# International Multidisciplinary Research Journal

# Indian Streams Research Journal

Executive Editor Ashok Yakkaldevi Editor-in-Chief H.N.Jagtap

#### Welcome to ISRJ

#### **RNI MAHMUL/2011/38595**

#### **ISSN No.2230-7850**

Indian Streams Research Journal is a multidisciplinary research journal, published monthly in English, Hindi & Marathi Language. All research papers submitted to the journal will be double - blind peer reviewed referred by members of the editorial board. Readers will include investigator in universities, research institutes government and industry with research interest in the general subjects.

# International Advisory Board

Flávio de São Pedro Filho Federal University of Rondonia, Brazil

Kamani Perera Regional Center For Strategic Studies, Sri Lanka

Janaki Sinnasamy Librarian, University of Malaya

Romona Mihaila Spiru Haret University, Romania

Delia Serbescu Spiru Haret University, Bucharest, Romania

Anurag Misra DBS College, Kanpur

Titus PopPhD, Partium Christian University, Oradea, Romania

Mohammad Hailat Dept. of Mathematical Sciences, University of South Carolina Aiken

Abdullah Sabbagh Engineering Studies, Sydney

Ecaterina Patrascu Spiru Haret University, Bucharest

Loredana Bosca Spiru Haret University, Romania

Fabricio Moraes de Almeida Federal University of Rondonia, Brazil

George - Calin SERITAN Faculty of Philosophy and Socio-Political Sciences Al. I. Cuza University, Iasi

Hasan Baktir English Language and Literature Department, Kayseri

Ghayoor Abbas Chotana Dept of Chemistry, Lahore University of Management Sciences[PK]

Anna Maria Constantinovici AL. I. Cuza University, Romania

Ilie Pintea. Spiru Haret University, Romania

Xiaohua Yang PhD. USA

.....More

# Editorial Board

Pratap Vyamktrao Naikwade Iresh Swami ASP College Devrukh, Ratnagiri, MS India Ex - VC. Solapur University, Solapur

R. R. Patil Head Geology Department Solapur University, Solapur

Rama Bhosale Prin. and Jt. Director Higher Education, Panvel

Salve R. N. Department of Sociology, Shivaji University,Kolhapur

Govind P. Shinde Bharati Vidvapeeth School of Distance Education Center, Navi Mumbai

Chakane Sanjay Dnyaneshwar Arts, Science & Commerce College, Indapur, Pune

Awadhesh Kumar Shirotriya Secretary, Play India Play, Meerut(U.P.) N.S. Dhaygude Ex. Prin. Dayanand College, Solapur

Narendra Kadu Jt. Director Higher Education, Pune

K. M. Bhandarkar Praful Patel College of Education, Gondia

Sonal Singh Vikram University, Ujjain

G. P. Patankar

Maj. S. Bakhtiar Choudhary Director, Hyderabad AP India.

S.Parvathi Devi Ph.D.-University of Allahabad

Sonal Singh, Vikram University, Ujjain

Rajendra Shendge Director, B.C.U.D. Solapur University, Solapur

R. R. Yalikar Director Managment Institute, Solapur

Umesh Rajderkar Head Humanities & Social Science YCMOU,Nashik

S. R. Pandya Head Education Dept. Mumbai University, Mumbai

Alka Darshan Shrivastava S. D. M. Degree College, Honavar, Karnataka Shaskiya Snatkottar Mahavidyalaya, Dhar

> Rahul Shriram Sudke Devi Ahilya Vishwavidyalaya, Indore

S.KANNAN Annamalai University, TN

Satish Kumar Kalhotra Maulana Azad National Urdu University

Address:-Ashok Yakkaldevi 258/34, Raviwar Peth, Solapur - 413 005 Maharashtra, India Cell: 9595 359 435, Ph No: 02172372010 Email: ayisrj@yahoo.in Website: www.isrj.org

# International Recognized Double-Blind Peer Reviewed Multidisciplinary Research Journal Indian Streams Research Journal

ISSN 2230-7850 Volume - 5 | Issue - 5 | June - 2015 Impact Factor : 3.1560(UIF) Available online at www.isrj.org

# EFFECT OF SIC/CARBON REINFORCED PARTICULATE ON CARBON COMPOSITES

## L. M. Manocha

Department of Materials Science, Sardar Patel University, Vallabh Vidyanagar.

## Short Profile

L. M. Manocha was a former Head of Department of Materials Science, Sardar Patel University, Vallabh Vidyanagar, Gujarat.

## Co - Author Details :

<sup>2</sup>Parul Sheth and <sup>3</sup>Chetan Sheth
<sup>2</sup>Department of Materials Science, Sardar Patel University ,Vallabh Vidyanagar .
<sup>3</sup>Department of Electrical Engg.,GCET , Vallabh Vidyanagar .



