

Fabrication and Evaluation of Low Density Glass-Epoxy Composites for Microwave Absorption Applications

I. Srikanth^{*,@}, P.S.N.S.R. Srikar¹, K. Anusha[&], S.K. Majee[@], H. Bhusan Baskey[^],
P. Rama Subba Reddy[#], and P. Ghosal[#]

[@]Advanced Systems Laboratory, Hyderabad - 500 058, India

¹Central University of Karnataka, Gulbarga - 585 367, India

[&]Rajiv Gandhi University of Knowledge Technologies, Nuzvid - 521 202, India

[^]Defence Materials Stores Research Development Establishment, Kanpur - 208 013, India

[#]Defence Metallurgical Research Laboratory, Hyderabad - 500 058, India

^{*}E-mail: i_srikanth@yahoo.co.uk

ABSTRACT

In the present work, fabrication and evaluation of low density glass – epoxy (LDGE) composites suitable for absorbing minimum 80 per cent of incident microwave energy in 8 GHz to 12 GHz (X-band) is reported. LDGE composites having different densities were fabricated using a novel method of partially replacing conventional S-glass fabric with low density glass (LDG) layers as the reinforcement materials. Flexural strength, inter laminar shear strength and impact strength of the prepared LDGE composites were evaluated and compared with conventional High density glass-epoxy (HDGE) composites to understand the changes in these properties due to replacement of S-glass fabrics with LDG layers. To convert LDGE structures to radar absorbing structures controlled quantities of milled carbon fibers were impregnated as these conducting milled carbon fibers can act as dielectric lossy materials which could absorb the incident microwave energy by interfacial polarisation. Electromagnetic properties namely loss tangent and reflection loss of carbon fiber impregnated LDGE composites were evaluated in 8 GHz -12 GHz frequency region and compared with HDGE composites. It was observed that both LDGE and HDGE composites have shown loss tangent values more than 1.1 and minimum 80 per cent absorption of incident microwave energy. Thus the results indicates that, LDGE composites can show EM properties on par with HDGE composites. Furthermore these LDGE composite could successfully withstand the low velocity impacts (4.5 m/s) with 50 J incident energy. Due to their ability to show good mechanical properties and light weight, LDGE composites can be used as a replacement for conventional HDGE composites to realise radar absorbing structures.

Keywords: Light weight composites; Mechanical properties; Fracture morphology; electromagnetic properties; Radar absorbing structures; RAS

1. INTRODUCTION

Glass fiber reinforced polymer matrix composites (GFRPs) are most widely used in both defence and civilian applications due to their durability, low cost, reasonable specific strength and good impact resistance. They are used to realise both impact resistant structures as well as microwave (MW) absorbing structures¹⁻³. One disadvantage with GFRPs is, their density is very high as compared to carbon fiber reinforced composites (CFRPs). Hence, to deploy GFRPs for aerospace applications it is essential to reduce their weight to maximum possible levels. Weight reduction of GFRPs can be achieved by replacing some of the conventional high density glass fabric layers with LDG layers. Such changes in the reinforcement can affect the mechanical properties, impact resistance. Hence, it is required to study the effect of introducing LDG into GFRPs.

Glass fiber reinforced polymer matrix composites are also known as good dielectric materials which are widely used for realising RAS. When specific additives like ferrites or

carbonaceous materials are introduced to GFRPs in controlled quantities, they acquire MW absorbing property⁴. However, very high amount of ferrites (approximately 80 wt per cent or above) are to be added to polymer matrix to achieve good MW absorption leading to heavy weight which is not intended for aerospace applications⁵. Carbonaceous materials like milled carbon fibers, carbon black, do not add extra weight yet give required MW absorption ability by acting as dielectric lossy material². However, with carbonaceous materials, it is difficult to attain good MW absorption for GFRPs at lower thickness^{2,6-8}. Hence, in practical applications, higher thickness GFRPs are to be fabricated with controlled addition of carbonaceous material to obtain good MW absorption. However, GFRPs having higher thickness possess more weight and thus limits their application in aerospace systems. Hence, weight reduction of the GFRPs without compromise in the thickness and MW absorption properties is one of the major requirements for advanced aerospace systems. Some researchers reported introducing PVC foam, poly urethane foam in GFRPs to realise light weight structures^{9,10}. Further studies on alternate fabrication processes

