Influence of Zinc Oxide and Silicon Carbide Micro fillers on Impact Strength and Hardness in E-Glass/Polyester Composites: Fabrication and Testing

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Abstract – **Now-a-days, polymer matrix composite plays a vital role in industries namely automotive, aerospace and marine. Polymer matrix modification is one of the approaches to develop new class of polymer structural materials. This modification can be done by addition of different ceramic powders of different sizes to achieve the required mechanical properties. In this paper polyester matrix was modified by SiO_2 and ZnO microparticles in glass fiber/polyester composite to improve mechanical properties. This paper involves fabrication of polyester resin composites using zinc oxide, silicon carbide with different proportion of ZnO and SiC along with GFRP. A mixing unit has been fabricated for making reinforcement mixtures by using hand lay-up technique. Mechanical testing like impact strength and hardness are conducted to know properties of fabricated composites. Results show composites with filler have higher strength as compared to composites with unfilled ones.**

Keywords: GFRP, Micro fillers, Zinc Oxide, Silicon Carbide, Impact strength, Hardness

I. INTRODUCTION

IN THE current quest for improved performance, which may be specified by numerous criteria comprising less weight, more strength and lower cost, currently used materials frequently reach the limit of their utility. Thus material researchers, engineers and scientists strive to produce either improved traditional materials or completely novel materials. Composites are an example of the second category. Over the last thirty years, composite materials, plastics and ceramics have been the prevailing emerging materials. The volume and numbers of applications of composite materials have developed steadily, penetrating and conquering new markets persistently. Modern composite materials establish a significant proportion of the engineered materials market ranging from everyday products to sophisticated niche applications.

Composites have already proven their worth as weight-saving materials; the current challenge is to make them cost effective. The hard work to produce economically attractive composite components resulted in several innovative manufacturing techniques currently being used in the composites industry. The composites industry recognizes that commercial applications of composites promise to offer much larger business opportunities than the aerospace sector due to the sheer size of transportation industry. India endowed with an ample availability of natural fiber such as Bamboo, Ramie, Jute, Sisal, Pineapple, Coir, Banana etc. focused on the improvement of natural fiber composites mainly to explore value-added application avenues. Such natural fiber composites are well matched as wood substitutes in the housing and building sector.

Development of natural fiber composites in India is based on two cleft strategy of preventing depletion of forest resources as well as ensuring good economic returns for cultivation of natural fibers. Developments in composite material after meeting the challenges of aerospace industry have poured down for catering to domestic and industrial applications. Composites, the spectacle material with light-weight; high strength-to-weight ratio and stiffness properties have come a long way in replacing the conventional materials like wood, metals etc.

Need of a composite: Paradigm in Materials Science: “smaller is stronger”. In the present stage of technological improvement, the strongest materials that can be easily handled are manufactured in the form of small diameter fibers (< 200 μm). The use of fibers in structural elements has the following limitations:

- Punching
- Surface damage (wear, abrasion and chemical attack)
- Buckling
- Optimum orientation.

These problems are overcome when the fibers are embedded in a continuum matrix. Composite materials were developed in parallel with manufacturing of new fibers with high stiffness and strength. Thus, the first reason to use composite materials is to take advantage of outstanding mechanical properties of fibers. Dispersion of reinforcements (either particles or fibers) can improve the matrix properties (toughness of ceramics, conductivity of polymers, high temperature mechanical properties of metals), leading to new applications.

The biggest advantage of modern composite materials is that they are light as well as strong. By choosing an appropriate combination of reinforcement material and matrix, a novel material can be made that exactly meets the requirements of a specific application. Composites also give design flexibility because many of them can be moulded into complex shapes.

Filler and Other Additives: The most common filler in polyester and vinyl ester resins is calcium carbonate. It not only reduces the cost but also lessens mould shrinkage. Examples of other fillers are clay, mica, and glass microspheres. Though fillers increase the modulus of an unreinforced matrix, they also tend to reduce its strength and impact resistance. The impact strength and crash resistance of brittle thermosetting polymers can be improved by mixing them with small amounts of elastic elastomeric toughness.

E-Glass Fiber: Fiber is the reinforcing phase of a composite material. The present research work, woven E-glass fiber is taken as the reinforcement in the epoxy resin and polyester resin to fabricate composites samples.

E-glass woven roving is a bi-directional fabric made by interweaving direct rovings and is compatible with many resin systems such as polyester, vinyl ester, epoxy and phenolic resins. These fibers are high-performance reinforcement widely used in hand lay-up and robot processes for the production of boats, vessels, plane and automotive parts, furniture and sports facilities. It is relatively low cost, the most common form of reinforcing fiber used in polymer matrix composites. "E" glass produced fibers are considered as predominant reinforcement for polymer matrix composites due to their:

- High electrical insulating properties,
- Low susceptibility to moisture,
- High mechanical properties, and
- Low cost.

