Research Article

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A comparative experimental study on damping properties of epoxy nanocomposite beams reinforced with carbon nanotubes and graphene nanoplatelets

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Abstract: Carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) are extremely ideal nanofillers for applications in damping polymer. This work explores the damping behavior of polymer nanocomposite beams made of epoxy resin reinforced with CNTs and GNPs experimentally. Beam specimens for the vibration tests together with dynamic mechanical analysis (DMA) are fabricated with different weight ratios of CNTs and GNPs, upon which DMA and free vibration tests are conducted. Scanning electron microscope images are also obtained to check the dispersion of nanofillers in microscale. It is found that the first-order loss factor of composite beam specimens shows a rise of 41.1% at 0.4 wt% CNT content compared with that of pure epoxy, while the first-order loss factor of composite beam specimens with 0.025 wt% GNP content increases up by 128.9%. The maximum value of the first-order loss factor of nanocomposite beams with GNP reinforcement is 62.2% higher than that with CNTs.

Keywords: CNT/GNP reinforcement, epoxy resin, damping characteristics, dynamic mechanical analysis, vibration test, scanning electron microscope

1 Introduction

Carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) have gained a lot of interest as high-performance composites in industries and academies. Ever since the discovery of CNTs in 1991, their exceedingly high aspect ratio, stiffness, elastic modulus, impressive strength, and low density make them greatly favorable reinforced materials for polymer matrices [1–3]. However, potential application of these one-dimensional nanoparticles is severely limited by the weak interfaces of CNTs and the polymer matrices. Additionally, their high aspect ratio results in aggregation of CNTs. To minimize the aggregation effect, high dispersive energies are required to properly disperse CNT into polymer matrices. This results in low thresholds of nanofiller concentration and failure of high aspect ratio, and finally limits the mechanical properties of composites. On account of the huge specific surface area and ultrahigh mechanical strength, GNPs exhibit desired application characteristics in the polymers [4–6]. Furthermore, surface modifications can enhance the dispersion of GNPs and realize the strong interface between GNPs and matrix, which improve the mechanical strength and Young’s modulus of nanocomposites [7,8]. As for the matrix, epoxy resin is a promising candidate considering its chemical resistance, specific strength, high stiffness, and dimensional stability [9–11]. By the addition of CNTs/GNPs, the mechanical properties of epoxy resin-based nanocomposites can be further improved. Hence, CNT/GNP-reinforced polymer nanocomposites have been widely used for the noise reduction and vibration control in various engineering sectors [12–16].

In the last decade, the damping properties of polymer nanocomposites reinforced by CNTs have been extensively studied both theoretically and experimentally [17–30]. Zhou et al. [23] investigated the damping ratio and loss factor of CNT-reinforced polymer nanocomposites considering
interfacial friction between CNTs and the resin. Experiments were carried out to verify the theoretical results. Khan et al. [24] explored the vibration damping properties of polymer composites reinforced by multiwall CNTs and carbon fiber (CF) through dynamic mechanical analysis (DMA), and free and forced vibration tests. DeValve and Pitchumani [25] experimentally studied the damping effects of CF and CNT-reinforced composites via vibration tests of cantilever beams in both the fixed frame and rotating frame. The experimental results revealed that 2 wt% of CNTs can increase the damping by more than 130% in a stationary composite beam. For the composite beam rotating at 500 rpm, it could increase more than 150%. Bandarian et al. [26] studied the acoustic damping behavior of open-cell flexible polyurethane foams reinforced with multiwall CNTs, and found that the acoustic damping behavior improved significantly by a small content of nanotubes.

Recently, researchers also carried out experimental studies to explore the damping performances of nanocomposites reinforced by GNP [31–34]. Chen et al. [31] investigated the surface energy and damping behaviors of GNP-reinforced polymer composites experimentally, and reported that GNP reinforcement could enhance the damping properties of petroleum resin and ethylene propylene rubber composites. The damping capability of recycled mortar reinforced with GNP was evaluated by DMA in ref. [32], where the recycled mortar with GNP exhibited a higher damping capability than the recycled mortar without GNP. Lu et al. [33] investigated GNP-reinforced sandwich polyurethane composites via DMA. It was found that in comparison with the baseline polyurethane beam, the dynamic and quasi-static damping properties of the composites with exceedingly low content of GNP markedly increased by 94 and 71%, respectively. The damping effects in multiscale GNPs/ fiberglass/epoxy composites were investigated via vibration tests by Rafee et al. [34], where it was observed that the damping ratio increased at higher contents of GNP nanoparticles.

