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Processing and Characterization of Jute–Epoxy Composites Reinforced with SiC Derived from Rice Husk

ALOK SATAPATHY AND ALOK KUMAR JHA*
Department of Mechanical Engineering, National Institute of Technology, Rourkela, India

SISIR MANTRY AND S. K. SINGH
Advanced Materials Department, Institute of Minerals and Materials Technology, Bhubhaneswar, India

AMAR PATNAIK
Department of Mechanical Engineering, National Institute of Technology, Hamirpur, India

ABSTRACT: This article depicts the processing and mechanical characterization of a new class of multi-phase composites consisting of epoxy resin reinforced with jute fiber and filled with silicon carbide (SiC) particulates. The SiC used as filler material in this work was prepared from rice husk through plasma-processing technique. The effect of filler in modifying the physical and mechanical properties of jute–epoxy composites has been studied. It is found that the incorporation of rice husk derived SiC modifies the tensile, flexural, and inter-laminar shear strengths of the jute–epoxy composites. The micro-hardness and density of the composites are also greatly influenced by the content of these fillers.

KEY WORDS: silicon carbide, rice husk, jute–epoxy, mechanical characterization.

INTRODUCTION

FIBER-REINFORCED POLYMER COMPOSITES are now considered as an important class of engineering materials. They offer outstanding mechanical properties, unique flexibility in design capability, and ease of fabrication. Additional advantages include light weight, corrosion, and impact resistance and excellent fatigue strength. Today, fiber composites are routinely used in diverse applications such as automobiles, aircraft, space vehicles, off-shore structures, containers and piping, sporting goods, electronics, and appliances. A fiber-reinforced composite is not simply a mass of fibers dispersed within a polymer. It consists of fibers embedded in or bonded to a polymer matrix with distinct interfaces between the two constituent phases. The fibers are usually of high strength and modulus

*Author to whom correspondence should be addressed. E-mail: alokjha.mech@gmail.com

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and serve as the principal load-carrying members. The matrix acts as the load-transfer medium between fibers and, in less ideal cases, where loads are complex, the matrix may even have to partly bear loads. The matrix also serves to protect the fibers from environmental damage before, during, and after composite processing. In a composite, both fibers and matrix largely retain their identities and yet result in many properties that cannot be achieved with either of the constituents acting alone. A wide variety of fibers are available for use in composites. The most commonly used fibers are various types of carbon, glass, and aramid fibers. Besides, natural fibers such as jute, sisal, and ceramic fibers like alumina, silicon carbide (SiC), mullite, and silicon nitride are also used in composite making. The unique combinations of properties available in these fibers provide the outstanding functional and structural characteristics such as high specific strength and specific stiffness to the fiber-reinforced composites.

A key feature of fiber composites that makes them so promising as engineering materials is the opportunity to tailor the material properties through the control of fiber and matrix combinations and the selection of processing techniques. In principle, an infinite range of composite types exists, 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 those of conventional metallic materials [1]. The physical and mechanical characteristics can further be modified by adding a solid filler phase to the matrix body during the composite preparation. It has been observed that by incorporating filler particles into fiber-reinforced composites, synergistic effects may be achieved in the form of higher modulus and reduced material cost, yet accompanied with decreased strength and impact toughness [2,3]. Garcia et al. [4,5] suggested this 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, whiskers, or micro-fibers is added to the matrix prior to resin impregnation. Jang et al. [6,7] found 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 composites, even up to three orders of magnitude [8].

The improved performance of polymers and their composites in industrial and structural applications by the addition of filler materials has shown great promise and so has lately been a subject of considerable interest. Various kinds of polymers and polymer matrix composites reinforced with metal particles have a wide range of industrial applications such as in heaters, electrodes [9], and composites with thermal durability at high temperature [10]. These engineering composites are desired due to their low density, high corrosion resistance, ease of fabrication, and low cost [11–13]. Similarly, ceramic-filled polymer composites have been the subject of extensive research in the last two decades. The inclusion of inorganic fillers into polymers for commercial applications is primarily aimed at the cost reduction and stiffness improvement [14,15]. Along with fiber-reinforced composites, the composites made with particulate fillers have been found to perform well in many real-operational conditions.

