Development of Carbon Fibers Reinforced Silicon Carbide Matrix Ceramic Composite

[1] Pramod Kumar H S, [1] Pujari Uday Kiran, [1] Mohankumar S L, [1]P S Shashidhar, [2] Dasharath S M

[1]Students and [2]Assistant Professor

School of Mechanical Engineering, REVA University, Bengaluru

***Abstract*—Silicon Carbide (SiC) powders as matrix, which is present in micro scale, is pulverised to nano scale using ball milling process. This process is carried out for 8 hours, with two 10mmϕ Tungsten Carbide balls. Microscopic study is done and material is examined using SEM. Carbon Fibers (Cf) are randomly mixed with the milled powders for better mechanical properties. Sintering process is carried out using machined graphite die and punch at 1900ºC and 2200ºC for two samples. The sintered materials are characterized by sintering temperatures. Comparing the samples we can understand the variation of mechanical properties of ceramics with sintering temperature.**

***Keywords*—Silicon Carbide, Carbon Fibers, Spark Plasma sintering, Hardness.**

1 INTRODUCTION

This project is based on fabrication ofCeramic composite material using Silicon Carbide as matrix and Carbon Fibers as reinforcement materials. Cer-amic materials all in all have an alluring bundle of properties: high quality and high solidness at exceptionally high temperatures, substance inactive-ness, low thickness, etc. This alluring bundle is defaced by one lethal imperfection, specifically, an articulate absence of toughness. They are inclined to cataclysmic failures within the sight of defects (surface or inward). They are very vulnerable to heat and are handily harmed during manufacture. It is therefore understandable that an overriding consideration in ceramic matrix composites (CMCs) is to strengthen the ceramics by adding fibers in them and thus enhance the useful high temperature strength and environmental resistance of ceramic materials without risking a catastrophic failure. It merits bringing up at the very start that there are sure fundamental contrasts among CMCs and different composites. The general way of thinking in CMC's is to have the fiber bear a more prominent extent of the applied burden. This heap apportioning relies upon the proportion of fiber and lattice flexible moduli, Ef/Em. In MMC's and PMC's, this proportion can be exceptionally high, while in CMCs, it is somewhat low and can be as low as solidarity; consider alumina fiber fortified alumina framework composite. Another unmistakable point with respect to CMCs is that in view of constrained lattice ductility and for the most part high creation temperature, temperature difference, between segments has a significant bearing on CMC execution. The issue of synthetic similarity between parts in CMCs has consequences like those in, like, MMCs. We initially depict a portrayal of some remarkable attributes of CMCs in regards to interface and mechanical properties and then the procedures and particular metod of SPS to fabricate the CMC is discussed. then the testing procedure is briefly explained.

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1.1 Properties of CMCs

A significant element of CMCs is framework miniaturized scale splitting, which doesn't have an equal in MMCs or PMCs. As referenced before, the relative versatile moduli estimations of fiber, Ef and lattice, Em are significant in CMCs. This proportion Ef/Em decides the chance of lattice miniaturized scale breaking. Normally, the strain-to-break estimation of an artistic network is extremely low. Consequently, in MMCs and in thermoplastic PMCs, the framework failure strain (ϵm) is significantly more noteworthy than that of strands.

Most unreinforced metals show up, a strain-to-split ϵm> 10% while most polymers break some place in the scope of 3 and 5% strain. As such, in both MMCs and PMCs fiber disillusionment strain controls the composite disappointment strain. Typically, fibers, for instance, boron, carbon, and silicon carbide show εm estimations of ~1–2%. Difference this and the failure strains of under 0.05% for most CMC materials.

The spark plasma sintering method (SPS) is a modern method of rapid pressurized sintering of a broad group of materials,

including materials classified as hard-sinter able. In this strategy, occasionally rehashed high-current driving forces of direct current enduring up to a few hundred milliseconds are utilized to warm packed powder. The warmth not just surpasses the softening temperature of the base material, yet in addition enables the particles to bond together. Whenever the spark strikes between reinforcement material and matrix phase, the current breaks the bond between SiC particles and because of liquefying temperature, bonds with the external surface of Carbon fiber. This bond stays strong after cooling. The reinforcement material bears maximum of the load.