to realise light weight structures are required as comprehensive data on mechanical and electromagnetic properties of light weight RAS is not reported so far by any research group. Present study proposes partial replacement of conventional high density glass layers with LDG layers to realise light weight hybrid composites which can offer significant weight saving to the end applications without compromise in the electromagnetic properties. Present study also includes mechanical properties as well as low velocity impact response evaluation of proposed lightweight composites along with the studies on their suitability for replacing HDGE composites in MW absorbing applications.

2. MATERIALS AND METHODS

2.1 Raw Materials

Diglycidyl ether of bisphenol - A (DGEBA) based epoxy resin with diethyl toluene diamine (DETDA) hardener was used as matrix system. S-glass fabric (M/s BGF Industries, USA) having aerial weight of 815 ± 10 grams per square meter (GSM) was used to realise HDGE composites whereas for fabricating LDGE composites LDG layers (Kaowool Paper of M/s Murugappa Morgan Thermal Ceramics Ltd, India) having 215 ± 10 GSM were used as the primary reinforcement. Milled carbon fibers were used as dielectric lossy material (DLM) as shown in Fig. 1. Source for the milled fibers is T-300 grade carbon fibers with diameter of 8 microns and carbon content >95 per cent. These carbon fibers were initially chopped to an approximate size of 2 mm to 4 mm (Figs. 1(a) and 1(b)) length which were subsequently ball milled to a size of 20 to 50 micrometers (Figs. 1(c) and 1(d)). SS balls taken in SS bowl of 500 ml was used for this purpose. Ball to fiber weight ratio is 4:1 and ball milling was carried out at 150 rpm. These milled fibers were added to impart MW absorption characteristics to composites. 8HS grade carbon fabric is used to realise a perfect electric conductor (PEC) layer.

2.2 Fabrication of Composites

Composites were fabricated in two stages. In the first stage (stage-1), 4 mm thick composites were fabricated with an aim to study the effect of partial replacement of S-glass layers with controlled quantities of LDG layers on the flexural strength and inter laminar shear strength (ILSS). In the second stage (stage-2), samples having higher thickness (10 mm) were fabricated to know whether controlled replacement of

the S-glass layers with LDG layers has got any negative effect on EM properties (loss tangent and MW absorption). Minimum impact resistance is essential for RAS to withstand accidental tool drops. Low velocity impact test can be used to evaluate ability of RAS to withstand such conditions. Hence samples fabricated in stage 2 were also evaluated for the low velocity impact resistance.

2.2.1 Stage 1: Fabrication of Composites For Mechanical Property Evaluation

For fabricating the HDGE composite, epoxy resin was mixed with hardener (in the weight ratio of 100:24) and this mixture was applied on S-glass layers and carbon fabric layer with a hand brush. Initially carbon fabric layer was laid up (which formed the bottom most layer of the composite) over which required number of S-glass fabric layers were stacked in a suitable die. These layers were compacted to a thickness of $4 (\pm 0.1)$ mm. Curing was carried out for two hours at 120°C followed by five hours at 160°C . Thus HDGE composites were realised.

For reducing the density of HDGE, (to realise LDGE) S-glass fabric layers were partly replaced with LDG layers. Different composites made, along with the variations in terms of S-glass to LDG layers weight ratios and the resultant densities of the fabricated composites is given in Table 1.

2.2.2 Stage 2: Fabrication of Composites for EM Property Evaluation

Based on the results obtained in stage 1 studies, two different compositions namely S-1 and S-4 were chosen for stage 2 fabrication. S-1 and S-4 represents two extremes of composite densities. S-4 was chosen over S-5, because it has shown reasonably good mechanical properties sufficient for radar absorbing structures where reasonable strength at lowest possible density is the primary criteria.

