Indian Journal of Engineering & Materials Sciences
Vol. , December 2022, pp. 817-825
DOI: 10.56042/ijems.v29i6.70296

Multi-Fillers GFRP Laminated Composite Plates: Fabrication & Properties

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Received: 9 September 2022; Accepted : 17 October 2022

It is a common practice to enhance the strength of composites; the constituent materials (fiber/matrix) are altered. The addition of filler materials such as graphene can fulfil the need. However, the cost of graphene limits its use as a common filler material. In this regard, some other substitutes of filler or scheme of material preparation needs to be explored. In this work, bi-directional woven E-glass fabric epoxy composite laminated plates (LCPs) are fabricated using the hand layup technique. Graphene and flyash is used as filler. Ten types of LCPs are fabricated by altering the fillers at the designated fabric layer. Various thermal, mechanical, and physical tests are conducted. It is observed, the interfacial chemical bonding, load transfer between fiber and matrix and surface quality is affected due to the presence of fillers. LCPs with graphene have superior physical and mechanical properties than the LCPs of flyash filler. However, adding graphene in targeted layers without adding to whole LCP or functionally-grading through layers can also enhance the strength of LCPs, e.g., graphene when added in outer layers of LCP makes hydrophobic, hard and thermally stable along with increasing tensile and flexural strengths.

Keywords*:* Composite, Fabrication, Fillers, Flyash, Graphene, LCP

1 Introduction

As a result of high strength, modulus, corrosion resistance and insulation, glass fibre reinforced epoxy composites are a popular choice for structural applications. Automobile bodies, industrial furnishings, household, and now decorative furnish materials all use glass fibre epoxy composite. There is an ancient art to preparing composites, and it is a necessity.

There are numerous ways of preparation of composite materials[1-3]. A hand layup technique is discussed for preparation of composite beams taking the E-glass fiber as reinforcement in an epoxy base[4]. Further, they have included bi-directional glass fiber as reinforcement and studied modal properties of fabricated beams by doing experimentation[5]. In general, to enhance the mechanical strength of conventional polymeric composites, filler materials are added. The use of graphene is a general practice. A number of review works depicts that use of graphene as a filler material enhances the material property of the conventional polymeric composites[6-10]. It is reported that the conventional way of laminated composite plates(LCPs) preparation can be re-modified to incorporate filler material in the designated layer making the fabrication process cost effective with little bit care[11-13]. The static responses such as normal and shear stresses in such LCPs with fillers reported to be superior as examined theoretically[14]. It is observed that the microstructure and material properties of glass-epoxy composite with graphene dispersion is studied by Papageorgiou et al.[9] and their superiority is reported. Graphene can be used as a coating material for strain measurement[15]. The failure mode and strength of composites containing graphene are reported by Chandrasekaran et al.[16]. The addition of graphene increases the fatigue life of the composites[17]. It is reported that the electrical and thermal conductivity of the composites increases by the inclusion of graphene[18].

The thermal stability, mechanical strength and storage modulus of epoxy-based poly ether-graphene composite is reported to be improved[20]. The dielectric properties of composites are reported to be improved by the addition of graphene as it causes fine dispersion in the matrix[21]. The addition of graphene in kevlar-Cocos nucifera sheath-epoxy hybrid composites increases the thermal stability and mechanical strength[22]. The use of multiple fibers can enhance the mechanical and thermal strength of composites[22-24]. The use of flyash as a filler to enhance the thermo-

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mechanical characteristics of composites are reported[25]. Nano-clay proved to be a substitute if the thermal stability of the composite material needs to improved[26-27]. The thermal conductivity, absorptivity, and stability analysis of the composite materials with fillers are conducted and reported to be superior[28-29]. The composite plate with filler material also exhibits superior mechanical stability in free and forced vibration[30]. The development of plate theories and their implication as a further part of study of static and dynamic responses are elaborated[31].

From the study, it is observed that addition of filler materials alters the interfacial bonding between the fiber and matrix. As a result, the composites exhibit superior mechanical strength and thermal stability. It is also reported that adding graphene to the composite material induces thermal conductivity. Use of graphene in the composites in practice aspect is limited keeping its cost in consideration. As a result, the replacement of graphene may be flyash or nano-clay[26-28]. The method of preparation is another aspect of concern to minimize the cost of composite. The novelty of this work contributes adoption of simple hand-layup fabrication process to obtain break trough of material strength and graphene use as graphene is used in the selected layers. Flyash is used as secondary filler materials along with graphene are used to improve the thermal and mechanical properties of epoxy-glass composite plates. Ten schemes of LCPs are prepared by altering the graphene and flyash content. Different material testing is conducted and reported. For each of the material testing, five readings are considered, and the averages of the experimented results are presented through the study.