2 Literature Review

1. This study discusses the potentials of spark plasma sintering (SPS) integrated with high temperature process that can enable sintering of SiC/Cf composites without any sintering aids

2. The composite with the 25% silicon carbide content exhibited the greatest hardness (81.2 HBW) and compression strength (315 MPa). In turn, the greatest tensile strength was achieved by the Al-SiC 20% composite material, in addition to better density. The pressure applied during the spark plasma sintering method should be increased in order to increase the apparent density of the produced composites.

3. This work indicates that low temperature hot pressing of nanosized powders provides great potential in preparation of carbon fiber reinforced ultra-high temperature ceramics with excellent properties.

4. According to this paper, the C/SiC-SiBCN composite fabricated by CVI + PI-OP method had the best oxidation resistance in air between 1200 ℃ and 1400℃.

5. In this work, SiC-based ceramics mixed with 1% by weight and 3% by weight Multi-Layer-Graphene (MLG), respectively, were fabricated by solid-state spark plasma sintering (SPS) at different temperatures. Author reports the processing of MLG/SiC composites, study their microstructure and mechanical properties and demonstrate the influence of MLG loading on the microstructure of sintered bodies.

6. It is shown that the initial punch-die clearance is a key SPS parameter to be specified when different SPS experiments or apparatus are carried out.

7. The methodology has the advantage to prepare the powders and the composites in a relatively shorter time and in an environmentally friendly way. This method bears the potential for extension to the synthesis of metal carbides and borides and their composites.

8. Materials have been found to attain unusual properties in their nano level sizes which are not found in their conventional form. Al-Si alloys containing other transition metals are used in the as cast condition for automotive and aerospace for the engine components.

Spark Plasma Sintering equipment is required to rapidly sinter conductive, non-conductive and/or composite powder materials to the required level of density, including full density. Rapid sintering/densification should take place by the generation of plasma due to sparking between the particles. Details of the SPS components follow.

Ceramic matrix composites (CMCs) can be processed either by conventional powder processing techniques used for making polycrystalline ceramics or by some new techniques developed specifically for making CMCs.

3 EXPERIMENTAL PROCEDURE

Step 1: Ball milling-SiC nano powders (avg powder size of 500 nm) and carbon fibers (5μm diameter) are used as starting materials. Carbon fibers, 1% by weight of SiC which are cut to 30μm in length, are used as reinforcement materials. Silicon Carbide is inserted into Spex mixer mill for 8 hours to reduce the size to nano scale. Carbon fibers are randomly mixed with Silicon Carbide repeteadly using the SPEX miller for 10 minutes.

Step 2: Spark Plasma Sintering- After mixing stage, the mixture is dried on a hot plate. In the wake of drying, the blended powders are embedded straightforwardly into a cylindrical graphite form with 20.5 mm diameter measurement. The sintering process is performed using SPS equipment with an initial pressure of 10MPa under vacuum condition (15 Pa) and followed by expanding pressure during the procedure up to conclusive weight of 50 MPa and completed at sintering temperatures of 1900ºC and 2200ºC for around 8 and 6 min as holding time. The surface layer is removed from the sintered samples by grinding. Microstructure characterization of sintered samples is carried out using Scanning Electron Microscopy (SEM).

Step 3: Vickers Hardness Testing- The Vickers hardness estimations is performed on the cleaned surface of the examples with 5 progressive space tests under the heap of 30 kgf for 10s at room temperature. The obtained diagonals of the indentations and break sizes were estimated utilizing the optical magnifying lens and SEM perceptions. The indenter utilized for Vicker's hardness test is Diamond indenter.

Procedure:

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-The polished scratch free surface of the specimen is kept on the Vicker's hardness apparatus.

-Check the area where indentation is to be made.

-The load to be applied and the dwell time are specified.

-Loading is commenced.

-With the help of the measurement scales, the hardness value is notedand tabulated.

The fracture toughness (KIC) is calculated as below:

$$KIC = 0.18(P/c^{1.5}) (E/Hv)^{0.5}$$

Where P is the load (N) and c is half of the average crack length, and E is Young's modulus, which is calculated using the formula

$$E=(Vf \times Ef)+(Vm \times Em)$$

Where, Ef is elastic modulus of fiber, Em is elastic modulus of matrix, Vf and Vm are volume fractions of fiber and matrix respectively, represented in%.