Since, MW absorption of composites is more effective for laminates having more thickness, high thickness ($10 \text{ mm} \pm 0.1$ thick) HDGE (Reinforcement sequence equivalent to S-1 with proportionate increase in the S-glass layers to obtain targeted thickness) and LDGE (Reinforcement sequence similar to S-4) were fabricated taking required number of reinforcement layers. Epoxy resin with hardener were added with 3wt per cent of milled carbon fibers. This mixture was subjected to ball milling using a planetary ball mill (M/s Insmart Systems, India).

Table 1. Different composites made with varying amounts of S-glass and LDG layers with Flexural strength and ILSS values

Sample ID	Sequence of reinforcement layers from top to bottom	Weight ratio of reinforcing layers		Density of fabricated composite (g/cc)	Flexural Strength (MPa)	Flexural Modulus (GPa)	ILSS (MPa)
		S-Glass	LDG				
S-1	1C,8S	100	0	1.84	534(40)	34	36(2.3)
S-2	1C,4LDG,6S	80	20	1.72	354(10)	29.4	28(2.1)
S-3	1C,9LDG,3S	50	50	1.63	276(3)	23.7	26(2.5)
S-4	1C,10LDG,2S	40	60	1.42	248(7)	19.5	21(0.6)
S-5	1C,14LDG	0	100	1.30	149(5)	6.6	13(0.5)

Note: Values in the parenthesis shows standard deviation. 'C': Carbon fabric' and 'S': S-glass fabric'.

SS balls taken in a SS bowl were used at a ball to fiber weight ratio of 2:1. Milling was carried out for 15 mins at a speed of 150 rpm and then this mixture was applied on the fabric layers. These fabric layers were stacked and compacted to realise the composites following the method discussed earlier.

2.3 Characterisation

Composites fabricated in Stage 1 were subjected to flexural strength and ILSS tests following ASTM D790 and ASTM D2344, respectively on a universal testing machine (United 50 KN, USA). The crosshead speed for both the test was 2 mm/min. For both flexural and ILSS tests, samples were loaded in UTM in such a manner that, carbon fabric layer is away from the loading point thus forming the bottom of the sample under testing. Crack propagation modes of the tested samples were analysed using SEM (ESEM, FEI Quanta 400, The Netherlands). Composites fabricated in the Stage 2 were subjected to low velocity impact strength as per ASTM D 3763 using a drop weight tester (Ceast - CEAST 9350) with 50 J incident energy. Diameter of the impactor nose is 12.7 mm with a weight of 5.266 Kg. It impacted the samples at a velocity of 4.53 m/s. Samples were placed in such manner that, the carbon fabric layer is away from the impactor nose. To know the extent of damage, thermography analysis was carried out on impacted samples using IR camera (Model Thermo CAM SC 3000, Germany) supplied by M/s FLIR systems. Electromagnetic properties of composite specimens fabricated in stage-2 were evaluated using free space measurement setup (FSMS-Agilent VNA, E8364B)¹¹. Sample holder is mounted between two horn lens antenna at a distance of 30 cm from the source antenna. Measurement of scattering parameters was carried out after calibration of FSMS using thru-reflect and line (TRL) method. The typical accuracy of the measured scattering co-efficient is ± 0.05 dB and phase ± 2 per cent. Samples were mounted in such a way that, carbon fabric layer which forms the perfect electrical conducting layer (PEC layer) is away from the loading point.