2 Materials and Methods

As part of this study, polymeric LCPs are constructed by hand layup process. Fiber glass (bi-directional wove fabric, 800 g/m^2) of size 600 × 600 mm^2 is used as reinforcement in epoxy resin (L56 epoxy with MS91hardener) base. To enhance the mechanical and thermal properties graphene and flyash is used as the filler material. Graphene nano-platelets are collected from the supplier directly while flyash is received from the nearby thermal power plants. To remove oxides and acids from the flyash, it is alkali treated. In alkaline water, the flyash is soaked for about an hour. Then, under direct sunlight, the soaked flyash is allowed to dry. It is then grinded to powder from in a mixture grinder. The grinded flyash is then kept in an induction furnace for 30min to remove the moisture if any present inside the flyash. A sieve shaker with a mesh of 320 is then used to screen the flyash to the desired size.

2.1 Preparation of LCP

For the preparation of LCP specimens with graphene and flyash filler, an open hand layup process is followed according to ASTM standard (D5687). For good surface finish of the LCPs, toughened thick mirror polished granite plate is used as the base plate and 600×600mm^2 square frame made of 20×20mm^2 M.S. square bar is used as side wall. The frame is held in position over the granite slab using four C-clamps. A thin layer of mansion polish wax is applied to the granite surface to make it easier to remove the cured LCP. Multi-purpose grease is used as a sealant between the granite plate and the square frame to prevent the epoxy from leaking out.

To prepare the mixture for every 100 gm of epoxy, 10 gm of an amine-based hardener is added as an accelerator. Then Graphene and fly ash are added to the mixture in a disposable polyethylene container and stirred for another fifteen minutes. Using a toothed roller, a first fibre layer is laid down, and epoxy is then evenly spread over it. Then, the second layer is laid. Each fibre layer is rolled by with a teethed steel roller to remove trapped air bubbles and voids in the resin. This process is repeated until all twelve fabric layers have been positioned. The casted specimen is left for 48-72 hours to cure properly and then removed. A fine graded carborundum paper and smooth cotton are used to clean the granite plate after each use. In the composite, 60 percent of the weight is made up of reinforcement. Nevertheless, the weight percentages of filler materials and the composition have been changed and ten schemes of LCPs are prepared and the details of each one is presented in Table 1.

2.2 Testing and characterization

To find the mechanical and thermal properties of the fabricated LCPs, different material testing is conducted. The LCPs are cut into required dimensions as of need of individual tests from the main sheet. The test specimens are machined using a FERM profile cutter following ASTM standard D5687 at room temperature. Five samples are prepared for each test type. The average of the experimented result of each test type is taken and presented.

Table 1. — Different wt. % of ingredient considered in laminated composite plate

Sl. No.	Weight Percentage of constituents				Stacking Morphology	Nomenclature of LCP variant
	Fiber	Resin	Graphene	Flyash		
1	60	40	---	---	Neat epoxy and glass	N-LCP
2	57.5	40	2.5	0	All Layer graphene	G-LCP1
3	55	40	5	0	All Layer graphene	G-LCP2
4	50	40	10	0	All Layer graphene	G-LCP3
5	57.5	40	0	2.5	All Layer flyash	FLCP1
6	55	40	0	5	All Layer flyash	F-LCP2
7	50	40	0	10	All Layer flyash	F-LCP3
8	55	40	0-5	5-0	Functionally Graded (L_{12} rich in Graphene and L_1 rich in Flyash)	FG-LCP
9	55	40	5	5	L_1 and L_{12} graphene, other flyash	O-LCP
10	55	40	5	5	L_6 and L_7 graphene, other flyash	C-LCP

2.2.1 Tensile test

UTM model INSTRON 3382, available at material testing laboratory, VSSUT, Burla, is used to find the tensile strength of the prepared LCP samples. Test samples of 150×20×6 mm width with dog bone shape at mid-span according to the ASTM standard are prepared. 80 mm of holding length at the two ends are left for holding of the UTM jaws. The samples are fitted to the jaws and a feed rate of 1 mm/min is maintained. The yield strength and rate of strain is recorded by the personal computer attached to the machine.

