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# Evaluation of Nanomaterial Approaches to Damping in Epoxy Resin and Carbon Fiber/Epoxy Composite Structures by Dynamic Mechanical Analysis

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## **Abstract**

Vibration mitigation in composite structures has been demonstrated through widely varying methods which include both active and passive damping. Recently, nanomaterials have been investigated as a viable approach to composite vibration damping due to the large surface available to generate energy dissipation through friction. This work evaluates the influence of dispersed nanoparticles on the damping ratio of an epoxy matrix. Limited benefit was observed through dispersion methods, however nanoparticle application as a coating resulting in up to a three-fold increase in damping.

## **Introduction**

Vibration damping is an essential property to study for structural materials used in environments where vibrations and oscillations are present. Such a preview can predict potential material performance issues; such as micro-cracking or a complete material failure. There are several bodies of literature available which describe methods to reduce damping in composite materials. These include: application of a coating, (Ref. 1) incorporation of a viscoelastic material, (Refs. 2 and 3) and variation in composite ply configuration (Ref. 4). Numerous reports also describe nanoparticle dispersion as a viable technique to improve the damping characteristics of a polymer or composite (Ref. 5).

For each method available to increase material damping capability, numerous variables and large test matrices can be envisioned. A useful technique to analyze material damping is through beam vibration testing which yields damping measurements over a range of bending mode resonance frequencies, as described in ASTM Standard E 756-05, "Standard Test Method for Measuring Vibration- Damping Properties of Materials". However, the calculations associated with this measurement are cumbersome and a reliable screening tool would provide valuable insight to down-select candidate materials.

Dynamic mechanical analysis (DMA) is a technique that uses small samples to quickly characterize the viscoelastic damping properties of a material. The technique provides a measure of material loss modulus ( $E''$ ) and the storage modulus ( $E'$ ), respectively describing the energy dissipated and energy

stored. The ratio of these properties ( $E''/E'$ ) is the damping factor,  $\tan \delta$ , thus energy dissipated over energy stored. This value can be tracked by DMA to identify trends and damping mechanisms. DMA uses small rectangular coupons representative of a larger structure. Given the small sample size, factors such as friction between sample and fixture clamps or variation of sample dimensions may have a large impact on the damping data.

The initial portion of this study was conducted to gain confidence in the reproducibility of  $\tan \delta$  characterization by DMA. Epon 862/W epoxy resin was selected as the baseline material. The samples varied in depth, width, and length to show a full spectrum of size; with the purpose of identifying an optimum sample size to use as a screening tool for further beam testing. From the DMA an increase in  $\tan \delta$  could be recognized as greater damping within the material and select samples would be moved forward for full beam vibration tests.

The second component of this study included an investigation of material damping enhancement through nanomaterial based approaches. A series of nanomaterials were either dispersed into the baseline 862/W resin, or applied as a coating. The materials were characterized by DMA to evaluate their influence on damping, and methods showing significant benefit were selected for beam testing.

## **Experimental Procedure**

### **Materials**

The baseline resin was composed of Epon 862 and W curing agent, received from Miller-Stephenson. A mix ratio of 100:26.4, epoxy to curing agent was used.

Nanoparticles included:

- a. Cloisite 30B, purchased from Southern Clay Product, (2 wt% loading)
- b. Graphene, purchased from Vorbeck, Inc., (0.5 wt% loading).
- c. Epoxy functionalized graphite, purchased from Adherent Technologies, Inc., (0.5 wt% loading).
- d. PR19 Carbon nanofiber, purchased from Applied Sciences, Inc., (0.1 to 1.5 wt% loading).
- e. Single Wall Carbon Nanotubes (SWCNT), Double Wall Carbon Nanotubes (DWCNT), and Multiwall Carbon Nanotubes (MWCNT), were purchased from Nanoshel and used in the range from 0.1 to 1.5 wt% loading.

All materials were used as received.

### **Sample Preparation**

#### **Coupons to Evaluate Reproducibility of DMA Damping Measurements**

Four plaques of 862/W epoxy resin were prepared with the amount of material calculated for plaque thicknesses of 1.5, 3.0, and 6.0 mm. These were chosen to portray a range of dimensions for the DMA testing as suggested by ASTM standard for Dynamic Mechanical Properties, ASTM D4065-06.