# **ABSTRACT:**

Carbon-ceramic particulate composites contain the phenolic resin as a matrix precursor and SiC, graphite powder, carbon black, fly ash, coke etc. as reinforceing materials. Composite samples were prepared to evaluate the effect on various properties of the samples heat treated at 1000°C. Powder metallurgical method was used for processing of the various composites/samples. Changes in physical properties of the

composites like weight loss, volume shrinkage, density, % porosity etc. and many other properties were measured and monitored. Optical microscopy was used to study the distribution of SiC and graphite powder in the carbon matrix and to observe other parameters. It shows that the reinforcing particles are well distributed in the carbon matrix and also exhibited good bonding between the matrix and reinforcing particles. SEM images confirmed dense nature of carbon-ceramic composites.

# **KEYWORDS**

Composite, Carbon, Phenolic resin.

| Article Index | ked in :       |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |

## 1.INTRODUCTION :

Carbon materials (including composite materials containing carbon) are well-known for their structural applications, especially as electrodes for metallurgy; high temperature dies, for aircraft brakes, structures, adsorbents and lubrication etc. These are also known for thermal applications relating heat conduction as well as thermal insulation, environmental applications in the form of activated carbon and biomedical applications as implants. This is because of its unique characteristics such as light weight, high temperature stability (without loss of strength) up to ~ 2500°C in non-oxidizing atmospheres, low coefficient of thermal expansion and controlled friction, good electrical and thermal conductivities, and high thermal shock resistance [1].

Synthetic carbons (also known as laboratory-created carbon, laboratory-grown carbon, cultured carbon or cultivated carbon) are carbon produced in an artificial process. Carbon-ceramic composites have added improved mechanical & thermal properties with good combination of properties of both materials. For any material used for structural applications, it is essential to have knowledge of its microstructure due to direct dependence of properties and ultimate performance of that material on its microstructure.

A commercial carbon or graphite product usually contains more than 10 different constituents. They are often categorized into four main classes: - binders, fillers, modifiers, and abrasives. Selections of the constituents are often based on experience or a trial and error method to make new formulation. Phenolic resins are among the most commonly used binders in these materials. In the class of fillers, graphite powder, coke, fly ash etc. are used [2]. They replace metals/alloys without deleterious loss of desirable properties. Abrasives like alumina, oxides, iron oxide, silica, SiC, and boron carbide can be incorporated to improve the mechanical properties. In this work, for that SiC is incorporated. For further improvement of the mechanical and thermal properties of carbon composites, addition of various kinds of fibers as reinforcements could be most cost effective. Mostly a combination of fibers such as synthetic fibers (e.g., aramid), metallic (e.g., steel wool), ceramic (e.g., chopped glass fibers), mineral fibers (e.g., rock wool) and natural fibers (e.g., cellulose)) can be used. Carbon fibers possessing high strength and high modulus have significant effect on their characteristics. [3-4] Addition of fibrous reinforcement plays a major role in maintaining strength, stiffness, and thermal stability of the composite material. These materials can be classified as organic (polymeric), carbon-based, asbestosbased, metallic and phenolic based materials. From the literature it is proven that phenolic based composites are becoming a promising candidate for different automobile and brake lining industries. Therefore, studies have been undertaken to develop different types of carbon based composites with different formulations for their characterization w.r.t. mechanical, electrical and thermal properties and constituents, to study the effect of above mentioned constituents on their microstructural properties [5-6].

In present study, carbon-ceramic was prepared using phenolic resin as matrix precursor and silicon micro particles, graphite powder, carbon black, fly ash, and coke as reinforcement. Study of physical properties and microstrutural analysis of composites before and after pyrolysis were carried out. All the constituents were mixed in predefined proportion using ball milling prior to hot pressing of mixture at 150°C temperatures and 200Kg. pressures. The prepared green composites were then carbonized at 1000°C temperature in  $N_2$  atmosphere with slow heating and cooling in order to avoid any crack formation or chipping of composites. Density, porosity and microstructure of green and along

| Article Index | ed in :        |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |

with these other significant properties of carbonized composites were determined. On carbonization, green composites showed increase in density with all compositions. Microstructural analysis showed good packing of constituents. For comparison and detailed analysis, commercially available carbon products were also included in the present studies.

## 2. RAW MATERIALS AND SAMPLE PREPARATION

## (a) Phenolic resin as matrix

The matrix precursor used in this research work was phenolic resin (Novolac type) mixed with 12 % hardener manufactured by Polymers and Polyols Chemicals, Vapi. The novolac phenolic was used as precursor for composite preparation due to its good dimensional stability and high yield. Prime function of phenolic resin was to bind or hold all the ingredients [7]. Phenolic resin is a common binder for resin-based carbon materials. Thermal applications of pure phenolic resins derived carbons are usually limited due to their relatively poor strength. Therefore, it is imperative to incorporate various reinforcing and ?lling constituents such as reinforcing ?bers and ?llers into phenolic resin-based composites with the purpose of increasing their strength and stability [8-9].