Hv is Vickers hardness, which is calculated using

$$HV = 2 \times \sin(136/2) \times (P/ d^2)$$

Where, P = force (load) in Kgf, d = Arithmetic mean of the two diagonals, d1 and d2 in mm.

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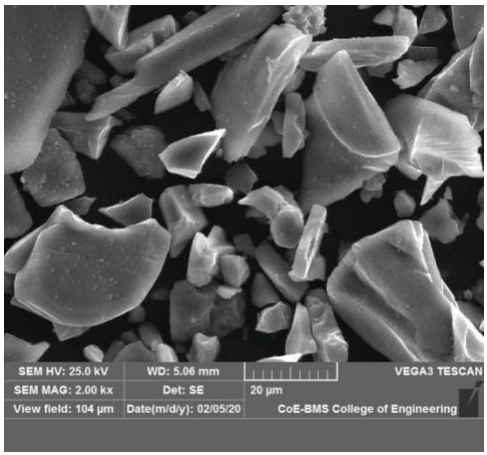
4 RESULTS AND DISCUSSIONS

4.1 Ball Milling

SiC Nano powders (37µm) and carbon fibers (5µm diameter) are used as matrix and reinforcement materials respectively. SiC powders are further run in a ball milling machine for 8 hours with relax time of 15 minutes for every 1.5 hour, in order to avoid amalgamation of SiC powders. The ball milling is initially done for sample of 10grams to identify potential problems. Following which, rest 100 grams are milled for 8 hours. Milling is carried out using two Tungsten Carbide (WC) metal balls each of 10mmφ. Following images Fig4.1 (a), (b) are SEM images of sample before ball milling process.



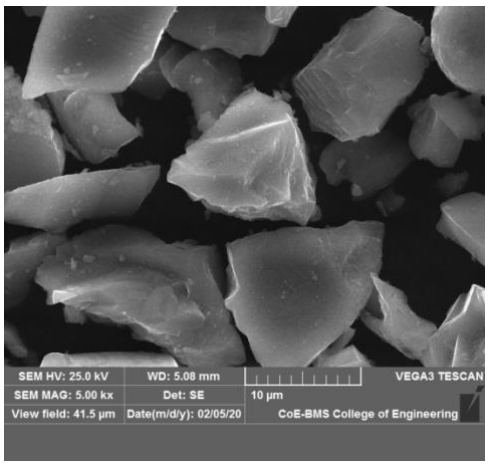
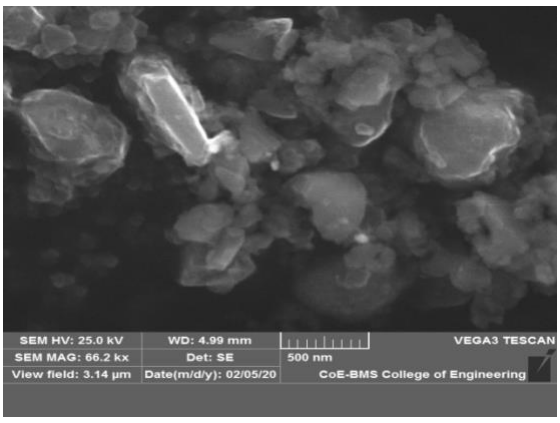
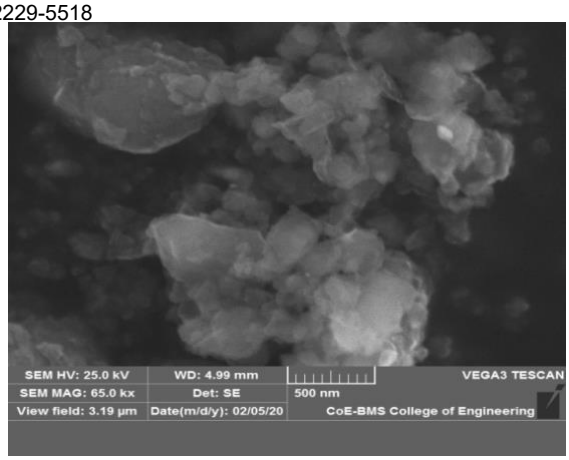


Fig 4.1(a), (b), SiC particles (37µm) before ball milling

During ball milling, the balls are lifted up on the rising side of the container and they fall down. While this happens, the powders present in between the WC balls are pulverised due to impact. If the machine is run without rest, the amalgamation of particles takes place due to heat between the powders, which can result them to combine together, which will be more difficult to separate. Therefore repetitive cooling and distribution of particles is to be followed for certain time period. The images can be compared with Fig 4.2 (a), (b).



