

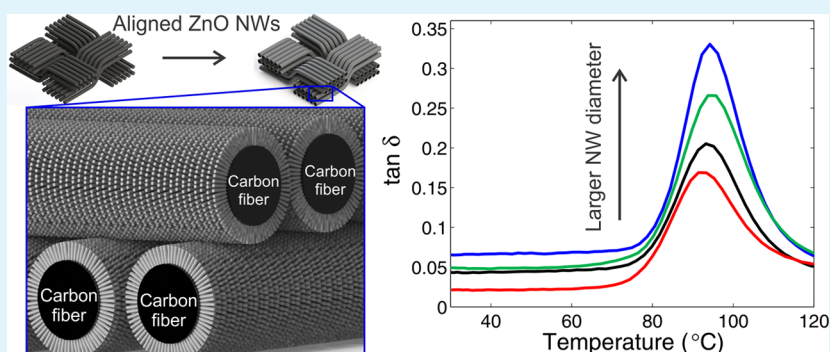
Morphology-Controlled ZnO Nanowire Arrays for Tailored Hybrid Composites with High Damping

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Supporting Information



ABSTRACT: Hybrid fiber reinforced composites using a nanoscale reinforcement of the interface have not reached their optimal performance in practical applications due to their complex design and the challenging assembly of their multiscale components. One promising approach to the fabrication of hybrid composites is the growth of zinc oxide (ZnO) nanowire arrays on the surface of carbon fibers to provide improved interfacial strength and out of plane reinforcement. However, this approach has been demonstrated mainly on fibers and thus still requires complex processing conditions. Here we demonstrate a simple approach to the fabrication of such composites through the growth of the nanowires on the fabric. The fabricated composites with nanostructured graded interphase not only exhibit remarkable damping enhancement but also stiffness improvement. It is demonstrated that these two extremely important properties of the composite can be controlled by tuning the morphology of the ZnO nanowires at the interface. Higher damping and flexural rigidity of these composites over traditional ones offer practical high-performance composites.

KEYWORDS: ZnO nanowire, morphology control, damping, nanostructured interface, hybrid composites

INTRODUCTION

Energy dissipation regulates the dynamic stability and load-bearing properties of composites, which are essential for structural applications in the aerospace, automotive and sports equipment industries. The induced strain energy in a composite material subjected to dynamic loading is absorbed through a variety of damping mechanisms. Regardless of the viscoelastic nature of polymer matrix, which is the main contributing factor in energy dissipation,¹ the interphase region^{2–4} and damaged area⁵ can affect the damping behavior of composites. Recently, it was shown that assembled carbon nanotube (CNT) forests on carbon fiber increase the damping coefficient of continuous fiber reinforced polymer (CFRP) composites by 514% whereas dispersed CNTs show only 53% enhancement.^{6–8} In nanocomposites, it is postulated that the improved damping response relies on increasing the friction area at the interface between the CNTs and matrix phase.^{9–11} Nevertheless, the damping performance of dispersed CNTs within the matrix phase of a polymer based composite is compromised by complex fabrication processes and reduction in stiffness.¹²

Thus, the distribution and alignment process of CNTs during CFRP composite fabrication have not achieved their optimum efficiency yet.¹³ For instance, a problem arises in vacuum assisted resin transfer molding (VARTM) where the CNTs can be unintentionally filtered by becoming trapped between the fiber tows resulting in a solution with too high viscosity to be infused between fibers.¹⁴ Although the concentration and homogeneity of the nanofillers significantly affect the mechanical properties directly, the dispersion of CNTs for concentrations above 1% is very challenging.¹⁴ Thus, the damping enhancement of carbon fiber composites using CNTs, specifically in large scales, appears unrealistic.