Pyrolysis behavior of phenolic resin was performed in N2 atmosphere at 950°C with heating rate of 20°C/hr as shown in Fig.2.1. During carbonization stage, non-carbon elements in the cured resin come out as volatiles, such as  $H_2O$ , CO,  $CO_2$ ,  $H_2$  and other gases. The reactions were due to the condensation of aromatic ribbon molecules in the cured resin and the volatilization of low molecular weight species. This results in shrinkage and weight loss in the cured resin. The carbon yield of the phenolic resin was found to be (~ 56.74%) which shows it is a good precursor for the carbon-ceramic products.

#### (b) Silicon carbide powder as filler

SiC was procured from Carbonrundum, Thiruvanathpuram, having purity of 99.9 %. The use of SiC material is to enhance the mechanical property of the end product. It has been found that the ceramic particles improve both the room and high temperature strength.

#### (c) Graphite powder

The natural graphite powder (particle size 1-10 µm) was obtained from M/s Hindustan Electro graphite, Bhopal, India and was used as reinforcing filler.

Natural and synthetic graphite often used to construct the anode of all major battery technologies [9]. The lithium-ion battery utilizes roughly twice more graphite than lithium carbonate [10]. Natural graphite has found uses in zinc-carbon batteries, in electric motor brushes, and various specialized electrical applications. Graphite is also commonly used in the form of powders, and sticks for the purpose of writing or drawing. Graphite of various hardness or softness results in different qualities and properties.

The carbon-ceramic/carbon composites were developed using powder metallurgical method which involves mixing of reinforcements i.e. silicon carbide and graphite powder with thermosetting

| Article Index | ked in :       |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |

resin (phenolic resin) with varying quantities.

In the group silicon carbide compositions varied as 40% and 60%, which are identified as 4P4S2G and 6P4S and graphite powder compositions varied as 20%, 50% and 80% respectively which are identified as 8P2G, 5P5G and 2P8G Composition for C-particulate composites is as shown in Table 2.1.

| ID     | Phenolic resin | SiC  | Graphite powder |
|--------|----------------|------|-----------------|
| 6P4S   | 60 %           | 40 % |                 |
| 2P8G   | 20%            |      | 80 %            |
| 5P5G   | 50 %           |      | 50 %            |
| 8P2G   | 80 %           |      | 20 %            |
| 4P4S2G | 40 %           | 40 % | 20 %            |

Table 2.1: Composition for carbon- particulate composites.

#### 3. Result and discussion

3.1 Physical properties of carbon particulate composites

# 3.1.1 Weight loss and volume shrinkage of carbon particulate composites during heat treatment at 1000°C

Weight loss and volume shrinkage graphs for all C-particulate composites vs heat-treatment temperatures are presented in Fig. 3.1 (a) and (b). During carbonization, process of pyrolysis of phenolic resin occurs which leads to weight loss and volume shrinkage in the composites. Other constituents like graphite powder and silicon carbide are already calcined at high temperature. In C-particulate composites , phenolic resin act as a source of carbon for further synthesis of silicon carbide/graphite powder. Fig.3.1 (a) and (b) shows the change in weight loss and volume shrinkage in SiC/carbon-particulate composites. As the % of phenolic resin increases, weight loss and volume shrinkage simultaneously increases. The behavior of C-particulate composites with silicon carbide as reinforcement is different as compared to composites with fly ash. In other case, the weight loss and volume shrinkage decreases with increase in phenolic resin content. This may be due to formation of composites of phenolic resin with high oxides in fly ash.

Heat treatment of composites up to 1000°C involves pyrolysis of phenolic resin matrix. Therefore, the weight loss of composites is primarily due to pyrolysis of the phenolic resin. SiC and graphite powders are already calcined materials and have been processed at temperature more than 1000°C. Pyrolysis of the phenolic resin takes place in different temperature zones, predominantly below 700°C. Above 700°C, reorganization of carbon chains in phenolic chars takes place [11]. Initially

| Article Index | ed in :        |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |

at around 200°C - 400°C temperature, there is a release of volatiles such as unreacted phenols, water vapors,  $CO_{2}$ ,  $H_{2}O$  etc. This results in the shrinkage of the cured phenolic resin. There is a steep weight loss between 400-600°C temperature ranges. In this temperature range, hydrocarbon molecular chains undergo thermal cracking and recombination. After which the rearrangement of the molecular chains occur, which results in further shrinkage [12]. If this rearrangement phenomenon is not controlled properly, it may erupt the structure of the composites. Also due to different heat capacity and coefficient of thermal expansion of the SiC and the matrix materials, the thermal mismatch is experienced by the composites, which leads to the chipping of the composites. So, in order to avoid eruption, chipping and bending; slow heating /cooling rates were applied during carbonization.



