December 2009 Volume 5 Issue 4

Trade Science Inc.

Materials Science

An Indian Journal

Full Paper

MSAIJ, 5(4), 2009 [483-496]

Evaluation of mechanical and tribological properties of E-glass fiber reinforced vinylester composites filled with SiC, Al_2O_3 and fly ash particulates

S.R.Chauhan[1,*], Anoop Kumar[1], I.Singh[2]

[1]Department of Mechanical Engineering National Institute of Technology, Hamirpur (H.P.)-177005, (INDIA)

[2]Department of Mechanical and Industrial Engineering I.I.T Roorkee, (INDIA)

E-mail : srchauhan@nitham.ac.in

Received: 18th October, 2009 ; Accepted: 28th October, 2009

ABSTRACT

The behavior of bi-directional woven E- glass fiber reinforced vinylester composites filled with three different ceramic fillers aluminum oxide (Al_2O_3), silicon carbide (SiC) and fly ash had been investigated for mechanical and tribological characterization. The effects of these three fillers on mechanical and tribological properties of the composites investigated were compared with composite without filler. The analysis revealed that the tensile strength of composites decreased significantly by inclusion of these fillers. The flexural properties, interlaminar shear strength, compressive strength, density and hardness were also affected by the type of content of filler material. It has been observed that presence of the SiC improve the hardness of the glass vinylester composites where as other two fillers showed marginal effect. Investigation revealed that the reduction in tensile strength was lower in case of fly ash among fillers. The toughness was also improved for SiC and Fly ash with 10% addition. Tensile modulus was improved for SiC and Fly ash based glass-vinylester composite with10% and 20% addition by weight. Filler addition of SiC and fly ash make these composites potential materials in tribological applications.

KEYWORDS

Ceramic filler;
Mechanical properties;
Particulate;
Vinylester resin.

INTRODUCTION

The composite materials have largely replaced metals in various engineering applications owing to their numerous advantages, like high strength-to-weight ratio, low cost etc. There is always an increasing demand for use of these materials in defense applications like naval ships, warplanes, armor vehicles re-entry vehicles etc.[1]. The key feature of fiber composites that make them so attractive as engineering materials is the capability to tailor the materials properties through the control of fiber and matrix combinations and the selection of processing techniques. There is a wide range of composites type exist, from randomly oriented chopped fiber based materials at the low property end to continuous, unidirectional fiber composites at the high performance end. A judicious selection of matrix and the reinforcing phase can lead to a composite with a combination of strength and modulus comparable to or even better than that conventional metallic material[2]. Composite materials consist of resin and a reinforcement chosen according to the desired mechanical properties and the application. Fibers are the principal constituents in a fiber reinforced composite material. They occupy the largest volume fraction in a composite laminate and share the major portion of the load acting on a composite structure. Among the fiber reinforcements, glass, carbon and aramid fibers are widely employed. Polymer composites reinforced with these fibers are usually one to four times stronger and stiffer than their unfilled matrices. Glass fibers are widely used in light aircraft and for bodywork of buses, mobile homes and high performance sailplanes. Their main advantages are good corrosion resistance, lightweight, and better damping characteristics than metals[3]. Glass fiber is also used for the bodies of specialty and sports cars. Glass fibers are produced, also available in woven form and varying density woven glass fabrics determine the mechanical properties of fabrics[4]. The bi-directional woven fabric type of composites is gaining popularity in because of their balanced properties in the fiber plane, as well as their ease of handling during fabrication. Two dimensional (2D) fabrics have been relatively well developed in terms of production, analysis and application and some of them have long been used in structural applications. However most of the 2D composites retain the inherent weakness of laminated composites that are susceptible to delamination[5,6]. The role of polymer as matrix in a fiber-reinforced composite is to transfer stresses between the fibers, protect fibers against an adverse environment and to protect the surface of the fibers from mechanical abrasion. Among thermoset resins polyester, epoxy, phenolic, silicone and polyamide resins are widely employed. Epoxy resins are generally known as products used in structural components, adhesives and protective plating on account of their very good mechanical properties, chemical resistant and electrical characteristics. When epoxy resins are reinforced with high strengthened glass fibers, the product obtained is used in structural applications for requirement of high strength and low weight. However, the matrix materials also play an important role as is the case for thermoset resin matrix composites which can be designed for speci?c applications by properly changing the polymer used as matrix. In recent years, much research has been devoted to exploring the potential advantage of thermoset matrix for composite applications[3,5,7-9].

One such matrix is vinylester which was introduced commercially in early 1960s.Vinyl ester has found place in family, comprising the thermoset engineering polymers. Today vinylester resins have become one of the most important thermosetting materials due to its excellent mechanical properties with good chemical/corrosion resistance to wide variety of commonly encounter chemical environment[1]. Vinylester is a hybrid form of polyester resin which has been toughened with epoxy molecules within the main molecular structure. Vinylester resins are stronger than polyester resins and cheaper than epoxy resins. Vinylester resins utilize a polyester resin type of cross-linking molecules in the bonding process. Vinylester resins offer better resistance to moisture absorption than polyester resins. It is also known that vinylester resins bond very well to fiber glass. These resins are used to fabricate variety of reinforced structure including pipes, scrubber and ducts. They are the prime candidates for use in composites for transportation and/or infrastructure[1,6].

The physical and mechanical characteristics of glass fiber polymer composites further can be modified by adding a solid filler phase to the matrix during the composite preparation. This has been suggested the kind of multi-phase composite technique for improving the matrix dominated properties of continuous fiber reinforced composites. In this technique a supplementary reinforcement such as particulates, whisker, or micro fibers is added to the matrix prior to resin impregnation. It has been found that there is a significant improvement in impact energy of hybrid composites incorporating either particulates or ceramic whiskers. Hard particulate fillers consisting of ceramic or metal particles and fiber fillers made of glass are being used these days to dramatically improve the wear resistance of the composites, even up to three orders of magnitude[10,11].The improved performance of polymers and their composites in industrial and structural applications by the addition of filler materials has shown a great promise. Various kinds of polymers and polymer matrix composites reinforced with metal particles have a wide range of industrial application such as heaters, electrode and composite with thermal durability at high temperature etc. These engineering composites are preferred due to their low density, high corrosion resistance, ease of fabrication, and low cost[12-15]. Similarly, ceramic filler polymer composites have been the subject of extensive research in last few decades. The inclusion of inorganic fillers into polymers for commercial applications is primarily aimed at the cost reduction and stiffness improvement[16,17].

On account of good combination of properties fiber reinforced polymer composites (FRPCs) are used to produce many mechanical components such as gears, cams, wheels, brakes, clutches, bearings and seals. Most of these are subjected to tribological loading conditions[18]. In order to improve the friction and wear properties many researchers modified polymers using different fillers[19-27]. It was reported that the wear rate of high-density polyethylene (HDPE) was reduced with the addition of inorganic fillers, such as CuO and Pb_3O_4[19]. In the study[20] it was concluded that the wear rate of polytetrofluroethylene (PTFE) was reduced when filled with ZrO_2 and TiO_2. In the investigations it was found that the compounds of copper such as CuO and CuS were very effective in reducing the wear rate of PEEK, PTFE, Nylon and HDPE. The study of the influence of sliding velocity and load on the friction and wear behavior of G-E composite, filled with either rubber or oxide particles, and reported that the wear loss increased with increase in load/speed[21-24]. Solid lubricants such as graphite and MoS_2 when added to polymers proved to be effective in reducing the coefficient of friction and wear rate of composites[25,26]. The use of graphite as a particulate filler has been reported to improve tribological behavior in metal matrix composites (MMCs)[27]. Most of the above findings are based on either randomly oriented or unidirectional oriented fiber composites. Woven fabric reinforced composites are gaining popularity because of their balanced properties in the fabric plane as well as their ease of handling during fabrication[28]. The researchers have shown that the simultaneous existence of parallel and anti-parallel oriented carbon fibers in a woven configuration leads to a synergistic effect on the enhancement of the wear resistance of the composite[29].

