

STUDY OF THE EFFECTS OF CARBON AND GLASS FIBRE REINFORCEMENT AND OTHER FILLERS ON ELEVATED TEMPERATURE RESISTANT PROPERTIES OF ER MATRIX COMPOSITES

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Abstract

In the present study, composite materials required for elevated temperature applications were fabricated using vacuum bagging technique. Epoxy Resin (ER-VP401) was used as the matrix and Glass fibre was used as reinforcement. SiC, Al₂O₃ and others were used as fillers to bring in elevated temperature resistance. These composites were subjected to mechanical tests like Tensile, Hardness and Impact test. Tribological tests like two body abrasion and Pin on disc (POD) were carried out. Tensile strength, hardness and impact energy were improved with increase in fillers content. Wear resistance also improved with increase in percentage of fillers substantially. SEM micrographs are used to explain the mechanism of the material strengthening at elevated temperatures.

Keywords: Epoxy resin, Glass Fiber (GF), Al₂O₃, SiC, Elevated Temperature Resistance.

1. INTRODUCTION

The materials used to manufacture various products are supposed to fulfill several criteria before being approved. Some of the criteria are the results of regulation and legislation with the environmental and safety concerns and some are the requirements of the customers. Composite material is a combination of two or more materials to form a new material system with enhanced material properties. After being solely used for their electromagnetic properties (insulators and radar-domes), using composites to improve the structural performance of spacecraft and military aircraft became popular in the last two decades. Nowadays, cost reduction during manufacturing and operation are the main technology drivers. Fibers or particles embedded in matrix of another material are the best example of modern-day composite materials.

For most structural applications in the current aircraft designs, polymer composites has been adequately used and implemented for a wide range of applications in areas where high temperatures are encountered. The usage of such composites, even for primary loadbearing structures in military fighters, transport aircraft, satellites and space vehicles has been beneficially realized. Attention is now focused on expanding the usage of such composites to other areas where temperatures could be higher in the range of 200-400°C [1]. Multi-Walled Carbon Nanotubes functionalized with amino groups (MWCNT-NH₂) via chemical modification of the carboxyl groups introduced on the surface of MWCNT. The thermal diffusivity and

conductivity of all of the composites continuously improved with increasing content of fillers [2]. The behavior of Glass Fiber-Reinforced Polymer (GFRP) bars subjected to extreme temperatures is very critical for industrial applications. They evaluated the variation of mechanical properties of sand-coated GFRP reinforcing bars subjected to low temperatures (ranging from 0 to -100°C) and elevated temperatures (ranging from 23 to 315°C). Tensile, shear and flexural properties improved as the glass fiber content increased [3]. The effect of addition of Silicon Carbide (SiC) filler in different weight percentages on physical properties, mechanical properties, and thermal properties of chopped glass fiber-reinforced epoxy composites has been investigated. The result showed that the physical and mechanical properties of SiC-filled glass fiber-reinforced epoxy composites were better than unfilled glass fiber-reinforced epoxy composites [4]. [5] Presented results of an experimental and analytical study about the mechanical behavior at elevated temperatures of Glass Fiber Reinforced Polymer (GFRP) pultruded profiles made of polyester resin and E-glass fibers. [6] observed the mechanical properties of Vapor Grown Carbon Nano fiber (VGCNF)polymer composites. They studied the structural and intrinsic mechanical properties of VGCNFs. Then the major factors (filler dispersion and distribution, filler aspect ratio, adhesion and interface between filler and polymer matrix) affecting the mechanical properties of VGCNF/polymer composites were presented. The effect of fiber content and fiber orientation on the strength of composites was studied to estimate the tensile strength out of fibre orientation and fibre content [7].