Zinc oxide is an environmentally friendly material with piezoelectric and semiconducting properties.^{15–17} Due to the outstanding properties and simple synthesis process of one-dimensional ZnO nanostructures, they have been utilized in the

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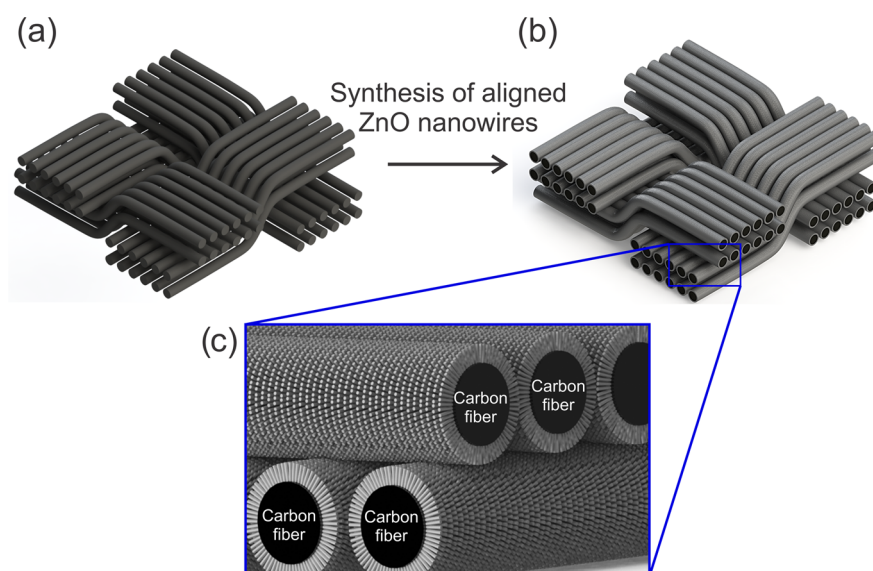


Figure 1. Schematic illustration of the carbon fabric modification for hybrid composites: (a) bare carbon fabric (b) carbon fabric covered by ZnO nanowires (c) close view of vertically aligned ZnO nanowires on carbon fibers.