It is found from literature review that some experimental studies have been working on the damping characteristics of polymer nanocomposites reinforced by CNTs and GNP separately; little research has been conducted to compare the damping behaviors of polymer nanocomposites reinforced by CNTs/GNPs. In this work, the damping performances of epoxy nanocomposite beams reinforced by CNT/GNP are experimentally investigated. Specimens of vibration tests and DMA beams initially are fabricated by epoxy reinforced by CNTs/GNPs. Then, DMA is carried out on CNT/GNP-reinforced epoxy nanocomposite beam specimens to determine the frequency/thermal-dependent damping behavior. Following DMA, vibration tests are conducted to investigate the damping behavior of nanocomposite beams associated with the first three modes. Microstructure imaging of scanning electron microscope (SEM) is also employed to reveal the CNT/GNP dispersion and explain the damping behaviors observed in vibration tests. Finally, based on those measured data, comparison investigations are performed to assess the damping effects of GNP and CNT reinforcements on epoxy resin.

2 Fabrication

2.1 Materials

E51 epoxy resin YT-CC301HhPqc is used as the matrix for this work, while multiwall CNTs JCGMT-999-25-20-COOH prepared via the process of chemical vapor deposition under 2,800°C temperature and GNPs JCG-1-150n-COOH produced by chemical reduction are used as the filling materials. According to its manufacturers, the mixing weight rate between curing agent and epoxy resin is 1:3. Tables 1–3, respectively, exhibit the material properties of CNTs/GNPs together with mechanical performances of the epoxy resin.

2.2 Specimens’ fabrication

The generation of polymer nanocomposite beam specimens reinforced by CNTs/GNPs is exhibited in Figure 1. The epoxy matrix is added with GNPs and multiwall CNTs at certain amounts of weight ratio. A magnetic stirrer with a power of 20 W is utilized to stir the mixture for 30 min, which is then sonicated for half an hour by using ultrasonic disperser with a power of 600 W. Afterward, an
epoxy resin curing agent is added at a weight ratio of 3/1 of the resin to curing agent. The mixture is stirred for 10 min through applying a magnetic stirrer and subsequently poured into the polytetrafluoroethylene (PTFE) molds with a size of 30 mm × 10 mm × 2 mm to prepare the specimens for vibration test. Before pouring the mixture of CNTs/GNPs and epoxy, the beam-shaped cavities of PTFE molds are carefully cleaned by absolute ethanol via the ultrasonic disperser, and a mold release agent is applied for 30 min. Ultimately, the molds are stored in a vacuum oven at an ambient temperature of 25°C for 6 h. Repeat the above steps to acquire epoxy composite beams having different weight ratios of CNT/GNP for three-point bending DMA tests. In those specimens, the CNT weight ratios can be adjusted at 0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, and 0.7%, while the weight ratios of GNPs are 0.0125, 0.0375, 0.0625, 0.0875, 0.1, 0.2, 0.3, and 0.4%. Following the same procedure, the vibration test specimens are also produced in a PTFE mold with the size of 200 mm × 15 mm × 3 mm, of which CNT and GNP weight ratios are identical with specimens for DMA tests, and three more specimens with 0.025 wt% GNPs for vibration tests are fabricated to achieve higher damping ratio. Figure 2 reveals the specimens of epoxy beams reinforced by CNTs for the vibration tests and DMA.

3 Experiments

To evaluate the damping effects of CNTs and GNPs on epoxy resin, DMA, vibration tests, and SEM are conducted. Comparison studies are carried out on the test results of epoxy specimens reinforced by CNTs and GNPs.
3.1 DMA

DMA can separate the elastic and viscous responses of materials when simple harmonic strain, for instance sinusoidal strain, is applied. The operating mechanism includes employing sinusoidal deformation and detecting its force, or utilizing sinusoidal force to the sample and testing its deformation, or even employing the constant deformation or force and detecting its relaxation or creep modulus. The tests of three-point bending DMA are performed for the characterization of the frequency-dependent damping behavior of epoxy resin reinforced by CNTs, as revealed in Figure 3. The specimens can be excited under 25°C temperature utilizing the constant displacement amplitude-isothermal-frequency scanning program between 4 and 200 Hz and a constant displacement amplitude-isofrequency-thermal scanning program between 30 and 180°C with a frequency of 20 Hz.