The resin samples for DMA testing were prepared by stirring Epon 862 epoxy and W curing agent with a spatula until completely mixed. The mixture was transferred to a mold that had been coated with a mold release agent, Figure 1. Once in the mold, the liquid resin was degassed in a vacuum oven at 65 °C for approximately 1.5 hr.

The sample cure profile included preheating a hydraulic press 121 °C, placing the material in the press, and maintaining for 1 hr. No pressure was applied during the low temperature dwell. The temperature was increased to 177 °C over 15 min followed by a 2.5 hr dwell. During this hold, following gelation, a top plate was positioned on the resin to ensure a flat, level top surface. The mold was then slowly cooled to ambient temperature.

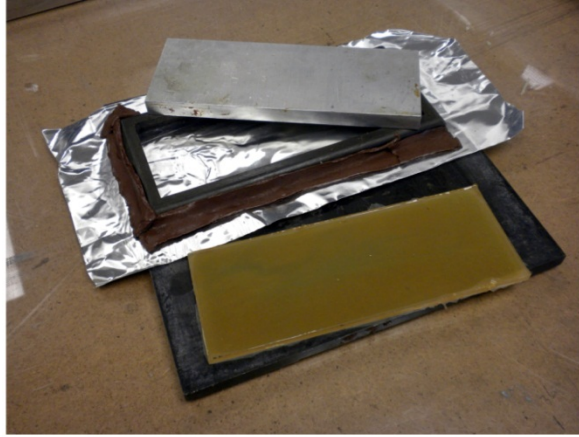


Figure 1.—Mold used to fabricate 862/W resin plaques.

TABLE 1.—SAMPLE DIMENSIONS FOR  
DMA REPRODUCIBILITY STUDY

Sample	Depth	Width	Length
1	1.5 mm (0.03 in.)	9.4 mm (0.38 in.)	50 mm (2.0 in.)
2	3 mm (0.12 in.)	7.6 mm (0.3 in.)	38.1 mm (1.5 in.)
2a	3 mm (0.12 in.)	9.4 mm (0.38 in.)	38.1 mm (1.5 in.)
2b	3 mm (0.12 in.)	9.4 mm (0.38 in.)	50 mm (2.0 in.)
3	6 mm (0.24 in.)	7.6 mm (0.3 in.)	38.1 mm (1.5 in.)

### Coupons to Evaluate Influence of Nanomaterials on Damping

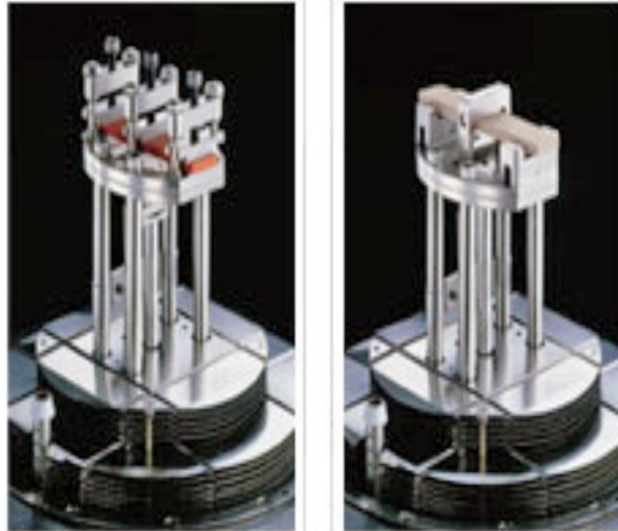
Resin plaques containing dispersed nanoparticles were prepared as 8 by 3 in. sheets of 3 mm thickness. Each nanomaterial was separately added at the earlier specified loading of 862 resin. The materials were mixed in a Thinky Mixer for up to 20 min. The resin was cooled, and W curing agent was added. The mixture was then added to the mold and processed as previously described.

### Testing by DMA

Samples were tested on a TA Instruments 2980 DMA set at a 1 Hz frequency and 20  $\mu$ m amplitude. A 5  $^{\circ}$ C per minute ramp rate was used.

Numerous references site recommended sample dimensions for DMA characterization (Ref. 6). The 862/W samples of varying thickness were cut to create a reasonable range for the DMA characterization. Table 1 displays the dimensions selected from recommendations in ASTM D4065-06 and D5023-07.