2.2.2 Flexural Test

Three-point bending test of the prepared LCP samples are performed in INSTRON 3382 UTM at VSSUT Burla material testing laboratory. For flexural test, test samples of 150×20×6 mm are prepared following ASTM D790 standard. Due to downward movement with a constant rate (0.01mm/min) of upper jaw the specimens bent and fail. The flexural properties of the test specimens such as strength, load at failure, stress vs. strain curve etc. are recorded.

2.2.3 Micro Hardness Test

Hardness is the resistance to indention of LCPs. In the current work, Vickers hardness test method is adopted. The specimens prepared are cut into smaller size of 20 ×20 mm. A ball diamond indenter fitted is indented till the required impression load is achieved. In this work, 0.025 Kg light load is applied to find the impression on composite materials. The hardness value is displayed by the display-cum-optical measurement system, installed on micro-Vickers hardness Tester (MMT-X) after a dwell period of 15Sec.

2.2.4 Impact Test

The impact test specimens are prepared according to the machine standard of AIT300D impact testing machine. Five test samples of size 80 ×10×6 mm are cut for each variant of LCP. The impact strength and impact energy of the test LCPs are recorded at the time of fracture of specimens due to impact force released by the pendulum.

2.2.5 Morphological Analysis

A Scanning Electron Microscope is used to study the surface morphology of the LCP samples after tensile testing. In order to avoid electrostatic charges during examination, fracture ends were mounted on aluminum stubs and coated with a thin layer of metal. The bonding of constituent materials, presence of voids and cracks are examined through SEM graph.

2.2.6 Water Absorption Test

Water absorption testing is performed as per the ASTM standard. The LCP samples are cut into small pieces of size 100×100 mm and dehumidified at an elevated temperature of 50˚C. The weight of each sample is then measured using a metered weight balance (AJ150 with least count of ±1mg) and immerged in the containers full of distilled water at room temperature inside the laboratory. The water absorptivity of the LCP samples is calculated using following formula $Wt.\%\, of water Absorption = \frac{Wt. of LCPs dipped in water - Wt. of dry LCPs}{Wt. of dry LCP Sample}$ … (1)

2.2.7 Thermal Conductivity Test

To define thermal behavior of a material, thermal conductivity test is used. It is reported that the chemical bonding of interfaces of matrix ad fiber in a composite decides the thermal conductivity and resistivity[29]. By this method, thermal conductivity of a bad conductor is determined using Lees Apparatus. The samples are cut into circular shape with diameter of 50mm from the main sheet. The samples are then kept inside the Lee's apparatus and the thermal conductivity is calculated using following relation

$$K = \frac{m \times c \times d \left({}^{dT}\!/_{dt} \right)}{A(T_1 - T_2)} \qquad \ldots (2)$$

Where K= Thermal conductivity of the LCP in W/m- K, m= sample mass in Kg, c= specific heat of a given material in J/Kg- K, d= Distance which the heat travels in meters, dT/dt= Rate of cooling in K/S, A=area of the bad conductor T_1, T_2= Temperature in K.

2.2.8 Thermography Analysis

The fabricated LCPs are supposed to be used as structural member which is most of the time exposed to direct Sunlight[32]. Hence Thermal absorptivity of the LCPs under direct sunlight is examined. In this study, thermal absorptivity of the LCP specimens is analyzed by FLIR E85 24° thermography analyzer. During thermography analysis, the LCPs are kept under direct sun light for 2-3 hour, the thermal image taken by the device quantifies the heat absorbed of the LCPs by their temperature difference.

2.2.9 Thermo-gravimetric Analysis

The LCP samples of weight 50mg are tested in the temperature range of 30–600 °C at a constant rate of 10 °C per minute in the closed chamber of nitrogen. Thermo gravimetric analysis (TGA) measurements is carried out to get thermal stability of the LCPs and expressed as the remaining of weight percentage with operating temperature.

3. Result discussion

It is observed from the experimentations that addition of filler to LCPs alters the mechanical and thermal properties of LCPs. Figure. 1(a) shows the stress strain curve of the LCPs obtained from the tensile test. Graphene facilitates load transfer between fiber and matrix[19-20] as a result, it is observed LCPs with graphene filler such as G-LCP1 (Graphene in LCP with 2.5% weight content), and G-LCP2 (Graphene in LCP with 5% weight content) & G-LCP3 (Graphene in LCP with 10% weight content) have superior tensile property due to the presence of graphene. Further, it is observed that FG-LCP (Functionally graded LCP; top layer rich of graphene and bottom layer rich in flyash with 5% weight content) have superior tensile strength than other types of LCPs. C-LCPs (LCP with two core layers filled with graphene and others with flyash of 5% weight content) and O-LCPs (LCP with two outer layers filled with graphene and others with flyash of 5% weight content) may be a suitable choice as of a replacement of G-LCP1 as shown in Fig. 1(b) where the whole layers of LCPs need not to be reinforced with graphene.