The F584/PW Pre-impregnated materials (prepregs) presented the highest values of tensile strength while the highest modulus results were obtained for the 8HS composite laminates [8]. [9] Conducted a research on epoxy resin polymers reinforced with natural fibers like Sisal, Banana and Roselle and three hybrid combinations of any two fibers. Less elongation and fiber pull out and brittle nature of fracture were observed in fiber based composites while more elongation, fiber pull out and partial brittle nature of fracture were observed in hybrid composites. It was seen from the results that well dispersed Nano particles of CaCO₃ up to the weight percentage of 15 increases the impact strength of the composite [10]. [11] Conducted impact tests on aluminum filled milled (carbon and glass) fiber reinforced epoxy based polymer composites. The milled fiber addition slightly increased the impact resistance of the composites. [12] Studied the impact resistance of epoxy based composites reinforced with fiber and hybrid of sisal, banana and Roselle fibers. The results showed that the hybrid composites absorbed more impact energy before fracture. The greater level of fiber pull out observed in specimens fabricated using hybrid reinforcement, leads to superior impact strength. The effect of the reinforcement of thermosetting polyester with short glass fibers on mechanical properties and tribological behavior was studied. The friction and wear-behavior as a function of sliding speed and fiber-glass proportion (0 to 50%). Wear resistance behavior increased with increase in glass fiber and filler content [13]. [14] Investigated that the tribological behavior was found to depend on the filler materials in the tested composites and better results were obtained for the composite containing solid lubricants (Polytetrafluoroethylene (PTFE) and graphite). [15] Observed that the wear loss increases with increase in normal load. The optimum wear reduction was obtained with 40% fiber content.

The objective of the present study is to investigate the effect of filler materials on the mechanical properties of the selected polymer matrix composite at elevated temperature. The composition of the specimens is given in table-1.

2. MATERIALS AND METHODS

Table-1: composition of the specimens

Components' percentage by weight	Specimen No				
	1	2	3	4	5
Epoxy Resin	50	50	50	50	50
Activated Carbon Powder	10	10	10	10	10
Chopped E - Glass fiber	10	10	10	10	10
Sodium Sulphide (Na ₂ S)	5	5	5	5	5
Sintered Clay	5	5	5	5	5
Silicon Carbide (SiC)	20	15	10	5	0
Aluminium Oxide(Al ₂ O ₃)	0	5	10	15	20

2.2 Specimen Preparation and Experimental Set Up

Table I gives the materials used in the present study. Epoxy resin is kept constant at 50 Wt%. Activated carbon powder and chopped E glass fibre were kept at 10 Wt% respectively, sodium sulphide and sintered clay were used as fillers to

bring in elevated temperature resistance properties and they were kept at 5 Wt% each. Silicon carbide (SiC) and Aluminum oxide (Al₂O₃) were varied from 0-20 Wt% each.

First the materials are weighed as required, and then they are put together and mixed well. The mixture is then poured into a prepared mould of the required thickness. It should be noted that the epoxy resin and hardener start to set i.e. start solidifying after 30 minutes of mixing and hence, the mixture should be poured into the mould before the setting time. The mixture is poured in excess and suitable weights are applied on it. Similarly, five different compositions are poured in separate moulds by varying the SiC and Al₂O₃ content while keeping all the other weight percentages constant. The content of SiC and Al₂O₃ are varied in steps of five percent in such a way that in any composition, the sum of SiC and Al₂O₃ is 20 percent of the total weight. The moulds are then left for 24 hours to solidify and cure at room temperature. After solidification, the specimens are removed from the mould and post cured at 100°C for 2 hours in a hot air oven. The specimens are then taken out and labeled. The specimens are then marked as per the test standards. Specimens were prepared for tensile, impact strength and wear tests. Figure-1 shows the specimen preparation pictures.



(a) Weighing the constituents (b) Mixing the constituents in right Proportion



(c) Pouring the mixture into the (d) Specimens Moulds
Fig-1- Preparation of Specimens

To determine the mechanical properties of the material the following tests were conducted.

- A. *Tensile test-* Tensile tests were conducted according to the ASTM D-638. Computerized Universal Testing Machine (UTM) used for this purpose and the loading arrangement is shown in Figure. 2(a). Specifications are also mentioned. The dimension of the tensile

specimen was 165 mm x 19 mm x 3.2 mm. Gauge length was 50 mm. Results were used to calculate the tensile strength of composite samples.