Two separate DMA fixtures were used to evaluate damping reproducibility. They included the Single Cantilever Beam (SCB) and Three Point Bend (TPB) fixtures as shown in Figure 2. The SCB fixture clamps the sample on both ends; potentially creating friction which could influence the material damping characteristics. With a small sample size the effect of the friction in the damping could be significant. In the three-point bend test, the samples are not clamped; instead the beam rests on platforms and a preset load is applied to set the sample in place.



Single/dual  
cantilever bending

Three-point  
bend

Figure 2.—Specimen fixture options for DMA.

## Results on Variation in $\tan \delta$ Measured by DMA

The purpose of these tests were to identify the variability of  $\tan \delta$  values as a function of fixture and sample dimension changes. DMA results demonstrated that variation in sample length and width had little to no influence on the  $\tan \delta$  values. The material thickness proved to be the relevant parameter regardless of the fixture used.

### Variations in $\tan \delta$ With Sample Thickness Using a Single Cantilever Beam Fixture

The temperature of interest for the damping value is below 50 °C; ideally room temperature. Within the SCB tests, specimen thicknesses of 1.5 and 3 mm led to low variation in  $\tan \delta$ ; 5 and 0 percent, respectively *within* each thickness set, Figure 3. The variation *between* the 1.5 and 3 mm thick samples was approximately 15 percent.

Increasing thickness to 6 mm increased the variability to almost 30 percent, Figure 4. Of most concern with these samples however was the large variation in  $T_g$ . Again, samples of each thickness were machined from a single stock panel. The 15 °C variation in  $T_g$  was not anticipated.

### Variations in $\tan \delta$ With Sample Thickness Using a Three Point Bend Fixture

There was considerable variation the  $\tan \delta$  values measured using the three point bend test.  $\tan \delta$  values were taken at 50 °C. This again was not anticipated due to the minimal clamping of the sample. Results from these tests are shown in Figure 5. In this case, variation in  $\tan \delta$  value *within* the 3 mm thick sample set was up to 50 percent, as compared to no variation using the single cantilever beam fixture.

According to the ASTM D4065-94 and D5023-94 Standards,  $E'$  data generated, like other DMA property data, are only intended to indicate relative rather than absolute values since the measurements are influenced by experimental conditions and instrumentation compliance; meaning results can vary depending on the instrument used.



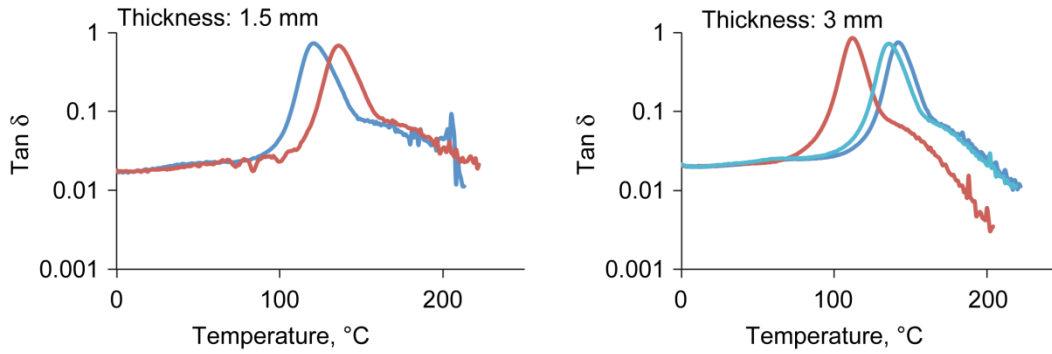


Figure 3.—Tan  $\delta$  plots of 1.5 and 3.0 mm thick epoxy plaques, measured in SCB fixture.

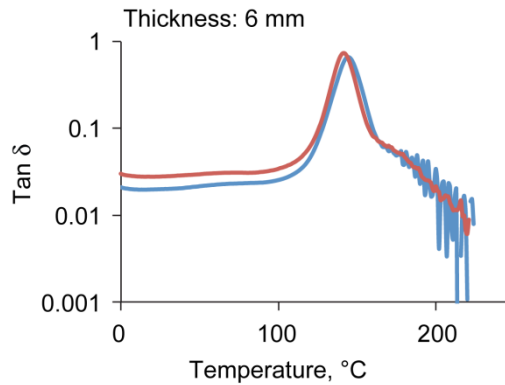


Figure 4.—Tan  $\delta$  plots of 6.0 mm thick epoxy plaques, measured in SCB fixture.