Fig.3.1: (a) Weight loss and (b) Volume shrinkage of various % of phenolic resin during heat treatment up to 1000°C.

#### 3.1.2 Pyrolysis of phenolic resin to carbon char

Pyrolysis is one of the critical processes in the manufacture of carbon or related composites with phenol-formaldehyde resin. The pyrolysis reaction converts the resin matrix into amorphous carbon. The potential damage during this process is serious because of the thermal stresses that develop in the composites and high pressures from the evolution of many gaseous products. The whole mechanism of pyrolysis can be series of both simultaneous and sequential reactions of condensation, oxidation, dehydration, decomposition, formation of additional crosslink, thescission of crosslink, and polycyclic aromatization. Fig.3.2 (a) and (b) shows a general sequence of probable chemical reactions occurring during pyrolysis of phenol-formaldehyde polymer [13]. As shown in Fig.3.2 (a) and (b), during pyrolysis various dissociation/condensation reactions takes place concurrently. Therefore, weight loss of the polymers during pyrolysis is accompanied by volumetric shrinkage. Matrix shrinkage has a dominating effect on the microstructure and performance of carbon based composites. Excessive shrinkage during carbonization generates stresses at the reinforcement/matrix interface and often results in matrix cracks. A systematic analysis of the pyrolysis mechanism and TGA analysis of resin matrices help in choosing optimum carbonization conditions, especially the rate of heating in particular

| Article Index | ed in :        |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |

temperature region. The solid-state pyrolysis of highly cross linked polymers results in turbo static carbon with aromatic systems angularly displaced. These resins yield low density non-graphitizable carbons with closed porosity, which limits the density of carbon based composites [14].

The slight weight loss in first stage i.e. up to 200°C is due to the loss of moisture, and the second stage, up to 400°C, may be attributed to the loss of some volatiles, such as unreacted phenol and formaldehyde etc.

According to the pyrolysis chemistry of PF resin, phenolic hydroxyl is active in pyrolysis but there is no direct relationship on the decomposition of PF resin. In contrast, its reaction with methylene forms an additional crosslink in polymer chain backbone at initial pyrolysis stage, which may strengthen the thermal stability [15-16]. Besides reacting with phenolic hydroxyl; methylene can also be oxidized or scissored out of polymer chain backbone. According to the Fig. 3.2 (a) and (b), phenol and its methyl derivatives are the main volatiles when PF resin is pyrolyzed at 450°C temperature. This means some methylenes are scissored as the pyrolysis reactions proceed [17].

Further pyrolysis chemistry of phenol formaldehyde suggests that phenol and its methyl derivatives are always the majority volatiles whenever PF resin is pyrolyzed at 450, 550, 650 or 750°C. Therefore, it can be inferred that methylene scission results in the damage of polymer chain backbone. It should be also noted that some volatiles such as benzene and its methyl derivatives are released at temperature above 650°C. These volatiles propose that some phenolic hydroxyls are apparently stripped off, and hydroxyl radicals are generated correspondingly. There are almost no carboxyl compounds in the pyrolysis products which can be explained by the difference in thermal stability. The carboxyl located at the terminal chain, is deboned upon the formation of carbon dioxide at high temperatures. The carbonyl located between aromatic rings, has a higher stability for the conjugation effect. It is not removed until the temperature is sufficiently high and carbon monoxide is released. Therefore, carbon dioxide evolves before carbon monoxide.

In the whole pyrolysis process, an additional crosslink is firstly formed between ethylene and phenolic hydroxyl. Second, some methylene are scissored, thereby generating some volatiles including phenol and its methyl derivatives [18]. Third, some phenolic hydroxyls are stripped off with the formation of hydroxyl radical. Hydroxyl radicals react with methylene and hydroxymethyl, leading to the release of carbon dioxide and carbon monoxide. Finally, the resin is gradually transformed into amorphous carbon by polycyclic reactions including those of polyaromatics.



Fig.3.2 (a) & (b): Pyrolysis chemistry of phenol-formaldehyde polymer [10].

| Article Indexed in | ו:             |             |
|--------------------|----------------|-------------|
| DOAJ               | Google Scholar | DRJI        |
| BASE               | EBSCO          | Open J-Gate |