- B. **Impact test** - Izod impact tests were conducted on V-notched composite specimen according to ASTM D256. A Pendulum impact tester, shown in Figure 2(b) was used for this purpose. Dimension of the specimen were 64 mm x 12.5 mm x 3.2 mm. The pendulum impact testing machine ascertains the impact strength of the material by shattering the specimen with a pendulum hammer, measuring the spent energy and relating it to the cross section of the specimen. The respective values of impact energy of different specimen are recorded directly from the digital indicator and reported.
- C. **Hardness test** - Shore-D hardness tests were conducted on specimen according to ASTM D2240 using Durometer shown in Figure 2(c). The hardness tester is placed on the specimen and pressure is applied so that the flats underneath the tester touch the surface of the specimen. The readings are taken directly from the dial. The specimens are then heated to different temperatures and the readings are taken to determine the variation in the hardness of the specimen with respect to temperature.
- D. **Wear test** - Wear tests were conducted according to the ASTM using Pin on Disc Machine (POD). The machine and its specifications are given in Figure 2(d). Dry sliding tests were conducted at ambient conditions with the loads of 4 Kg, 8 Kg, 12 Kg and 16 Kg. Disc speed was kept at 1000rpm with track diameter of 40mm, resulting in a sliding velocity of 3.92 m/s. The tests were conducted for two minutes or the length of time until the specimen withstands the maximum load and failed whichever being the earliest. The temperature of the tip of the specimen was registered using the optical pyrometer. Wear of the materials considered was measured by loss in length which is then converted into wear volume using the measured cross-sectional area data. The specific Wear rate (K_s) was calculated from the below equation (1).

$$K_s = \frac{\Delta V}{P \times D} \text{ in } \frac{m^3}{N - m}$$

Where, ΔV is the volume loss in m^3 , P is the load in Newton; D is the sliding distance in meters.



(a) Tensile testing machine setup (UTM) (b) Computerized impact testing machine



(c) Shore-D hardness testing machine (d) Pin on disc machine

Fig-2- Testing machines

3. RESULTS AND DISCUSSIONS

Physical and mechanical properties describe the behavior of materials when they are used in practical applications. The properties such as hardness describe the physical state of the system. The mechanical property of the material is a measure of the behavior of the material under different loading conditions. Tests were done to notice the effect of variation of filler content on the physical and mechanical properties and the optimum filler loading of carbon-glass reinforced polymer composites at which specific Specific Wear rate is least.

3.1 Effect of SiC and Al₂O₃ on Tensile Strength of Carbon-Glass Reinforced Polymer Composites

The tensile strength is an engineering value that is calculated by dividing the maximum load on the material by the initial cross sectional area of the test specimen. The Table II shows the results obtained during the tensile test conducted on all the five specimens.

Table-:2 tensile strength of cgrp composites

Specimen No.	Filler Content	Ultimate Tensile Strength (MPa)
1	20% SiC, 0% Al ₂ O ₃	15.38
2	15% SiC, 05% Al ₂ O ₃	17.31
3	10% SiC, 10% Al ₂ O ₃	19.35
4	05% SiC, 15% Al ₂ O ₃	20.98
5	0% SiC, 20% Al ₂ O ₃	25.28