3. RESULTS AND DISCUSSION

3.1 Flexural Strength and ILSS

Table 1 shows the obtained results. Load vs displacement curves of the samples tested for flexural strength is shown in Fig. 2. It can be observed that, when composites were fabricated exclusively with either S-glass (S-1) or LDG layers (S-5), failure has occurred in a more ductile manner with significant strain associated with the samples prior to the failure whereas the hybrid samples (S-2, S-3, S-4) failed in a brittle manner. This could be attributed to the fact that, in unifiber (single type of fiber/fabric) reinforced composites, load distribution occurs homogeneously throughout the area of the test specimen which is subjected to bending/flexural loads. Failure initiation under flexural loads for a unifiber composites is generally by compressive failure under the loading point of the three point bend test¹². Kinking of the fibers precedes the compressive failures. In the unifiber composites these kinks were observed to occur uniformly throughout the composite which can prohibit transverse crack propagation. Hence, crack propagation was observed to proceed through the thickness of

the composites (Fig. 3(a)). This resulted in a typical graceful failure that fibrous material reinforced composites generally display.

The hybrid samples (S-2, S-3, and S-4) were found to display more inter laminar delamination (Fig. 3(b)). From Fig. 3(c), it can be seen that, before S-glass layers can respond completely to the compressive loads (evidenced by insignificant kinking) transverse cracks across the interface of the S-glass layers to the LDG layers got generated. This could be understood from the fact that, S-glass-epoxy portion of S-2, S-3, and S-4 composites which have got a flexural modulus of 34 GPa (measured from load – displacement curves shown in Fig. 2) will undergo less strain whereas the LDG-epoxy portion of these composites which have a flexural modulus of 6.6 GPa (Table 1) will undergo more strain. Thus when two different reinforcements having significant difference in their strength and stiffness are present in a single composite, differential stiffness of the of the composite layers across the thickness gives shear force at the interface leading to generation of inter-facial cracks which results in a brittle failure of hybrid composites. It can be seen from the micrographs that, crack propagation from the LDG layers to the carbon fabric layer at the bottom was not observed (Figs. 3(c) and 3(d)). This is because of the low stress intensity factors (K_{Ic}) of foam based materials (LDG layers in present case) which can be of the order of 0.5 MPa m^{1/2} where crack initiation and propagation is easy¹³. However, such low stress intensity factor at the tip of the LDG layers may not be sufficient to rupture and propagate through the high strength layers (Carbon fabric layer present at the bottom) which need minimum three fold higher values of stress intensity factors as compared to LDG layers to rupture^{14,15}. This is leading to under utilisation of the strength of the top S-glass layers as well as bottom carbon layer there by leading to overall reduction in strength.

ILSS of S-1 was found to be better because the orthogonal weaving pattern in the S-glass gives the locking of matrix rich interface zones (crimp) with the adjacent S-glass layers. Moreover, the crimps zones increases the interface area leading to better ILSS. When S-glass layers were replaced with LDG layers (which is planar without any crimps), overall inter-facial area will come down. This resulted in reduction of ILSS for the samples fabricated with LDG layers.

3.2 Impact Strength

Typical force vs time, energy vs time, displacement vs time curves and the thermography images of the impacted samples are shown in Fig. 4. The impact resistance of the laminate can be derived from the maximum force beyond which laminate will fail. Force vs time curves are showing reduction in the peak force for the LDGE samples as compared to HDGE samples. This indicates low impact strength for LDGE. However, force has not dropped to zero, rather sustained on par with the HDGE composites. This indicates the suitability of the LDGE composites against low velocity impacts. In the force vs time curves, slope of the curve indicates the contact stiffness^{16,17}. Slope of the curves remained more or less same for HDGE and LDGE samples. From this, it can be inferred that, top few