The fiber-reinforced composites made with particulates fillers have been found to perform well in many actual operational conditions. However such multi phase hybrid composites form complex systems and there is inadequate information available about phenomena behind the properties changes due to the addition of particulate fillers to the fiber reinforced vinylester components. The objective of this paper is to know how the incorporation of different ceramics fillers effects on the mechanical and tribological properties of glass fiber reinforced vinylester composites. Silicon carbide (SiC), Aluminum oxide (Al_2O_3) and Fly ash are the three filler materials taken for this study and an exhaustive comparison of their influence on the composite characteristics is presented. Silicon carbide (SiC) is ceramic material that has the potential to be used as filler in various polymer matrices. It is an excellent abrasive used in grinding wheels and other abrasive products for over one hundred years. This material has been developed into a high quality technical grade ceramics with very good mechanical properties. It is used in abrasives, thermal conductivity coupled with low thermal expansion and high strength gives this material exceptional thermal expansion, high elastic modulus, high strength, high hardness, and superior chemical inertness. Fly ash is a finely divided powder generated in huge quantities during power generation in coal based plants. It is a mixture of ceramics such as SiO_2, Fe_2O_3, Al_2O_3 and TiO_2 etc. Aluminum oxide (Al_2O_3) commonly referred to as ‘alumina’ is the most effective and widely used materials in the family of engineering ceramics.

This study reported here is aimed to characterize the mechanical and sliding wear behavior of bi-directional woven E-glass fiber reinforced vinylester composite laminates under conditions such as varying ceramic filler content. Uniaxial Tension, Compression, Flexural (three point bending) and Short Beam Shear tests, Hardness and Impact tests have been carried out. Elastic properties, failure strength, toughness, failure modes and mechanisms are analyzed. Friction and sliding wear behavior of vinylester composites under dry sliding conditions is also explored.

EXPERIMENTAL

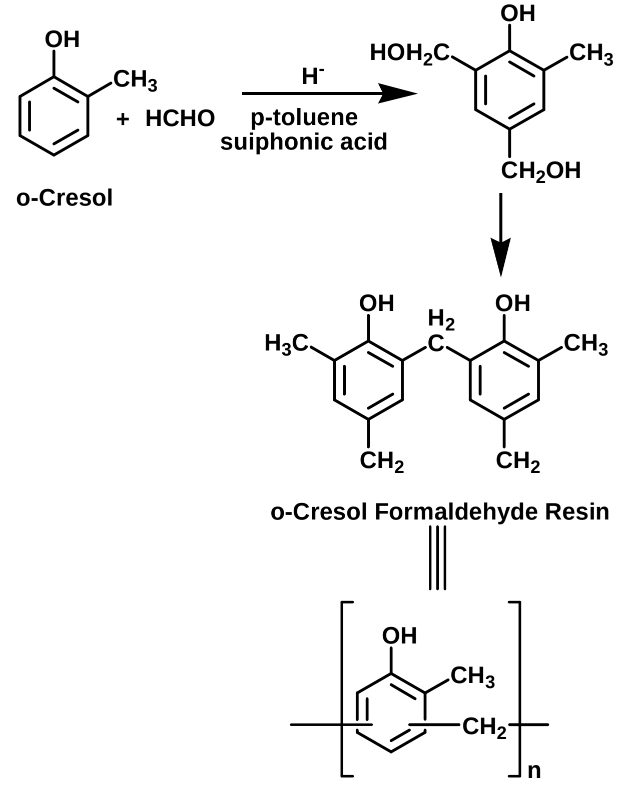
Preparation of vinylester resin

Vinylester resins are addition products of various epoxide resins and unsaturated monocarboxylic acids, most commonly methacrylic acid. It is common that vinylester oligomers is diluted with a low molecular weight co-monomer, such as styrene, vinyl-toluene, or methyl methacrylate, to reduce the room temperature viscosity of the mixture and yield a solution with a typical viscosity in the range of 200-2000cps. In many industrial products, vinylester resins are comprised of 40-50wt% styrene. The reaction to form the vinylester oligomers is usually catalyzed by tertiary amines, phosphines and alkali salts. Typical reaction conditions are 120°C for 4-5h, and hydroquinone is commonly used as the initiator. The conversion of the reaction is 90-95%[30]. o-Cresol (S.D Fine Chemicals) and formaldehyde [37-41% solution, S.D. Fine Chemicals] were used for the preparation of novolac. Resorcinol (CHD), triphenyl phosphine (CHD), imidazole (CHD), Epoxy novolac resin (EPN-1138, Ciba-Giegy), methacrylic acid (Merck), triphenylphophine (Fluka AG), butyl acrylate (Merck) and styrene (Ranbaxy), are used in the present study. Epichlorohydrin L.R. grade (CDH), sodium hydroxide pellets (Merck), used for the preparation of epoxy novolac.

Synthesis of o-cresol formaldehyde novolac

o-Cresol and formaldehyde in the molar ratio of 1:0.7 were used for the preparation of novolac resin. Weighed amount of o-Cresol (108gm) was dissolved in 6ml of water and its pH was adjusted to 1.6 with concentrated H_2SO_4 under a pH scan 3+ double junction pH meter. This solution was charged into a 550ml capacity, three-necked flask fitted with a Teflon coated blade-stirrer, a mercury thermometer and leibig condenser. The solution was heated to 82°C and maintained at this temperature with constant stirring and formaldehyde solution (54.3ml) was added in a drop wise manner over a period of 4 hours. Heating was continued for an additional half an hour after the completion of the addition of formaldehyde solution. 40ml of 10% sodium bicarbonate was added to neutralize the H_2SO_4 and arrest the reaction. The reaction mixture from the flask was then transferred to the separating funnel and washed with warm water to neutralize the pH removing excess sodium bicarbonate salt and 3-4 washings were generally adequate for the purpose. The resin was finally dried at 82°C under reduced pressure (35±5mm Hg).

