification cycles
R	Resistance (Ω)
R_a	Arithmetic means of roughness values
t	Test duration (s)
t_h	Total process time till HP method (h)
t_i	Total process time till ITC method (h)
u_{m1}	Volume fraction of PCM in compact after HP method
u_r	Volume fraction of reinforcement in compact after HP method
v_{m1}	Volume of PCM in compact after HP method (cm^3)
v_r	Volume of reinforcement in compact after HP method (cm^3)
V	Volume of the compact (cm^3)
V_p	Volume of gas permeates during test (cm^3)
w	Weight of the compact (g)
w_{as}	Weight of sample after stabilization (g)
w_{bs}	Weight of sample before stabilization (g)
w_c	Weight of the compact after HP method (g)
w_{m1}	Weight of PCM in compact after HP method (g)

w_{pi} Weight of PMP taken initially (g)

w_{pv} Weight loss of PMP during vacuum moulding (g)

w_r Weight of reinforcement in the compact after HP method (g)

w_{ri} Weight of reinforcement taken initially (g)

w_{rp} Weight loss of reinforcement during processing (g)

y_i Yield impact (%)

y_p Yield of PMP (%)

y_r Yield rate (%/h)

y_s Yield of SMP (%)

y_v Densification efficiency (%)

Y_l Overall liquid (SMP) impregnation efficiency (%)

AFC Alkaline fuel cell

BP Bipolar plate

C/C Carbon/carbon

CCL Carbon and ceramics laboratory

C/P Carbon/polymer

C/SiC Carbon/silicon carbide composite

CHN Carbon hydrogen nitrogen

CHNS Carbon hydrogen nitrogen sulphur

CNT Carbon nanotube

CTP Coal tar pitch

CVD Chemical vapor deposition

CVI Chemical vapor infiltration

DC Densification cycle

DMFC	Direct methanol fuel cell
DOE	Department of energy
EDM	Electron discharge machining
FESEM	Field emission scanning electron microscope
FTIR	Fourier transform infrared
GDL	Gas diffusion layer
GE	General electric
HIP	Hot isostatic pressing
HIPIC	Hot isostatic pressure impregnation carbonization
HP	Hot-pressing
HSXD	High speed xenon discharge
HTT	Heat treatment temperature
ISRO	Indian space research organisation
ITC	Impregnation-thermosetting-carbonization
K	Group of 1000 filaments
LPI	Liquid phase impregnation
MCFC	Molten carbonate fuel cell
MEA	Membrane electrode assembly
MWCNT	Multi wall carbon nanotube
NASA	National aeronautics and space administration
OMG	Oxygen mass gain
PAFC	Phosphoric acid fuel cell
PAN	Polyacrylonitrile
PCM	Primary carbon matrix
PEEK	Polyether ether ketone

PEI	Polyethylene imine
PEM	Proton exchange membrane
PEMFC	Proton exchange membrane fuel cell
PI	Polyimide
PMP	Primary matrix precursor
PP	Petroleum pitch
PR	Phenolic resin
SCM	Secondary carbon matrix
SEM	Scanning electron microscope
SMP	Secondary matrix precursor
SOFC	Solid oxide fuel cell
SWCNT	Single wall carbon nanotube
TGA	Thermogravimetric analysis
vol	Volume
VSSC	Vikram sarbhai space centre
wt	Weight
XRD	X-ray diffraction

Content