Effect of microcapsules addition on impact properties of carbon fiber reinforced polymer and glass fiber reinforced polymer composites

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Abstract: Fiber reinforced composites are ubiquitous in the aerospace and automobile industries due to their significant superior characteristics such as strength to weight ratio, corrosion resistance, fatigue resistance, specific strength and specific modulus. Impact is a common kind of damage found in aircraft and automobile structures. The cause of damage may be a sudden strike from a bird, falling of tool during servicing or striking of debris during transportation. Hence, predicting the behavior of composites under impact is necessary and also some mechanism to repair these damages should be found. So, this work aims at determining the effect of inclusion of microcapsules containing healing agents on impact behavior of Fiber Reinforced Polymer (FRP) composite. The resin stuffed and hardener stuffed microcapsules were prepared by in-situ polymerization. These capsules of diameter ranging from 0.19 µm to 0.30 µm were mixed in epoxy resin system during the manufacturing of FRP laminates. Experimental investigation was performed under ambient conditions to observe the variation in impact properties of Carbon Fiber Reinforced Polymer (CFRP) and Glass Fiber Reinforced Polymer (GFRP) composites. It was observed that value of peak load was boosted with addition of capsules and more energy was absorbed during elastic deformation and less during plastic deformation. Also, partial healing was observed due to addition of capsules at 2.5 wt. %.

Keywords: self-healing, low velocity impact, composites, healing agents, microcapsules.

1. Introduction

FRP composites are applied in structures of many components that require use of advanced materials to satiate the demand of superiority in multiple properties through a single material. Such components are from industries like marine, civil, aerospace etc. The FRP composite materials are formed by combination of polymer matrix and fiber reinforcements. The matrix transfers the load to reinforcements and reinforcements aligned in various directions carry load in corresponding directions [1-5]. The structures made from such materials are anisotropic in nature, so damage detection, its prediction and its repair are highly complicated. It not only requires skilled professionals and advanced machineries but also demands a lot of time and effort.

Damage occurring due to impact by any foreign matter in case of composite structures can lead to their failure that might be or might not be visible [6-8]. Parts with detected damages can be repaired or replaced. But if the damage remains undetected, it can result in catastrophic failure of structure and that can be a threat to the personnel working around the structure. So, the behavior of composites under impact
has been investigated by many researchers. Impact energy and specimen thickness plays a critical role in composites subjected to impact. Composite undergoes lower damage at lower impact energy and damage increases with the increase in impact energy. Plates with higher thickness have higher resistance to impact and they also redistribute the loads better than plates with lower thickness [9]. Many factors like fiber volume fraction, exposure temperature and exposure time can also influence impact resistance. Due to variation in fiber volume fraction and exposure temperature, the increase or decrease in impact strength of composite mainly depends on the source of failure i.e. fiber or matrix [10]. Many researchers included additives like CNTs, CNF and MCWNTs in required amount in the matrix system to improve the impact resistance of the composites [11, 12, 13]. Addition of nanofillers in the resin system requires proper dispersion to provide better properties. Methods like ultrasonication can be used for obtaining such fine dispersion [14]. The inclusion of Shape Memory Alloy wires in basalt fiber reinforced composite improved the impact resistance due to hybridization [15].

Most of the research is focused on improving impact resistance. But once the damage has occurred, providing permanent solutions is necessary. Traditional way of detecting damages and repairing them requires a lot of time and skill and is also a costly affair. So, many researchers focused on finding new solutions like implementation of self-healing technology in composites. Such materials can restore their original properties by healing themselves. Self-healing can be included in FRP composites through various techniques. These techniques can be subdivided into two groups i.e. intrinsic and extrinsic healing. Intrinsic self-healing completes by reversibility of cross-linking of bonds whereas extrinsic self-healing uses some capsules or vascular network that breaks by damage and as a result, releases the encapsulated healing agents [16-18]. Inclusion of such capsules can modify the properties of both epoxy system as well as composite materials. Such capsules containing healing agent and curing agent when dispersed into the resin system to produce glass fiber epoxy composite subjected to damage caused by indentation resulted in 80% recovery of the indented zone [19]. This work has been done to understand the change in behavior of CFRP and GFRP laminate under impact load upon inclusion of microcapsules in them during their fabrication. Laminates without microcapsules were also prepared for easy comparison to notice the change. The damage behavior of all the laminates was observed visually by capturing the images after damage.