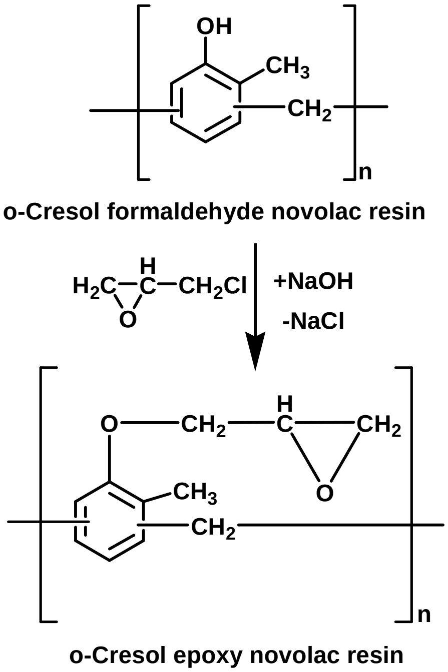
Preparation of o-cresol formaldehyde novolac resin



Synthesis of epoxy novolac from o-Cresol formaldehyde novolac resin

Epoxy novolac resin was prepared by the reaction of o-Cresol formaldehyde novolac resin with epichlorohydrin. Sodium hydroxide in the mole ration with epichlorohydrin of 0.2:1.0 was used as catalyst. The apparatus used for the preparation of epoxy novolac consisted of an oil bath, a 3-necked flask fitted with a glass stirrer having Teflon coated blade in the central neck, Dean and Stark assembly in one side neck and in another side neck a thermometer pocket and a dropping funnel to deliver sodium hydroxide at a regular rate by the side of thermometer pocket. Novolac resin was with epichlorohydrin was charged into the flask and heated under stirring to temperature of 112°C±1°C. While maintaining this temperature for epoxidation to proceed, 16.3934g of sodium hydroxide (40% w/w) were added gradually to the reactants in the flask over a period of three and half hours. Heating was continued for an additional 15 minutes, after which the contents were dissolved in toluene and the solution filtered using Whatman filter paper no. 42 to remove the salts. Toluene was then removed by heating under reduced pressure.

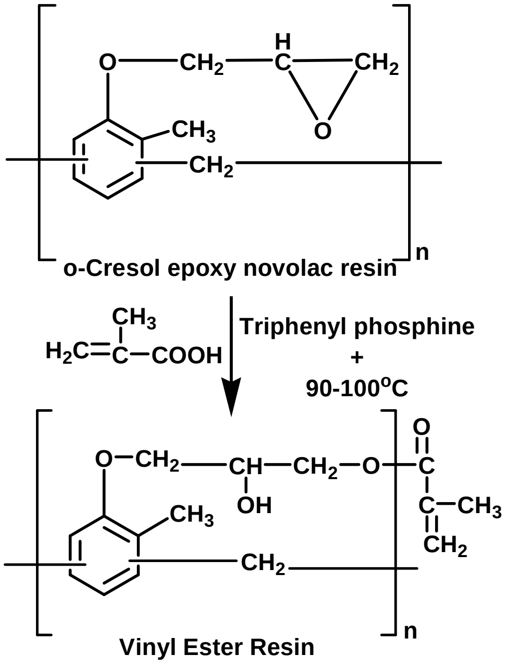
Preparation of o-cresol epoxy novolac resin



Synthesis of vinylester resin form o-cresol novolac epoxy resin

Vinylester resin was prepared using 1:0.9 mole ratios of o-Cresol epoxy novolac resin prepared and methacrylic acid in the presence of imidazole (1 phr by weight of the epoxy resin) and hydroquinone (200ppm) at 86° C±1°C.

Preparation of vinyl ester resin based on o-cresol novolac resin

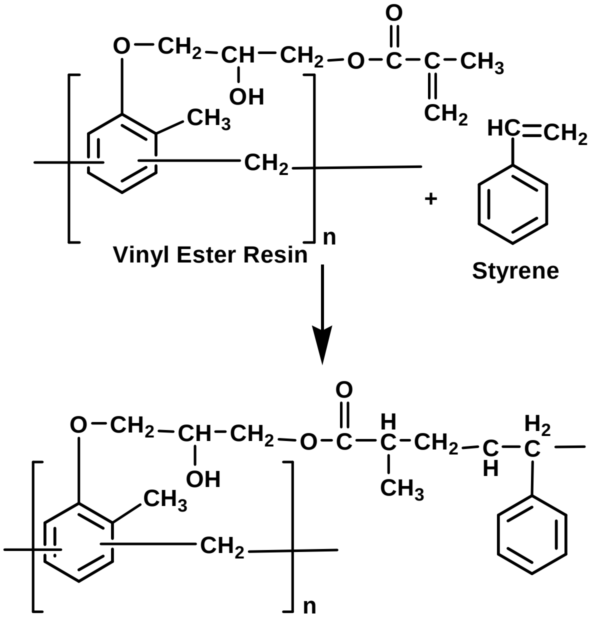


The etherification reaction was carried out for a three hours to obtain a product with an acid value of 20 mg KOH/gms solids determined. The VER samples were stored in a refrigerator at 10°C.

Cross linking reaction with styrene as comonomer

Vinylester resins are cross linked by free radical copolymerization of methacrylate end groups with styrene. It is a system of copolymerization of vinyl/divinyl monomers. Gelation occurs when a three-dimensional network or an infinite molecular weight polymer (gel) is formed. Increasing the concentration of the initiator and/or accelerators and the use of elevated temperatures will shorten the time to onset of the gel. In this work Styrene as monomers. The ratio of resin to co monomer (reactive diluent) is kept 10:4 by weight.

Cross linking of vinylester with styrene as comonomer



COMPOSITE FABRICATION

The resin used in this work is vinylester prepared from o-cresol formaldehyde novolac resin (density 1.42gm/cc) and reinforcing phase E-glass fibers (modulus 72.4GPa, density 2.54g/cm^3) were supplied by Northern Polymer Pvt. Ltd. New Delhi. Methyl ethyl ketone peroxide (MEKP-1%), Cobalt Naphthenate (1.5%) was used as catalyst and accelerator respectively. Seven different types of composites were prepared for this study. Three different ceramic powders Silicon Carbide (SiC), Alumina (Al_2O_3) and Fly ash were used as filler materials (10% and 20% by weight) in this study. Raw fly ash (size 80-100μm) was collected from the power plant of NALCO, located in India. Aluminum oxide and Silicon carbide powders of similar size range were obtained from NICE Ltd. India. The chemical compositions/formulae properties such as density and hardness of these particulate fillers are given in TABLE 1.

TABLE 1 : Chemical composition and physical properties of filler materials

Filler	Composition/Chemical Formulae	Mean hardness (Hv)	Density(gm/cc)
Silicon Carbide	SiC	2800	3.22
Alumina	Al_2O_3	1175	3.89
Fly Ash	SiO_2 (48.3%), Al_2O_3 (20.25%), Fe_2O_3, TiO_2(1.9%)	725	3.15

Conventional hand layup technique was used for making of the composite laminates. The cobalt Naphthenate 1.5% is mixed thoroughly in vinylester resin and then 1% methyl ethyl ketone-peroxide (MEKP) was mixed in the resins prior to reinforcement. The fiber loading (weight fraction of glass fiber in the composite) is kept 50 wt% for all the samples. The stacking procedure consists of placing the fabric one above the other with the resin mix well spread between the fabrics on a mould release sheet. A porous Te?on ?lm was again used to complete the stack. To ensure uniform thickness of the sample, a 3mm spacer was used. The mould plates were coated with release agent in order to aid the ease of separation on curing. This procedure was repeated in all cases unless thickness of 3mm was obtained. A metal roller was used so that uniform thickness and compactness could obtain. The whole assembly is placed in the compression molding machine at a pressure of 60Kgf/cm^{2}and allowed to cure at room temperature for 24hrs. The laminate sheets of sizes 300X300X3mm were prepared. Specimens of suitable dimensions were cut using a diamond cutter for physical characterization, mechanical and wear testing as per ASTM standard.

