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RESEARCH ARTICLE

Evaluation of Curing Process for Bi-directional S-Glass (5HS)/Epoxy (780E +782H) Composites Fabricated by Vacuum Infusion Process for Wind Energy Blades

A. Chennakesava Reddy

Professor, Department of Mechanical Engineering, JNTUH College of Engineering, Kukatpally, Hyderabad, Telangana State, India.

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Abstract

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*Corresponding Author

A. Chennakesava Reddy

This article has been carried out to fabricate the wind energy blades S-glass/epoxy composites cured under vacuum. The tensile and flexural strengths of S-glass/epoxy composites were 3590.55 MPa and 1602.14 MPa respectively cured under vacuum at reliability of 90%. The tensile and flexural moduli of vacuum cured S-glass/epoxy composites were found to be 56.29 GPa and 43.95 GPa respectively at reliability of 90%. The interlaminar shear strength tensile strength of S-glass/epoxy composites was 79.08 MPa at 90% of reliability. The fiber dominated fracture was observed in the S-glass/epoxy composites having high volume fraction of fiber content.

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INTRODUCTION

Polymer matrix reinforced by fiber is probably the most commonly used form of composites in structural application, such as air craft, boats, automobiles etc. S-glass fibers are of magnesium alumino-silicate produced for textile substrates or reinforcements in composite applications. S-glass was originally developed for military applications in the 1960s, and a lower cost version, S-glass, was later developed for commercial applications. A comparison of the chemical composition and physical, mechanical and other properties of S-glass, E-glass and various other commercially available glass fibers has been published elsewhere (Hartman, et al., 2001). Takeda et al. (1994) have examined the thermo-mechanical behavior of cracked G-11 woven glass/epoxy composites with temperature dependent material properties under tension at cryogenic temperatures. They found that residual stresses have no significant effect on young's modulus of G-11 woven composites. The use of textile reinforcement is to take advantage of through-the-thickness arrangement of fibers to enhance interlaminar strength and toughness, compressive strength (Broek, 1986). Reinforcing fibers in the thickness direction improves stiffness and strength in that direction. Delamination is dominated by the resin property; as the resin gets tougher, the delamination becomes less brittle, which may limit the linear analysis of toughness (Ghassemieh and Nassehi, 2001). In the load-deflection curve, crack extension is sometimes indicated by a sudden drop in load, under displacement controlled testing. The effective properties of the fiber reinforced composites strongly depend upon the geometrical arrangement of the fibers within the matrix (Megel, et al., 2001). This arrangement is characterized by the volume fraction, fiber aspect ratio, fiber spacing parameters and orientation angles of fibers. The initiation of stress corrosion cracking (SCC) in the composites based on E-glass fibers can be controlled by the water/acid diffusion through a polymer matrix (Tang et al., 1987). Determination of cure cycle is one of the important factors for successful fabrication of thick epoxy product with dependable quality. Prepreg lay-up and autoclave cure is one of several important processes used in obtaining high quality fiber reinforced thermoset composite structures. High temperature and high pressure are

usually used to reduce the resin viscosity and to suppress voids during the consolidation of the composite (Kotiveerachari and Chennakesava, 1999).

The aim of this study is to determine the mechanical properties for S-glass/epoxy composites with different process parameters to improve fiber volume fraction for wind energy blades as shown in figure 1. The reasons to choose S-2 glass in the present work are as follows:

- S-glass is approximately 40-70% stronger than E-glass.
- E-glass and S-type glass lose about 50% of their tensile strength at 540° C; their strength at high temperatures is still considered good.
- Another temperature-related property to consider is the coefficient of thermal expansion (CTE). Fibers with a high CTE expand more as temperatures increase. S-type glass has a much lower CTE than either E-glass or C-glass.
- Both C-glass and S-glass offer good corrosion resistance when exposed to hydrochloric or sulfuric acid. E-glass and S-glass resist sodium carbonate solution better than C-glass.

The reasons to choose epoxy resin as the matrix material in the present work are as follows:

- Epoxy resin is used in structural aerospace applications
- Epoxy resin has less shrinkage and higher strength/stiffness at moderate temperatures.
- Epoxy resin is corrosion-resistant to solvents, alkalis, and some acids.

1. Material and Methods

All the testing procedures were as per ASTM standards. For every test three samples were used and the average value was reported for the results and discussion.