It is also observed that increase of filler weight content in LCPs alters the tensile strength as shown in Fig. 2. Increase of graphene weight content in LCP

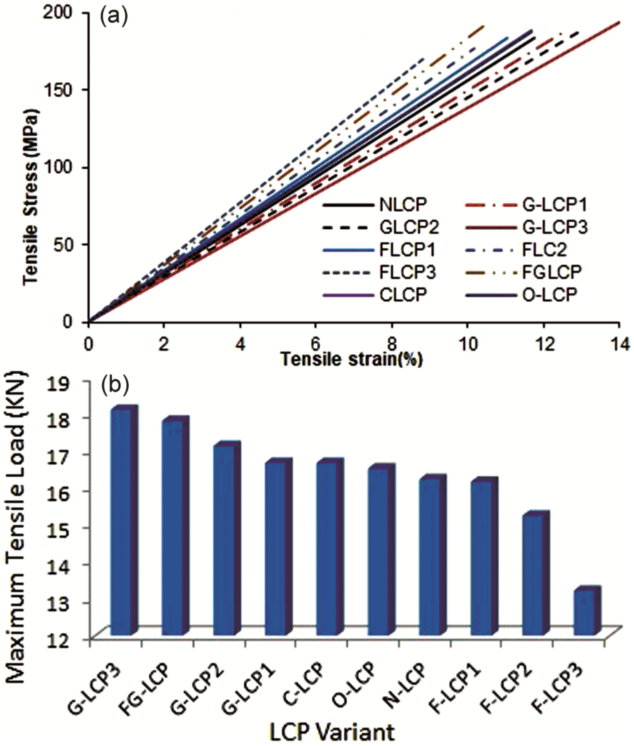


Fig.1. — (a) Tensile stress vs. strain (b) Maximum tensile load carried by LCPs at breaking.

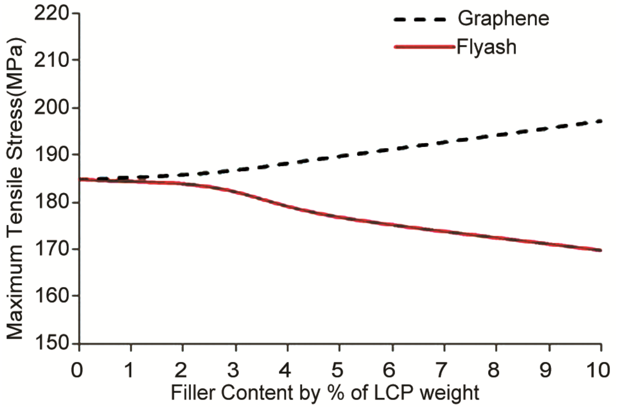


Fig.2. — Variation of maximum tensile stress with filler weight percentage.

increases tensile strength while increase of flyash weight content in LCPs decreases it. The graphene has superior elastic modulus over epoxy and glass as a result increase of graphene weight content increases tensile strength while flyash have lower elastic modulus thus causes decrease of tensile strength of F-LCPs (flyash dispersed LCPs).

Reinforcement of graphene develops a smooth coherent interface between the matrix and reinforcement as a result the load transfer becomes smoother, and the G-LCPs (LCPs with Graphene filler) withstand to higher tensile load. This phenomenon is least observed in case of F-LCPs such as F-LCP1 (Flyash in LCP with 2.5% weight content), and F-LCP3 (Flyash in LCP with 10% weight content) though addition of flyash develops hardness of F-LCPs.

A sample load vs. extension curve obtained from tensile test in UTM is depicted in Fig. 3. Increase of flyash content more stiffness the LCP than the content of graphene. It is observed that G-LCPs show maximum extensions at failure than F-LCPs.

Like tensile properties the flexural stress strain behaviour of LCPs are examined and plotted in Fig. 4(a). It is observed that, G-LCPs have better flexural strength as shown in Fig. 4(b). It is because, the overall stiffness-to-weight of the LCPs are increased due to addition of graphene in contrast to the addition of flyash. The tensile and flexural strength of FG-LCP, C-LCP, and O-LCP lies in between the G-LCPs and F-LCP. Addition of both flyash and graphene to LCPs improves the hardness. The surface area and density of the graphene is much higher than flyash; thus, LCP with graphene filler have superior hardness than that of LCPs with flyash.

