A Review on the Methods of Interlaminar Properties Using Nanomaterials to Reinforce Carbon Fiber Epoxy Composites

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Abstract. Carbon fiber reinforced epoxy composites is playing a role in reducing structural weight and costs, saving resources and optimizing processes. However, the low properties of epoxy matrix and the weak interfacial bonding between carbon fiber and epoxy lead to the poor interlaminar performance for carbon fiber or fabric reinforced epoxy composites. The application of nanomaterials is an effective way to improve or enhance the interlaminar properties of composite materials. Therefore, this paper summarized the research on the methods of nanomaterials reinforce resin matrix, and nanomaterials-carbon fibers multi-scale reinforcement to enhance the interlaminar performance, and nanomaterials to distribute the surface of prepreg uniformly, and nanomaterials to promote interfacial binding energy between carbon fiber and epoxy and nanomaterials macrostructure to improve the interlaminar properties of composites.

1. Introduction
Carbon fiber-reinforced epoxy composites in structural components has increased significantly, and its application has become more extensive. However, the composites has insufficient interlaminar properties and low impact damage resistance and has been the focus on the research for a long time. The currently widely used toughening method is to add rubber or thermoplastic resin to the thermosetting matrix. Although the toughness of the resin system can be effectively improved, the additive of toughening components not only increases the chemical complexity of the resin system but may also cause toughness, such as toughening. The compatibility of the agent with the original resin matrix change the process conditions, and impact on the resin modulus, wet/heat performance and other issues. Moreover, the viscosity of the resin system is greatly increased after being toughened and modified by a rubber or a thermoplastic resin, which cannot be applied to a place where the viscosity of the resin is required to be high. The application of nanomaterials to improve or enhance the interlaminar properties of composites is an effective way. Therefore, this article summarizes research on nanomaterials in recent years to enhance the interlaminar properties of carbon fiber epoxy resin composites.

2. Research Progress on the Methods of Improving Interlaminar Performance Utilize the Nanomaterials Reinforce Carbon Fiber Epoxy Resin Composites
This paper reviews the interlaminar properties of nanomaterial strong carbon fiber epoxy resin composite materials in recent years based on five aspects including the resin matrix, the resin-fiber interface, the continuous carbon fiber or fabric, the prepreg and the nanomaterial macrostructures.
Attention was paid to the methods and application forms of nanomaterials. The summary is shown in Figure 1.

![Figure 1. Several applications of nanomaterials in carbon fiber reinforced epoxy composites](image)

### 2.1. Nanomaterials Enhanced or Toughening Resin Properties

It is the most quick and the easiest way to add nanomaterials directly to the epoxy resin matrix by ultra-sonic, high-speed shear dispersion, three-roller grinding etc. The advantages of this method are simple addition[1–3], low process complexity, and easy and fast implementation. However, the nanomaterial is added in such a manner that the particles contents are low[4–6], and tends to cause problems such as a large increase in the viscosity of the resin, a decrease in dispersibility, and a deterioration in processability. The results of other researchers using nanomaterials improving the interlaminar performance of the composites are shown in Table 1.