The other composite sample with particulate fillers of fixed weights (10% and 20%) percentage is fabricated by the same technique. The fillers are mixed thoroughly in the vinylester resin mechanically before the glass fiber mats are reinforced in the matrix body. Composites GV_2 and GV_3 contain SiC particles in 10wt% and 20 wt% proportions respectively. Similarly GV_4, GV_5, GV_6 and GV_7 are the composites containing alumina and fly ash fillers respectively along with 50% of glass fiber in them. The composites prepared for this study are designated as GV_1, GV_2, GV_3, GV_4, GV_5, GV_6 and GV_7 respectively. The detailed compositions along with the designation are presented in TABLE 2.

TABLE 2 : Designation, detail composition and physical properties of filler based composite materials

Designation	Composite composition	Fiber Volume fraction(%)	Fiber void fraction(%)
GV_1	Vinylester+50wt% glass fiber	0.547	4.6
GV_2	Vinylester+50wt% glass fiber+10wt% SiC	0.540	5.87
GV_3	Vinylester+50wt% glass fiber+20wt% SiC	0.655	3.19
GV_4	Vinylester+50wt% glass fiber+10wt% Alumina	0.539	9.38
GV_5	Vinylester+50wt% glass fiber+20wt% Alumina	0.523	13.30
GV_6	Vinylester+50wt% glass fiber+10wt% fly ash	0.572	6.85
GV_7	Vinylester+50wt% glass fiber+20wt% fly ash	0.512	9.09

MECHANICAL MEASUREMENTS AND WEAR TESTING

Density

The composites under investigations has three components namely matrix, fiber and fillers. Therefore density of composite can be obtained from the rule of mixture as shown in the following expression:

$$\rho_{ct} = \frac{1}{(w_m/\rho_m)+(w_f/\rho_f)+(w_p+\rho_p)} \qquad (1)$$

Where w and ρ represent the weight fraction and density respectively. The suffix p, m, f, ct stand for particulate, matrix, fiber and composite. The actual density (ρ_{ce}) of the composites however can be determined experimentally by simple water immersion technique. The volume fraction of voids (V_v) in the composites is calculated using the following equations:

$$Vv = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \times 100 \qquad (2)$$

Volume fraction is an important parameter that gives a fair amount of idea about the degree of reinforcement that has been done by the addition of fibers.

Tensile strength and compression strength

The Uniaxial tensile tests were carried out in the laboratory at temperature 27°C and 60% of Humidity. The tests were conducted using a computer controlled Hounse field-25K (25KN capacity) Universal testing machine. The tension tests were carried out on the tension test specimens at a speed of 2mm/min. The mechanical grips were used for holding the specimen without end tabs. The tests were carried as per ASTM D3039-76. Instantaneous load and displacement were recorded at the rate of one set per second. The tests were carried out at least three times for each specimen and the results were averaged arithmetically. Ultimate Tensile strength, modulus of elasticity, percentage elongation was determined by the tension tests used most frequently when determining the mechanical properties of polymer composites.

Similarly, the compression test was carried out to determine the compressive strength using plain faced mechanical grips without tabs. Three specimens were tested for each sample. The displacement during the test was monitored using a load cell transducer and a load-displacement curve was obtained for each specimen. From the test record, the compressive modulus and the compressive strength corresponding to the maximum load at failure could be determined. The compression tests were carried on the Hounse field-25K (25KN) Universal testing machine as per the ASTM D3410M-95.

Flexural and inter-iaminar shear strength

The flexural and short beam shear tests (SBS) were also conducted on the same machine as per ASTM D790 and ASTM D2344 at room temperature. The flexural test using a three-point bending fixture having radius of the loading rollers 5 mm and the test speed 2.54mm/min was carried. The span length used was kept at 50 mm and therefore the span length to thickness ratio was about 16:1. The flexural strength of any composite specimen is determined using the equation:

$$F.S.=3PL/2bt^2 \qquad (3)$$

Where P -is maximum load, b- is width of specimen, t- is thickness of the specimen and L- is span length.

For Short beam shear test the radius of the loading edges was about 2.5 mm and the test speed was 2.54mm/min. The span length was set at 20 mm, corresponding to a span length/thickness ratio of about 6: 1.Instantaneous load P and crosshead displacement was measured by a load cell were recorded by a computerized data acquisition system at one second intervals. The ILSS values are calculated as:

$$ILSS=3P/4bt \qquad (4)$$

Where P is maximum load, b- is width of specimen and t-is thickness of the specimen

Micro hardness

Micro Hardness value of the composite material ws determined using Vicker's hardness test according to ASTM D785-89. Micro-hardness measurement is done using a Leitz micro-hardness tester. A diamond indenter, in the form of a right pyramid with a square base and an angle 136° between opposite faces, is forced into the material under a load F. The two diagonals X and Y of the indentation left on the surface of the material after removal of the load are measured and their arithmetic mean L is calculated. In the present study, the load considered F=24.54N and Vickers hardness number is calculated using the following equation.

$$Hv = 0.1889\ F/L^2 \qquad (5)$$

$$L = (X+Y)/2$$

Where F is the applied load (N), L is the diagonal of square impression (mm), X is the horizontal length (mm), and Y is the vertical length (mm).

Impact strength

Impact tests are carried out on composite specimens. The tests are done as per ASTM D256 using an impact tester. The pendulum impact testing machine ascertains the notch impact strength of the material by shattering the V-notched specimen with a pendulum hammer, measuring the spent energy and relating to the cross section of the specimen. The standard specimen for ASTM D256 is 50×10×2 mm and depth under the notch is 2mm.The machine is adjusted such that the blade on the free-hanging pendulum just barely contacts the specimen (zero position). Since there are practically no losses due to bearing friction, etc. (<0.3%), the testing conditions may be regarded as ideal. The specimens are clamped in a square support and are struck at their central point by a hemispherical bolt of diameter 5 mm. Raising the pendulum gives it the requisite energy of deformation (potential energy-E_p) as the product of reaction force (F) times the relative height (h).

$e_p = F.h = F.L(1-\cos\alpha)[Nm]$

The reaction force (F) is measured with the pendulum in the precisely horizontal position at a distance L from the fixed point. The height of fall, h:

$h = L(1-\cos\alpha)[m]$

The length L is the distance between the pendulum axis and the point of impact of the hammer's blades at the center of the specimen. Upon release of the pendulum, the potential energy is converted into kinetic energy by acceleration due to gravity. At the moment it passes the zero position, the Pendulum constitutes the potential energy (e_p) engraved on the hammer. As it shatters a specimen, the pendulum is braked by the impact, losing part of its velocity and, hence, part of its potential energy (E_p), namely the impact-energy component (e_1).

$e_1 = F.h_1 = F.L(1-\cos\alpha)[Joules]$

The amount of impact energy (e_1) thusly consumed is the difference between the potential energy (e_p) and the surplus energy (e_s).

$e_1 = e_p - e_s [Joules]$ (6)

The respective values of impact energy of different specimens are recorded directly by the data acquisition system attached to the machine.