2. Experimental work details

2.1 Materials Used and Methods of formation

Plain weave fabrics made up of Toray T300 Carbon fibers and E-glass fibers, having fibers woven in two directions 0º and 90º were used along with DGEBA epoxy (LY 556) and amine hardener (HY951) for this study. Materials applied in microcapsule synthesis were melamine, ethylene maleic anhydride, formaldehyde, ammonium chloride, styrene, citric acid, sodium hydroxide and resorcinol. Fabrication of composite laminates with microcapsule infusion to it was procured in the following three steps.

2.1.1 Formation of resin and hardener filled microcapsules

Both resin filled capsules and hardener filled capsules were prepared by in-situ polymerization in a similar way as described in (17) with minute modifications. Borosilicate glass beaker of 2000 ml capacity was used for synthesis of these capsules. Firstly, 31.25 g of melamine and 67.75 g of formaldehyde were poured in a 200 ml beaker and kept in an hot air oven where melamine and formaldehyde mixture was heated at a constant temperature of 70 ºC for half an hour in order to form the cross links between melamine and formaldehyde. The pH of the mixture was kept between 9-10 to maintain the basic state while heating, which was achieved by addition of triethanolamine. After 30 minutes the mixture was removed from hot air oven and cooled at atmospheric temperature of 32 ºC. For encapsulation of DGEBA based epoxy, 600 ml distilled water was poured in 2000 ml beaker and 200 g of DGEBA based epoxy, 1.5
g of ammonium chloride, 1.5 g of resorcinol and 6 g of styrene were added to it. The mixture was stirred with low shear propeller for 5 minutes at 500 rpm. Melamine formaldehyde blend was added to it and mixture was continuously stirred in ambient conditions for one hour at 800 rpm. The formed capsules were washed, filtered and dried at 60 °C in hot air oven. In the similar way, hardener was filled in melamine formaldehyde capsules. The hardener filled capsules were dipped in catalyst overnight so that catalyst permeates through shell walls. Hardener capsules were rinsed and dried at room temperature. Both the capsules were dried for 48 hours. Both the resin and hardener filled capsules formed were white in colour and are shown in Figure 1.

![Figure 1](image)

(a) Resin Microcapsules (b) Hardener microcapsules

2.1.2 Preparation of resin system
After successful drying of capsules, the capsules were mixed in weight percentage of 2.5% in epoxy resin through continuous stirring at room temperature with mechanical stirring equipment at 400 rpm until the capsules were mixed in the resin properly. Both resin filled capsules and hardener filled capsules were added in ratio of 50:50.

2.1.3 Fabrication of Carbon fiber/epoxy and Glass fiber/epoxy Composite Laminate
Both Carbon fiber (Toray T300) reinforced epoxy laminate and E-glass fiber reinforced epoxy laminate were created by Hand Lay-up Process. Fabrication was done on a granite table and surface was cleaned with a paint thinner solution to remove any dirt or grease present to ensure that the surface was clean and even. A square area of 400 mm * 400 mm was marked on the surface. Two 100 micron Mylar sheets were cut into same dimension (400 mm * 400 mm) and kept aside. The 5000 mm * 5000 mm plain weave T300 carbon fabric was cut into dimension of 350 mm*350 mm to obtain 12 layers of same dimension from it. One Mylar sheet was placed on the surface. The surface of the Mylar sheet was cleaned and wax was applied. Wax acted as surface release agent that led to easy removal of the laminate after curing and also it provided good surface finish. The LY556 resin was mixed with HY 951 hardener in the ratio 10:1 in a 1000 ml beaker. Once the wax was dried, first layer of resin/hardener mixture was laid on the surface followed by laying of T300 carbon fabrics. The second layer of resin was applied on the surface and a metal roller was used to wet the surface of fibers with resin. In a similar fashion, all 12 layers were applied. On the top layer, a Mylar sheet with waxed surface was attached and rolled out for proper sticking and smooth surface finish. The surface was pressed with heavy metal plates. The laminate was allowed to cure for 24 hours. In the similar way, four laminates were created i.e. Carbon Fiber Reinforced Polymer with Microcapsules (CFRPWC), Carbon Fiber Reinforced Polymer with no Microcapsules.
(CFRPNC), Glass Fiber Reinforced Polymer with Microcapsules (GFRPWC), and Glass Fiber Reinforced Polymer with no Microcapsules (GFRPNC).