The graph for the tensile strength is shown in Figure 3.1

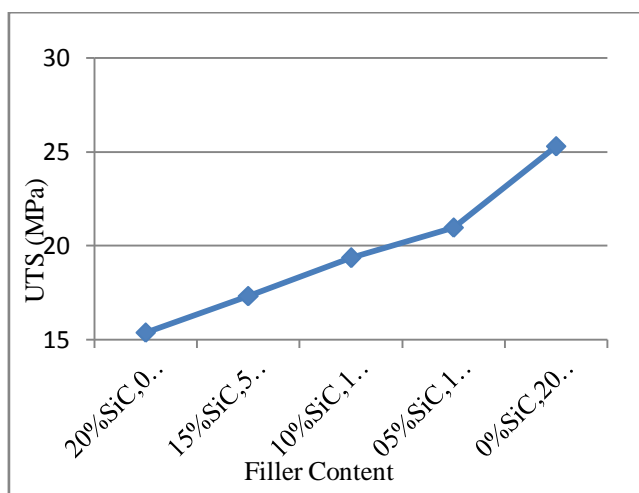


Fig 3.1 – Effect of SiC and Al₂O₃ on Ultimate Tensile Strength (UTS) on CGRP Composites

From the graph 3.1, it can be seen that the tensile strength is highest in the composite with 20% Al₂O₃ and no SiC, and lowest in that with 20% SiC and no Al₂O₃. The tensile strength increases proportionally with the increase in Al₂O₃ content and decrease in SiC content since the tensile strength of Al₂O₃ is higher than that of SiC. But complete absence of SiC in the fifth composite specimen results in a higher tensile strength which is not in proportion with the tensile strengths of other specimens. This is due to the presence of higher percentage of Al₂O₃ alone and no SiC. The Al₂O₃ particles which fill the composite, due to their higher tensile strength can withstand more loads and transfer it to the adjacent particle at the same time, thus reducing the load concentration at a single point which in turn reduces the stress concentration thereby increasing the ultimate tensile strength of the specimen. Also, the Al₂O₃ particles which are smaller than the SiC particles have a higher density compared to the SiC density. The higher density results in a uniform and continuous distribution of the filler and lower bonding surface resulting in increase in bonding strength which also results in increase in the tensile strength of the specimen.

The specimen with 20% SiC and 0% Al₂O₃ has the least tensile strength of the five compositions. This is due to the low tensile strength of SiC and its larger particle size. While the low tensile strength of SiC hampers the tensile strength of the specimen directly by failing at lower loads, the larger particle size of SiC results in a discrete distribution of filler along with an increase in bonding surface area which decreases the binding strength, thereby decreases the tensile strength of the specimen.

3.2 Effect of SiC and Al₂O₃ on Impact Strength of Carbon-Glass Reinforced Polymer Composites

The material’s resistance to fracture is known as toughness. It is the energy absorbed by the material before fracture and is expressed in terms of the same. A ductile material can absorb considerable amount of energy before fracture while a brittle material absorbs very little energy before fracture.

Table III shows the results obtained during the impact test conducted on all the five specimens.

Table-3: Impact Strength Of Cgrp Composites

Specimen No.	Filler Content	Impact Energy (J)
1	20% SiC, 0% Al ₂ O ₃	1
2	15% SiC, 05% Al ₂ O ₃	0.45
3	10% SiC, 10% Al ₂ O ₃	0.45
4	05% SiC, 15% Al ₂ O ₃	1.3
5	0% SiC, 20% Al ₂ O ₃	0.6

The graph for the impact strength is shown in Figure 3.2

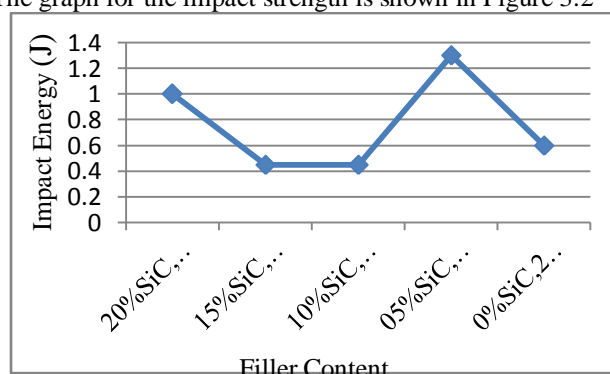


Fig. 3.2 - Effect of SiC and Al₂O₃ impact strength on CGRP Composites

From table III, it is observed that the impact energy is the highest for the composite having 05%SiC and 15%Al₂O₃. The fracture toughness for SiC is 1.5 times greater than that of Al₂O₃. Glass fiber has least fracture toughness, at the highest percentage of silicon impact strength is 1J, but it has decreased to 0.45J. It has remained the same for the further decrease in SiC, indicating the fracture toughness of both SiC and Al₂O₃ are contributing to the increase in impact strength, contribution from Al₂O₃ being higher. This emphasizes the further increase in Al₂O₃ percentage has increased the impact strength significantly (up to 15% of Al₂O₃).