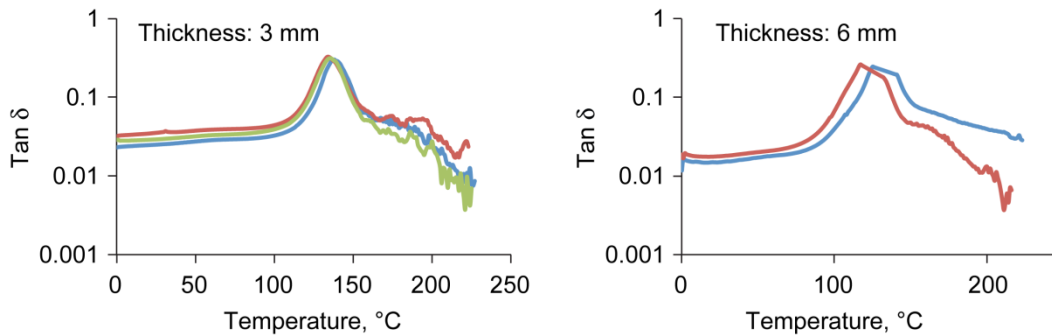


Figure 5.—Tan  $\delta$  plots of 3.0 and 6.0 mm thick epoxy plaques, measured in TPB fixture.

Sullivan and Dykeman have reported that the value for  $E'$  is influenced by the sample span-to-thickness ratio, drive amplitude, and loading clamp used (Ref. 7). Their investigations to determine the parameters for improved repeatability and reproducibility of  $E'$  measurements used the three-point bending clamp. The three point bend mode was selected because the deformation is considered to be more pure than either the single/dual cantilever or tension modes since clamping effects are eliminated.

Within this DMA sample study, a variation in the measured  $\tan \delta$  value was observed within samples ranging from 1.5 to 6 mm in thickness. These measurements also demonstrated variation in  $T_g$ .

While the variation in  $\tan \delta$  data collected with the three point bend fixture was greater, the  $T_g$  was consistent. It was concluded that the three point bend fixture would be used for material screening by DMA. Due to the 50 percent scatter noted in the measured  $\tan \delta$ , initial screening of materials for damping enhancements would require a  $\tan \delta$  increase greater than 50 percent to be considered reasonable for continued characterization by beam testing.

## Results on Damping Through Nanoparticle Dispersion

Nanocomposite samples were prepared as previously described. Each plaque was machined to provide: (a) two beams for vibration tests; 0.75 in. W by 8 in. L, and (b) three beams for dynamic mechanical analysis; 0.3 in. W by 1.5 in. L. Representative images are shown in Figure 6.

Unfortunately, of the nanoparticles investigated, none lead to a greater than 50 percent increase in material damping over the baseline resin.  $\tan \delta$  curves are shown in Figure 7 of nanocomposites composed of 862/W resin and: (a) Cloisite 30B (2 wt% loading), (b) Graphene (0.5 wt% loading), and (c) Epoxy functionalized graphite (0.5 wt% loading).

$\tan \delta$  is calculated by the ratio of  $E''/E'$  (energy dissipated over energy stored). For nanocomposites fabricated from the above listed nanoparticles, a reduction of both  $E'$  and  $E''$  was observed, therefore the ratio of these properties remained approximately unchanged. As a result, the damping coefficient was reduced or maintained relative to the baseline material.