However, such multi-component hybrid composites form complex systems and there is inadequate data available about phenomena behind the property changes due to the addition of particulate fillers to the fiber-reinforced thermoplastic components. Hence the objective of this article is to know how the incorporation of SiC (derived from rice husk)

particulates affects the mechanical properties of jute fiber-reinforced epoxy composites. SiC is a 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 100 years. Today, the material has been developed into a high-quality technical grade ceramic with very good mechanical properties. It is used in abrasives, refractories, ceramics, and numerous high-performance applications. The high thermal conductivity coupled with low thermal expansion and high strength gives this material exceptional thermal shock-resistant qualities. Moreover, SiC has low density, low thermal expansion, high elastic modulus, high strength, high hardness, and superior chemical inertness. Although the effect of SiC (produced from mineral sources) as a filler material has been investigated earlier [16] in glass–polyester composites, there is no report available on the potential of SiC particles derived from a bio-resource like rice husk in jute fiber-reinforced polymer composites. In this investigation, SiC produced from rice husk by plasma-processing route has been used. The details of formation of SiC from rice husk by this route are described elsewhere [17].

EXPERIMENTAL DETAILS

Matrix Material

Epoxy LY 556 is the resin which is used as the matrix material. Its common name is bisphenol-A-diglycidyl-ether and it chemically belongs to the 'epoxide' family. The epoxy resin and the hardener are supplied by Ciba Geigy India Ltd.

Fiber Material

Jute is a long, soft, shiny vegetable fiber that can be spun into coarse, strong threads. It is produced from plants in the genus *Corchorus*, family Tiliaceae. Jute is one of the cheapest natural fibers and is second only to cotton in amount produced and variety of uses. Jute fibers are composed primarily of the plant materials cellulose (major component of plant fiber) and lignin (major components wood fiber). It is thus a ligno-cellulosic fiber that is partially a textile fiber and partially wood. It falls into the bast fiber category (fiber collected from bast or skin of the plant) along with kenaf, industrial hemp, flax (linen), ramie, etc. Cross-plied woven mats of this jute fiber have been used as the reinforcing phase in the composites used in this work.

Composite Fabrication

Cross-plied jute fibers are reinforced in epoxy resin in three different weight proportions (20 wt%, 30 wt%, and 40 wt%) to prepare the composites A_1, B_1, and C_1 respectively. Jute fibers and epoxy resin have modulus of about 55 GPa and 3.42 GPa respectively and possess density of 1300 kg/m^3 and 1100 kg/m^3 respectively. No particulate filler is used in these composites.

The other composite samples C_2 and C_3 with SiC fillers of fixed weight percentage are fabricated by the same technique. The low temperature curing epoxy resin and corresponding hardener (HY951) are mixed in a ratio of 10 : 1 by weight as recommended. The mix is stirred manually to disperse the particulate fillers in the matrix. The mixing is done thoroughly before the jute-fiber mats (40 wt%) are reinforced in the matrix body.

Table 1. Designation and detailed composition of the composites.

Designation	Composition
A_1	Epoxy + 20 wt% jute fiber
B_1	Epoxy + 30 wt% jute fiber
C_1	Epoxy + 40 wt% jute fiber
C_2	Epoxy + 40 wt% jute fiber + 10 wt% SiC
C_3	Epoxy + 40 wt% jute fiber + 20 wt% SiC

Composites C_2 and C_3 contain SiC particles in 10 wt% and 20 wt% proportions respectively. Each ply of jute-fiber is of dimension 200 mm × 200 mm. The composite slabs are made by conventional hand-lay-up technique followed by light compression-molding technique. A stainless steel mold having dimensions of $210 \times 210 \times 40\,mm^3$ is used. A releasing agent (Silicon spray) is used to facilitate easy removal of the composite from the mold after curing. Care is taken to ensure a uniform sample since particles have a tendency to clump and tangle together when mixed. The cast of each composite is cured under a load of about 25 kg for 24 h before it is removed from the mold. Then this cast is post-cured in the air for another 24 h after removing out of the mould. Specimens of suitable dimension are cut using a diamond cutter for physical characterization and mechanical testing. Utmost care has been taken to maintain uniformity and homogeneity of the composite. The designation and detailed composition of the composites are given in Table 1.

MECHANICAL CHARACTERIZATION

Density and Void Fraction

The composites under this investigation consists of three components namely matrix, fiber, and particulate filler. The theoretical density of composites in terms of weight fraction can easily be obtained as per the following equation:

$$\rho_{ct} = \frac{1}{(W_f/\rho_f) + (W_m/\rho_m) + (W_p/\rho_p)} \tag{1}$$

where, W and ρ represent the weight fraction and density respectively. The suffix f, m, and ct stand for the fiber, matrix, and the composite materials respectively. The suffix 'p' indicates the particulate filler materials.