The conclusion is that relatively lesser percentage of SiC is a must for increase in impact strength irrespective of increase in Al₂O₃. This observation is evident as the impact strength has decreased drastically for 0% of SiC.

3.3 Effect of SiC and Al₂O₃ on Wear Resistance of Carbon-Glass Reinforced Polymer Composites

Wear is the sideways erosion of material on a solid surface due to the action of another surface. A material is said to possess good wear properties when less amount of material gets eroded due to the friction. Table 3.3 shows the results obtained during the wear test conducted on all the specimens.

Speed of the disc = 1000 rpm = 4.18 m/s
 Radius of the disc = 40 mm
 Diameter of each specimen = 6 mm
 Length of each specimen = 30mm

Table 4: wear properties of cgrp composites

Specimen No.		Load(N)		
		39.24	78.48	117.72
1	Specific Wear rate (mm ³ /Nm)	2.722 x 10 ⁻⁵	7.23 x 10 ⁻⁵	13.19 x 10 ⁻⁵
2	Specific Wear rate (mm ³ /Nm)	9.88 x 10 ⁻⁵	8.814 x 10 ⁻⁵	8.743 x 10 ⁻⁵
3	Specific Wear rate (mm ³ /Nm)	3.14 x 10 ⁻⁵	6.28 x 10 ⁻⁵	8.743 x 10 ⁻⁵
4	Specific Wear rate (mm ³ /Nm)	2.28 x 10 ⁻⁵	7.57 x 10 ⁻⁵	5.712 x 10 ⁻⁵
5	Specific Wear rate (mm ³ /Nm)	2.86 x 10 ⁻⁵	5.589 x 10 ⁻⁵	4.52 x 10 ⁻⁵

Figure 3.3 shows the variation of Specific Wear rate with respect to weight graphically

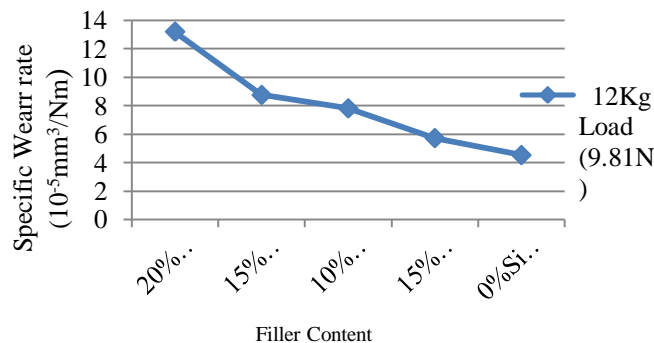


Fig. 3.3 – Effect of SiC and Al₂O₃ on Wear resistance of CGRP Composite at maximum load

3.4 Effect of Temperature on Hardness of Carbon-Glass Reinforced Polymer Composites

Hardness is a measure of how resistant a solid matter is to various kinds of permanent shape change when a force is applied. The hardness readings of the specimens were obtained directly from the Shore-D hardness tester. The specimens were heated to six different temperatures by a hot air oven and the readings were taken. Table V shows the readings thus obtained.