2.1.4 Preparation of specimens
The specimens of required dimensions (150mm *100mm) were cut from laminates by using water jet cutting. Water jet cutting with suitable velocity is preferred over conventional methods like cutting by hand saw and advanced methods like cutting by laser to avoid micro-cracks and damage to surface due to dissipation of heat while cutting. Water jet cutting also provides good surface finish.

3. Details of Specimen Testing

3.1 Scanning Electron Microscopy (SEM)
Scanning Electron Microscopy was used to determine the size of the capsule formed. Scanning electron microscope imaged the capsules by using focused beam of electrons of short wavelength, which in turn gave high resolution. The SEM test setup is shown in Figure 2.

Figure 2. SEM test set-up.

Scanning Electron Microscope images of both resin filled capsules and hardener filled capsules are shown in Figure 3. It was observed from the Figure 3 that size of resin filled microcapsules and hardener filled microcapsules ranged from 0.19µm to 0.30µm and 0.21µm to 0.28µm respectively.

Figure 3. SEM image of (a) resin filled microcapsules and (b) hardener filled microcapsules
3.2 Drop weight impact test
As drop weight impact tester can impact the specimens at higher energy than pendulum and can also rupture specimens of different shape and materials, it was used to test fiber reinforced composite laminate specimens. Firstly, the specimen was fixed between the clamps to avoid its movement during the test. Then the parameters like impact velocity, impactor height, impactor mass; test type and testing standard were given as input and values of parameters are shown in Table 1. The impactor was set at the vertical distance of 1593.21 mm from the specimen, so that when it hits the specimen by free fall under gravity, the kinetic energy will be 30 J. The impactor was set at the center of the clamps to get a central impact on the specimen. All the specimens were tested in a similar way. Drop weight impact tester is shown in Figure 4. Impact tests were conducted in accordance with ASTM D5628.

| S.No. | Name of parameter         | Value          |
|-------|---------------------------|----------------|
| 1.    | Impact Velocity           | 5.59 m/s       |
| 2.    | Impact Height             | 1593.21 mm     |
| 3.    | Carriage Mass             | 1.3 Kg         |
| 4.    | Total Mass                | 1.926 Kg       |
| 5.    | Impactor shape            | Hemispherical  |
| 6.    | Impactor diameter         | 12.7 mm        |
| 7.    | Test type                 | Biaxial        |

Figure 4. Drop weight impact test set-up

4. Results and Discussion
This section deals with the results of drop weight impact test of Carbon Fiber Reinforced Polymers and Glass Fiber Reinforced Polymers. The sub-sections scrutinize the impact force response, energy response and deformation response of the CFRP and GFRP laminates. In total, 12 specimens were tested, that were divided into the following four categories (a) CFRPNC-Carbon Fiber Reinforced Polymer with no Capsules (b) CFRPWC- Carbon Fiber Reinforced Polymer with Capsules (c) GFRPNC-
Glass Fiber Reinforced Polymer with no Capsules (d) GFRPWC- Glass Fiber Reinforced Polymer with Capsules. Three specimens from each category were tested under the impact loading at room temperature.

4.1 Impact force response of CFRP and GFRP laminates
Impact force response of carbon fiber reinforced laminates with no capsules (CFRPNC) and with capsules (CFRPWC) is shown in Figure 5. Impact force response of glass fiber reinforced laminates with no capsules (GFRPNC) and with capsules (GFRPWC) is shown in Figure 6. As all the specimens in the corresponding category shown the similar curves, from each category impact force response for one specimen is shown to understand the load Vs time curve.