In this work, the Kelvin–Voigt model is adopted to extract the parameters of viscoelasticity, in which the loss and storage moduli $E''$ and $E'$ are, respectively, defined as the stresses needed per unit of viscous strain and elastic strain. Hence, the expressions of $E'$, $E''$, and loss factor $\tan \delta$ are as follows:

$$
E' = \frac{\sigma_0(t)}{\varepsilon(t)} = \frac{\sigma_0}{\varepsilon_0} \cos(\delta),
$$

$$
E'' = \frac{\sigma_{n}(t)}{\dot{\varepsilon}(t)} = \frac{\sigma_0}{\varepsilon_0} \sin(\delta),
$$

$$
\tan \delta = \frac{E''}{E'},
$$

where $\varepsilon(t)$, $\dot{\varepsilon}(t)$, and $\varepsilon_0$ are the applied strain, strain rate, and strain amplitude, respectively; $\sigma_0(t)$, $\sigma_0$, and $\sigma_{n}(t)$ are the stress in phase with the strain, the stress amplitude, and the stress out of phase with the strain, respectively; $\omega$ is the frequency; $\delta$ is the phase angle in the frequency domain. The obtained storage and loss moduli, respectively, represent the responses of the spring and dashpot in the model of Kelvin–Voigt. The loss factor $\tan \delta$ represents the ratio of applied strain energy distribution between the viscous and elastic components.

3.2 Vibration tests

As exhibited in Figure 4, the vibration tests are implemented at 25°C to determine the vibration response for the composite beam specimens being fixed at one end with 180 mm free length. The specimens for cantilever beam are excited via the impact hammer, and at the free end, the beam response is tracked through the portable digital vibration meter. The signals of laser vibration meter and impact hammer are acquired by LMS system. The resolution is 0.125 Hz and the frequency range is between 0 and 2,048 Hz.

Three specimens are prepared and tested for different weight ratios of CNTs/GNPs, and each test with certain specimen and loading conditions is repeated three times to ensure the accuracy of measurement. LMS Test Lab is employed to analyze the determined frequency response functions (FRFs) to obtain the damping ratio and natural frequency related to different modes. The damping ratio is obtained from FRFs via half-power bandwidth approach, as exhibited below:

$$
\zeta = \frac{\Delta f}{2f_n},
$$
where $\zeta$ represents the damping ratio, $\Delta f$ is the frequency difference at 3 dB points around the natural frequencies, and $f_n$ represents the nth order natural frequency.

### 3.3 SEM

For SEM imaging, the samples with a length of 15 mm, a width of 3 mm, and a thickness of 3 mm are cut from the beam specimens for vibration tests. Then, the specimens are cleaned via oscillation in ethyl alcohol using an ultrasonic disperser and plated gold powder for 300 s to increase conductivity before imaging. The standard metal stubs are mounted with fractured specimen for imaging. Thermionic source emission microscope is applied to acquire SEM images. With the aim of achieving imaging with high resolution, the acceleration voltage is maintained below 2 keV in order to minimize the electron beam interaction volume in sample.
4 Results and discussion

4.1 DMA results

The variations of loss factors for the epoxy nanocomposite beam specimens reinforced by CNTs/GNPs along with excitation temperature and frequency are acquired via DMA as reflected in Figures 5–8, respectively. Table 4 lists the glass transition temperature for both pure epoxy and CNT/GNP-reinforced epoxy materials. The DMA outcomes in Figure 5 and Table 4 suggest that the glass transition temperature of neat epoxy specimens is almost 90°C. For specimens with 0.1, 0.3, 0.4, 0.5, 0.6, and 0.7 wt% CNT content, the glass transition temperature changes little; whereas, the glass transition temperature of specimens reinforced by 0.2 wt% CNT content decreases to 82°C with the decrement of loss factor at glass transition temperature. It can be seen in Figure 6 and Table 4 that as for GNP-reinforced epoxy nanocomposites, the glass transition temperature decreases with little GNP content. To be specific, the glass transition temperatures of specimens with 0.0125, 0.0375, 0.0625, and 0.0875 wt% GNP content are close to 80°C. However, as the weight ratio of GNPs continues to increase and becomes higher than 0.1 wt%, the glass transition temperatures return back to 88°C. Compared with pure epoxy, the loss factor of specimens at glass transition temperature with little GNP

Figure 5: Temperature-dependent loss factor of CNT-reinforced composites by DMA tests.

Figure 6: Temperature-dependent loss factor of GNP-reinforced composites by DMA tests.

Figure 7: Frequency-dependent loss factor of CNT-reinforced composites by DMA tests.
content is increased, while the increment of loss factor at glass transition temperature with high weight fractions of GNP is insignificant. Based on Figures 5 and 6, when the temperature becomes higher than 120°C, the loss factors with a large amount of GNP content show a sudden increase, which indicates that the epoxy is getting to melt. However, for composites incorporating with little GNP content or CNT content, the loss factor decreases smoothly when the temperature increases higher than the glass transition temperature.