The actual density (ρ_{ce}) of the composite, 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 equation:

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \tag{2}$$

The theoretical and measured densities of the composites along with the corresponding volume fraction of voids are presented in Table 2. It may be noted that the composite density values calculated theoretically from weight fractions using Equation (2) are not equal to the experimentally measured values. This difference is a measure of voids and

Table 2. Measured and theoretical densities of the composites.

Composites	Measured density (gm/cc)	Theoretical density (gm/cc)	Volume fraction of voids (%)
A_1	1.127	1.135	0.71
B_1	1.139	1.153	1.35
C_1	1.157	1.172	1.28
C_2	1.199	1.258	4.68
C_3	1.287	1.358	5.22

pores present in the composites. It is clearly seen that with the increase in fiber content from 20 wt% to 40 wt%, there is an increase in the void fraction. However, in all the three composites A_1, B_1, and C_1, the volume fractions of voids are reasonably small ($< 1.5\%$) and this can be attributed to the absence of particulate fillers in these composites. With the addition of SiC as the filler material, more voids are found in the composites. As the filler content increases from 0 wt% to 10 wt% and subsequently from 10 wt% to 20 wt% the volume fraction of voids is found to be increase. This trend is observed in both the particulate filled composites (C_2 and C_3).

Density of a composite 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 content is the cause for the difference between the values of true density and the theoretically calculated one. The voids significantly affect some of the mechanical properties and even the performance of composites in the workplace. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration, and weathering [18]. The knowledge of void content is desirable for estimation of the quality of the composites. It is understandable that a good composite should have fewer voids. However, the presence of void is unavoidable in composite making particularly through hand-lay-up route.

Micro-hardness

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 of 24.54 N. The variation of composite micro-hardness with the weight fraction of jute fiber and SiC particulates is shown in Figure 1. For the composite A_1 (20 wt% of JF), the micro-hardness value is recorded as 57 Hv while for C_1 (40 wt% of GF) this value is 63 Hv. It is thus seen that with the increase in fiber content in the composite, the hardness improves although the increment is marginal. Similarly, with the incorporation of filler particulates into the composites, the mean hardness is seen to have improved.

Tensile, Flexural, and Inter-laminar Shear Strength

The tensile test is generally performed on flat specimens. The commonly used specimens for tensile test are the dog-bone type and the straight-side type with end tabs. During the test, a uniaxial load is applied through both the ends of the specimen. The ASTM standard test method for tensile properties of fiber resin composites has the designation D 3039-76. The length of the test section should be 200 mm. The tensile test is performed in the

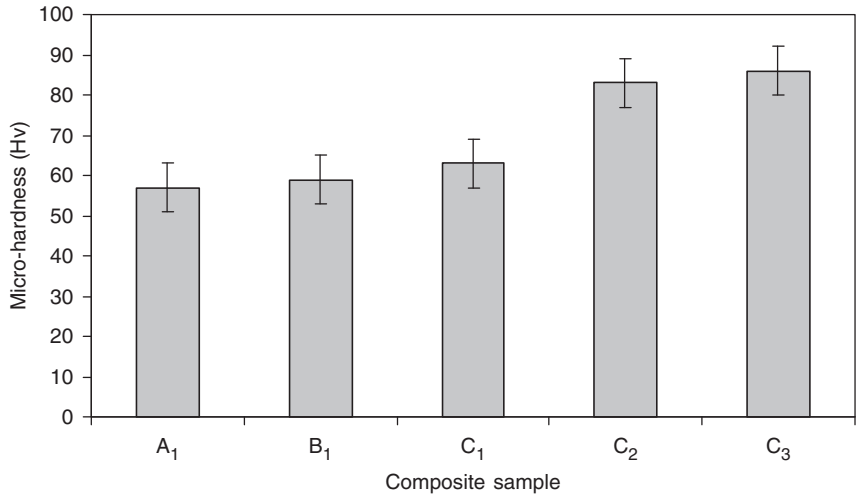

Figure 1. *Micro-hardness values of composites with different fiber and filler content.*

universal testing machine (UTM) Instron 1195 and results are analyzed to calculate the tensile strength of composite samples.

The short-beam shear (SBS) tests are performed on the composite samples at room temperature to evaluate the value of inter-laminar shear strength (ILSS). It is a three-point bend test, which generally promotes failure by inter-laminar shear. The SBS test is conducted as per ASTM standard (D2344-84) using the same UTM. Span length of 40 mm and the cross-head speed of 10 mm/min are maintained. The ILSS values are calculated as follows:

$$\text{ILSS} = \frac{3P}{4b \cdot t} \tag{3}$$

where, P is maximum load, b the width of specimen, and t the thickness of specimen.