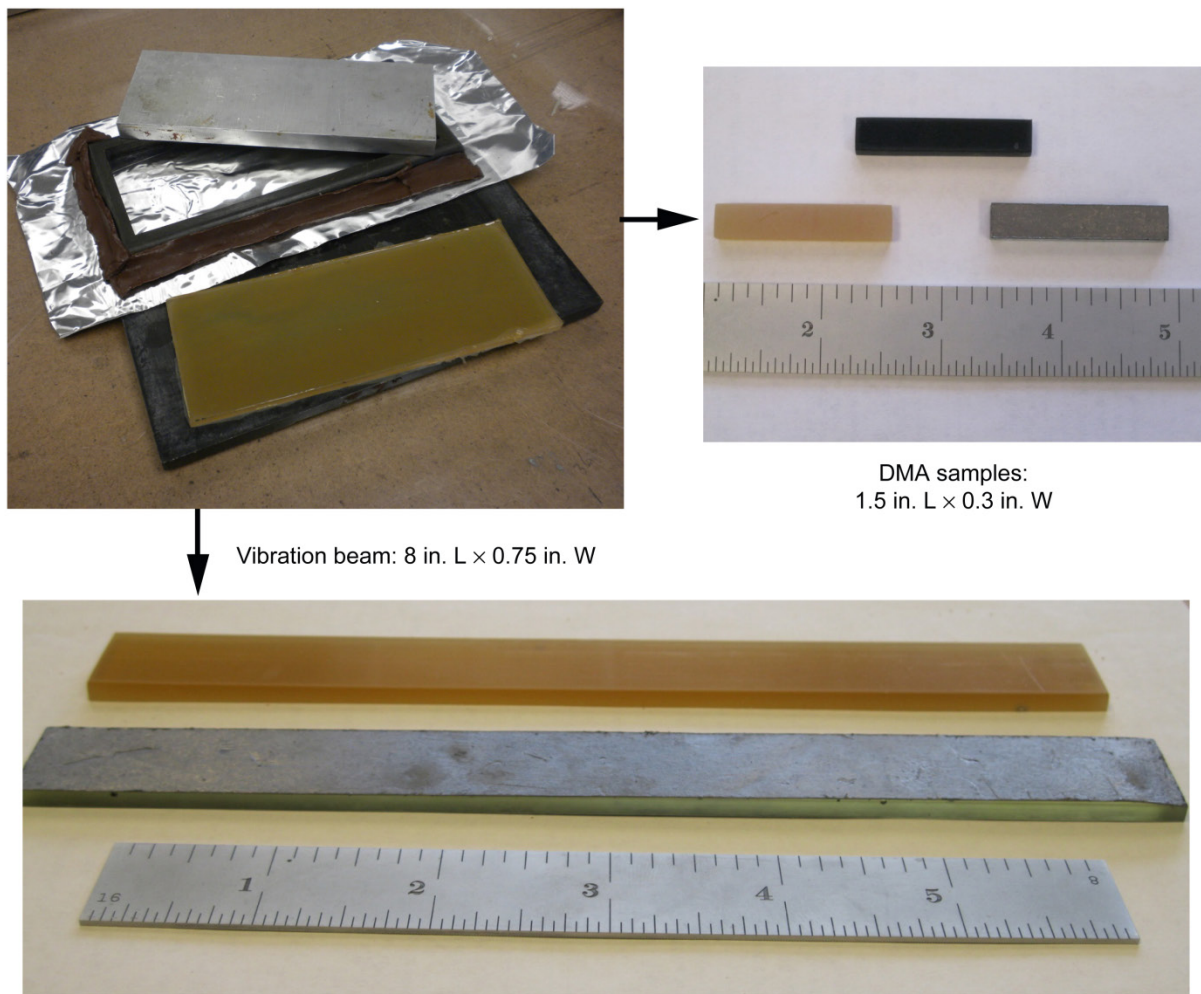


Figure 6.—Photos of parent resin plaque, DMA samples, and samples cut for beam testing.

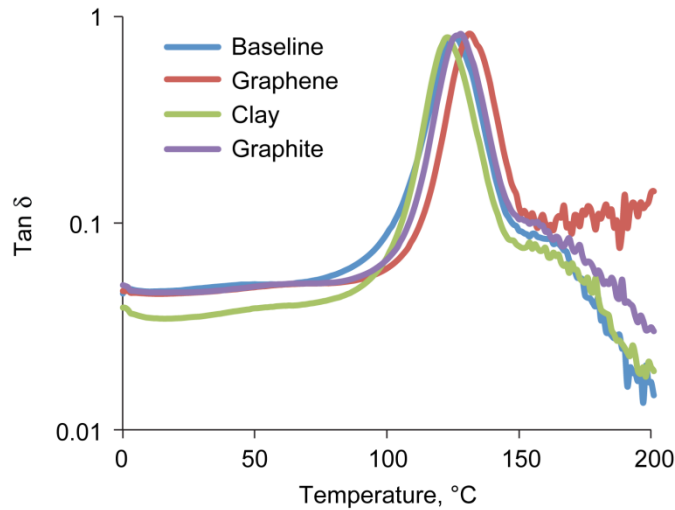


Figure 7.—Tan  $\delta$  plots of nanocomposites prepared by nanoparticle dispersion.