2.1 Materials

In the present study, epoxy resin (AIRSTONE 780E) was used as a matrix material. The hardener was AIRSTONE 782H. The S-glass (5-harness satin) weave) bi-directional fiber was used as reinforcing material. In the 5-harness satin (5-HS) weave the fill yarn floats over four warp yarns and under one. It is more pliable than the plain weave and easily conforms to curved surfaces like wind energy blades. The thickness and width were 0.031mm and 965mm. The structure of plain weave S-glass is shown in figure 2.

2.2 Composite Preparation

The vacuum infusion process (VIP) was used to fabricate the composites. S-glass fiber of 320mm x 320mm was taken from the fabric role. Epoxy resin (780E) and hardener (782H) were mixed in the 100:31 (parts by weight) ratio in a beaker and the solution was applied on the bi-directional S-glass fiber. The moulds were cleaned with acetone and the wax was applied to the moulds for easy removal of the cured composite. Resin impregnated S-glass fiber layers were placed in the mould by hand layup technique. The resin coated S-glass fibers were stacked to get 3mm thickness of the composite in the mould. The composites were prepared by different process parameters such as:

- No vacuum and pressure
- With vacuum
- With vacuum and pressure.

The corresponding cure cycles, vacuum levels and pressure application steps were assigned as cure cycle-1, cure cycle-2 and cure cycle-3.

Cure cycle-1:

- Component temperature at 140°C for 4 hrs
- Vacuum is -960 mbar

• Pressure at 140°C, 1.0 bar up to 2 hrs and 2.0 bar up to 4 hrs.

Cure cycle-2:

- Component temperature at 140°C for 4 hours
- Vacuum is -960 mbar
- Pressure at 140°C, 0.5 bar up to 1 hr, 1.0 bar up to 2 hrs, 1.5 bar up to 3 hrs and 2.0 bar up to 4 hrs.

Cure cycle-3:

(1)

- Component temperature at 140°C for 4 hrs
- Vacuum is -960 mbar
- Pressure is nil.

2.3 Testing Procedures

Fiber volume fraction was determined by acid digestion method. In this testing method, the digestion of resin matrix was carried out in a hot digestion medium like nitric acid which did not attack the fibers extremely. The specimens were cut in the required dimensions as per the ASTM standards using a diamond wheel cutting machine. The tensile test was carried out on universal testing machine (UTM) of Instron make, 1185 model, and 100KN load capacity. The tensile specimens were prepared as per ASTM D3039 standard. The three-point flexural test was conducted on the flexural specimens prepared as per ASTM D790 standard.

A bar of rectangular cross-section was used for interlaminar shear strength (ILSS) test specimen as shown in figure 3a. As shown in figure 3b three-point loading system, with center loading in a simply supported beam, was used. The specimen was rest on two supports and was loaded by means of loading nose between the supports as shown in figure 3c. The ILSS test was conducted as per ASTM D2344.

Longitudinal tensile strength, $\sigma_s = P_A$

where P is the maximum load at failure, N and A is the minimum cross-sectional area, mm^2 .

Tensile modulus,
$$E_t = \frac{\Delta P}{A_P}$$
 (2)

where ΔP is the load with in elastic limit, N and *e* is the strain.

Flexural strength,
$$E = \frac{3PL}{2bd^2}$$
 (3)

where P is the maximum load at failure, N and L is the support span, mm, b is the width of specimen, mm, d is the thickness of specimen, mm.

Flexural modulus,
$$E_f = \left(\frac{L^3}{4bd^3}\right) \times \left(\frac{\Delta P}{\Delta l}\right)$$
 (4)

where ΔP is the change in load at failure, N and Δl is the deflection, mm.

The stresses acting on the interface of two adjacent lamina are called interlaminar stresses. The interlaminar stresses are illustrated in figure 4. σ_t is the interlaminar normal stress on plane ABCD; τ_{tl} and τ_{tt} are the interlaminar shear stresses. These cause relative deformations between the lamina 1 and 2. If these stresses are sufficiently high, they will cause failure along the plane ABCD. It is of considerable interest to evaluate interlaminar shear strength through tests in which failure of composites initiates in a shear (delamination) mode.