It is well known that the strength properties of composites are mainly determined by the fiber content and the fiber strength. So variation in composite strength with different fiber loading is obvious. These variations in tensile and flexural strengths of the composites A_1, B_1, and C_1 are presented in Table 2 and are shown in Figure 2. A gradual increase in both tensile strength as well as flexural strength with the fiber-weight fraction is noticed. It clearly indicates that inclusion of jute fiber improves the load-bearing capacity and the ability to withstand bending of the composites. Similar observations have been reported by Harsha et al. [19] for fiber-reinforced thermoplastics such as poly-aryl-ether-ketone composites. It may be mentioned here that both tensile and flexural strengths are important for recommending any composite as a candidate for structural applications.

The test results for tensile and flexural strengths for the particulate filled composites C_1, C_2, and C_3 are shown in Figure 3. It is seen that the tensile strength of the composite decreases with increase in the filler content. The unfilled jute–epoxy composite has a strength of 349.6 MPa in tension and it is seen that this value drops to 304.5 MPa and 279.4 MPa with addition of 10 wt% and 20 wt% of SiC respectively. Similar trend is observed in case of flexural strength of these composites.

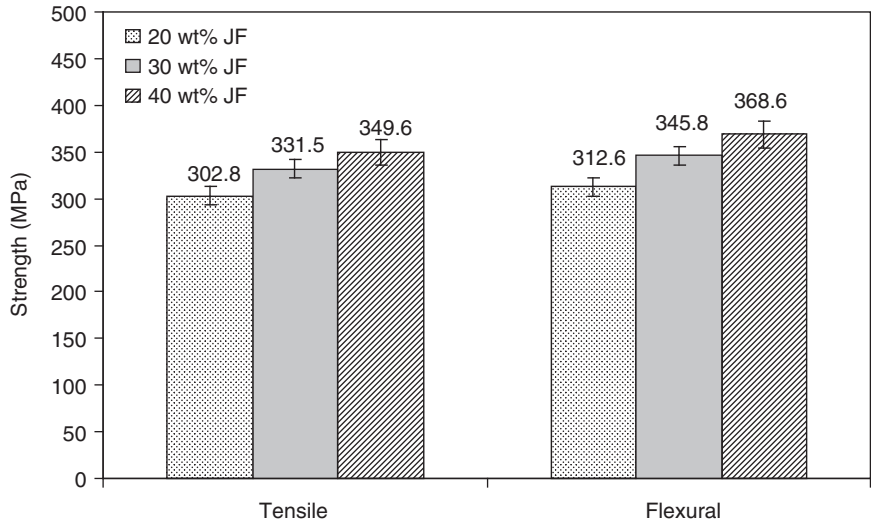

Figure 2. *Effect of fiber loading on tensile and flexural strength of jute fiber–epoxy composites.*

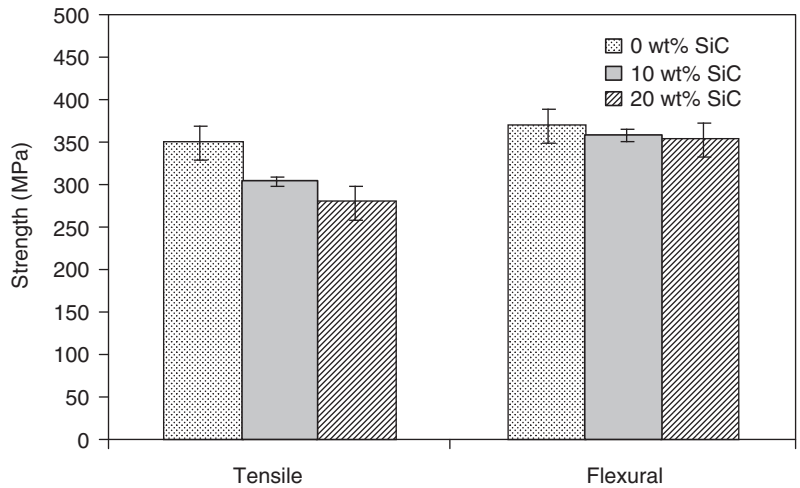

Figure 3. *Effect of filler content on tensile and flexural strength of jute fiber–epoxy composites.*

By incorporating these particulate fillers into the jute fiber-reinforced epoxy, synergistic effects, as expected were achieved in the form of modified mechanical properties and improved erosion wear resistance. Inclusion of jute fiber in neat epoxy improved the load-bearing capacity (tensile strength) and the ability to withstand bending (flexural strength) of the composites. But with the incorporation of SiC fillers, the tensile strengths of the composites were found to be less. There can be two reasons for this decline in tensile strength of these particulate-filled composites compared to the unfilled one. One possibility is that the chemical reaction at the interface between the filler particles 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 epoxy matrix.