Friction and wear measurements

To evaluate the friction and sliding wear performance of glass fiber reinforced composites prepared with different ceramic filler under dry sliding condition, wear tests were carried out in a pin-on-disc type friction and wear monitoring test rig (DUCOM) as per ASTM G 99. The counter body is a disc made of hardened ground steel (EN-32, hardness 72 HRC, surface roughness 0.6 μ Ra). The specimen was held stationary and the disc was rotated while a normal force was applied through a lever mechanism. During the test, friction force was measured by transducer mounted on the loading arm. The friction force readings were taken as the average of 100readings every 40seconds for the required period. For this purpose a microprocessor controlled data acquisition system was used. The average mass loss was used to calculate the specific wear rate. The tests are conducted with sliding velocity of 2m/s under constant normal loading of 30N. Sliding wear data reported here is the average of at least three runs. The material loss from the composite surface is measured using a precision electronic balance with an accuracy of ±0.001mg.

The specific wear rate ($mm^3N^{-1}mm^{-1}$) is then expressed on 'volume loss' basis

$K_S = \Delta m/L \rho F_N$

Where K_S is the specific wear rate ($mm^3N^{-1}mm^{-1}$), Δm-is the mass loss in the test duration (gm), ρ-is the density of the composite (gm/mm^3), t -is the test duration (sec),V_s -is the sliding velocity (m/sec), F_N- is the average normal load (N), L-is sliding distance

Scanning electron microscopy

The surfaces of the specimen were examined directly by scanning electron microscope JEOL JSM-6480LV.The composite samples were mounted on stubs with silver paste. To enhance the conductivity of the samples, a thin film of platinum was vacuum evaporated onto them before the photomicrographs were taken.

RESULTS AND DISCUSSION

The investigation of mechanical characterization and failure analysis of composites reveal that inclusion of the particulate filler has significant influence on the physical and mechanical properties of composites. The modified properties with inclusion of fillers under this investigation are presented and compared against the unfilled glass vinylester composites in TABLE 3.

Density

The values for theoretical and measured densities are presented in the TABLE 2.The composite density values calculated theoretically from weight fractions are not in agreement with the experimentally obtained values. The difference in these densities is the measure of voids or pores present in the composites. It can be seen from the TABLE 2 that in the sample GV_1 volume fraction of voids is lowest due to absence of particulate filler. However it is observed from these results that with the addition of particulate filler in matrix more voids and pores are found in the composites. As the filler content is increased from 10% to 20% the volume fraction of voids increased proportionately. This trend is observed in all the particulate filled composites from GV_2 to GV_7 but percentage volume of voids varies from composite to composite. This may be due to the reaction of the particulate with the vinylester resin and/or due to the processing factors while making laminates.

Density is one of the important physical property of polymer composite and it depends on the relative proportion of matrix and reinforcing materials and this is one of the most important factors determining the properties of the composites. The void fraction is the cause for the difference between the values of the actual density obtained by simple water immersion technique and the theoretically calculated value. The voids significantly affect the mechanical properties and even the performance of composites in the place of use. Higher the void content lower is fatigue resistance, greater susceptibility to water penetration and weathering. The knowledge of void content is desirable for correct estimation of the quality of the composite. It is understandable that a good composite should have fewer voids. However, presence of voids is unavoidable in composite making particularly through wet hand layup technique but proper and careful process can reduce the void content.

Micro hardness

The measured hardness values (Hv) of all the seven composites are presented in the TABLE 3. It is observed that the hardness is affected marginally by the addition of alumina particles in composites GV_4 and GV_5. It is further observed that fly ash addition of 10% also shows a minor change in the hardness. The composite GV_7 with 20wt% fly ash content found to exhibit some improvement in hardness compared to the unfilled composite GV_1. The hardness of fly ash as well as alumina particles are generally high like any other oxide ceramic however the low or marginal effect of these particulates fillers on composite hardness may be due to the presence of pores or voids. However, as expected the hardness values of SiC-GF-vinylester composites (GV_2 and GV_3) are found to be higher than that of composite (GV_1).

Here the hardness is seen to have improved with increase in SiC content Figure1.and so with the inclusion of SiC in the composite high hardness values commensurate with the theory.

Tensile strength

The test results for uniaxial tensile strength and moduli are presented in the TABLE 3. It can be seen irrespective of the filler material the tensile strength of all the samples of the composites decreases with increase in filler content. The unfilled glass vinylester composite has a strength of 282.38MPa in tension and this value drops to 269.75MPa and 261.4MPa for GV_6 and GV_2, with 10wt% addition fly ash and SiC respectively. For other composite sample Al_2O_3 the 20% addition the tensile strength reduces to 198.30MPa.

It is observed that when filler content is increased from 10% to 20% the tensile strength is reduced for all samples. Among the three fillers taken in this study, the

TABLE 3: Mechanical properties of the glass vinylester composites

Composites	Micro Hardness (Hv)	Tensile strength (MPa)	Tensile Modulus (GPa)	Compressive strength (MPa)	Flexural strength (MPa)	ILSS (MPa)	Impact Energy (J)
GV_1	35	282.38	11.72	29.85	288.68	174.52	1.1290
GV_2	46	261.25	11.76	10.73	301.20	151.53	1.2060
GV_3	48	202.38	12.92	16.32	245.20	154.4	1.069
GV_4	37	220.49	8.280	12.25	316.15	125.9	1.135
GV_5	38	198.30	9.209	32.09	268.23	159.4	0.979
GV_6	39	269.75	10.53	20.69	398.15	100.56	1.502
GV_7	41	228.33	12.862	33.10	256.13	120.48	0.924

inclusion of alumina causes maximum reduction in the composite strength. There can be two reasons for this decline in the strength properties of these particulate filled composites, compared to the unfilled ones. One possibility is that the chemical reaction at the interface between the filler and the matrix may be too weak to transfer the tensile stress; the other is that the corner points of the irregular shaped particulates result in stress concentration in the vinylester matrix[2]. The alumina particles seem to be less compatible to vinylester resin than that of fly ash and SiC. As a result of which the percentage reduction in tensile strength is highest in the former case. The tensile modulus of alumina filled composites GV_4 and $GV_5$are also found to be less than of the unfilled ones. On the contrary the experimental findings Figure 2 suggest that with addition of fly ash and SiC the tensile moduli of the glass vinylester composites is improved reasonably compared to Al_2O_3 and unfilled composites. This improvement is attributed to the relatively lower strain rates of composites GV_2, GV_3, GV_6 and GV_7during the tensile test. The reduction in tensile strength of these composites may be due the reasons of poor bond strength at interface of the matrix and fiber or matrix and particulate. It may be due to the chemical reaction between resin and filler or lack of adhesiveness between the filler particulate and glass fiber. The reduction in tensile strength with the incorporation of filler can be explained that under the action of a tensile force the filler matrix interface is vulnerable to de bonding depending on interfacial bond strength and this may lead to a break in the composite. Similar property modification has been previously reported for Al_2O_3 particles reinforcement in polyurethane matrix[2,4].