Table 5: Effect Of Temperature On Hardness Of Cgrp Composites

Specimen No.	Temperature (°C)						
	27	57	87	117	147	177	207
	7	7	7	7	7	7	7
Shore-D Hardness no.							
1 - 20% SiC, 0% Al ₂ O ₃	90	88	85	80	71	71	58
2 - 15% SiC, 05% Al ₂ O ₃	88	88	87	86	86	80	70
3 - 10% SiC, 10% Al ₂ O ₃	88	84	84	82	78	73	58
4 - 05% SiC, 15% Al ₂ O ₃	88	88	88	86	72	65	63
5 - 0% SiC, 20% Al ₂ O ₃	88	87	84	80	78	65	64
Average Shore-D Hardness no	88	87	86	83	77	71	63

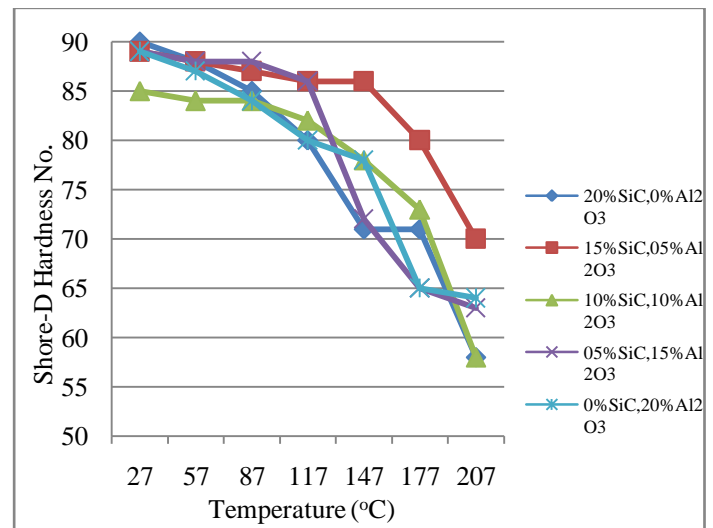


Fig. 3.4 – Variation of Shore-D hardness of CGRP Composites with temperature

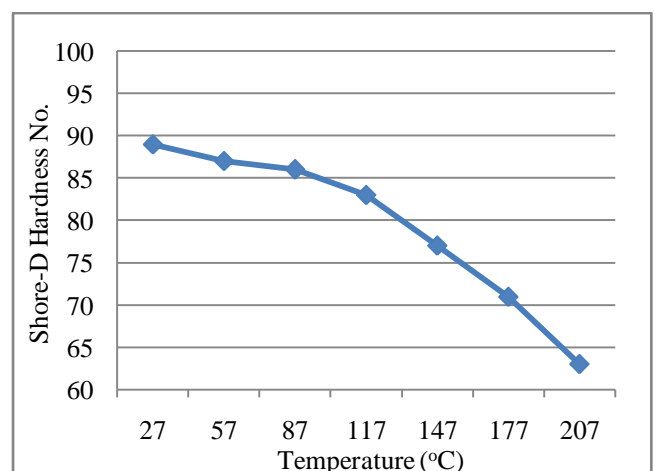


Fig. 3.5 – Variation of average hardness of CGRP Composites with temperature

It could be seen from the Table V and Fig 3.5 the average Shore-D hardness value obtained at room temperature for the entire specimen considered irrespective of the percentage by weight of SiC and Al₂O₃ is 89. Similarly, the average values of hardness for considered specimen at temperatures greater than the room temperature in steps of 30°C upto 207°C are 87,86,83,77,71,63.

From 27°C (room temperature) to 117°C, the hardness value has decreased by 6.7%, which is not very significant. From 147°C to 207°C, the percentage decreased in Shore-D hardness is 18.2%, which is significant. This means that the considered specimen can retain their hardness up to 120°C. Hardness of the SiC is 2.3 times greater than that of Al₂O₃, the thermal conductivity of SiC is 6.6 times greater than that of Al₂O₃. Whereas, the coefficient of thermal expansion of Al₂O₃ is 2 times greater than that of SiC.

At room temperature, the average value of Shore-D hardness is about 89. This value has decreased slightly up to 87°C, from 87°C up to 117°C the decrease in value is about 3.48%. Further increase in temperature in steps of 30°C up to 207°C, the percentage decrease is 6.89%.