Hardness values have been found to have improved for the particulate-filled composites. The reduction in tensile strength and the improvement in hardness with the incorporation of fillers can be explained as follows: under the action of a tensile force, the filler-matrix interface is vulnerable to debonding depending on interfacial bond strength and this may lead to a break in the composite. But in case of hardness test, a compression or pressing stress is in action. So the polymeric matrix phase and the solid filler phase would be pressed together and touch each other more tightly. Thus, the interface can transfer pressure more effectively although the interfacial bond may be poor. This might have resulted in an enhancement of hardness.

The stresses acting on the interface of the two adjacent laminae in a layered composite are called inter-laminar stresses. These stresses cause relative deformations between the consecutive laminae and if these are sufficiently high they may cause failure along the mid-plane between two adjacent laminae. It is therefore of considerable interest to evaluate ILSS through tests in which failure of the laminates of the composite initiates in a shear (delamination) mode. In the present work, the ILSS values are measured for unfilled jute-epoxy composites A_1, B_1, and C_1 and no improvement is recorded in the ILSS of the composites with increase in the fiber content in them.

The ILSS values of the particulate-filled composites are shown along with that of the unfilled jute epoxy composite (C_1) in the same Figure 4. It is seen that there is improvement of ILSS of jute–epoxy composites with particulate filling. Incorporation of SiC is seen to have caused the substantial increase in the ILSS. In the present investigation, during flexural test, the span length is very short (40 mm). A large span–to-depth ratio in bending test increases the maximum normal stress without affecting the inter-laminar shear stress and thereby increases the tendency for longitudinal failure. If the span is short enough, failure initiates and propagates by inter-laminar shear failure. The maximum shear stress in a beam occurs at the mid-plane. So in the shear test, failure consists of a crack running along the mid-plane of the beam so that crack plane is parallel to the longitudinal plane.

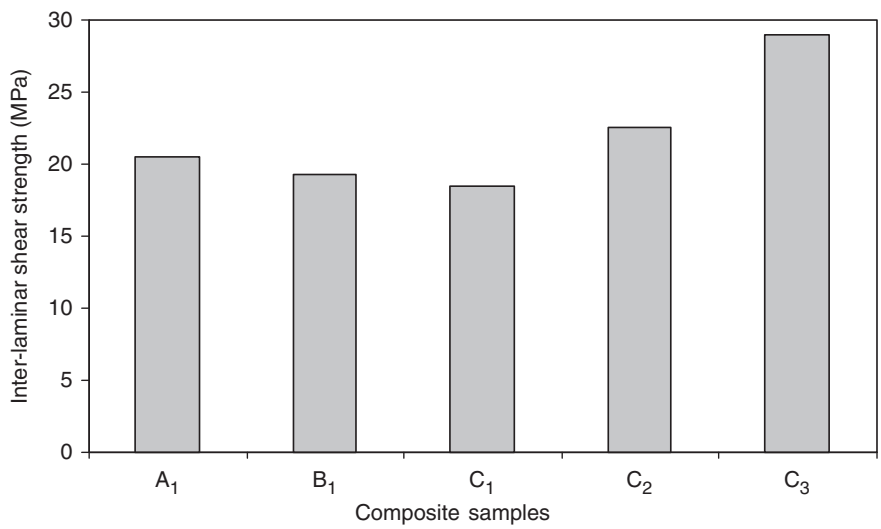

Figure 4. *Comparison of inter-laminar shear strength of different composites.*

Surface Morphology of Composite Samples

The surfaces of the specimens are examined directly by scanning electron microscope JEOL JSM-6480LV. The composite samples are mounted on stubs with silver paste. To enhance the conductivity of the samples, a thin film of platinum is vacuum-evaporated onto them before the photomicrographs are taken. The surface micro-structures of some of the composite samples are observed under scanning electron microscope and the surfaces are found to be reasonably homogeneous. No cracks are seen although some voids and pores are visible even at this lower magnification.

CONCLUSIONS

Successful fabrication of jute–epoxy composites with reinforcement of SiC derived from rice husk is possible. Incorporation of these fillers modifies the tensile, flexural, and ILSS of the jute–epoxy composites. The micro-hardness and density of the composites are also greatly influenced by the content of fillers. Hence, 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.

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