Due to the above promising characteristic it is widely adopted in Aviation industry, E-glass fiber has been taken as reinforcement for this work. The type of E-Glass fiber which is used in this study is woven rovings. This fiber type has good mechanical properties as compared to chopped mat and it is used when higher strength part is required. The typical picture of plain woven E-glass fiber is shown below.

Figure 1. Rolled Woven Fabrics.

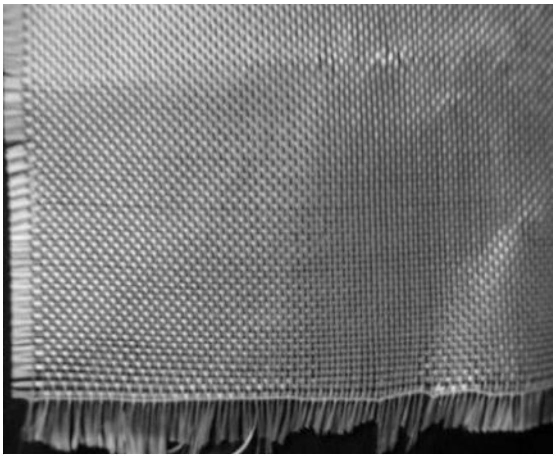

Figure 2. Woven Fabrics E-glass fiber.

Polyester Resin and its Catalyst: It is a low viscosity for fast wet-out, styrene suppressed, high thixotropic index to prevent draining on vertical surfaces. It exhibits good mechanical and electrical properties together with good chemical resistance compared to general purpose resins.

Catalyst

Polyester resin is cured by adding a catalyst, which causes a chemical reaction without changing its own composition. The catalyst initiates the chemical reaction of the unsaturated polyester and monomer ingredient from liquid to a solid state. When used as a curing agent, catalysts are referred to as catalytic hardeners. The curing agent applied for the liquid resin is Hardener (MEKP).

II. LITERATURE SURVEY

Nafisa Gull *et al.* [1] studied that mechanical and thermal properties of GFRP composite are improved with incorporation of ZnO filler. Flexural modulus and flexural strength are enhanced by raising the filler loading up to 3 wt.% of ZnO and then decreased. Impact energy is enhanced gradually by increasing the filler concentration in fiber reinforced composites. Hardness also shows similar increasing behavior. Degradation temperature of ZnO filled composite has been found increased but amount of residue decreased up to 3 wt.%. Beyond this concentration negative effect was observed. Hence, it is concluded that ZnO is a promising filler for GFRP composite as it is responsible for the incremental modification in its mechanical and thermal behavior.

S.Srinivasa Moorthy *et al.* [2] investigated the composite specimens to determine the tensile strength, impact strength, hardness and chemical resistance. It was observed that the inclusion of titanium oxide and increase of fiber length resulted in composites with increased tensile strength, impact strength, hardness and chemical resistance. The effect of pull load on the composites was studied with scanning electron microscope images.

Andrzej *et al.* [3] investigated influence of type of reinforcing fiber, fiber and micro void content on mechanical properties of composites. Increasing the fiber content leads to increase in the impact strength and shear modulus. Though, increasing the micro void content in the matrix results in decreased impact strength and shear modulus.

Oboh *et al.* [4] reported the potentialities of Luffa cylindrical crop that is virtually found around the world in the areas such as medicine, agriculture, science, biotechnology and engineering and has been discussed on recent major advances and discoveries. The authors conclude that in the context of the morph synthesis, the ability of replication of the luffa sponge unties the chances of the use of biodiversity in obtaining new materials. This emerging cash crop will expand the economies of many nations in the near future because of its numerous potentials.

Lassad Ghali *et al.* **[5]** studied effect of chemical modification of luffa fibers on mechanical and hydrothermal behaviors of polyester composites. They reported that acetylation treatment improved mechanical properties of composites. The process decreased hydrophilic behavior of the luffa fibers, improving their adhesion to polyester matrix. Chemical alterations at the surface of the luffa fibers also decreased the diffusion coefficient and the maximum amount of water absorbed by these fibers. The diffusion process was found to be affected by external loads applied on the exposed composite materials.

K. Trinath *et al.* [6] studied stir processing method used to increase the strength of polymer with the addition of reinforcements such as micro and nano silica, ZnO and chitin powder. They concluded that by adding ZnO nano particles with micro silica and chitin, the tensile property of polyester composite was increased. The hardness increased with the addition of Silica (micro and nano) and ZnO whereas, it decreased with the addition of chitin powder.