![Figure 5. Load Vs Time Response of (a) CFRPNC specimen and (b) CFRPWC specimen](image1)

![Figure 6. Load Vs Time Response of (a) GFRPNC specimen (b) GFRPWC specimen](image2)
Load Vs time curves of all carbon fiber reinforced epoxy laminates show three important load points i.e. First damage (FD), Incipient load (IL) and Peak load (PL). The first damage (FD) corresponds to the first drop in load. It is the point where the first damage occurred, but the laminate was able to take more loads and its effect was negligible on load bearing capacity of laminates. Incipient load (IL) is load corresponds to the point where damage appears majorly after which damage propagates very fast, in curves it can be implied by first intense drop in load which is followed by oscillations in the curve. Peak load (PL) is load corresponds to the point where material will not be able to take any more load, in curves it is represented by highest point. The curve did not witness significant oscillations between the points FD and IL. The oscillations were witnessed corresponding to progression of damage in all the specimens following which specimens failed. The incipient load, peak load and load corresponds to first damage are displayed in Table 2.

| Specimen Details | Incipient load (N) | Peak load (N) | First damage load (N) |
|------------------|--------------------|---------------|-----------------------|
| CFRPNC 1         | 5215.89            | 5410.66       | 1463.83               |
| CFRPNC 2         | 4528.69            | 4972.13       | 1201.68               |
| CFRPNC 3         | 4306.97            | 5032.14       | 1194.33               |
| CFRPWC 1         | 3771.66            | 5808.78       | 1515.28               |
| CFRPWC 2         | 3891.70            | 5898.20       | 1287.44               |
| CFRPWC 3         | 3825.56            | 5879.82       | 1409.93               |
| GFRPNC 1         | -                  | 7794.44       | 1509.15               |
| GFRPNC 2         | -                  | 7382.85       | 1255.18               |
| GFRPNC 3         | -                  | 7638.87       | 1277.63               |
| GFRPWC 1         | -                  | 8617.62       | 1602.25               |
| GFRPWC 2         | -                  | 8740.11       | 1821.52               |
| GFRPWC 3         | -                  | 8560.04       | 1640.23               |

It was observed from the obtained values that addition of microcapsules increased the peak load and load where first damage occurred whereas incipient load decreased. Both categories of glass fiber reinforced epoxy laminates behaved differently from the carbon fiber reinforced epoxy laminates. The load Vs time curve shows two important points i.e. first damage (FD) and peak load (PL). The load Vs time curves of GFRPNC and GFRPWC category did not show incipient load point and there were no significant oscillations which proved that the damage growth happened in a stable way. It is clear from the Table 2 that specimens of both GFRPNC and GFRPWC groups have shown higher peak loads as compared to CFRPNC and CFRPWC group of specimens. Like carbon fiber epoxy laminates, peak load and first damage load values of glass fiber epoxy laminates boosted with addition of epoxy and hardener filled microcapsules.

4.2 Load Vs displacement response of CFRP and GFRP laminates.

The load Vs displacement curves obtained during impact test are shown in Figures 7 (a) and 7 (b). As the curves of all the specimens of a category showed the similar characteristics, load Vs displacement curve of one specimen from each category is displayed. Figure 7(a) indicates the load Vs displacement curve for CFRPNC and CFRPWC specimens and Figure 7 (b) depicts the load Vs displacement curve for GFRPNC and GFRPWC specimens. From Figure 7 (a), it is clear that both the curves contain two phases i.e. loading phase and unloading phase. Both the curves of “CFRPNC” and “CFRPWC” are linear till first damage (FD), at first damage there is minor fluctuation of load and also few load fluctuations were observed.
between FD and IL. This behavior shows that, the laminate was deformed continuously with very less damage progression. Once the peak load point was reached, the curve showed higher intensity fluctuations due to severe progression of damage, followed by failure. The unloading phase is different from the loading phase due to dissipation of energy during the impact. It was also observed that CFRPWC specimen has shown initially less displacement as compared to CFRPNC specimen. Due to addition of microcapsules, after the first damage, some capsules broke and delivered healing agents to fill the damage, which in turn reduced the overall damage and corresponding displacement. It can be seen from the Figure 7 (b) that the area between loading and unloading of curve GFRPWC is smaller than curve GFRPNC. This reduction in area corresponds to healing and less displacement. Also, curve GFRPWC shows more smoother profile till peak load than curve GFRPNC and after peak load both the curves show a smooth unloading phase. Like CFRP specimens there were no fluctuations witnessed after peak load. Few fluctuations correspond to stable damage growth.