Figure. 5(a) shows the hardness of test LCPs with a microscopic view of the FG-LCP surface after indentation is presented in Fig. 5(b). In the study, it is also revealed that O-LCP have higher hardness than the FG-LCP, C-LCP, N-LCP(Neat epoxy glass LCP) and F-LCPs. It is because when Graphene is given at the outer layer as filler, being the smallest particles with the relatively largest surface area cover-ups the entire LCP surface and resists a lot to indentation. The impact strength of the LCPs increases with increase of filler (both graphene and flyash) material. During impact failure, it is assumed that fibers got pulled out of the matrix. The mechanism of failure is governed by shearing failure and combined mechanism of tensile failure and shear failure as well.

It is reported presence of filler materials smoothens the load transfer between the fiber and matrix, and

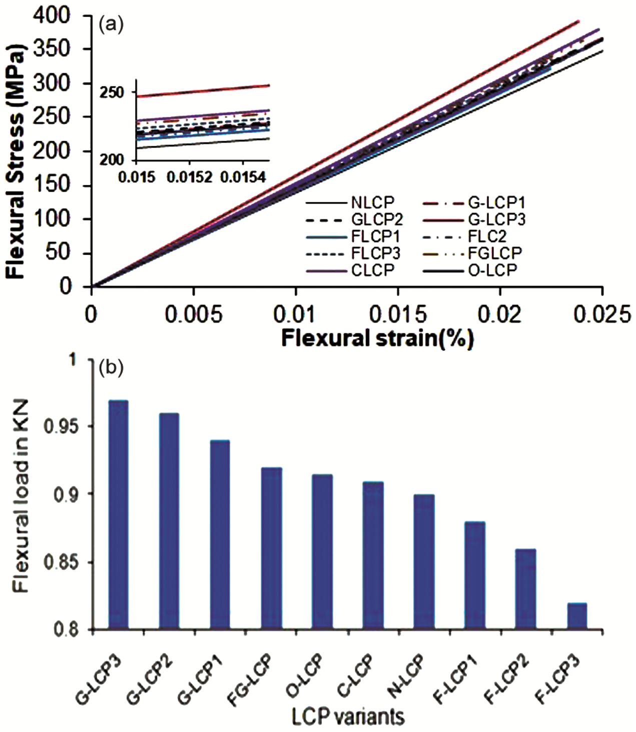


Fig.4 — (a) Flexural stress vs. Flexural strain , and (b) Maximum flexural strength of LCP variants.

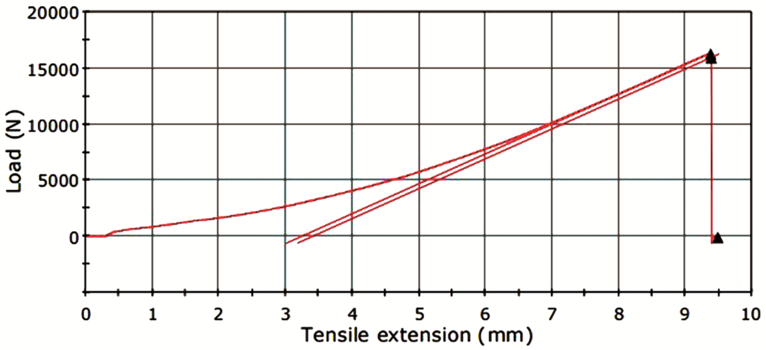


Fig. 3 — Tensile load vs. tensile extension.