#### 3.1.3 Density and porosity of C-particulate (I) composites

Density and kerosene porosity of green and carbonized C-particulate composites were measured. For group-I, these graphs are plotted in Fig.3.3. It is clear from Fig.3.3 that the density of the C-particulate composites increases with the heat treatment temperature. The densities of green composites were measured as 1.35 g/cm<sup>3</sup>, 1.48 g/cm<sup>3</sup>, 1.59 g/cm<sup>3</sup>, 1.73g/cm<sup>3</sup> and 1.75 g/cm<sup>3</sup> for 8P2G, 5P5G, 2P8G, 6P4S and 4P4S2G respectively. In case of carbonized C-particulate composites it is clear that density is increased with filler amount. This is because of the shrinkage in matrix (phenolic based carbon) during carbonization (heating) and reinforcements (SiC, graphite powder) get compacted in the carbon matrix to increase in the density at 1000°C [19]. There is difference in increase in density in case of 2P8G composites which is due to presence of more amount of total graphite powder content in C-particulate composites [Fig.3.3 (a) to (f)]. The sample 8P2G has 1.51g/cm<sup>3</sup> carbonized density whereas 2P8G has 1.63g/cm3carbonized density [Fig. 3.3(b)]. As from Table 3.1 it is known that 8P2G has 20% of total graphite content as compared to 2P8G which has 80% of total graphite content.

The Fig. 3.3(a) and 3.3(b) shows density and % porosity change in green and carbonized heat treated SiC/graphite particulate composites. The densities of carbonized SiC / graphite particulate composites were slightly increased with rise in pyrolysis temperature. The results show that the addition of silicon carbide, evidently improves the sinter ability of sample. Porosity of the C-particulate composites was found to be decreased with increase in percentage of SiC/graphite powder. Porosity also decreased with increase in heat treatment temperature. During carbonization the diameters of the pores were reduced, leading to decrease in porosity at heat treatment, which implies that during carbonization more graphite particles get compacted in the carbon matrix leading to high packing density and less porosity. Therefore, 2P8G has higher density as compared to 8P2G. Also it can be justified from its optical micrographs.

For SiC reinforced composite samples, also during carbonization more SiC particles get compacted in the carbon matrix leading to high packing density and less porosity. The SiC- carbon composite has higher density than graphite –carbon composite samples but addition of graphite particles in to the SiC reinforced carbon based sample decreases the packing density of these composites. Porosity of these samples has inverse nature as shown in Fig. 3.3 (b).



Fig.3.3 (a)& (b) : Variation in densities and Change in % porosity of C-particulate composites heat treated up to 1000°C.

| Article Indexed in | ו:             |             |
|--------------------|----------------|-------------|
| DOAJ               | Google Scholar | DRJI        |
| BASE               | EBSCO          | Open J-Gate |

#### 3.2 Macro / Microstructures studies

Microstructural analysis was done to see the microstructure as well as morphology for Cparticulate composites heat treated up to 1000°C. Both matrix as well as reinforcing agents (SiC, graphite powder) was observed under optical microscopy and SEM.

# 3.2.1 Optical microscopy of C-particulate (I) composites

The Optical micrographs illustrates the microstructures of C-particulate composites with 20 %, 50%, 80% and 40% weight fraction of graphite powder and SiC respectively at 50X magnification [Fig.3.4 (a) to (f)]. The purpose of microstructure examination was to investigate the particle size, shape morphology and distribution of the silicon carbide, graphite particles with their interfacial integrity between the matrix and reinforcement [20]. The microstructure consists of three main constituents. SiC particles are having round shape [Fig.3.5 (a) and (b)]. The large and small dark region represents the voids present in C-particulate composites. Remaining regions show the presence of carbon matrix. As the percentage of graphite particles increases, dispersion of reinforcement with carbon matrix becomes more homogeneous. The particle size of SiC and graphite powder as seen in Fig.3.4 and Fig. 3.5 is 5-10 microns and 2-10 microns respectively. As discussed above, after carbonization process the particles get compacted and thus their density increases, which is clearly seen in these optical micrographs.



8P2G before carbonization



**8P2G** after carbonization

# Fig. 3.4 (a) to (f): Optical micrographs of C-particulate composites.



4P4S2G before carbonization



4P4S2G after carbonization

# Fig 3.5(a) to (d): Optical micrographs of C-particulate composites (I).

The Fig.3.5 (a) to (d) shows optical micrographs of C-particulate composites heat treated up to 1000oC. It shows proper distribution of silicon carbide and graphite particles in the carbon matrix. The particle size of silicon carbide is smaller than graphite particles. Optical micrographs show that silicon carbide particles are irregular in shape whereas graphite particles are somewhat crystalline in shape. These figures also show that there is good compaction between matrix and reinforcing particles which leads to good density at higher temperature. Ball-milling can have pronounced effect on particle size of silicon carbide. If we increase milling time, there is probability of reduction in particle size of silicon carbide which may enhance the density of C-particulate composites.