Guirong Peng *et al.* [7] investigated degradation behavior of ZnO-glass fiber-unsaturated polyester composite under exposure to a metal halide lamp. Results show that the UV photons can increase the carbonyl group on the surface, but ZnO can decrease the carbonyl content and further contribute to the breaking of single bond of carbon to oxygen on the surface. All results indicate that for pure unsaturated polyester under the UV exposure in air, the major reaction is photo-initiated oxidation, whereas for the composites with ZnO, decarbonylation is the major effect. In addition, when the content of ZnO is not higher than 4 wt. %, the composites are still transparent, and its effects on the cross linking process and thermal stability are insignificant. ZnO can be used as a stabilizer of unsaturated polyester for the glass fiber reinforced polymer industry.

Surfsh J S *et al.* [8] studied mechanical characterizations of Epoxy and polyester based composites reinforced with Glass Fiber and filled with the natural filler, Arabic Gum Tree Coal Powder (A.C.P). The experimental results demonstrate that tensile strength (221.4Mpa) is more for the composite of designation Epoxy/Glass fiber. This is due to the strong interface adhesion between polyester resin and filler particles. Tensile modulus for the composite Epoxy/Glass fiber is maximum and is equal to 6.4GPa. This is because of good adhesion between glass fiber and polymer resin.52.5 wt% polyester and 40wt% G.F with 7.5wt% A.C.P composite exhibited maximum flexural strength.

Devendra *et al.* [9] studied that mechanical behavior of E-glass fiber reinforced epoxy composites filled with varying concentration of aluminum oxide (Al_2O_3), magnesium hydroxide ($Mg(OH)_2$) and silicon carbide(SiC). Experimental results show that composites filled by (10% Vol.) $Mg(OH)_2$ exhibited maximum ultimate tensile strength and SiC filled composites exhibited maximum impact strength, flexural strength and hardness.

S. Rajesh *et al.* [10] studied fabrication of epoxy and polyester resin composites using aluminium oxide, silicon carbide with different proportion of Al_2O_3 and SiC along with GFRP. They reported that GFRP composites with epoxy resin have very high tensile strength 36.53MPa. Also since adding of fillers in the composite, Silicon carbide the composite has provided very good stiffness and shrinkage has been reduced. The biaxial stresses are more in GFRP composite with polyester resin of 40.7MPa.The shear strength is also more in the GFRP composite with polyester resin of value 6.45kN.

III. MATERIALS AND METHODS

E-glass fiber: E-glass woven roving is a bi-directional fabric made by interweaving direct rovings and is compatible with many resin systems such as polyester, vinyl ester, epoxy and phenolic resins. These fibers are high-performance reinforcement widely used in hand lay-up and robot processes for the production of boats, vessels, plane and automotive parts, furniture and sports facilities.

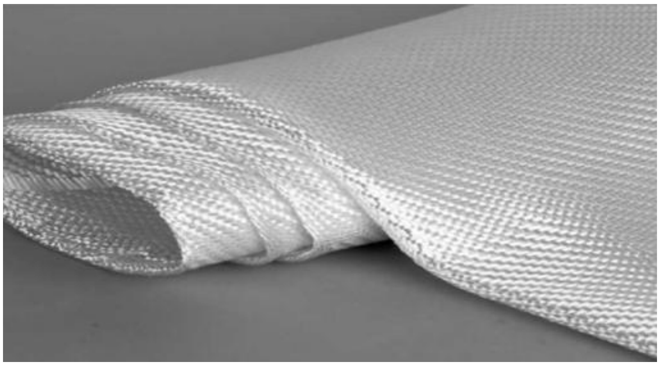

Figure 3. E-glass fiber.

TABLE 1–MECHANICAL PROPERTIES OF E-GLASS FIBER

S.N.	Property	E-Glass Fiber
1	Density (gm/cm^3)	2.55-2.6
2	Modulus of elasticity (GPa)	72-85
3	Tensile Strength (MPa)	1950-2050
4	Poisson's Ratio	0.21-0.23

Hardener

The hardener, MEKP (Methyl Ethyl Ketone Peroxide) is added to cure, or harden the resin. MEKP hardeners for polyester resin, are often referred to as catalyst.

Figure 4 (*a*). Polyester Resin with Hardener.

Figure 4 (*b*). Zinc Oxide.