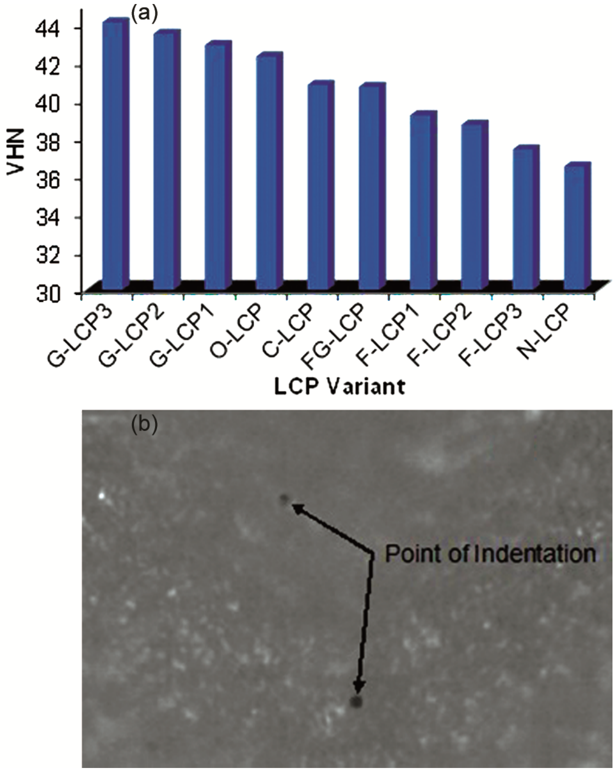


Fig. 5 — (a) Hardness of LCPs, and (b) A microscopic view of FG LCP after indentation.

thus compacts it to carry higher impact loads[27]. However, a noticeable rise of impact strength and energy is observed in LCPs with graphene as mentioned in the Fig. 6(a & b), respectively. It is due to the improvement of crosslink density of the LCPs with rise of graphene content. It is also observed that the impact strength increases 25% more than that of N-LCP with 2.5% addition of graphene. A dramatic rise of impact strength is observed for G-LCP3 with 10% of graphene content where the impact strength is found to 150% of impact strength of NLCP.

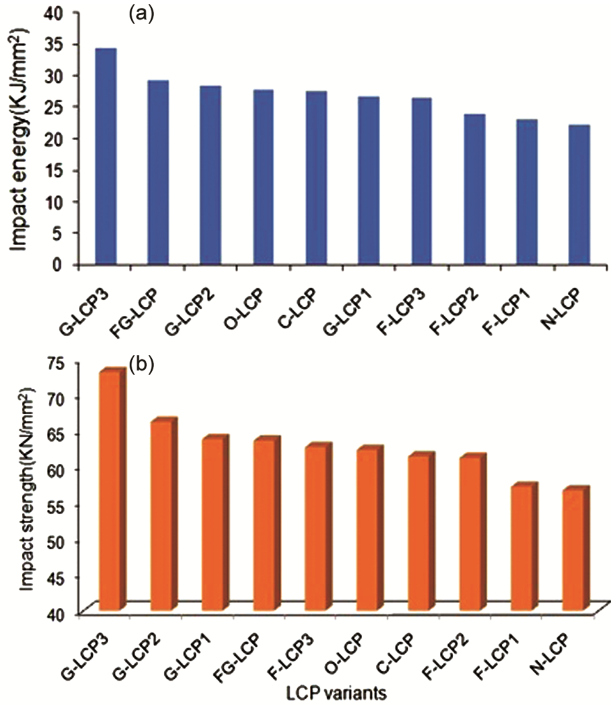


Fig. 6 — Impact Test Results (a) Impact energy in LCP, and (b) Impact strength in LCP.

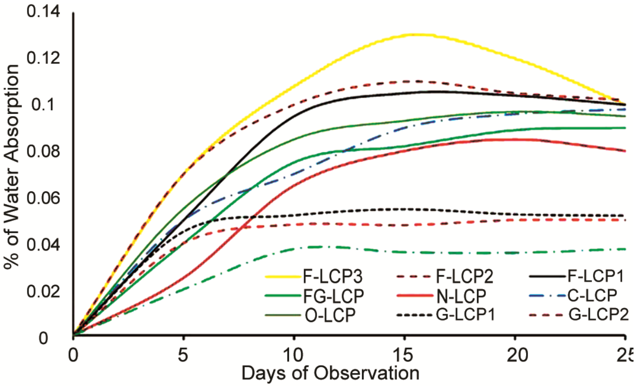


Fig. 7 — Water absorptive curves.

The water absorptivity of the LCPs prepared are observed and presented in Fig 7. It is observed that the impetration of water particle to LCPs increases the weight of the LCPs initially. The rate of water absorption seems to be saturated after 10 to 15 days. Flyash being porous and hydroponic in nature has the general tendency to retain moisture, thus LCPs with flash have higher tendency to retain moisture in the same working condition. Whereas the graphene has hydrophobic nature and the LCPs containing graphene as filler have lower water absorption rate. It is also observed that O-LCPs shows superior hydrophobic property than C-LCP as graphene in the outer layer increases the surface quality make it compact and void free.