TABLE 2.—DMA RESULTS OF CARBON NANOFIBER AND CARBON NANOTUBE SAMPLES

Material	Loading, wt%	E', MPa	E'', MPa	Tan $\delta$
CNF (PR19)	0	1388	31.5	0.0227
	0.05	1694	31.9	0.0188
	0.5	1533	32.6	0.0208
	1	1909	37.6	0.0197
	1.5	1536	28.9	0.0188
SWCNT	0	1343	34.3	0.0256
	0.05	1693	38.2	0.0226
	0.5	1896	47.6	0.0251
	1	2302	56.6	0.0246
	1.5	1026	26.3	0.0256
DWCNT	0	1343	34.3	0.0256
	0.05	1385	35.6	0.0257
	0.5	1570	36.9	0.0235
	1	1858	46.7	0.0250
	1.5	1213	33.8	0.0279
MWCNT	0	1343	34.3	0.0256
	0.05	1793	38.0	0.0212
	0.5	1808	37.2	0.0206
	1	2111	44.5	0.0211
	1.5	2442	48.42	0.0198

Often, an increase in  $E'$  is observed in nanocomposite materials; due to the higher stiffness of the nanoparticle relative to the polymer matrix. As shown in Table 2, within the CNF nanocomposites, storage modulus was increased, as was loss modulus. To realize an increase in damping however, the increase in  $E''$  would have to more than compensate for the increased  $E'$ . This was not observed in these nanocomposites, and as a result,  $\tan \delta$  was reduced relative to the baseline material.

Within the CNT nanocomposites,  $\tan \delta$  was maintained regardless of nanotube loading or structure (single wall, double wall, multiwall). In all cases, the increase in  $E'$  was accompanied by a comparable increase in loss modulus. As a result, there was not a large change in damping noted by DMA, none of the nanocomposite materials moved forward to beam testing.

The data listed in Table 2 also shows a drop in storage modulus for most materials as loading reached 1.5 wt%. This was attributed to disruption of the crosslinked network due to increased material viscosity at the higher loading (Ref. 8).

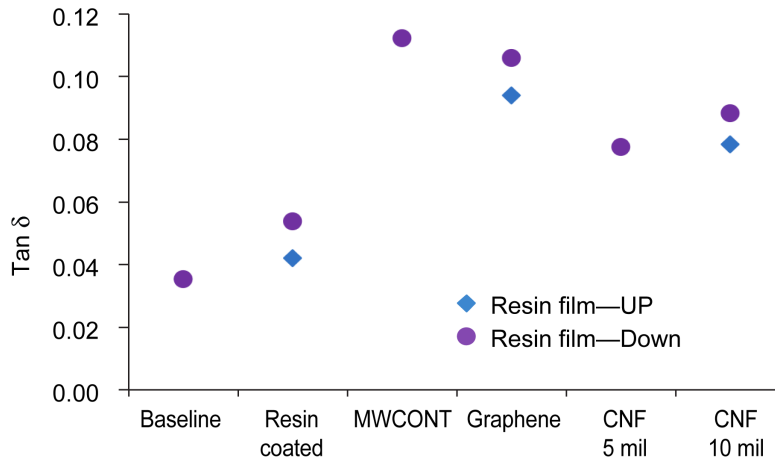


Figure 8.—Measured tan  $\delta$  of baseline composite and coated coupons.

## Results of Damping Enhancement Through Coatings

Nanoparticle based sheets were purchased from both Applied Sciences Inc. and Buckeye Composites. These sheets included: (a) graphene sheet (80 percent graphene, 20 percent MWCNT), (b) multiwall carbon nanotube sheet, and (c) carbon nanofiber sheets; 5 and 10 mil thick. The nanoparticle sheets did not contain a binder material.