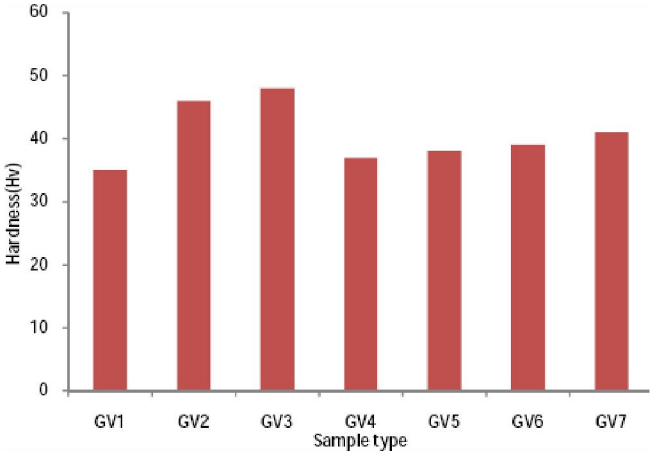


Figure 1: Comparison of Hardness of composites

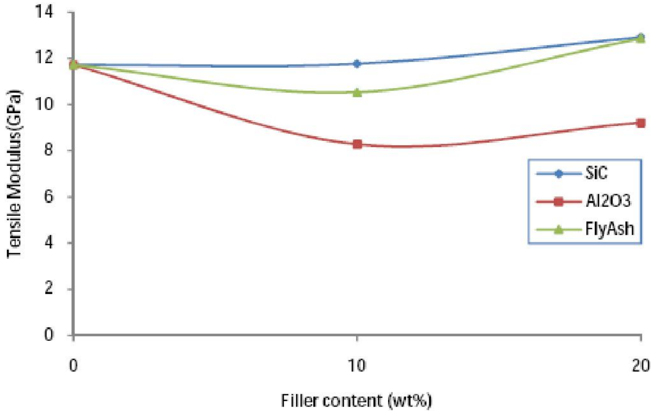


Figure 2 : Variation of tensile modulus of composites with filler type and content

Compressive strength

The compressive strengths for the samples GV_1, GV_2, GV_3, GV_4, GV_5, GV_6 and GV_7 are shown in the TABLE 3. The compressive strengths values for all samples are much lower than their respective tensile strength and flexural strength values. This is due to the highly anisotropic nature of the glass fiber (poor in transverse direction). The compressive strengths are lower than their tensile and flexural values, also due to fiber crimps and fiber anisotropy. From the TABLE 3 it is observed that Fly ash and Al_2O_3 particulates show improvement in the compressive strengths. The maximum compressive strength is obtained for GV_7 (33.10MPa) followed by that of GV_5 (32.09MPa). It is also observed that compressive strength improves by increasing the filler contents in vinylester composites from 10% to 20%. This observation shows that in compression, matrix also bears some of the load.

Flexural strength response

Figure 3 shows the comparison of flexural strengths of the composites obtained experimentally form the three point bend tests. It is interesting to note that addition of small amount (10wt %) fly ash as well as alumina improves the flexural strength of glass vinylester composites structure. But addition of (20wt %) these fillers lower the flexural strength values.

This trend is found in the entire composites. The flexural properties have great importance for any structural element. Composite materials used in structures are prone to fail in bending and therefore the development of new composites with improved flexural characteristics is essential. From the results it may be sug-

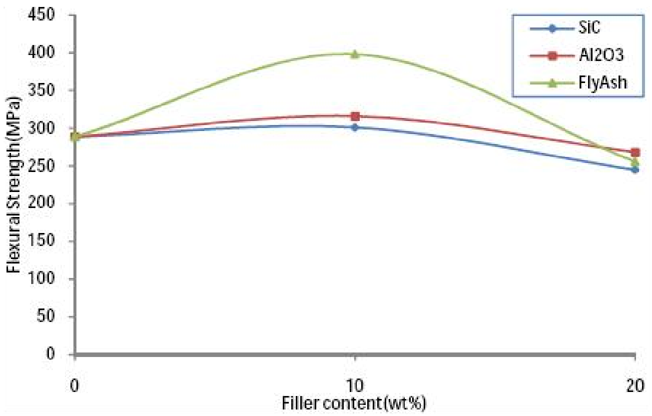


Figure 3 : Variation of flexural strength of composites with filler type and content

gested that alumina and fly ash (10%) are potential candidates to be used as fillers in making high flexural strength composites.

Inter laminar shear strength (ILSS)

When a short beam is subjected to three point bending, the maximum shear stress (Interlaminar shear stress) occurs in the beam mid plane (neutral plane) where normal stresses are zero. This results in combination of failure modes, such as fiber rupture, micro buckling and interlaminar shear cracking. The maximum bending stresses (compression and tensile) occur at the beam upper and lower surfaces respectively. The ratio (maximum shear stress/maximum bending stresses), increases as the beam span length to thickness ratio decreases, and thus the beam is more likely to fail in shear. An isotropic material in bending will fail in shear if (maximum shear stress/maximum bending stress), exceeds 0.58 according to the VonMises criterion. Anisotropic materials may fail in shear at a lower ratio[6]. The interlaminar shear strength of the particulate filled composites are shown along with that of the unfilled glass vinylester composite GV_1 in TABLE 3. It is seen that there is reduction of ILSS of the composites with particulate filling addition when compared with unfilled composite GV_1.Incorporation of silicon carbide is seen to have caused improve the strength, compared to the composite GV_1. It has been noticed that with increase in filler content from 10%wt. to 20%wt. there is small increase in the interlaminar shear strength.

Impact strength response

The impact energy values of different composites recorded during the impact tests are given in TABLE 3. It is observed that the resistance to impact loading of glass vinylester composites improves with 10% addition of particulate filler. However this improvement in GV_2 and GV_6 is very significant. It is seen that with incorporation of filler particles, the impact strength of glass fiber vinylester composite GV_2 and GV_6 increases by about 10-35%. However increasing filler contents from 10% to 20% the impact strength decreases for all samples. High strain rates or impact loads may be expected in many engineering applications of composite materials. The suitability of a composite for such application should therefore be determined not only by usual design parameters, but by its impact or energy absorbing properties. Thus it is important to have a good understanding of this impact behavior of composites for both safe and efficient design of structural and to develop new composites having good impact properties. The result of impact tests in the present study reveals that fly ash (10% addition) can be a promising filler material that would enhance the impact characteristics of polymer composites and at same time it is cost effective.

Friction and wear behavior

The variation of friction of seven composites with sliding distance is shown in the Figure4.The sliding velocity is kept constant 2m/s and normal load of 30N throughout the test duration.

It is observed that for all the composites coefficient of friction increases continuously. However at higher sliding distance rate of change becomes almost constant. It is also observed that composite specimen without filler (GV_1) show higher friction than the ceramic

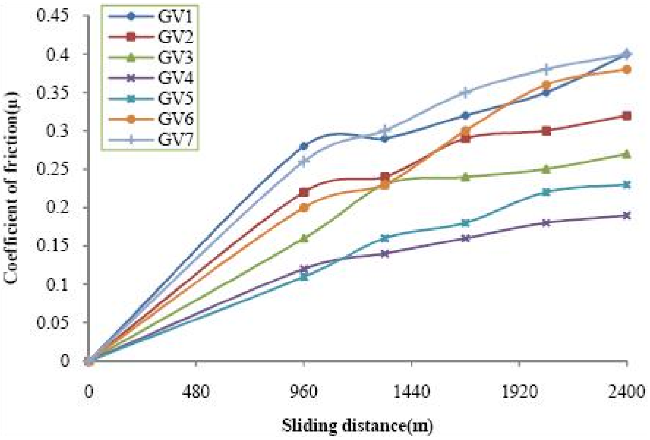


Figure 4 : Variation in coefficient of friction of composites with sliding distance

filled glass vinylester composites. Among the various ceramic filled composites SiC and fly ash filled composites show higher values of friction coefficients. It is observed that among all composites Alumina filled composites show the lowest values for friction coefficients. Figure 5 represents the variation of specific wear rate of the composites with sliding distance. The sliding velocity and normal applied loads are 2m/s and 30N respectively during the experimentation. It is observed that specific wear rate increases initially with time and sliding distance and after certain duration there is decrease in the specific wear rate. Further increasing the sliding distance, almost steady values for specific wear rate are obtained. It is also observed that Alumina filled glass vinylester composites show maximum specific wear rate. Whereas SiC and Fly ash filled composites show lowest specific wear rate and improved wear resistance. It is worth noting that wear rate initially increases when there is contact between the composite and counter surface, the composite is sheared and ploughed.