It is reported that the chemical bonding of graphene and epoxy is the key factor that decides the thermal conductivity and resistivity of the LCPs containing graphene filler[29]. In general, LCPs are bad conductor of heat. The thermal conductivity of the LCPs is observed to be very much negligible as compared to other materials. However, it is experimented that by adding filler material, increases thermal conductivity as shown in Fig. 8. LCPs with graphene (G-LCP1, G-LCP2 and G-LCP3) have higher thermal conductivity followed by the LCPs with flyash filler.

It is because graphene isfiner particle than flyash creates much more effective interfacial bonding.

The thermal absorptivity of LCPs is examined through the thermography analysis and presented in Fig. 9. The thermal absorptivity of any material depends upon the surface morphology and quality[33]. It is evident that graphene has a superior surface area and conductivity; as a result, it is observed that the LCPs with Graphene have maximum heat retention capacity followed by O-LCP and FG-LCP. Also, addition of flyash increases the thermal absorptivity of the LCPs.

The continuity of the bonding among the constituent materials is studied by SEM. Fig. 10 (a) presents the morphology of FG LCP surface, where the distribution of fiber and filler at fracture surface is showen in Fig. 10 (b). It is observed the distribution of constituent material is uniform.

The thermal stability of the LCP variants is examined through TGA and presented through Fig. 11.

It is observed initially at a temperature of 90-130,C, all the LCPs loses weight about 1-5% due to evaporation of moisture present inside the LCP. Flyash being hydroponic in nature, LCPs containing flyash as filler such as F-LCP1, F-LCP2, F-LCP3, FG-LCP, C-LCP and O-LCP retains maximum moisture and the initial weight loss of an around 5% is observed. In other hand, graphene is hydrophobic in nature; as a result, LCPs with graphene filler (G-LCP1, G-LCP2 and G-LCP3) and N-LCP retains lower moisture and minimum weight loss (about 2%) is observed. In the second stage of weight loss occurred at 300-350,C, the compound of epoxy starts to break, and degradation of epoxy matrix starts.

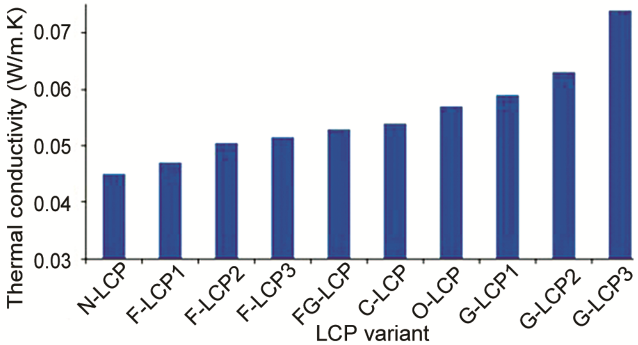


Fig. 8 — Thermal conductivity of LCPs.

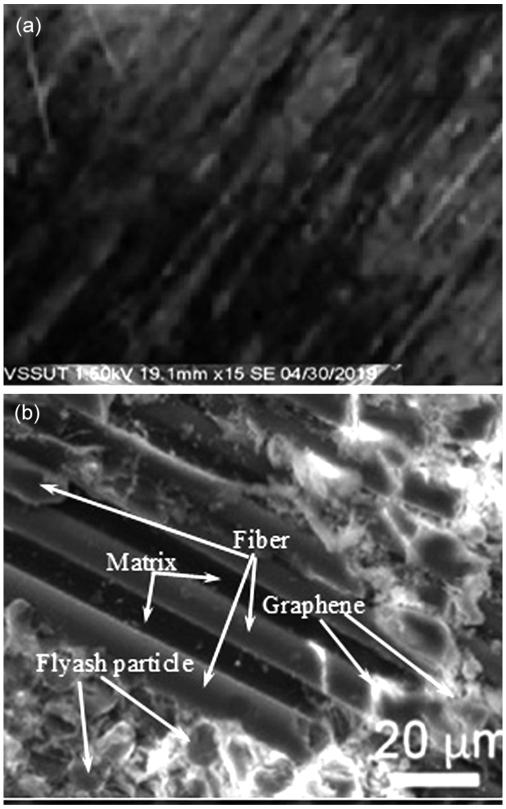
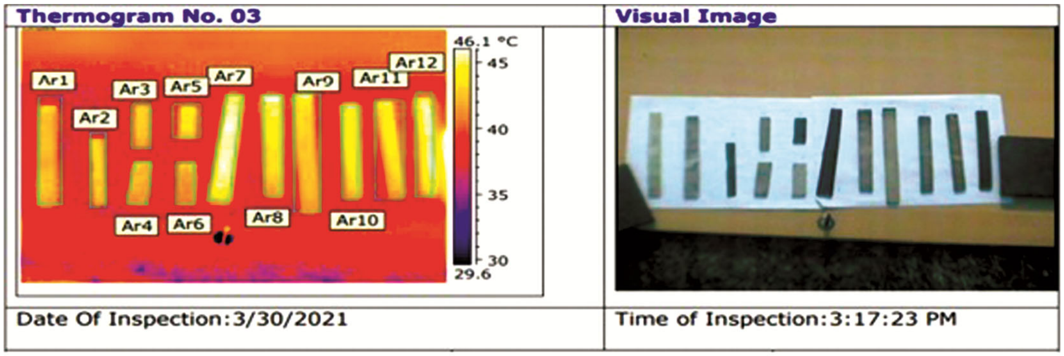