Interlaminar shear strength, $ILSS = {}^{3P_{r}}/_{4bd}$ (5) where P_{r} is the rupture load, N and L is the support span, mm, b is the width of specimen, mm, d is the thickness of

where P_r is the rupture load, N and L is the support span, mm, b is the width of specimen, mm, d is the thickness o specimen, mm.

2. Results and Discussion

The purpose of determination of mechanical properties like tensile, flexural and interlaminar shear strength for bidirectional S-glass/epoxy (780E+782H) composites with different curing process parameters was to improve fiber volume fraction for the wind energy blades. The reaction between epoxy resins and amine hardeners is exothermic. The time dependent failure stress of S-glass is shown in figure 5a. The failure stress of S-glass is higher than that of E-glass. The viscosity of various blends of epoxy blends is shown in figure 5b. The blend, 780E/782H which has high viscosity was chosen for the present work to fabricate the wind energy blades.

3.1 Physical Properties of Composites

The physical properties of S-glass/epoxy composites are shown in figure 6. The curing cycle-1 results in very high volume fraction (figure 6a) of fiber in the composite and superior composite density (figure 6b) as compared to two other two cycles. The curing cycle-3 results in very low volume fraction of fiber in the composite and density. This was because of lack of compactness on account of vacuum only.

3.2 Mechanical Properties of Composites

The influence of fiber content in the composite on tensile and flexural strengths is shown in figure 7a. The tensile and flexural strengths increase with an increase in the volume fraction of fiber in the composite. The flexural strengths were lower than tensile strengths. The composites produced through curing cycle-1 have low tensile and flexural strengths. The layers in the fibers were formed by strong covalent bonds. The sheet-like aggregations readily allow the propagation of cracks. Improved tensile and flexural strengths of composites might be also due to contribution of increased stiffness for both S-glass fabric and epoxy matrix after vacuum conditioning (curing cycle-3). For composites of curing cycle-1 and -2 lower strength at applied pressure was resulted owing to the presence of more interfaces leading to the generation of large amount of residual stresses which were not easy to accommodate in the strong interface. It was resulted in the interfacial microscopic cracks, which would transform to macroscopic level by coalesce, and debonding phenomena to discharge the developed stresses. From figure 7b, it is observed that at reliability 0.90 the survival tensile strength of S-glass/epoxy composites for curing cycle-1 is 3338.39 MPa, for curing cycle-2 is 3434.44 MPa, and for curing cycle-3 is 3590.55 MPa. From figure 7c, it is also detected that at reliability 0.90 the survival flexural strength of S-glass/epoxy composites for curing cycle-1 is 1458.96 MPa, for curing cycle-2 is 1502.49 MPa, and for curing cycle-3 is 1602.14 MPa.

The influence of fiber content in the composite on tensile and flexural moduli is shown in figure 8a. The tensile and flexural moduli increase with an increase in the volume fraction of S-glass fiber. The ability of a sample to store energy, i.e. its elasticity was enhanced with curing cycle-1 and 2. Energy storage would occur as molecules were distorted from their equilibrium position by application of a pressure. The occurrence of drop in flexural modulus was probably attributed to the fracture of the 0/90° fibers of the surface lamina. From figure 8b, it is viewed that at reliability 0.90 the reliable tensile modulus of S-glass/epoxy composites for curing cycle-1 is 50.09 GPa, for curing cycle-2 is 54.86 GPa, and for curing cycle-3 is 56.29 GPa. From figure 8c, it is also noticed that at reliability 0.90 the reliable flexural strength of S-glass/epoxy composites for curing cycle-1 38.39 GPa, for curing cycle-2 is 41.71 GPa, and for curing cycle-3 is 43.95 GPa. The value of coefficient of non-linear elasticity was lower in fibers of higher modulus and it would decrease with decreasing interlayer spacing of the fiber crystallite structure. In S-glass/epoxy composite, the elastic modulus of the S-glass (~86.9GPa) was eight times higher than that of the epoxy matrix (~10.5GPa) so as the volume fraction of fibers was increased; the elastic modulus of the composite (measured parallel to the fibers) was increased linearly.

The effect of volume fraction of the epoxy resin on the interlaminar shear strength is shown in figure 9a. The effect of curing cycle on interlaminar shear strength was marginal because it was resin dependent property. The interlaminar shear strength would depend on the interfacial strength among the fiber layers only. From figure 9b, it is observed that at reliability 0.90 the reliable interlaminar shear strength tensile strength of S-glass/epoxy composites for curing cycle-1 is 72.43 MPa, for curing cycle-2 is 74.63 MPa, and for curing cycle-3 is 79.08 MPa.