# 3.2.2 Scanning electron microscopy of C-particulate (I) composites

DRJ

**Open J-Gate** 

The microstructure, as revealed by scanning electron microscopy (SEM), is shown in Fig.3.6 (a) to (d) for the C-particulate composites heat treated at 1000°C. SEM images were obtained to understand the matrix-reinforcement interactions. The dark regions are the carbon matrix whereas the black regions are pores. Observations of the composites containing both SiC and graphite particles have been performed. These micrographs confirm that there was a uniform distribution of graphite particles in the base matrix and it clearly shows that there are less voids and discontinuities. Also SiC composite has a proper distribution of all constituents. The surface micro-porosity does not alter at different places of the C-particulate composites. SiC and graphite particles remain entrapped in the structure of the carbon matrix. A high level of the bonding between carbon matrix and reinforcements (SiC and graphite particles) may be achieved during mechano –chemical mixing and carbonization process [21].



Fig.3.6 (a) to (d): SEM micrographs of C-particulate composites (I).

# 3.3 Mechanical properties

Evaluation of mechanical properties for C-particulate composites was determined by means of Rockwell hardness and compressive measurement.

# 3.3.1 Rockwell hardness of C-particulate (I) composites heat treated to 1000°C

The hardness of the C-particulate composites was evaluated using Rockwell hardness testing machine with 100 kg load and 0.5 mm diameter steel ball indenter. The indentation time for hardness measurement was 5 seconds. Addition of SiC particles enhances hardness, as these particles are harder than carbon matrix which renders their inherent property of hardness to soft matrix. As the amount of the silicon carbide is increased the hardness of the composite is found to increase. The average hardness values of 6P4S and 4P4S2G samples were measured to be 129.2 and 122.6 HRL respectively [Fig.3.7]. The enhancement of hardness by adding the SiC particles mainly results from the presence of extremely harder SiC particles in the carbon matrix and higher constraint to the localized matrix deformation during indentation [7-9]. C-particulate composites with higher hardness could be achieved which may be due to the fact that silicon carbide particles act as obstacles to the motion of dislocation. [22]. Therefore, from Rockwell hardness study it is evidently indicated that 40 wt% of Silicon carbide have higher Rockwell hardness value.

Natural graphite particles are lubricative in nature due to weak bonding of Van der Waals forces

| Article Index | ked in :       |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |

between layers of these particles so tends to slip from one another means layers are little bit slippery in nature. This characteristic makes them ductile in nature when load is applied to bulk of these composites. Also natural graphite is soft compared to SiC particles. Therefore as the weight fraction of natural graphite in polymer matrix increases, bulk hardness of these samples tends to decreases. The average hardness values of 8P2G, 5P5G, 2P8G and 6P4S samples were measured to be 122.3, 118.3, 111.8 and 128 HRL respectively (Fig.3.7).



Fig. 3.7: Bulk hardness of C-particulate composites (I).

For SiC composite sample, there is an increase in hardness as the pyrolysis temperature increases beyond 1000°C. The rise in Rockwell hardness value may be attributed to the densification and enhancement in the formation of SiC at higher temperatures [23]. The maximum Rockwell hardness value (128 HRL) was achieved at 1000°C for carbon-SiC composites containing 40% of SiC.

#### 3.3.2 Compressive strength measurement

Compression tests were carried out on rectangular specimens of C-particulate composites of (25 x 15 x 3-5) mm3 dimensions. For first group, the values vary from 144-290MPa. When SiC particles were added the strength was higher as compared to graphite particles reinforced phenolic resin based particulate composites. This increase is more for higher amount of Silicon carbide (Fig.3.8). This shows that the addition of silicon carbide leads to improvement in the compressive strength of the C-particulate composites. So, improvement in the compressive strength by introducing the SiC particles could be explained by the homogeneous distribution of the SiC particles in carbon matrix. This homogenous distribution gets better and better with increase in SiC particles as seen from Fig.3.8. Increase in homogeneity decreases the pores present in C-particulate composites which finally increases the compressive strength. The variations in compressive strength with the different composites were consistent with the trend of that with Rockwell hardness.

| Article Index | ked in :       |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |



Fig. 3.9: Effect of the SiC particles on compressive strength of the C-particulate composites

It is evident from Fig.3.8 that the compressive strength of the SiC composites increases as the content of silicon increases which enhances the formation of silicon carbide. Dense SiC reinforced composites demonstrated high compressive strength. Compressive strength is higher for SiC composites heat treated at 1000°C having higher amount of silicon carbide (40% silicon).

# 3.4 Electrical properties

The electrical resistivity/conductivity is property which concerns the transport of quasiparticles such as electrons, holes and phonons. The carbon/carbon composites are electrical conductor in the in-plane direction due to the in-plane delocalization of the Pz electrons. However, the out-ofplane conductivity is low, due to the Van der Waals bonding in this direction. Therefore, the electrical conductivity of carbon composites depends greatly on the degree of preferred orientation of the carbon layers. This degree is enhanced by heat treatment, high graphitizability of the precursor materials and alignment of the carbon units (such as the carbon fibers in a composite material) during processing. As a result, carbon composites exhibit a wide range of electrical conductivity, thereby allowing a variety of electrical conduction applications [24].