Coatings were applied to an IM7/8552-1, unidirectional, 16 ply laminate through co-cure of a resin film which bonded the coating to the composite. A sample coated with solely resin film was fabricated for comparison and composite coupons were sectioned for DMA. DMA was run with samples mounted as either ‘coating up’ in contact with the central fixture, or ‘down’. The coated coupons demonstrated up to a 300 percent increase in damping, Figure 8, with neither the orientation of the sample in the fixture nor the addition of resin influencing tan  $\delta$ . The suspected mechanism is energy dissipation through friction generated as the nanoparticles within the coating slide against neighboring particles. The large interfacial area of the nanoparticles would contribute to the significant increase in energy dissipation.

The primary concern with application of these coatings relates to their thickness, lack of binder, and therefore weak bonding of the outermost nanoparticles. This led to nanoparticle release and potential health concerns. However, this issue may be mitigated through incorporation of a binder or a reduction in coating thickness. A photomicrograph of a graphene sheet bonded to the composite substrate is shown in Figure 9. The resin adhesive partially penetrated the coating, but non-bonded particulate is visible. In addition, voids are visible between the coating and substrate, which would contribute to poor bonding.

## Beam Testing

Beam vibration testing was performed on the coated composites as the DMA results showed an improvement to damping beyond the 50 percent margin of scatter. Beam vibration testing was conducted at around 1500 Hz, according to ASTM E-756-05, “Standard Test Method for Measuring Vibration-Damping Properties of Materials.” The trend in material damping was opposite that measured by DMA. Here we see a reduction in damping ratio in the coated coupons relative to the baseline material. The damping ratio  $\zeta$  shown in Figure 10 is equivalent to  $1/2 \tan \delta$ . The differences among the beams may have been within the range of experimental error, since the damping was so low, and there was only a single beam measured of each type. These values are much lower than those measured by DMA (tan  $\delta$  of  $\sim 0.006$  compared to  $>0.02$  by DMA). Damping can depend on vibration frequency, so it was decided that further DMA testing of material samples at various frequencies should be done.

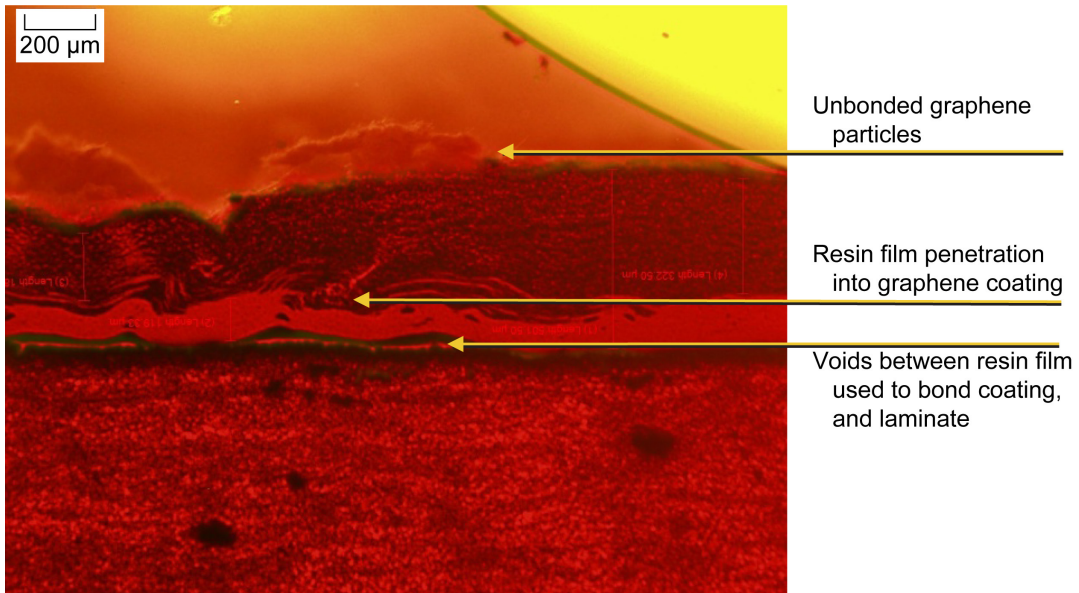


Figure 9.—Graphene sheet bonded to composite with resin film.