layers of S-glass present in the hybrid samples are dictating the resultant strain against impact. From thermography studies, it can be seen that, significant damage was not observed for HDGE samples whereas LDGE has shown an indentation mark Fig. 4. In force vs time curves oscillations are an indication of damage which occurred in the laminate in the form of surface splitting, delamination, failure at interface between the fibers and matrix and fiber breakage^{18,19}. Significant oscillations can be seen in the case of LDGE composite which indicates that the sample has undergone considerable damage where as such oscillations are not observed in case of HDGE composites. Hence, it can be inferred that, actual impact strength of the HDGE samples is much higher than the impact conditions that were used in the low velocity impact test. Hence, it has not shown any significant damage as observed from the thermography images. On the other hand LDGE has shown indications of damage. However both HDGE and LDGE laminates have shown rebound effects as seen in the force vs displacement curves. This indicates that the impact energy is below the laminate threshold energy for both LDGE and HDGE. From the force vs displacement data, contact stiffness is measured as the ratio of peak force to the corresponding displacement (Table 2)²⁰. Contact stiffness of HDGE composites is found to be higher as compared to the LDGE composites. It can be seen that, the addition of milled carbon fibers has not affected the contact stiffness of both LDGE and HDGE composites significantly. Energy vs time curves are indicating that both LDGE and HDGE samples could absorb almost similar amount of impact energy. This indicates that, laminates which may experience low velocity impacts (with energy values lower than 50 J) during service can be manufactured with limited amount of S-glass coupled with controlled additions of LDG layers without compromise in the impact energy absorption.

Table 2. Peak force and contact stiffness values of the composites

Sample ID	Displacement (mm)	Peak force (N)	Contact stiffness (N/mm)
S-1 (HDGE-B)	4.24	17827	4224
S-1 (HDGE)	4.3	17054	3966
S-4 (LDGE-B)	6.02	11980	2153
S-4 (LDGE)	6.04	11800	1919

3.3 Electromagnetic Properties

Loss tangent represents the loss properties of incident electromagnetic wave. As carbon fibers are nonmagnetic, their MW absorption capability in the composite originates either from polarisation losses, or from multiple scattering^{2,21}. Orientation polarisation and space charge polarisation plays a dominant role in imparting lossy character to the materials in MW frequency range²². The distribution of conducting carbonaceous material in the dielectric medium results into modified phase boundaries in terms of charge carrier delocalisation which changes the intrinsic properties of the composite within each region²³. In response to the incident waves these regions will generate inductive current which

will be dissipated by the dielectric medium as heat there by absorbing the impinged MW energy. Since both LDGE and HDGE composites having same thickness were impregnated with same quantity of milled carbon fibers, losses associated with polarisations and multiple internal scatterings should be same. Similar EM properties displayed by LDGE and HDGE composites also indicates the fact that, LDG layers are behaving same like S-glass fabric layers interms of their EM properties. This is possible because both LDG and HDG layers at atomic/molecular level consists of same composition of aluminosilicate based glass. This enables LDGE to behave same like HDGE in terms of EM properties both in unimpregnated (without milled carbon fibers) and impregnated (with milled carbon fibers) conditions. From the return loss values (in dB) percentage reflected power can be calculated as

$$\text{Reflected powder} = 100 \times \Gamma^2 \quad (1)$$

where Γ is reflection co-efficient.

$$\text{In turn}^{24} \quad \Gamma = 10^{(-\text{returnloss}/20)} \quad (2)$$

From the return loss/reflection loss data (shown in Fig. 5) it can be seen that minimum -8 dB loss is observed for both LDGE and HDGE composites. From this return loss data using Eqns. (1) and (2) it is found that minimum 15.8 per cent of the incident power is reflected back which indicates that around 84 per cent of the incident energy is absorbed by the LDGE/HDGE composites impregnated with milled carbon fibers.

However, the MW attenuation property of the composites changes with the change in the dielectric medium of host composite (LDG or HDG). This is because different dielectrics have got different ability to dissipate the inductive current⁷. In the present study, EM properties of both HDGE and LDGE composites are found to be similar as shown in Fig. 5. This indicates that, replacement of S-glass layers with LDG layers has not resulted in change of EM properties of composites. When these composites were modified with controlled addition of carbon fibers, loss tangent and RL values were observed to increase by approximately same magnitude and their pattern of change as function of frequency was also observed to be following similar trend which indicates that LDG layers are not interfering with the EM performance of the composites.