(a)



(b)

Fig 4.2(a), (b), SiC particles (500nm) after ball milling

4.2 Sintering Process

The sintering process is performed using SPS(SPARK PLASMA SINTERING) with an initial pressure of 50Mpa under vacuum condition and heating rate of 100°C/min followed by increasing pressure during the process and finishing at sintering temperature of 1900°C and 2200°C for about 8 and 6 min as holding time.

The Sintering process takes place in a gradual manner. Initially the samples are kept inside the die and vacuum is maintained (about 15-20Pa). The temperature is progressively increased alongside the pressure on the die with the help of spacers, starting with 10MPa.

Heat through electrode is sent to matrix-reinforcement interface, which is elevated to temperatures, sufficient to break the bond with the fiber. The pressure is then gradually increased to 50MPa and temperature will rise at a rate of 100ºC/min till maximum temperature is reached (1900ºC or 2200ºC). The primary oxidation issue in sintering conduct of SiC based composite particularly fortified with carbon based materials have been wiped out at high temperatures. A process prompts positive development in punch dislodging for the two examples at the temperature up to about 1200ºC. This pattern is seen until temperature up to about 1600ºC, and at this temperature, the sintering procedure would be begun for the two samples.

The all out development of test sintered at 1900ºC is higher than that of the example sintered at 2200ºC which can be clarified by traditional sintering system and higher applied current which prompted the higher heating rate for the 2200ºC example. It can be observed that this "traditional" quick heating rate to arrive at 2200ºC with practically equivalent sintering time prompted abbreviate development as opposed to sintering process by production of higher measures of heat between SiC nanoparticles for test sintered at 2200ºC compared to 1900ºC.

4.2.1 Graphite Dies

Graphite dies are to be used in order to heat the materials to such high temperatures, as shown in the below figure Fig 4.3.

The punches are held on both the sides of the die and pressure is applied onto the die through the pair of punches. In between the pair of punches lies the powder, which is compressed to required pressure calculated from density ($3.21 g/cm^3$). This can be understood using the image shown below (Fig4.4).

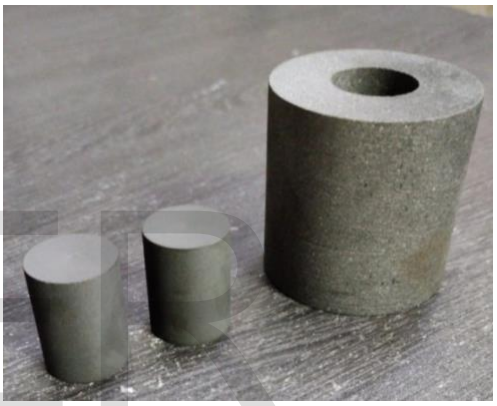


Fig 4.3: Graphite die 20.5mmϕ, 50 mm length along with two punches 20mmϕ, 27mm length.

Graphite is regularly utilized in SPS equipments as a viable contact material, especially in sliding conditions, because of its high electric and therm-al conductivity, oxidation and wear re-sistance. Its stacking limit is anyway restricted to 80– 150MPa. During SPS heating, elastic and plastic deformation of the contact roughness may initiate reversible or irreversible changes in the contact zone. Fig3.4 appears (a) sketch of a free and (b) close leeway along the (vertical) punch/die contact interface. Too little freedom may cause unreasonable seizing. On the other hand, too huge freedom may result in too much contact obstruction. Hence maintaining optimum clearance is necessary.