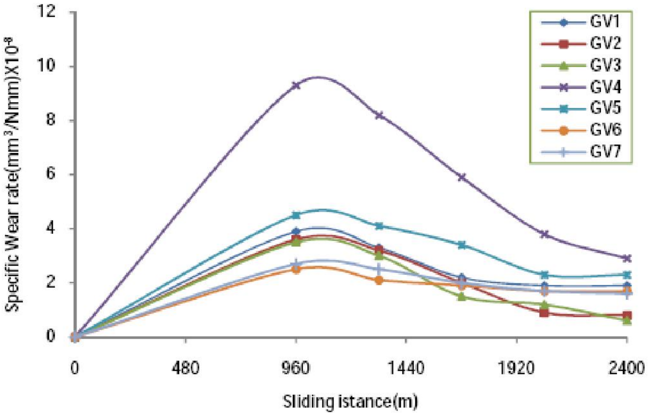


Figure 5 : Variation in Specific wear rate of composites with sliding distance

This leads to mass loss at relatively at faster rate[1,19]. With increase in test duration and sliding distance there seems formation of film which reduces the wear rate and hence material loss becomes steady[20] .

Further the Figure 6 (a)-(d) are the SEM micrographs for the composites at sliding distance of 2400m.The Figure6 (a) shows the wear mechanisms in unfilled specimen (GV_1). It is observed that there are patches of debris formation and matrix is broken and exposes the glass fiber. The debris which consists of fragments of glass fiber and matrix is observed. The Figure 6(b) and 6(c) represent the SEM of SiC filled

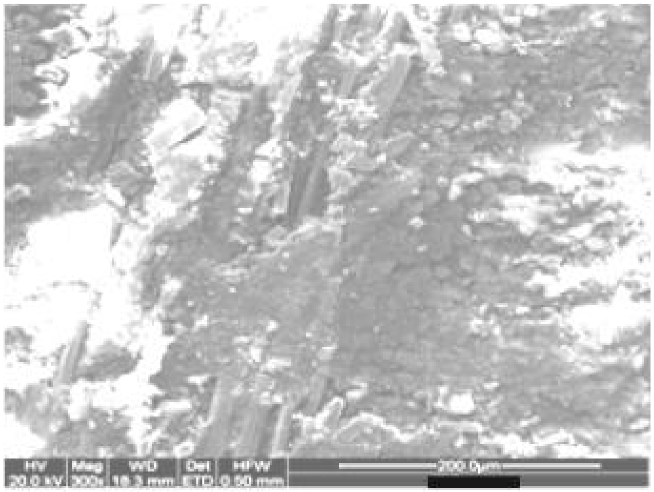


Figure 6(a) : SEM of Composite (GV_1) at load 30N & speed 2m/s

Figure 6(b) : SEM of(GV_2) at load 30N&speed 2m/s

Figure 6(c) : SEM of (GV_3) at load 30N&speed 2m/s

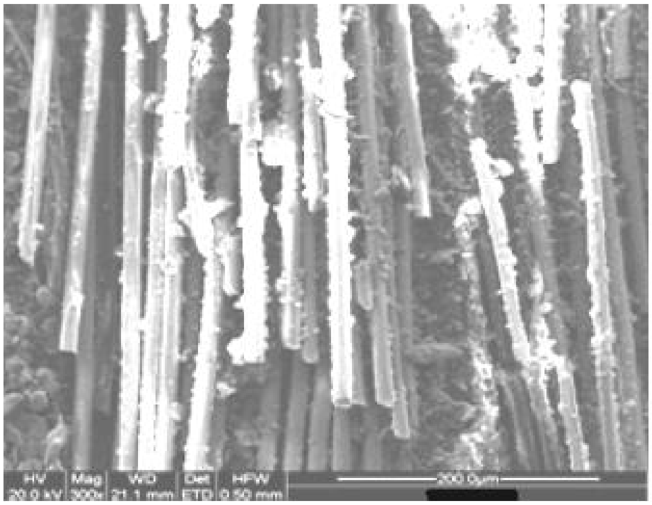


Figure 6(d) : SEM of(GV_4)at load 30N&speed 2m/s

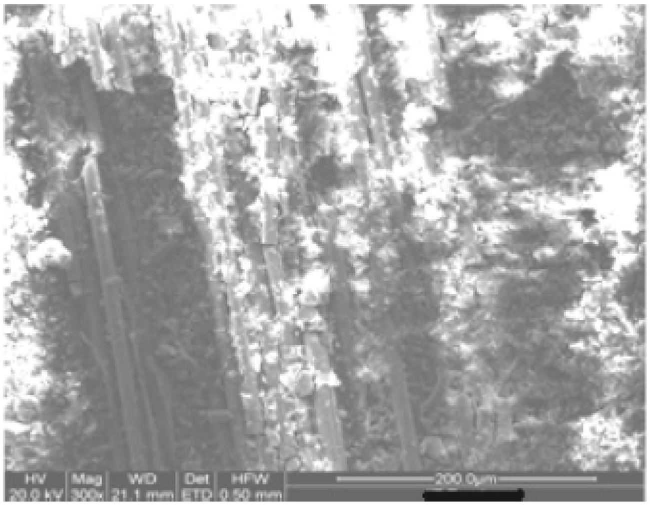


Figure 6(e) : SEM of(GV_5) at load 30N&speed2m/s

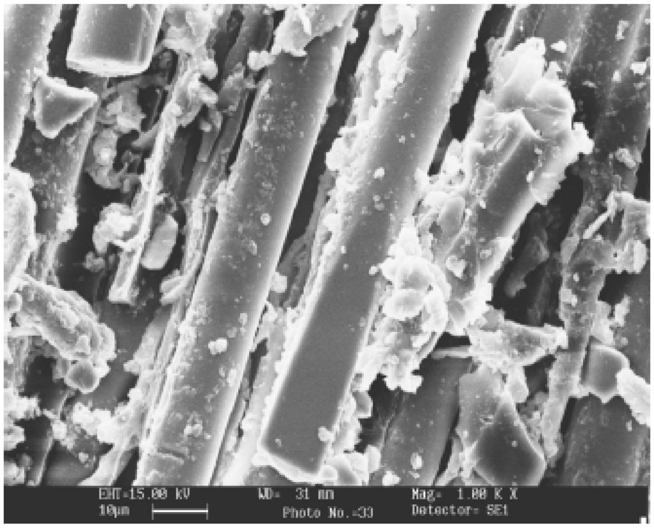


Figure 6(f) : SEM of (GV_6) at load 30N&speed2m/s

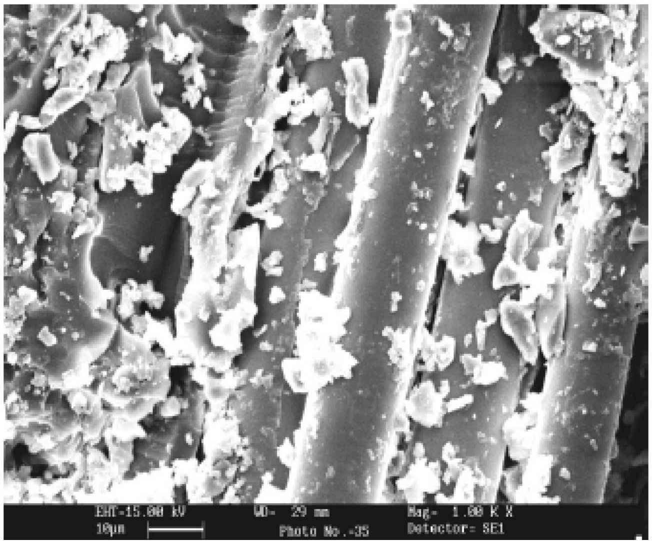


Figure 6(g) : SEM of(GV_7) at load 30N& speed2m/s

Figure 6 : The SEMs of composites at sliding distance of 2400m and load 30N, Sliding speed 2m/s