Voids are one of the most common types of manufacturing process induced defects in composite materials that have detrimental effect on the material properties. The void content can be reduced by carefully chosen process parameters, such as pressure and temperature. The formation of voids/micro porosity in the composite prepared by the curing cycle-3 is shown in figure 10a. The curing cycle-2 has imparted fewer voids in the composites as compared the other two curing cycles (figure 10b). Macro porosities were mainly present during low viscosity impregnation of the reinforcement as opposed to micro pores which were the majority when the flow was governed by capillarity (high viscosity). If the volume fraction was in between 0.5 % and 1 %, the porosity did not influence on the behavior of the S-glass fiber reinforced composite mechanical characteristics. However, for higher levels of porosity, mechanical properties of S-glass fiber resin composite were significantly affected, particularly the interlaminar shear. In fact, the interlaminar shear strength (ILSS) was very responsive to the presence of these gas inclusions. The average reduction in ILSS is estimated to average 6% per unit volume of porosity for carbon / epoxy composites (Chennakesava and Vidya Sagar, 2010) (Sreenivasulu and Chennakesava, 2014) (Liu, et al., 2006) (Wisnom, et al., 1996).

3.3 Fracture of Composites

The fiber packing is shown in figure 11a. At high volume fractions of fibers, the reinforcement constitutes the major load bearing section and the addition of matrix gradually decreases the strength as the applied load is partitioned between the fibers and the matrix. The tensile strengths of S-glass fiber and epoxy are 4445 MPa and 85 MPa respectively. When the strain in the composite reaches the fracture strain of the matrix, the matrix will fail. All of the load will then transfer instantly to the fibers, which occupying such a small fraction of the sample area will see a large jump in stress and they too will fail. After the matrix breaks only the fibers remain to carry the load and the

stress in the fiber jumps by. If this increase takes the stress in the fiber above its fracture strength then the fibers too will snap. This is most likely to happen when the volume fraction of fibers is small and when the strength of the matrix is large. This is called matrix controlled fracture. However, if the jump in stress is not sufficient to break the fibers then the load can be increased until the fibers break. This is the fiber controlled fracture. The fractures specimens are shown in figure 11b during tensile testing. The fractured top surface (figure 11c) and the bottom surface (figure 11d) under flexural loading indicate the fiber controlled fracture.



Figure 1: Goal of the present work to fabricate wind energy blades.



Figure 2: Materials used (a) 5H satin weave (bi-directional) S-glass and (b) Epoxy resin.



Fig. 3. Interlaminar shear strength test



Fig. 4. Interlaminar shear stresses







Figure 6. Physical properties of S-glass/epoxy composites.



Figure 7. Effect of fiber content on tensile and flexural strengths (a) Weibull reliability of tensile strength (b) and Weibull reliability of flexural strength (c)



Figure 8. Effect of fiber content on tensile and flexural moduli (a) Weibull reliability of tensile modulus (b) and Weibull reliability of flexural modulus (c).



Figure 9. Effect of fiber content on interlaminar shear strength (a) and reliability of laminar shear strength (b).



Figure 10: Voids/micro porosity in S-glass/epoxy composites



Figure 11: Fabricated composite (a), tensile tested fractured specimens (b) top surface of fractured flexural specimen and (c) bottom surface of fractured flexural specimen of composited prepared by curing cycle-3.

3. CONCLUSIONS

The S-glass/epoxy composites cured under vacuum exhibit excellent mechanical properties. The tensile strength is higher than the flexural strength. At reliability 0.90 the survival tensile and flexural strengths of S-glass/epoxy composites for curing cycle-3 were 3590.55 MPa and 1602.14 MPa respectively. At reliability 0.90 the reliable tensile and flexural moduli of S-glass/epoxy composites for cycle-3 were in that order 56.29 GPa and 43.95 GPa. At reliability 0.90 the reliable interlaminar shear strength tensile strength of S-glass/epoxy composites for curing cycle-3 was 79.08 MPa. The S-glass/epoxy composites cured under vacuum can be used to fabricate the wind energy blades for long service life.

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