| Researchers       | Nanomaterials                               | Strengthening and toughening methods                           | Enhanced the interlaminar performance |
|-------------------|---------------------------------------------|----------------------------------------------------------------|----------------------------------------|
| Mirjalili V et al | multi-walled carbon nanotubes               | dispersed in the epoxy resin                                    | the mode I fracture toughness of the composite was increased by 48%, and the Mode II fracture toughness was increased by 143% |
| Li Y T et al      | rod-shaped nano-iron oxides (FeOOH, \( \text{Fe}_2\text{O}_3 \)) | dispersed in the epoxy                                          | SBS shear strength of FeOOH/CF/EP composites is 58.85MPa, which is 41% higher than CF/EP composites. \( G_{\text{IC}} \) and \( G_{\text{II C}} \) are 0.685 kJ/m² and 2.28kJ/m², respectively, which are higher than CF/EP composites, increase by 99% and 29% |
| Liu B Y[9]        | multi-walled carbon nanotubes               | multi-step chemical grafting method                             | the interlaminar shear strength of CF/EP unidirectional composites increased by 7.5%, 9.3%, 9.9%, and 15.3%, respectively. |
| Zhao X F[10]      | carbon nanotubes                            | dispersed in the epoxy                                          | the interlaminar shear performance (ILSS) of the composite material increased from 79MPa to 86.3MPa. |
| Shen X J[11] et al| graphene oxide with magnetic nanoparticles nano-hydroxy aluminum oxide sheet | dispersed in the epoxy and oriented by the magnetic field       | enhance the interlaminar shear properties |
| Liu X[12] et al   | nano-rubber, carbon nanotubes               | dispersed in the epoxy                                          | the interlaminar fracture toughness improved |
| Xu F[13]          |                                              |                                                                | the toughening efficiency of CFRP laminates reaches the highest |
Researchers       Nanomaterials                  Strengthening and toughening methods                  Enhanced the interlaminar performance

Geng L Y[14] et al, Chen Q F[15]       nano-SiO₂ dispersed in the epoxy                  the interlaminar shear strength of composite unidirectional laminates have been increased

Chen Q F[15]       carbon nanotube dispersed in the epoxy                  the composite shear strength reach the 99.2MPa

Wang Q and Liu L[16]       carboxylated multi-walled carbon nanotubes dispersed in the epoxy                  ILSS, G_{II} increased by about 14%, 27%

Zeng Y[17]       silica nanoparticles dispersed in the epoxy                  the interlaminar toughness value is highest in epoxy resin filled with equal amounts of nano-silica and nano rubber

2.2. Nanomaterial-CF Multi-Scale Reinforcement Enhance Interlaminar Properties of Carbon Fiber Epoxy Resin Composites

It is also a research hotspot in recent years to combine the nanomaterials with carbon fibers by physical[18,19] or chemical methods[20–23] to prepare nanomaterials/carbon fiber multi-scale reinforcements to improve the interlaminar properties of composite materials.

Neisiany R E[24] et al produced styrene-acrylonitrile (SAN) nanofibers through electrospinning to improve the out-of-plane and impact properties of conventional carbon fiber/epoxy composites. The prepared SAN nanofibers were directly deposited on the surface of a conventional carbon fiber fabric. Vacuum assisted resin transfer molding (VARTM) is used to make carbon/epoxy composites. The study of the mechanical properties of composites shows that embedding SAN nanofibers in hybrid composites can increase the interlaminar shear strength by 27%.

Kim J J[25] et al firstly grafted 3-aminopropyltriethoxysilane(APS)-functionalized silica nanoparticles (SiO₂-APS) directly onto the surface of CF by covalent bonding Reinforcement (CF-g-SiO₂). SiO₂-APS nanoparticles are evenly distributed on the fiber surface, which can significantly increase surface polarity and roughness. The ILSS and IFSS increases by 53.27% and 40.92%, respectively, compared with untreated composites.

Some other researches onnanomaterials-carbon fibers multi-scale reinforcement to enhance the interlaminar performance are listed in Table 2.

Table 2. A table with other researches on nanomaterial-CF multi-scale reinforcement enhance interlaminar properties of carbon fiber epoxy resin composites