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(a)

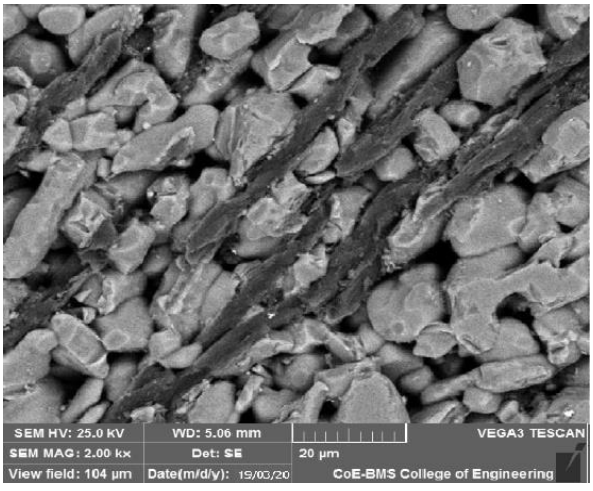
(b)

Fig 4.5: Samples sintered at (a) 1900ºC and (b) 2200ºC

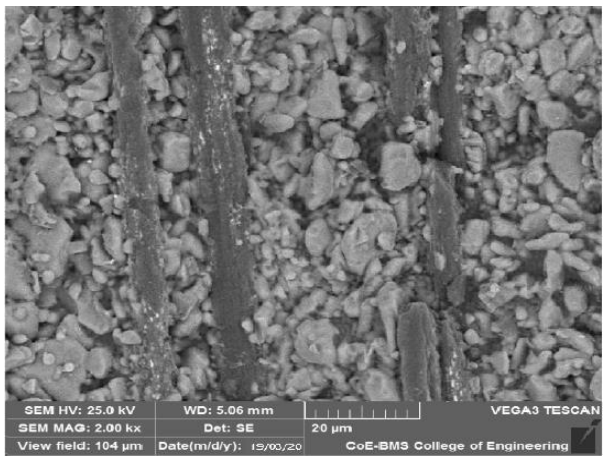
After the process is completed, the displacement is returned to first stage. The pressure is increased gradually, until the die retains its properties after bearing such high temperatures. This process is done until soaking time is reached to avoid graphite die fracture.

4.2.2 Testing

After removing the material from die and punch, the SEM image is taken out. Following figures, Fig4.5 (a), (b) are SEM images of samples taken after sintering.



(a)



(b)

Fig 4.6(a), (b): SEM images of samples sintered at 1900ºC and 2200ºC respectively

4.3 Vickers Hardness Test

Vickers Hardness is a famous test, which is portrayed by a square based Diamond pyramid indenter. The indenter is set precisely ground to a standard structure with 136 degrees between inverse faces and used to leave an imprint in metal under a correctly applied power by taking consideration to stay away from sway. The diagonals of the impression must be estimated utilizing an appropriate magnifying instrument and the outcomes are determined utilizing given formula:

$$H_V = 1.8544 \times (P / d^2)$$

Where, P = force (load) in Kgf

d = Arithmetic mean of the two diagonals, d1 and d2 (in mm).

Regularly utilized loads are 10, 15, 30 and 50 kg, however the range can be augmented if vital. In principle the hardness number ought to be autonomous of the load utilized, however practically speaking, as contrasts can be discovered, one should consistently report the load utilized.

Least depth of at least 10 times the profundity of the in-dentation is to be estimated, yet it is simpler to decrease it, just by choosing a lighter force. As common no imprints ought to be seen on the contrary surface.

The Vickers hardness measurement is carried out on the polished surface of specimen for 30kgf load. Five successive indentations were carried out. The suitably adapted diagonals

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of the indentation and crack sizes are measured using the optical microscope and SEM observation.

The Fracture toughness (K_{IC}) is calculated using the formula

$$K_{IC} = 0.18(P/c^{1.5})\ (E/Hv)^{0.5}$$

Where P is the load (N) and c is half of the average crack length (m), and E is Young's modulus, and H_V is Vicker's Hardness.

Temperature(°C)	Vicker's Hardness (H_v)	Fracture Tiughness (K_{IC})(MPam$^{1/2}$)
1900°C	2150	2.8
2200°C	2981	4.2

5 Conclusion

In our work, SiC/carbon fiber composites have been devoleped effectively by sp-ark plasma sintering at increased temperatures (1900ºC and 2200ºC). The examination uncovered that the densification begins at around1600ºC. The microstructure examinations indicated uniform placements of carbon fibers in SiC framework. The mechanical properties (the hardness of 2981Vickers (2981HV30/10), and failure toughness of 4.2 MPa m$^{1/2}$) were acquired for SiC/Cf test sintered at 2200ºC which was higher contrasted with test sintered at 1900ºC (2150 Vicker's (2150HV30/10), and failure toughness of 2.8 MPa m$^{1/2}$)

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