composites. It is observed that GV_3 show less wear than GV_2. In Figure 6(b) there is more breakage and exposure of fibres. The patches of debris are also observed which are absent in Figure 6(c).The matrix which protects the fibers is seen in Figure (c). These observations corroborate with the experimental results one to one. Similarly the Figure 6(e) and Figure 6(f) show the SEM of GV_4 and GV_5 composites.

The Figure 6(e) shows more fibre exposure and there is no debris formation .However in Figure 6(f) it is observed that some of the part is covered by matrix and small amount of fibres are exposed. The observations matches to the experimental results in Figure 5 in which GV_4 show the maximum wear followed by GV_5.The Figure 6(f) and Figure 6(g) are the SEM of GV_6 and GV_7. It is observed that fiber exposure is almost identical. Small part of uniform distribution of matrix is seen. The observations corroborate the experimental observations. Fibre exposure is lesser that Alumina filled composites but higher than SiC filled composites. This verifies the wear trend followed in experimental results in Figure 5.

CONCLUSIONS

This work shows the successful fabrication of glass reinforced vinylester composites with ceramics fillers such as SiC, Fly ash Al_2O_3 using wet hand layup technique. An industrial waste like fly ash can also be gainfully utilized for the composite making purpose which can reduce the cost significantly.

Incorporation of ceramic fillers such as SiC, Fly ash Al_2O_3 modify the tensile, compressive, flexural, impact and inter laminar shear strengths of the glass vinylester composites. A steady decline in the tensile strength is noticed in the ceramic filled composites whereas the presence of these particulate matters has caused improvement in impact and interlaminar shear strength of the composites. The hardness, density and flexural properties of the composites are also greatly influenced by the type and content of fillers. Compressive strength using SiC and Fly ash filler in glass vinylester composite system could be improved as a prospective property.

Alumina and Fly ash based glass vinylester composites can be the potential material for structural applications. The SiC and fly ash filled glass fiber vinylester composites are potential materials for Tribological applications.

While fabricating a composite of specific requirements, there is a need for the choice of appropriate filler material and for optimizing its content in the composite system.

REFERENCES

[1] S.Chauhan, A.Kumar, A.Patnaik, A.Satapathy, I.Singh; Journal Reinforced Plastics & Composites. **(2008)**.

[2] Amar Patnaik, Alok Satapathy, S.S.Mahapatra, R.R.Dash; Reinforced Plastics and Composites, **28(11)**, 1305-1318 **(2009)**.

[3] H.Pýhtýlý, N.Tosun; Composite Science and Technology, **62(3)**, 367-370 **(2002)**.

[4] M.M.Schwartz; Composite Materials Handbook, McGraw-Hill, New York, USA, **(1984)**.

[5] B.Suresha, G.Chandramohan, Siddaramaiah, P.Sampathkumaran, S.Seetharamu; Material Science & Engineering (A), **443**, 285-291 **(2007)**.

[6] Youjiang Wang, Jian Li, Dogming Zhao; Composite Engineering, **5(9)**, 1159-1175 **(1995)**.

[7] P.Kishore et al.; Reinforced Plastics and Composites, **18(1)**, 55-62 **(1999)**.

[8] P.Kishore,et al.; Wear, **237**, 20- 27 **(2000)**.

[9] N.S.El-Tayep, R.M.Gadelrap; Journal of Wear, **192**, 112-117 **(1996)**.

[10] J.L.Acosta, E.Morales, M.C.Ojeda, A.Linares; Angew Makromol Chem, **138**, 103-110 **(1986)**.

[11] W.Sawyer Gregory, D.Freudenberg Kevin, P.Bhimaraj, Schadler, S.Linda; Wear, **254**, 573-580 **(2003)**.

[12] Jung-il Kim, Phil Hyun Kang, Young Chang Nho; Appl.Poly.Sci., **69**, 2593-2598 **(2004)**.

[13] K.Zhu, S.Schmauder; Computational material Sci., **28**, 743-748 **(2003)**.

[14] M.Rusu, N.Sofian et al.; Polymer Testing, **20**, 409-417 **(2001)**.

[15] H.Serkan Tekce, Dilek Kumlutas; Reinforced Plastics and Composites, **26(1)**, 113-121 **(2007)**.

[16] R.N.Rothon; Mineral Fillers in Thermoplastics: Filler Manufacture, J.Adhesion, **64**, 87-109 **(1997)**.

[17] R.N.Rothon; Advance Polymer Science, **139**, 67 **(1995)**.

[18] B.Suresha, G.Chandramohan, J.N.Prakash, V.Balusamy,. K.Sankaranarayanasamy; Material and Material Characterization and Engineering, **5(1)**, 87-101 **(2006)**.

[19] B.J.Briscoe, A.K.Pogosion, D.Tabor; Wear, **27**, 19-34 **(1974)**.

[20] K.Tanaka; 'Effect of various fillers on the friction and wear of PTFE-based composites', In: Friction and Wear of Polymer composites, (K.Friedrich editor), Elsevier, Amsterdam, **205**, 137-174 **(1986)**.

[21] S.Bahadur, Q.Fu, D.Gong; Wear, **178**, 123-130 **(1994)**.

[22] Y.Yamaguchi; 'Tribology of Plastic Materials'; Tribology Series Elsevier, Newyork, **16**, **(1990)**.

[23] S.Bahadur, D.Gong, J.W.Anderegg; Wear, **154**, 207-223 **(1992)**.

[24] S.Bahadur; Wear, **245**, 92 **(2000)**.

[25] Kishore, P.Sampathkumaran, S.Seetharamu, P.Thomas, M.A.Janardhana; Wear, **259**, 634-641 **(2005)**.

[26] J.Wang, M.Gu, Songhao, S.Ge; Wear, **255**, 774-779 **(2003)**.

[27] S.Basavarajappa, G.C.Chandramohan; Material Science and Technology, **21(6)**, 348-350 **(2005)**.

[28] B.Viswanath, A.P.Verma, C.V.S.Kameswara Rao; Composite Science Technology, **44**, 77-86 **(1992)**.

[29] P.B.Mody, T.W.Chou, K.Friedrich; Material Science, **23**, 4319-4330 **(1988)**.

[30] S.R.Chauhan, Anoop Kumar, I.Singh; Material Science, **44**, 6338-6347 **(2009)**.