TABLE 2–MECHANICAL PROPERTIES OF POLYESTER RESIN

S. N.	Property	Polyester rasin
1	Density(gm/cm$^{3)}$	1.1-1.4
2	Modulus of elasticity (GPa)	1.3-4.5
3	Tensile Strength (MPa)	40-85
4	Poisson's Ratio	0.33

Zinc Oxide

Zinc oxide, an inorganic compound is a white powder that is insoluble in water, and widely used as an additive in numerous materials and products including rubber, plastics, ceramics, glass, cement, lubricants, paints, ointments, adhesives, sealants, pigments, foods, batteries, ferrites, fire retardants, and first-aid tapes. ZnO is a relatively soft material with approximate hardness of 4.5 on the Mohs scale. Though it occurs naturally as the mineral zincite, most zinc oxide is produced synthetically. Mechanical properties of ZnO are given in table 3.

TABLE 3–MECHANICAL PROPERTIES OF ZINC OXIDE

S.N.	Property	Polyester rasin
1	Density (gm/cm^3)	5.606
2	Modulus of elasticity (GPa)	0.22-5.4
3	Tensile Strength (MPa)	0.32-5.3
4	Poisson's Ratio	0.358

Silicon Carbide

Silicon carbide (SiC), also known as carborundum occurs in nature as the extremely rare mineral moissanite. Synthetic silicon carbide powder has been mass-produced since 1893 for use as an abrasive. Grains of silicon carbide can be bonded together by sintering to form very hard ceramics that are widely used in applications requiring high endurance, such as car brakes, car clutches and ceramic plates in bullet-proof vests. Mechanical properties of SiC are in Table 4.

TABLE 4–MECHANICAL PROPERTIES OF SILICON CARBIDE

S.N.	Property	Silicon Carbide
1	Density (gm/cm^3)	3.21
2	Modulus of elasticity (GPa)	410
3	Poisson's Ratio	0.45

Sample Preparation: The composite fiber is prepared by hand lay-up technique. It is used to fabricate E-Glass reinforced polyester based composites filled with ZnO and SiC at different weight fractions (0, 3 & 5 wt %). The designation and composition of Polyester, Glass fabric & fillers are shown in Table 1. Then it is properly cut to appropriate size. Wooden moulds with dimensions of 140 x 70 × 5 mm^3 were prepared for composite fabrication. For different volume fraction of fibers, calculated amount of polyester resin and hardener (ratio of 10:1 by weight) was thoroughly mixed in a glass jar. For quick and easy removal of composite, mold release sheet was put over the glass plate and a mold release spray was applied at the inner surface of the mold.

After keeping the mold on a ply board, a thin layer of the mixture was applied with brush. Then the fiber lamina was distributed on the mixture. Then again resin was applied over the fiber laminate and the procedure was repeated to get the desired thickness. Care was taken to avoid formation of air bubbles. Pressure was then applied from the top and the mold was allowed to preserve at room temperature for 72 hrs. During application of pressure, some amount of mixture of polyester and hardener squeezes out. Care has been taken to consider this loss during manufacturing of composite sheets. After 72 hrs, the samples were taken out of the mold.

Figure 5. Hand lay-up procedure.

TABLE 5–DIFFERENT COMPOSITION OF COMPOSITE MATERIALS

S.N.	Designation	Composition
C1	Pure GFRP	55 wt% polyester + 45 wt% glass fiber
C2	GFRP+5%SiC	50 wt% polyester+ 45 wt % glass fiber + 5 wt % SiC
C3	GFRP+5%ZnO	50 wt% polyester+ 45 wt % glass fiber + 5 wt % ZnO
C4	GFRP+3% SiC	52 wt% polyester + 45 wt % glass fiber +3 wt % SiC
C5	GFRP+3% ZnO	52 wt% polyester + 45 wt % glass fiber +3 wt % ZnO

Cutting of laminates into samples of desired dimensions: A wire hacksaw blade was used to cut each laminate into smaller pieces, for various experiments.

- IMPACT TEST - Sample was cut into flat shape (63.5 x 12.7 x 10) mm.
- HARDNESS TEST - Sample was cut into flat shape (25 x 25 x 3.5) mm, in accordance with ASTM standards.

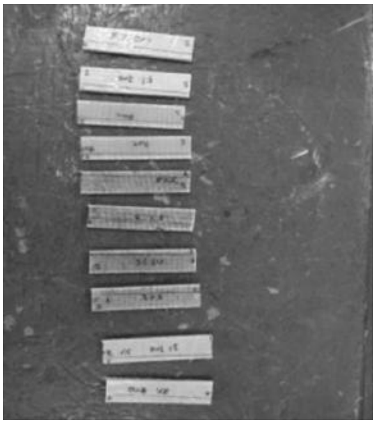

Figure 6 (*a*). Impact test sample.