| Researchers | Nanomaterial         | nanomaterial-CF multi-scale reinforcement method | Enhanced the interlaminar performance |
|-------------|----------------------|--------------------------------------------------|--------------------------------------|
| Wang Z[26]  et al | nano-Al₂O₃          | sprayed the modified nano-Al₂O₃ on the surface of unidirectional carbon fiber | mode II fracture toughness is 557J/m² and 162.5kJ/m² the interlaminar shear strength will be reduced if CNTs toolong |
| Liang X[27] | carbon nanotubes     | carbon nanotubes grown on carbon fiber fabrics |                                        |
| Yao H W[28] | carbon nanotubes     | electrophoretic deposition method to deposit CNTs on the surface of carbon fibers | The interfacial shear strength, interlaminar shear strength increased by 33.0%, 10.5% |
| Qiang Q[29] | carbon nanofibers    | a precursor of polyacrylonitrile nanofiber-carbon fiber multi-scale reinforcement is subjected to high-temperature carbonization treatment to get a reinforced carbon fiber-carbon nanofiber multi-scale preform. | The shear strength has been improved by 30% to 50% |
| Xu F[13]    | carbon nanotubes     | grow carbon nanotubes on carbon fibers          | the growth of carbon nanotubes on the fiber can effectively improve the interfacial strength the interlaminar shear strength of the composite prepared after vapor deposition at 500° for 10 min was improved by 11.0% and 26.5% |
| Fan W X[30] | carbon nanotubes     | grow carbon nanotubes on carbon fibers          |                                        |
2.3. Interlaminar Properties OF Nano-Materials Dispersed on the Surface of Prepreg to Strengthen Carbon Fiber Epoxy Resin Composites

Nanomaterials are currently added to the surface of prepreg composed of fibers and resin. The nanomaterial is filled between the fibers or layers due to the resin flow in the curing process. This is also a way of improving composites interlaminar properties. The method of adding nanomaterials to the resin to prepare the prepreg is essentially the content discussed in section 2.1 of this article and is beyond the scope of this section.

This method can effectively make the nanomaterials distribute between fibers or layers, but this research is still relatively rare and has not attracted enough attention. These studies are summarized in Table 3.

| Researchers     | Nanomaterial                                           | The methods of nano-materials on the surface of prepreg                                                                 | Enhanced the interlaminar performance                                                                 |
|-----------------|--------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------|
| Xue YH[31]      | amino-grafted multi-walled carbon nanotubes/(PEI)      | the amino-grafted multi-walled carbon nanotubes (MWCNTs)/polyetherimide (PEI) electrospun nanofiber films                | improve the impact toughness, the interlaminar fracture toughness of the composite.                |
| Fan YJ[32]      | carbon nanotubes                                       | adding carbon nanotubes (CNTs) to the resin and spraying CNTs on the surface of the prepregs                          | directly adding CNTs to the resin on the interlaminar fracture toughness of the CNTs-CF/epoxy composites is lower than that of CNTs sprayed on the surface of CF/epoxy prepregs |
| Zhang H[33] et al | carbon nanotube                                        | spraying carbon nanotubes (CNTs) onto carbon fiber prepregs                                                            | When the CNT loading is 0.047 wt.%, The mode I fracture toughness is improved by about 50%.         |
| Mujika F[34] et al | carbon nanotubes                                       | Use spray gun to make carbon nanotube onto carbon fiber prepregs to improve the interlaminar performance              | 22% maximum increase in initial fracture toughness.                                               |
| Chaudhry M S[35] et al | carbon nanotubes                                    | uniform distribution of carbon nanotubes on the surface of carbon fiber prepregs                                         | When the CNT load is 1.0g/m2, the fracture toughness is improved by 32%.                             |

2.4. Improving Interlaminar Performance of Carbon Fiber Epoxy Composites by Enhancing Interfacial Performance through Nanomaterial

Some researchers have loaded nanomaterials chemically or physically on the fiber surface or added them to carbon fiber sizing agents to improve the interfacial properties between resins and carbon fibers, there by enhencing the interlaminary properties of composite materials.

Wang B C[36] et al used hexamethylene diisocyanate (HDI) as a coupling agent to chemically graft carboxylated carbon nanotubes on the surface of activated carbon fibers by oxygen plasma, and prepared carbon nanotubes/carbon fibers/epoxy resin by a molding process. The interlaminar shear strength of carbon fiber composites with chemically grafted carbon nanotubes on the surface is increased by 32.1% compared with the untreated carbon fiber composites.