The frequency-dependent loss factors of CNT/GNP-reinforced epoxy nanocomposites are illustrated in Figures 7 and 8, where no loss factor values are observed in the frequency region from 50 to 80 Hz due to the fact that the beam samples are in the resonant state, and the loss factors cannot be measured exactly by DMA tests in this frequency region. According to Figure 7, the highest loss factors are achieved at 0.1 wt% CNTs. When the weight ratio continues to increase, the loss factor starts to reduce and even lower than the loss factor of epoxy resin without reinforcement of CNTs. It is considered that this behavior principally results from CNT aggregation for high weight ratio. Figure 8 shows that the maximum loss factors are achieved at 0.0625 wt% GNP in the frequency range 4–160 Hz, and 0.05 wt% GNP in the frequency range 160–200 Hz. With the increasing weight ratio, the variation trends of loss factors for GNP-reinforced epoxy specimens are consistent with those of specimens with CNT reinforcements, that is, the value of loss factors continue to reduce and is lower than the loss factors of pure epoxy resin. However, the reason for the decrease of loss factors with GNP reinforcement is the lubricative effect of GNP with high weight ratio, which is different from that of CNT reinforcement. In Figures 7 and 8, with the increasing frequency of excitation, the loss factor values also increase, suggesting that the damping effect of epoxy resin composites reinforced by CNTs/GNP is enhanced.

### 4.2 Vibration test results

Through vibration tests, the damping ratios together with standard deviations of the first three modes of polymer nanocomposites reinforced by CNTs/GNP with different weight ratios of CNTs/GNP are acquired as shown in Figures 9 and 10. It can be observed from Figure 9 that the damping ratios related to the second mode are always less than those in the first mode for CNT-reinforced composites. In the third mode, the damping ratio is higher than that in the first mode when the content of CNT is less

| CNT wt% | Glass transition temperature (°C) | GNP (wt%) | Glass transition temperature (°C) |
|---------|----------------------------------|-----------|----------------------------------|
| 0       | 88.57                            | 0.0125    | 80.52                            |
| 0.1     | 88.38                            | 0.0375    | 80.05                            |
| 0.2     | 81.67                            | 0.0625    | 79.56                            |
| 0.3     | 88.08                            | 0.0875    | 81.11                            |
| 0.4     | 87.84                            | 0.1       | 87.45                            |
| 0.5     | 87.29                            | 0.2       | 89.26                            |
| 0.6     | 88.06                            | 0.3       | 88.63                            |
| 0.7     | 89.59                            | 0.4       | 85.81                            |

**Figure 8:** Frequency-dependent loss factor of GNP-reinforced composites by DMA tests.

**Figure 9:** Damping ratios of the composite beams reinforced by CNT via vibration tests.
than 0.2 wt%; when it increases to 0.2 wt%, the damping ratio is almost the same with that in the first mode; then, as the CNT content continues to increase, the damping ratio is always lower than that in the first mode. For the first three modes, the maximum damping ratios always occur with 0.4 wt% CNT content. As shown in Figure 10, the damping ratios of the first mode are higher than those of the second and third modes for GNP-reinforced composites with the exception of 0.2 wt% GNP content in the second mode and 0.3 wt% GNP content in the third mode. In conclusion, the damping ratios related to the first mode are higher than those in second and third modes in most cases for both CNT- and GNP-reinforced nanocomposites. The standard deviations of damping ratios for GNP-reinforced composites are higher than that for CNT-reinforced composites. Because of the flocculent structure and low density of GNPs, it would be difficult to control the added mass of GNPs in the nanocomposites. Besides, the damping ratios are more sensitive for GNPs as nanofillers than CNTs. To be specific, 0.025 wt% GNP content can increase 128.9% of damping ratio for nanocomposites.