Fig. 10 — SEM image (a) LCP surface, and (b) after failure.



Thermogram No. 03

Visual Image

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Label	Ar6	Ar10	Ar7	Ar8	Ar2	Ar1	Ar5	Ar11	Ar12	Ar3
LCP variant	N-LCP	G-LCP1	G-LCP2	G-LCP3	F-LCP1	F-LCP2	F-LCP3	FG-LCP	O-LCP	C-LCP
Temperature (°C)	43.1	45.9	46.1	47.1	43.4	44.3	44.7	45.5	45.8	43.5

Fig. 9 — Thermography Image of LCP samples.

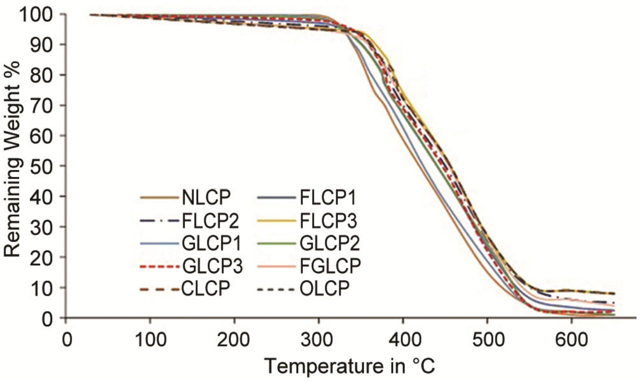

Fig. 11 — TGA curve of LCPs.

LCPs prepared with fillers exhibit superior thermal stability as compared to N-LCP. This is due to the lower rate of degradation of epoxy due to filer inclusion that affects the rate of pyrolysis of oxygen molecule present in the long chain of functional carbon groups[28-29]. It is observed the third stage of degradation of glass fiber due to pyrolysis begins on an around 445,C and completed around 550,C. LCPs with fillers degrades little it later than NLCP due to agglomeration of fillers to the fibers and matrix. It is also observed that LCPs with graphene filler (G-LCP1, G-LCP2 and G-LCP3) degrades faster than LCPs with flyash. It is understood that Graphene being compound of carbon is to burn at higher temperature hence minimum remaining of LCPs mass is observed. While flyash is nonvolatile in nature as no remaining of unburnt carbon particle is there hence becomes fire retardant. As a result, LCPs with flyash filler shows greater existence of degraded particles at the end. It is also observed that C-LCP shows better thermal stability than O-LCP and FG-LCP as flyash present in outer layer act as a fire-retardant layer.

4 Conclusion

Hand-layup fabrication of LCPs with filler material is a time-consuming process, but it can be done with a little bit of patience. If possible, it should be calibrated so that it doesn't affect the composition of adjacent layers. In this regard, a perfect cure time prediction is helpful. Following conclusion are drawn from the material testing.

- LCPs with Graphene filler exhibits increased tensile and flexural strength. LCPs with flyash filler.
- Most of the time Hardness and impact strength of LCPs are increased by addition of filler. The surface area and density of the graphene is much higher than flyash; thus, LCP with graphene filler have superior hardness than that of LCPs with flyash.
- The presence of filler materials smoothens the load transfer between the fiber and matrix and thus noticeable rise of impact strength and energy is observed in LCPs with graphene
- FLCPs being porous are hydroponic in nature, Whereas GLCPs have lower water absorption rate.
- From TGA it is observed that filler material added to LCP increases the thermal stability. C-LCP shows better thermal stability over FG-LCP and O-LCP.

Acknowledgement

The authors would like to thank the Department of Production Engineering, VSSUT, Burla, for providing the research facilities for this study.

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