The reason is that higher percentage of SiC has contributed to the average hardness of all the specimens considered up till 117°C. Slight Decrease in hardness in this temperature range is attributed to increase in percentage of Al₂O₃, which has got comparatively higher hardness, beyond 117°C one must note that SiC percentage has decreased and Al₂O₃ percentage has increased.

By the order of magnitude of hardness of SiC and Al₂O₃ it is evident that the hardness has decreased quite drastically up to 207°C. The synergy of these fillers has come to play up to 117°C and it has ceased to exist from 117°C to 207°C.

4. SEM MORPHOLOGY

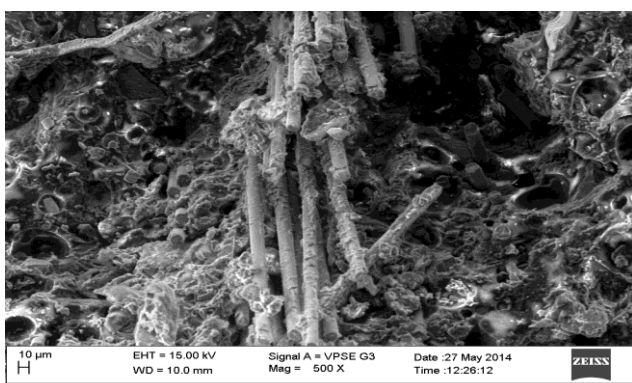


Fig 4.1 – SEM micrograph (500 X Magnification) of CGRP composite with subjected to tensile test

From the Table III, it is observed that tensile strength is increasing with increase in percentage of Al₂O₃, it is 64.36% increased which is very significant.

The variation is also linear; the increasing tensile strength up to 15% Al₂O₃, for every 5% addition of Al₂O₃ increasing in

tensile strength is 10.91%. Whereas, increase in tensile strength for 5% more addition of Al₂O₃, increases the tensile strength is 20.49%. One can observe that, the order of magnitude of increase in tensile strength for 5% more addition is almost twice.

SiC has 410GPa elastic modulus, whereas Al₂O₃ has 300GPa, it is evident that increase in tensile strength for the decrease in SiC is around 10% and increase in 5% of Al₂O₃ when SiC has become 0 is 20%. Hence, elastic modulus of SiC and Al₂O₃ are contributing to the increase in overall tensile strength.

These observations are also evident from SEM micrographs (Fig-4.1). As could be seen from the plate, GF have got pulled out from the matrix, whereas more GFs have broken in a brittle manner. These surfaces bonding energy between GF and resin is lesser when compared with that of Al₂O₃, SiC and other fillers. This means that the contribution of GF for the increase in tensile strength is not significant. It is the larger interfacial attractive forces between the fillers and their properties which have contributed to the increase in tensile strength. It can also be observed from SEM micrographs that very few of these particles are still in their location and these have not fractured.

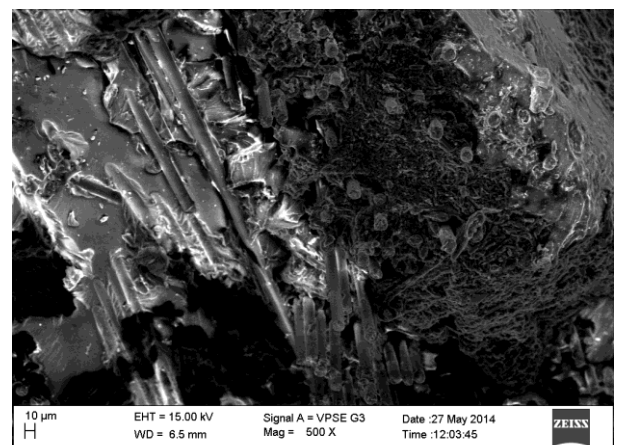


Fig 4.2 – SEM micrograph (500 X Magnification) of CGRP composite with subjected to Impact test

