

**PREPARATION AND CHARACTERIZATION OF CONTINUOUS
FIBER REINFORCED MULTIDIRECTIONAL C-SiC COMPOSITES
USING LIQUID-SILICON INFILTRATION METHOD**

by

SURESH KUMAR

Department of Chemical Engineering

submitted

in fulfillment of the requirements of the degree of Doctor of Philosophy

to the



Indian Institute of Technology, Delhi

April 2008



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**Dedicated to my
parents**

Certificate

This is to certify that the thesis entitled, "*Preparation and characterization of continuous-fiber reinforced multidirectional C-SiC composites using liquid-silicon infiltration method*", presented by **Mr. Suresh Kumar, Entry No. 2005CHZ8295** is worthy of consideration for the award of the degree of Doctor of Philosophy and is record of the original research work carried out by him under our guidance and supervision and that the results contained in it have not been submitted in part or in full to any other University or Institute for award of any degree.



G. Rohini Devi Sc 'G'

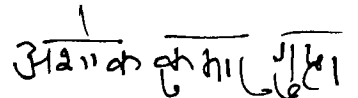
Technology Director
High Temperature Composite Centre
Advanced Systems Laboratory
Defence Research and Development Organization
Hyderabad-500058 (India)



Anupam Shukla

Asistant Professor

Dept. of Chemical Engineering
Indian Institute of Technology
New Delhi-110016 (India)



A K Gupta

Professor

Acknowledgement

I take this opportunity to express my profound gratitude to my research supervisors Smt. G. Rohini Devi Sc 'G' ASL DRDO, Prof. A. K. Gupta and Dr. Anupam Shukla, Chemical Engineering Department, IIT Delhi.

Mrs. G. Rohini Devi imbibed me with deep perception of the high end technologies, inculcated in me commitment and dedication for one's work, and inspired me to maintain the enthusiasm even when things around looked bleak.

Prof. A. K. Gupta is a mentor. He persistently motivated me to excel. I am left with no words to thank him for the pains he has taken to spend all weekends for reviewing progress of my work. In spite of his busy schedule he could spare sufficient time as and when required. His analytical interpretation of the results made me to have a feel of research.

I am indebted to Dr. Anupam Shukla for his guidance like a supervisor, support like a friend advice like a colleague. His quick suggestions on experimentation and result interpretation helped me immensely. I feel lucky to be associated with such a learned young faculty.

I express my sincere thanks to Shri Anil Kumar Division Head (CMCD/HTCC), whose persistent support and encouragement made this work to progress in an organized way.

I wish to express my sincere thanks to Prof. S. K. Gupta, Head of the Department of Chemical Engineering, IIT Delhi and Prof. B. K. Guha, a former Head for their generous help in administrative and technical matters.

I also take this opportunity to extend my sincere thanks to Prof. B. P. Mani, Dr. Ratan Mohan, Dr. V. V. Krishnan, Dr. Mrs. Sreedevi, Dr. S. Aravindan, Dr. K. K. Pant, and Dr. Shantanu Roy, whose technical suggestions during the presentations helped me immensely.

I express my heartfelt gratitude for the support extended by Shri Avinash Chander Director Advanced Systems Laboratory, who allowed me to carryout research in the area of C-SiC composites and Shri J. C. Chaudhary Sc 'F', Incharge of Carbon-Carbon composites Processing-Plant, by giving the time for this work. I also extend my sincere thanks to Shri K. H. Sinnur, Sc 'F' who helped me in characterization of composites, Dr. R. K. Jain, Sc 'F', Shri Rama Guru Sc 'F' and other colleagues at ASL whose valuable suggestions and comments inspired me to enrich the work. I also express my sincere thanks to Shri Murthy Reddy TO 'A', Shri Kamalakar STA 'A' and Shri Purna Chandra Rao STA 'A' who helped me in experimentation and testing part.

I express my gratitude to Shri M. Venkata Reddy, Sc 'C', G. Srinivas, Sc 'C' (DRDL) for their help in carrying out X-ray and Kinetic Heating studies. I also would like to thanks

Dr. J. Subrahmanyam Sc 'G' and Dr. V. Bhanu Prasad Sc 'F', DMRL for their suggestions on mechanical testing. I also express my sincere thanks to Mrs. Sweety Kumari Sc 'C', Dr. P. Ghoshal Sc 'E', Shri Rajneesh Sc 'C' of DMRL who have helped me in mechanical properties and microstructure characterization. I also express my thanks to Dr. R. M. Mohanthy, NFTDC, Dr. B. T. N. Sridhar, MIT Chennai and Dr. Roy Johnson of ARCI for their generous help in testing of samples.

I would like to express my sincere thanks to my friends Sharad, Srikanth, Jitendra, Ashok Babu, Manoj, Sambhav, Jyothsna, Alex, Manivannan and all others who have helped directly or indirectly during this work.

I also wish to thank the supporting staff Mr. Venkat, Mr. Ram Murthy, Mr. Hanumanth Rao, and Mr. Narsimha who spent many weekends and worked along with me from early mornings to late nights during siliconization experiments.

Without the support of my wife and daughter, from whom I took away important family time, this thesis would not have been completed in time.

I offer the fruits of my work at the Lotus feet of Almighty for providing me everything that was required to accomplish this gigantic task in minimum possible time.