4. CONCLUSIONS

Present study establishes that, for fabricating microwave absorbing composites, conventional high density glass layers can be partly replaced with other alternate materials like LDG which can result into around 20 per cent reduction in overall density of the systems. Fracture mode of hybrid composites was observed to be brittle with inter layer crack propagation as the main failure mechanism. These HDG and LDG hybrid composites can show impact resistance sufficient to withstand low velocity impacts (having impact strengths up to 50 J of incident energy). These hybrid composites can show reflection losses upto 80 per cent or above by controlled addition of milled carbon fibers. The MW absorption mechanisms like interfacial polarisation that operates in HDG reinforced composites can also operate in the similar manner in the LDG reinforced composites leading to no compromise in the EM properties for the LDG composite against the HDG based composites.

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CONTRIBUTORS

Dr I. Srikanth obtained MTech (Corrosion Science & Engineering) from IIT, Mumbai, in 2000 and MSc (Chemistry) from Andhra University Vizag, in 1998 and PhD from Metallurgical & Materials Engineering Department of IIT, Hyderabad, in 2014. Presently working as Scientist 'E' at Advanced Systems Laboratory, Hyderabad. He has published 25 papers, filed 4 Indian patents and one international patent. His areas of interest includes: Radar absorbing structures, ultra high temperature ceramics, and nano material added CFRP/GFRPs.

In this paper his contributions are planning the experimental work, executing it, compiling the data and interpreting the results.

Mr P.S.N.S.R. Srikar obtained MSc (Physics) from School of Physical Sciences, Central University of Karnataka, India in 2016. He worked as a project student for a brief period of time in 2016 at Advanced System Laboratories, Hyderabad. His research is centered on developing novel materials to study the microwave absorption of composites at varying frequencies. In this paper, he is involved in the fabrication and testing of the composites.

Mrs Anusha has graduated from Rajiv Gandhi University of Knowledge Technologies, Nuzvid, Andhra Preadesh. Currently associated with Advanced Systems Laboratory, DRDO. Her areas of interest are Polymer chemistry and composite materials. In this paper she contributed in fabrication, testing of composites and compilation data.

Dr Swarup Kumar Majee has completed his MSc (Physics) from Jadavpur University, Kolkata, in 2001 and PhD from Indian Association for the Cultivation of Science (Kolkata) and Presently working as a Scientist 'D' Presently working in non-destructive evaluation of composites structures using radiography, infrared thermography, ultrasonics. In this paper, he contributed towards thermographic studies of impacted composites.

Mr Himanshu Bhusan Baskey received MTech in RF & Microwave Engineering from the Indian Institute of Technology, Kanpur, India, in 2013. Currently, he is working towards the PhD, on radar cross section reduction using metamaterials/frequency selective surfaces and various dielectric and magnetic materials. Presently working as a scientist in the Defence Materials Stores Research Development Establishment, Kanpur, India. He is mainly involved in the electromagnetic characterisation of various types of advanced composites, nanocomposites, artificial dielectrics, electromagnetic absorbers, and radar cross section measurements for the defence stealth applications. In this paper, he contributed towards electromagnetic property evaluation of composites.

Mr P. Rama Subba Reddy is working as a Scientist in Armour Technology Center (ATC) at DMRL. His area of research includes: Development of composite armour materials and its ballistic evaluation. His specific contributions in present work is characterisation of composites using low velocity impact tester.

Dr P. Ghosal, received PhD in Metallurgical Engineering from IIT, Banaras Hindu University, in 1996. Currently, working as, Head, Electron Microscopy Group in DMRL, Hyderabad in the area of advanced characterisation techniques, along with SEM, EBSD, FIB, TEM, HRTEM and Raman-AFM systems. Current interests, includes in-situ mechanical testing and in-situ heating experiments of nano and advanced materials inside electron microscopes. In this paper, he contributed towards SEM characterisation of raw materials, fractured composites.