To further demonstrate the damping effects of CNT/GNP reinforcement, for nanocomposites filled with different CNT/GNP contents, the histogram and error bar of the damping ratios related to the first mode are shown in Figures 11 and 12. With the increase of the weight ratio of CNTs, the damping ratio of the first mode increases first and subsequently reduces, which has been approved by the polynomial regression model in ref. [35]. Overall, the damping ratio for the first mode with CNT reinforcement is always higher than that in the pure epoxy resin. Moreover, the damping ratio of 0.4 wt% CNTs to the first mode is 0.591%, which is 41.1% higher than the damping ratio in pure epoxy resin. The aggregation of CNTs in the epoxy resin matrix is obtained at a high weight ratio of CNTs, which can explain the reason why the damping ratio reduces when the weight ratio of CNTs further increases to more than 0.4%. These test outcomes support the view [36] that CNTs dispersed most uniformly with a weight ratio of 0.4%. It is shown in Figure 12 that the damping ratio for the first mode of GNP-reinforced composites has the similar variant trend with that of CNT-reinforced composites, and the damping ratio is always greater than that in the pure epoxy for the first mode.
Furthermore, the maximum damping ratio in the first mode is 0.901% at 0.025 wt% GNP, which shows a significant increase of 128.9% compared with pure epoxy. On the whole, the damping ratio first increases and then decreases with the increase of GNP/CNT content for composites, and there is a specific weight ratio of GNP/CNT content that reaches the maximum damping ratio.

4.3 Microstructure analysis

Figures 13 and 14 show the SEM micrographs of composites with 0.1, 0.4, and 0.6 wt% CNTs and 0.025 and 0.4 wt% GNP, respectively. As can be seen from the micrographs in Figure 13, some of the CNTs are attached on the surface of epoxy, whose longitudinal axis parallels to the fracture surface, and some are embedded CNTs, whose tips are sticking out of the cross section. In short, it can be seen that CNTs are evenly distributed on the fracture surface in Figure 13(c) and (d). Additionally, there are almost no agglomerates of CNTs at 0.4 wt% CNT content. CNT aggregation in the fracture surface for composites with 0.6 wt% CNTs can be observed in Figure 13(e) and (f), in which CNTs have greater waviness than the composites containing 0.1 and 0.4 wt% CNTs. The high level of homogeneous dispersion and distribution is the reason for the strong damping effect during vibration, which explains

![Figure Image]
why the damping ratio at a CNT content of 0.4 wt% is the highest in vibration tests.

It can be seen from the micrographs in Figure 14 that composites with 0.025 wt% GNPs show a rougher and patchier fracture surface than that with 0.4 wt% GNPs. This indicates stronger interactions between GNPs and epoxy surface. GNPs are homogeneously distributed on the fracture surface and form a strong bond with epoxy matrix. In addition, epoxy matrix has good affinity to GNPs, so they tend to exfoliate into epoxy matrices under mechanical loading. Therefore, the strong bond in GNP–epoxy interface and GNP exfoliation lead to high damping ratios. When GNP content is increased, the lubrication between GNP–epoxy interface and GNP–GNP interface tends to cause the decrease of damping ratios. This is the reason for the vibration test results, where the damping ratio at 0.025 wt% GNPs is maximum, and the damping ratio initially increases and then decreases.

5 Conclusion

This article focused on investigating the damping properties of polymer nanocomposite beams reinforced by CNTs/GNPs experimentally. CNT/GNP-reinforced composite beam specimens were fabricated, upon which the vibration tests and DMA were implemented for the exploration of damping mechanism, and the nanocomposite microstructure was confirmed by SEM tests. According to the comparisons of DMA, vibration tests results, and SEM images, the conclusions are listed as follows:

- The loss factors of CNT/GNP-reinforced epoxy beams increase along with the increment of excitation frequency. The glass transition temperature is decreasing by the dispersion of CNTs, but insignificantly affected by GNPs.
- With the increasing weight ratios of GNPs and CNTs, the damping ratios of the first mode increase first and subsequently reduce. The maximum first damping ratio of epoxy nanocomposite beams reinforced by CNTs of 0.4 wt% is 0.591%, which is 41.1% higher than that in pure epoxy. Correspondingly, the maximum value of 0.901% is reached for the first-order damping ratio of epoxy nanocomposite beams reinforced by GNPs when the GNP weight ratio is 0.025 wt%, in contrast to the epoxy resin with no GNPs, the damping ratio is increased by 128.9%.

Figure 14: Microstructure imaging of GNP-reinforced epoxy at 0.025 wt% GNPs at (a) 5,000× and (b) 20,000×; 0.4 wt% GNPs at (c) 5,000× and (d) 20,000×.
• The elastic–viscoelastic mechanism explains the increasing damping behavior of composites reinforced by CNTs. The strong bond between GNPs and epoxy explains the increasing damping behavior of GNP-reinforced composite.

Polymer nanocomposites reinforced by CNTs/GNPs have been extensively applied in different engineering sectors owing to their excellent mechanical properties. The work in this article focuses on the damping effects of CNT/GNP reinforcement, which benefits the design and application of polymer nanocomposites in harsh environment.

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