# 3.4.1 DC electrical conductivity

For measurement of electrical conductivity, three complete runs were taken to check repeatability of measurement for each specimen. Three identical samples were used having physical dimensions of (130x40x3) mm<sup>3</sup> with perfect flat surfaces. Samples were sandwiched between copper

| Article Index | ked in :       |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |

electrodes coated with silver paint on surfaces & mass to make proper electrical contact. Measurements were taken at room temperature (27°C to28°C) and carried out through 2400 Keithley source meter.

The Fig.3.9 shows variation in conductivity as a function of % weight fraction of graphite powder, indicating a typical non-ohmic behavior. Initially, the conductivity of phenolic/graphite composite samples seemed to reach a steady value [26]. With the increase of filler concentration, graphite particles were beneficial in forming a conductive network due to drastically reduced interparticle distance and hence, the conductivity for phenolic/graphite samples increases as shown in Fig.3.9. In the specimen of phenolic resin based SiC filler composite, SiC is hard and insulating material compare to graphite reinforced sample. So the conductivity of this sample is very low compared to graphite filled composite but with the addition of graphite particles in this sample (combination of Sic and graphite powder), the conductivity slightly increases because of conducting graphite filler as shown in Fig.3.9. The optical micrographs Fig.3.4, Fig.3.5 and SEM images of Fig.3.6 confirm these results.





The Fig.3.10 shows that, as the bulk DC conductivity of samples increases, there is a reduction in percentage porosity of specimens. This shows the formation of conductive paths of porous samples. This also conforms by observing the optical images. Reciprocal nature of electrical conductivity and porosity is shown in Fig. 3.10.

#### 4. CONCLUSION

Characterization of composites samples shows that process of carbonization improves the density and reduces the porosity that justified by its macro/micro studies. Addition of graphite powder decreases the composite hardness because of weak bonding of Van der Walls forces between the layers. Homogeneous distribution of SiC particles improves the hardness and compressive strength and

| Article Index | ked in :       |             |
|---------------|----------------|-------------|
| DOAJ          | Google Scholar | DRJI        |
| BASE          | EBSCO          | Open J-Gate |

simultaneously reduces the composite porosity. An electrical conductivity measurement shows that increasing the graphite powder enhances the conductivity but at the same time addition of SiC particles drastically reduces the electrical conductivity.

There is an increase of 4 to 5 % in the density of the SiC based composites, 3% in 8P2G, 6 to 7% in 5P5G and 11% in 2P8G in the density of graphite based composites respectively. But there is a decrease in the density of 0.5% in 3R4CB3F, 0.6 to 0.7 % in 3R7CB and 0.4% in 3R2CB3F2C heat treated at 1000°C. In case of graphite and phenolic resin based composite more weight percentage of graphite leads to higher density. Addition of 40 wt% of silicon carbide increases density up to 16 %. In 3R4CB3F composite when the % weight fraction of the fly ash increased to 30 %, compressive strength and hardness get decreased. Particulate composites made with phenolic resin as a matrix precursor (3R7CB, 3R4CB3F and 3R2CB3F2C) composites results in increase in thermal stability of fly ash based carbon-ceramic particulate composites.

# 5. REFERENCES

1. Fitzer E., Manocha L.M., "Carbon and carbon reinforcements", Chapter 3, IR@INFLIBNET, pp. 112-113, 1999.

2. Robert M. Jones, "Mechanics Of Composite Materials", Materials Science & Engineering Series, Hardcover ISBN-13: 978-1560327127, July 1, 1998.

3. Prevorsek DC, Li HL, "Carbon fiber-reinforced carbon composite material", US patent 5556704, 1996. 4. Lewis IC, "Chemistry of carbonization", Carbon; Vol. 20, Issue 6, pp.519–529, 1982.

5. Subcommittee Do2. Fo of Active Standard ASTM (838), "Standard Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes", ASTM, Vol.5, Issue 5, pp. 838-896, 2010.

6. T. William Lambe & Robert V. Whitman, "Chapter 3: Description of an Assemblage of Particles". Soil Mechanics (First ed.). John Wiley & Sons, Inc. pp. 553, ISBN 0-471-51192-7, 1969.

7.S. Sulaiman, R. Yunus, N. A. Ibrahim, F. Rezaei "Effect Of Hardener On Mechanical Properties Of Carbon Fibre Reinforced Phenolic Resin Composites", Journal Of Engineering Science And Technology, Vol. 3, Issue 1, pp.79–86, 2008.

8. Laza, J. M., Vilas, J. L., Garay, M. T., Rodri´Guez, M. and Leo´, N. L. M., "Dynamic mechanical properties of epoxy-phenolic mixtures", Journal of Polymer Science: Part B: Polymer Physics, Vol.43, pp. 1548–1555, 2005.