Li N[37] firstly prepared carboxylated multi-walled carbon nanotubes (MWCNTs) by using ‘Friedel-Crafts’ acylation reaction and mixed them with epoxy resin and acetone to make sizing agents containing MWCNTs and then prepared carbon nanotube/carbon fiber multi-scale reinforcing fiber impregnated with the sizing agent carbon fiber. Compared with unimpregnated carbon fibers, the ILSS of carbon fiber/epoxy resin composites impregnated with the carbon nanotube sizing agent increased by 34.33%. Other research results about are shown in Table 4.
Table 4. A table with researches on nanomaterials to promote interfacial binding energy between carbon fiber and epoxy

| Researchers      | Nanomaterial                                      | The methods of nanomaterials to promote interfacial binding energy                                      | Enhanced the interlaminar performance |
|------------------|---------------------------------------------------|--------------------------------------------------------------------------------------------------------|---------------------------------------|
| Wei Y R[38]      | Fe$_2$O$_3$/graphene nanosheets                   | magnetron coating and magnetron grafting on the surface of carbon fibers                              | the interfacial shear strength IFSS value increased by 20.43%. |
| Liu L[39] et al  | multi-walled carbon nanotubes                     | prepared multi-walled carbon nanotubes (MWN'Ts) grids with different thicknesses by using positive pressure filtration method | porous MWN'Ts grids increase the GIIC and ILSS of the composite by about 69% and 24% |
| Wu H M[40]       | SiO$_2$/polyethylene glycol shear thickening liquid | SiO$_2$/polyethylene glycol shear thickening liquid as a surface treatment agent                      | the interlaminar shear strength increased by 49.61% and 58.54%, respectively |
| Deng X[41]       | carbon nanotubes                                  | added carbon nanotubes to the sizing agent to size T800 carbon fibers while coating the carbon nanotubes with fiber surfaces physical coating method to modify carbon fibers and coated silanized nano-microcrystalline cellulose on the surface of carbon fibers | the interfacial shear strength from 50.31 MPa without carbon nanotubes to 62.35 MPa. |
| Zhang M J[42]    | carbon nanotubes                                  | used as an infiltrating agent to impregnate carbon fibers and dried                                   | The interfacial shear strength of the carbon fiber/epoxy composite is 79.3% higher than that of the untreated carbon fiber composite |

2.5. Preparation Carbon Fiber Reinforced Epoxy Composites Containing Nano-Materials

Macroscopic Inserted between Carbon Fiber Layers to Enhance Interlaminar Performance

The nanocomposites is prepared into a macroscopic nano-fiber film[44] or nanomaterials is directly prepared into a macroscopic film form[46], which were placed as an interlayer between carbon fibers or carbon fiber fabrics to improve the interlayer performance of the carbon fiber epoxy composite material.

Hamer S[47] et al fabricated laboratory-scale carbon/epoxy laminates which were interwoven with electrospin nylon 66 nanometer fibrils and reinforced with multi-walled carbon nanotubes (MWCNTs). Compared with non-staggered laminates, nanofibril interleaving results in mode I fracture energy fibers increased 3-fold, while the interleaving MWCNT reinforcement fiber nanofibrils leads to an increase of 4 times. Evaluation of mode II fracture energy indicated that the interweaving of nano fibrillar fibers increased by 40%, while the interweaving of MWCNT-reinforced nano fibrillar fibers increased by 60%.