![Figure 7](image.png)

**Figure 7.** Load Vs Displacement curve for (a) CFRPNC and CFRPWC specimens (b) GFRPNC and GFRPWC specimens

4.3 Energy Vs time response of CFRP and GFRP laminates

Energy Vs Time response of CFRPNC and CFRPWC is shown in Figure 8 (a) and Energy Vs Time response of GFRPNC and GFRPWC specimens is shown in Figure 8 (b). Two types of energy is marked in the curves i.e. Energy absorbed elastically (EEL) and Energy absorbed plastically (EAB). EEL represents elastic energy, elastic energy is the energy absorbed by the specimen during elastic deformation which results from hitting of specimen with impactor. Once the peak value of deformation is reached, this energy is released. When energy applied through impact is higher than EEL, the extra amount of energy is absorbed during plastic deformation and damage occurrence. This extra amount of energy absorbed is represented by EAB. From Figure 8, it is clearly depicted that CFRPWC and GFRPWC specimens shown higher values for EEL and lower values of EAB than CFRPNC and GFRPNC specimens. This shows that by addition of capsules, the energy absorption by elastic deformation is increased and energy absorbed during plastic phase and damage is decreased. The damage and plasticity is reduced by addition of capsules. The detail of EEL and EAB of all the specimens is shown in Table 3.
Figure 8. Energy Vs time curve for (a) CFRPNC and CFRPWC specimens (b) GFRPNC and GFRPWC specimens.

Table 3. Details of energy absorbed elastically (EEL) and energy absorbed plastically (EAB)

| Specimen Details | EEL (J) | EAB (J) |
|------------------|---------|---------|
| CFRPNC 1         | 6.83    | 21.37   |
| CFRPNC 2         | 5.89    | 22.66   |
| CFRPNC 3         | 5.90    | 22.65   |
| CFRPWC 1         | 9.16    | 19.25   |
| CFRPWC 2         | 9.41    | 18.91   |
| CFRPWC 3         | 9.47    | 18.82   |
| GFRPNC 1         | 13.26   | 14.71   |
| GFRPNC 2         | 12.96   | 15.26   |
| GFRPNC 3         | 12.81   | 15.67   |
| GFRPWC 1         | 14.07   | 12.47   |
| GFRPWC 2         | 14.10   | 12.50   |
| GFRPWC 3         | 13.97   | 12.38   |

4.4 Damage and Healing Assessment

The damaged area of the specimens was inspected visually. The region in the vicinity of the damage of CFRPNC and CFRPWC specimens and of GFRPNC and GFRPWC specimens is shown in Figure 9 and Figure 10 respectively. Figure 9 (a), (b) shows the front face of the CFRPNC and CFRPWC specimens, this face came into direct contact with the impactor. It can be seen from the photograph that CFRPNC specimen has shown an indentation along with cracks at the point of impact whereas the CFRPWC specimen has shown four cracks without any indentation. Figure 9 (c) and (d) shows the back face of CFRPNC specimen indicating a pyramidal damage and CFRPWC specimens indicating smooth surface with a single visible vertical crack respectively. This shows that the damage was healed partially. Figure 10 (a) and (b) shows the front face of GFRPNC and GFRPWC specimens. GFRPNC specimen shows less damage on the front face than GFRPWC specimens. Delamination and indentation is more on front face of GFRPWC specimen as compared to GFRPNC specimen. Figure 10 (c) and (d) shows back face of the GFRPNC and GFRPWC specimens. It is clear from the GFRPNC specimen that a pyramidal damage occurred whereas GFRPWC showed a smooth surface with delamination inside the layer (white in color).
As the amount of healing agents delivered at the damage site was less than required, so only partial healing happened.

**Figure 9.** Damage area of (a) front face of CFRPNC (b) front face of CFRPWC (c) back face of CFRPNC (d) back face of CFRPWC
5. Conclusion

Drop weight impact test was executed on Carbon Fiber Reinforced Epoxy Laminates and Glass Fiber Reinforced Epoxy Laminates. Four types of laminates namely CFRPNC, CFRPWC, GFRPNC and GFRPWC were tested. CFRPNC and GFRPNC did not contain any capsules and CFRPWC and GFRPWC contain capsules filled with resin and hardener. From the test results the following points were determined:

- Inclusion of capsules increased peak load in both CFRP and GFRP laminates.
- Inclusion of capsules reduced the energy absorbed during plastic deformation and damage progression whereas energy absorbed during elastic deformation was increased.
- Addition of 2.5 weight % capsules is not enough to heal the damage completely, only partial healing was achieved.

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