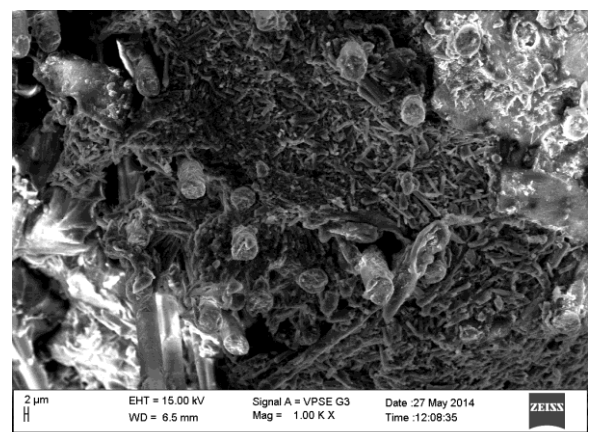


Fig 4.3 – SEM micrograph (1.0K X Magnification) of CGRP composite with subjected to Impact test

Figure 4.2 and 4.3 is showing SEM micrograph of the specimen having 5% SiC and 15% Al₂O₃, giving the highest value of impact strength. The main fractured constituent due to impact load are glass fibers which have got the lowest fracture toughness (1MPa m^{1/2}), these are indicated by the fractured glass fibers appearing as bright cylindrical columns. Most of the other particles have got embedded in the matrix including some broken GFs, this is appearing as the darker fibrous spot.

Obviously, the increase in impact strength is basically due to the presence of higher percentage of Al₂O₃ and lower percentage of SiC.

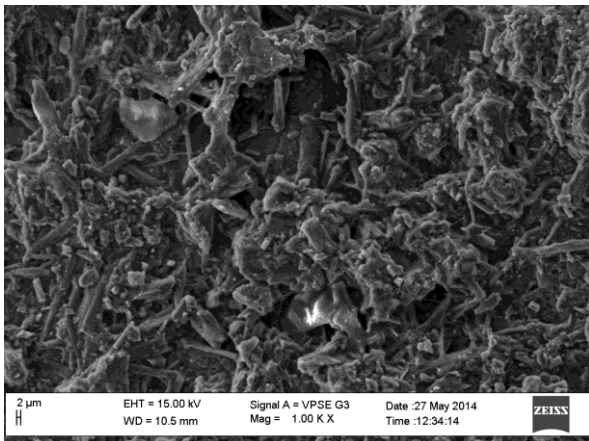


Fig 4.4– SEM micrograph (1.0K X Magnification) of CGRP composite with subjected to wear test

Figure 4.4 is showing SEM micrographs for the specimen having 10% SiC, 10% Al₂O₃ at the maximum load. The wear Debris of GFs appears as shorter brighter particles. Bright smaller dot represent Al₂O₃ and somewhat larger bright spots are representing SiC particles. The Graphite, carbon and sodium sulphide which have been used as lubricants do not appear predominantly for identification.

One can observe from the micrographs that the wear debris of glass fiber and the filler particles have got embedded on the plateaus formed on the surface of composite, the particles which are not worn are present in the dark portions of the SEM micrographs.

Higher thermal conductivity of SiC has softened the matrix which softened the matrix which facilitated the formation of plateaus, which have created a protective shield against wear.

5. CONCLUSIONS

The present study and analysis of results had led to the following conclusions

1. 10% Al₂O₃ and 10% SiC has shown minimum Specific Wear rate (4.39×10^{-4} mm³/Nm) and could be considered as optimum percentage of these fillers for further development.
2. The impact energy for the composite having 05% SiC and 15% Al₂O₃ has shown the maximum impact

strength, which is due to the fracture toughness of both SiC and Al₂O₃ are maximum in comparison with other fillers.

3. The tensile strength increased is 64.36%, which is very significant this is due to high tensile modulus of both SiC and Al₂O₃.
4. The general conclusion is that the addition of SiC and Al₂O₃ has significantly contributed to the improvement in wear resistance, impact and tensile strengths predominantly by Al₂O₃.

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