Lee S H[48] et al coated non-woven carbon paper (NWCT) with conventional carbon fibers and carbon nanotubes (CNT) to form a multi-scale reinforced interlaced layer that can be directly incorporated into the laminate of a composite process. The average $G_{IC}$ of the CNT-enhanced NWCT specimens was 353% higher than that of CFRP specimens, which was a significant improvement over NWCT specimens that were 5% lower than CFRP specimens. The average $G_{IC}$ of carbon nanotube-reinforced NWCT samples was 246% higher than that of CFRP samples, which were a significant improvement compared to the 194% increase in NWCT samples. The increase in $G_{IC}$ is mainly achieved through the crack bridging provided by the NWCT carbon fiber morphology.

In addition, some achievements in recent years are summarized in Table 5.
Table 5. A table with researches on nanomaterial macrostructure to improve the interlaminar properties of composites

| Researchers | Nanomaterial | The methods of nanomaterials to promote interfacial binding energy | Enhanced the interlaminar performance |
|-------------|--------------|---------------------------------------------------------------|--------------------------------------|
| Fang Q[49] et al | polyetherimide (PEI) nanofiber membranes | electrospinning to prepare polyetherimide (PEI) nanofiber membranes and applied them to interlaminar toughening as interlaminar modification material for applying liquid molding resin transfer molding process to prepare composites. | $G_k$ and $G_{IIk}$ of composite laminates increased by 45.0% and 48.9%, respectively |
| Liu G[50] et al | carbon nanotube film | using a high-voltage electrostatic spinning method | The mode II interlaminar fracture toughness increased from 1292J/m$^2$ of carbon fiber/epoxy composite to 2869J/m$^2$, an increase of 120% |
| Xue L W[51] et al | Nano-polyetherimide (PEI) nanofiber oriented film | by using the bubble electrospinning process | $G_k$ increased by 114.55% |
| Yang R R[52] | nano-fiber membranes | interlaminar toughening using the spray coating method and the pulling film method | the $G_{IIc}$ and $G_{IIIc}$ of the composite increased by 21% and 42% |
| Deng H Y[53] et al | carbon nanotube films | | |

3. Conclusions

The nano-material reinforced interlaminar performance method of carbon fiber epoxy resin composite materials has achieved the following progress.

(1) Nanomaterials enhance toughening resin properties. This method is the most straightforward and traditional addition method. The advantages of this method are easy to add, low process complexity, easy and fast implementation. However, the nano-material is added in such a manner that the content is low, and the nanoparticles are directly added to the resin, which tends to cause problems such as a large increase in the viscosity of the resin, a decrease in dispersibility, and a deterioration in processability. Nevertheless, this method still occupies an important research position, but there have also been some innovations in recent years, such as liquefying or activating of nano materials.

(2) The preparation of nano materials-CF multi-scale reinforcements to improve the interlaminar properties of carbon fiber epoxy resin composites is one of the current research hotspots. From recent years of research, the research on carbon nanotube-carbon fiber multi-scale reinforcements still occupies an important research position.

(3) Adding nanomaterials to prepregs composed of fibers and resin, resin flow during the resin curing process will induce nanomaterials to form dispersed nano-reinforced phases between carbon fibers or fabric layers, which is a relatively new research method. However, the areal density of nanomaterials on prepregs should be optimized, not the higher the better.

(4) The main methods of using nanomaterials to improve the interfacial properties of carbon fiber epoxy resin composite materials include physically or chemically grafting nanomaterials on the surface of carbon fibers or adding nanomaterials to carbon fiber as sizing agents and coating on the surface of carbon fibers to form a reinforced interface layer.

(5) The interlaminar performance of the carbon fiber epoxy composites can be effectively improved by using an intercalation layer containing a macromaterial of nano-materials to improve the interlaminar performance. The nano-structural macroscopic insert layer can be summarized as a tough nano-fiber insert layer and a rigid nano-fiber one. Tough nano-fiber inserts are mainly thermoplastic materials, including nanofiber films such as polyethersulfone, polyamide, and nylon. The rigid nano-fiber insertion layer is mainly composed of carbon nanotube film. Both of them can well improve the interlaminar mechanical properties of carbon fiber reinforced epoxy composites.
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