Suresh Kumar

Abstract

Multi-directional carbon fiber reinforced silicon carbide composites (C-SiC) were fabricated by liquid-silicon infiltration process (LSI) and tested for mechanical and thermal properties. Typical size jet-vanes were also prepared at best siliconization conditions and tested under a plume of aluminum based solid propelled Rocket Motor exhaust.

Three types of carbon-carbon (C-C) preforms were developed namely (i) bi-directional (2D) by polymeric resin (molding process), (ii) three-directional-stitched (3D-stitched) and (iii) 3D-woven by multiple coal-tar pitch impregnations, carbonizations and followed by high pressure impregnation carbonization (HIPIC) process. The C-C preforms of each type were siliconized using liquid silicon infiltration (LSI) method at 1450-1650°C in vacuum for 10-120 minutes. All the three types of C-SiC composites were characterized for flexural strength at room temperature. After preliminary siliconization experiments, it was found that 3D-stitched C-SiC composites gave better flexural strength than the other two fiber architectures.

Factorial design of experiments for siliconization was carried out to study the effect of four parameters viz. temperature, time, cross-sectional area and height of C-C preforms, on the density gain and β -SiC formation. Sixteen independent experiments were carried out by varying these four parameters at two levels (upper and lower). The levels of the parameters were chosen considering the intended product requirement and limitation of the siliconization furnace. Effect of individual parameters and their interaction were studied. Siliconization time and temperature are the two key parameters which affect density gain and β -SiC formation. Inferences drawn from factorial design of experiments were used to study the effect of time and temperature on the mechanical and thermal properties of the C-SiC composites made through stitching process.

3D-stitched-C-SiC composites made through LSI process consist of four distinct constituents viz. carbon-fibers, carbon-matrix, silicon-carbide (SiC) and un-reacted silicon, which have an influence on mechanical and thermal properties. C-SiC composites siliconized at different time and temperature were tested under bending (also termed as flexural), tensile and impact loading at room temperature. Flexural strength varied from 130-201 MPa while tensile strength varied from 60-101 MPa depending on siliconization conditions. Weibull-modulus approach was adopted to analyze the results of flexural strength. It was found to be 10.18 for composites siliconized at 1650°C for 120 minutes whereas it was only 6-7 for composites siliconized at 1450°C for 10 minutes and the corresponding characteristic strengths were 185.7 MPa and 120 MPa respectively.

Effect of testing temperature on the flexural strength was also studied upto 1200°C in air. Since the composites siliconized at 1650°C for 120 minutes offered highest strength at room temperature, only those were tested at 900°C and 1200°C as well. Flexural strength was found to be in the range of 150-155 MPa at 900°C and 120-130 MPa at 1200°C.

The composites siliconized at 1650°C for 120 minutes offered the highest and those siliconized at 1450°C for 10 minutes, showed the least tensile strength, the corresponding values were 90-101 MPa and 60-74 MPa respectively. The micro-hardness of the C-SiC composite phases was also measured using Knoop and Vickers hardness indenters at room temperature. The composites registered hardness values in the range of 2200-4200 kg/mm² by both the indentation methods.

Effect of siliconization conditions on thermal properties viz. thermal diffusivity and coefficient of thermal expansion (CTE) was also studied. Thermal diffusivity of composite samples in in-plane and through-thickness directions was determined from room temperature to 1500°C using Laser-Flash equipment. In the in-plane direction, it varied from 74 -15

mm²/s whereas in the through-thickness direction, it varied from 40-5 mm²/s. Thermal diffusivity also varied with siliconization conditions. CTE was determined in both the directions from room temperature to 1050°C. It was found to be higher in the through-thickness direction than in the in-plane direction. CTE was found to be the highest for composites siliconized at 1450°C for 120 minutes and the least for composites siliconized at 1650°C for 120 minutes. Also, CTE was found to increase with increase in residual silicon content in the composite.

Measurements using mercury porosity-meter, solvent capillary infiltration and permeability were used to characterize internal pore structure of the 3D-stitched C-C preforms. The effective mean pore radii were determined from each of these studies. A mathematical model was developed for capillary infiltration of organic solvents. Silicon infiltration was carried out at 1650°C in 10 C-C preform bars of size 150x17x50 mm³. Infiltration time was varied from 6 to 180 s. Infiltration heights were measured by X-ray images of siliconized bars.

Some of the C-SiC composite products prepared using the best siliconization conditions were tested simulating thermal environment by kinetic heating. Kinetic heat test was carried out using quartz heating lamps simulating a heat flux of 0.6 W/mm² for 10 minutes. The composites exhibited very good thermo-oxidative stability against sudden heating.

3D-stitched C-SiC jet-vanes of the density range of 2.25-2.3 g/cc were also prepared. These were subjected to aero-thermal testing in a solid propelled rocket motor exhaust with a plume velocity of about 3000 m/s and temperature 2500-3000K. The jet-vanes could withstand the thermal shock and also severe aero-thermal environment of rocket motor exhaust. Rate of erosion was found to be in the range of 1-2 mm/s.

The present study led to a very good understanding of the entire process of fabricating C-SiC composites including preforming involving 3D fiber architecture, rigidization with coal-tar

pitch precursor, siliconization, effect of various parameters of siliconization and their combined effect on the silicon uptake, density gain, silicon carbide formation, mechanical and thermal properties and erosion performance of the jet-vanes. All objectives set for this particular study were achieved successfully.

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