9. M. Sarikanat, K. Sever, E. Erbay, F. Güner, I. Tavman, A. Turgut, Y. Seki, I. Özdemir, "Preparation and mechanical properties of graphite filled HDPE nanocomposites", Archies of Materials Science and Engineering, Volume 50, Issue 2, pp. 120-124, 2011.

10. J. Stabik, A. Dybowska, "Electrical and tribological properties of gradient epoxy-graphite composites", Journal of Achievements in Materials and Manufacturing Engineering, Vol. 27, Issue1, pp. 39-42, 2007.

11. Encyclopedia of Polymer Science and Technology, John Wiley & Sons, Vol. 7, pp. 322-368.

12. Fitzer Schaffer, "The effect of cross linking on the formation of glass like carbons from thermosetting resins", Carbon; Vol. 8, Issue 3, pp. 353-364, 1970.

13. Encyclopedia of Polymer Science and Technology, John Wiley & Sons, Vol. 7, pp. 322-368.

14. Fitzer Schaffer, "The effect of cross linking on the formation of glass like carbons from thermosetting resins", Carbon; Vol. 8, Issue 3, pp. 353-364, 1970.

15. Trick K.A., Saliba T.E., "Mechanisms of the pyrolysis of phenolic resin in a carbon/ phenolic Composite", Carbon, Vol. 33, Issue 11, pp. 1509-1515, 1995.

16. Costa L., Montelera L.R., Camino G., Weilb E.D., Pearce E.M., "Structure-charring relationship in phenol-formaldehyde type resins", Polymer Degradation Stability, Vol. 56, Issue 1, pp.23-35, 1997.

17. Jackson W.M., Conley R.T., "High temperature oxidative degradation of phenol formaldehyde Polycondensates", Journal of Applied Polymer Science, Vol. 8, Issue 5, pp. 263-2193, 1964.

18.Lu L., Lim C.Y.H., Yeong W.M., "Effect of reinforcement on strength of Mg 9% Al Composite", Composites structure, Vol. 66, pp. 41-45, 2004.

19. Kimberly A. Trick, Tony E. Saliba, "Mechanisms of the pyrolysis of phenolic resin in a carbon/phenolic composite", Carbon, Volume 33, Issue 11, Pages 1509–1515, 1995.

20.Kulthe M. G. and Goyal R. K., "Micro hardness and electrical properties of PVC/Cu composites prepared by ball mill", Adv. Material Letter, Vol. 3, Issue 3, pp. 246-249, 2012.

21. Lin Y.J., Chen L.J., "Oxidation of SiC powders in SiC/alumina/zirconia compacts", Ceramic International, Vol. 26, pp. 593–598, 2000.

22.McKee D.W., "Borate treatment of carbon fibers and carbon/carbon composites with improved oxidation resistance", Carbon, Vol. 24, pp. 737-741, 1986.

23.Ehrburger P., Baranne P., Lahaye P., "Inhibition of oxidation of carbon/carbon composites by boronoxide", Carbon, Vol. 24, Issue 4, pp. 495-499, 1986.

24.Kobayashi K., Maeda K., Sano H., Uchiyama H., "Formation and oxidation resistance of the coating form on carbon materials composed of B4C-SiC powders", Carbon, Vol. 33, Isuue 4, pp. 397-403, 1995.

25.Raja R. Satheesh, K. Manisekar, Manikandan V., "Effect of fly ash filler size on mechanical properties of matrix composites", ISSN 2320–4060, (IJMMME), Vol. 1, Issue 1, 2013.

26.Luthra K.L., "Oxidation of carbon/carbon composites- A theoretical analysis", Carbon, Vol. 26, Issue 3, pp. 217-224, 1988.

# Publish Research Article International Level Multidisciplinary Research Journal For All Subjects

Dear Sir/Mam,

We invite unpublished Research Paper,Summary of Research Project,Theses,Books and Book Review for publication,you will be pleased to know that our journals are

# Associated and Indexed, India

- International Scientific Journal Consortium
- ★ OPEN J-GATE

# Associated and Indexed, USA

- Google Scholar
- EBSCO
- DOAJ
- Index Copernicus
- Publication Index
- Academic Journal Database
- Contemporary Research Index
- Academic Paper Databse
- Digital Journals Database
- Current Index to Scholarly Journals
- Elite Scientific Journal Archive
- Directory Of Academic Resources
- Scholar Journal Index
- Recent Science Index
- Scientific Resources Database
- Directory Of Research Journal Indexing

Indian Streams Research Journal 258/34 Raviwar Peth Solapur-413005,Maharashtra Contact-9595359435 E-Mail-ayisrj@yahoo.in/ayisrj2011@gmail.com Website : www.isrj.org