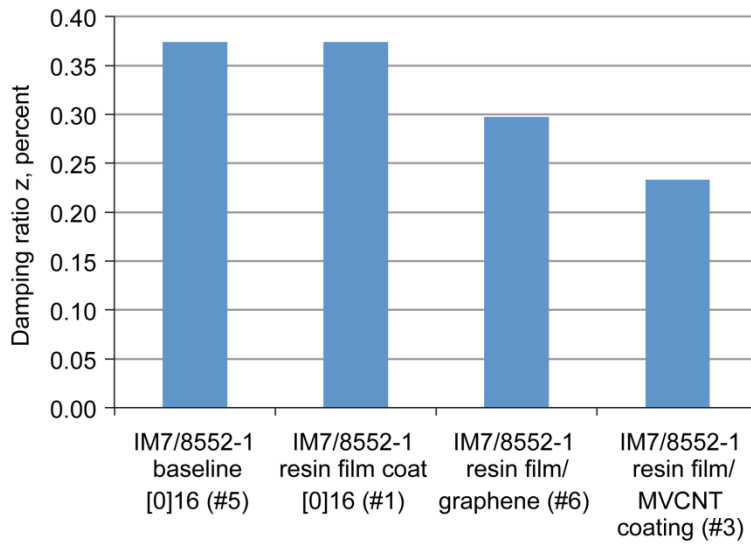


Figure 10.—Beam vibration damping results.

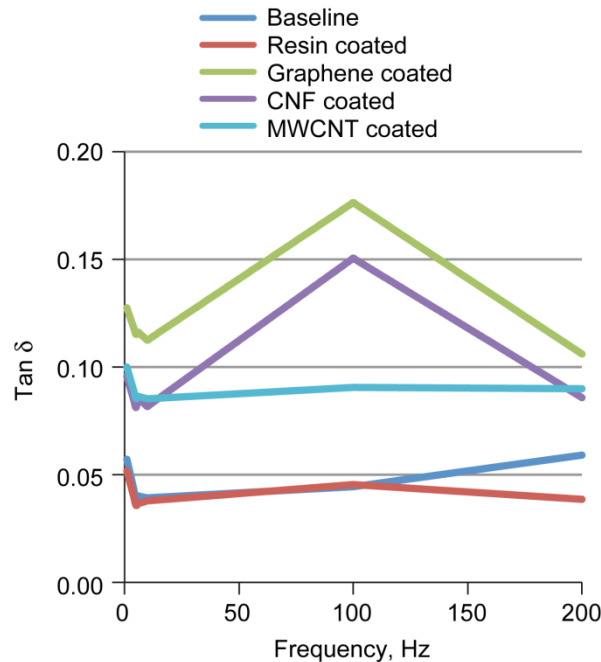


Figure 11.—Tan  $\delta$  of coated composite materials as a function of test frequency.

### Frequency Dependence Study

Based on the variation in results obtained from DMA and beam tests, a frequency dependence study was initiated. The frequency dependence of the coated composite damping performance was characterized by DMA. Frequency sweeps at room temperature provided data on coupon tan  $\delta$  at frequencies ranging from 1 to 200 Hz; Figure 11. At frequencies between 1 and 200 Hz, the damping factor of the coated coupons is greater than that of the baseline material. Near 100 Hz the frequency of the coated samples reaches a maximum and either levels off or declines. The opposite is true of the baseline material, where the damping factor is increased beyond 100 Hz. Elucidation of the mechanisms responsible for the change in damping is beyond the scope of this paper; however the data demonstrates a clear frequency dependence and offers explanation of the reduced damping observed in the beam tests which were conducted at 1500 Hz.

### Summary

DMA was used to evaluate damping in epoxy resin and composites coupons. A study demonstrated that the measured tan  $\delta$  value of samples taken from a single panel varied by up to 35 percent. Following this study, several nanoparticles were dispersed in an epoxy resin, including carbon nanotubes, carbon nanofibers, graphene, and layered silicate clay. A change in tan  $\delta$  greater than 50 percent was necessary to be considered significant. This was not observed following dispersion of any of the nanoparticles, due to off-setting increases in both the material storage and loss moduli. Application of the nanoparticles as a coating did not influence the inherent material properties and demonstrated a significant increase in damping by DMA. This damping increase was diminished as the test frequency was increased.



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