Figure 6 (*b*). Hardness test sample.

Impact test: The charpy impact strength of composites was tested using a standard impact machine as per ASTM D 256 standard. The standard test specimens 63.5x12.7x10 mm, having 450 V-notch and 2 mm deep were used for the test. Each test was repeated thrice and the average values were taken for calculating the impact strength.

Figure 7(*a*). Impact test machine.

Figure 7(*b*). Hardness test machine.

Hardness test: The Vickers hardness test method consists of indenting the test material with a diamond indenter, in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100 kgf. The full load is normally applied for 10 to 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the Kgf load by the square mm area of indentation.

IV. RESULTS AND DISCUSSION

*Impact Strength:*The test samples were cut to recommended shapes and sizes according to ASTM D 256 dimensions. The arm of the Charpy impact machine was allowed to swing freely to ensure freedom of movement and that the pointers were completely freed. The results of the impact energy are shown in Table 6 and Figure 8.

TABLE 6 - IMPACT TEST DATA

Samples	Composition	Impact Strength (J)
C_1	Pure GFRP	4
C_2	GFRP+5%SiC	6
C_3	GFRP+5%ZnO	5
C_4	GFRP+3%SiC	7
C_5	GFRP+3%ZnO	6

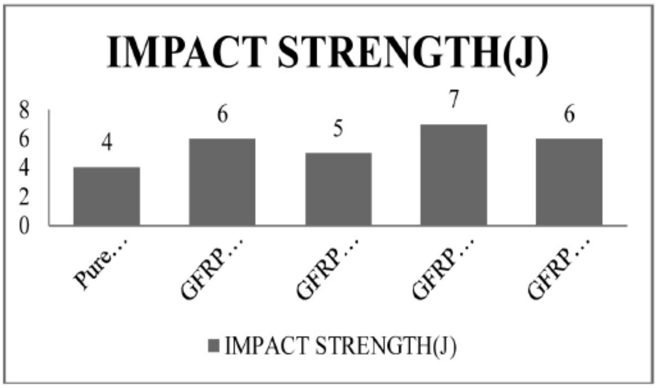

Figure 8. Impact Strength of ZnO and SiC filled composites.

It is observed that the highest value for impact strength is for GFRP with 3% SiC *i.e.*, 7 Joules. Due to the higher particle size compared to other fillers, it possessed highest energy. Compared to pure GFRP *i.e.*, the unfilled composites, the filled ones have improvement in impact energy.

*Hardness:*The Vickers hardness may be calculated from the formula when the mean diagonal of the indentation has been determined. The determined results in the same manner is plotted in Figure 9.

TABLE 7- HARDNESS TEST DATA

Samples	Composition	Hardness (HV)
C_1	Pure GFRP	28.75
C_2	GFRP+5%SiC	35.76
C_3	GFRP+5%ZnO	33.59
C4	GFRP+3%SiC	32.92
C_5	GFRP+3%ZnO	30.96

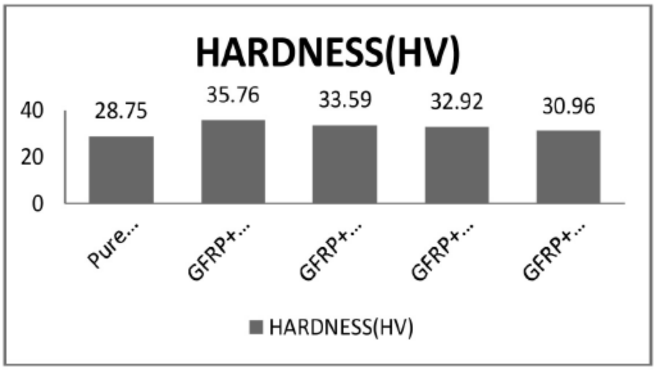

Figure 9. Hardness of ZnO and SiC filled composites.

Vickers hardness values have been depicted for the composites which has the highest value for the composite of 5% SiC with 35.76 Hv. It is observed that the hardness values of the composites of the filled ones got improved compared to the unfilled ones. It is due to the attraction of the fiber and filler material to the matrix.

V. CONCLUSION

The present work deals with the preparation of Glass fiber reinforced polyester composite with and without filler materials. Mechanical behavior of the composite leads to the following conclusions.

ZnO possessed lower values for impact and hardness tests whereas SiC got highest values for impact and hardness tests. Ultimately with further increase in the filler content, the impact strength got reduced whereas in other cases with the improvement of filler content, the test results were also been increased.

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