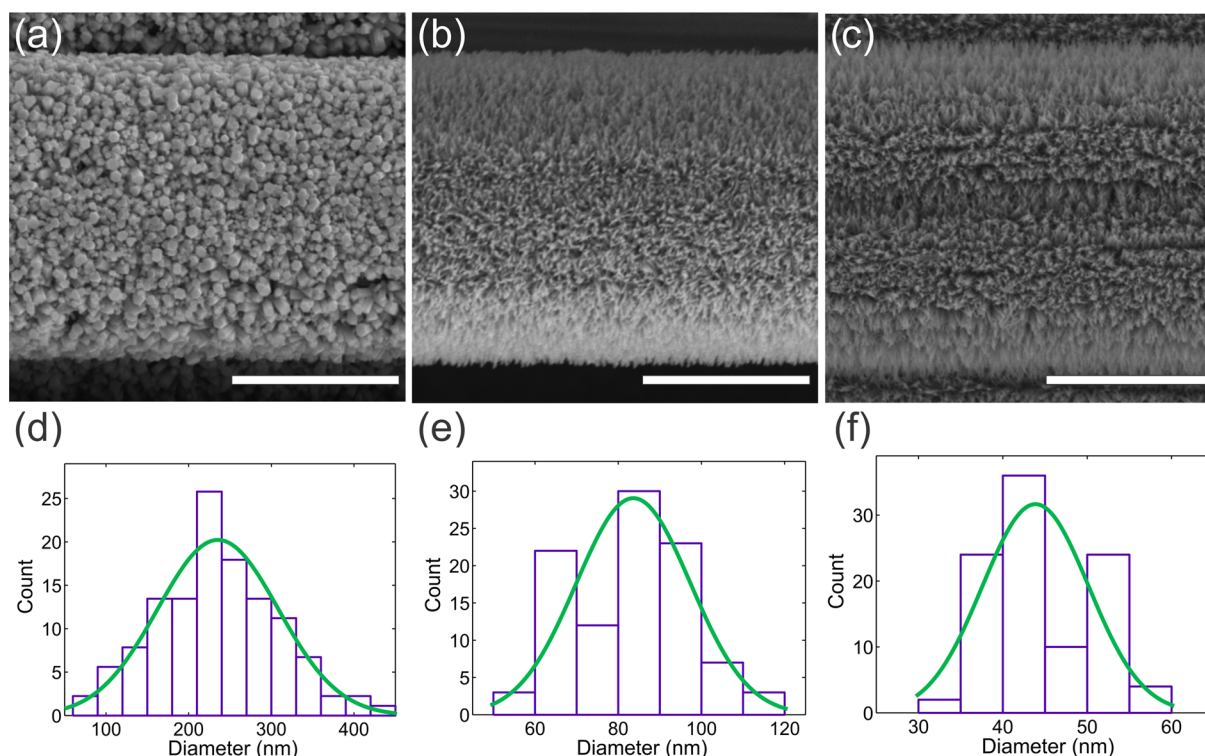


Figure 2. Micrograph of ZnO NW morphology for three different aspect ratios: (a) low AR, (b) intermediate AR and (c) high AR (scale bars are 4 μm). Diameter distribution of the corresponding ZnO nanowires for each case: (d) low AR, (e) intermediate AR and (f) high AR.

fabrication of many functional materials.^{18–20} ZnO nanowires (NWs) have also been used as a whisker interphase to enhance structural properties of CFRP composites.^{21–24} Lin et al. demonstrated that vertically aligned ZnO NWs on the surface of carbon fiber leads to a 113% increase in the interfacial shear strength of CFRP composites.²¹ Later, Galan et al. showed that tuning the ZnO NW interphase significantly affects the interfacial strength of carbon fiber composites.²³ Length and diameter of ZnO nanowires across the interface were considered as design parameters to tailor the interfacial strength. They successfully showed that the interfacial shear

strength can increase up to 3.28 times. The high mechanical interlocking through three-dimensional connectivity of nanowires and matrix allows for better load transfer from the reinforcement phase (carbon fiber) to the epoxy matrix. This nanowire interphase offers a unique load transfer mechanism across the interface region, which is one of the most critical regions for the load bearing properties of the composites. Moreover, utilizing this load transfer mechanism between fiber and matrix might enhance the energy dissipation ability of the CFRP composites. Recently, Skandani et al. showed that a ZnO nanorod interphase resulted in a 50% increase in damping

properties of bilayered composites.²⁵ They also reported a slight decrease in storage modulus while micromechanics modeling of composites with ZnO nanowire graded interphase indicated enhancement in flexural rigidity.²⁶ Because both stiffness and vibration damping are critical factors in composites for structural applications, further efforts are required to improve these properties of hybrid composites. Multiple layers of modified carbon fabrics with vertically aligned ZnO nanowires can be utilized to fabricate laminar composites and study the influence of nanowire interphase between the lamina.

This study aims to demonstrate the advantage of a ZnO nanowire interphase in CFRP composites for enhancing their damping behavior and elastic properties. As shown in Figure 1, carbon fabrics are modified by the radial growth of ZnO nanowires on the surface of carbon fibers. A low temperature (<90 °C) hydrothermal synthesis is employed to avoid damaging the carbon fibers during synthesis. The composites are fabricated by stacking modified woven carbon fabrics followed by epoxy resin infiltration using the VARTM technique. The effect of ZnO nanowire morphology on the amount of dissipated energy in the fabricated composites is shown. Furthermore, the storage modulus of the composites over a large temperature range is measured to confirm no loss in elastic modulus for the entire range.

RESULTS AND DISCUSSION

Tuning the morphology (length and diameter) of the nanowires was conducted by controlling the size of the deposited nanoparticles, reaction time, reaction temperature and level of impurities. For instance, larger ZnO nanoparticles and cadmium ion impurities both lead to larger diameter nanowires. In contrast, to obtain thinner or higher aspect ratio (AR) nanowires, higher concentrations of zinc nitrate hexahydrate, hexamethylene tetramine (HMTA) and ammonium hydroxide were used. Three different sets of reaction parameters were selected to synthesize three different cases of ZnO NWs with low, intermediate and high aspect ratios. The morphology of the nanowires in each case is shown in Figure 2. The synthesized NWs were observed by scanning electron microscopy (SEM) to measure the dimensions of the nanowires and also to ensure the uniformity of the NWs before composite fabrication.

To determine the mean length of nanowires in each case, half the total diameter of the fiber with coated nanowires as measured at 10 locations were subtracted from half the original diameter of the bare carbon fiber and averaged. The mean length of the NWs was kept similar for all morphologies which varied between ~680 nm to ~840 nm. Approximate average nanowire diameter was calculated by averaging the diameter of more than 100 individual NW tips grown on the fibers at high magnification. The average NW diameter varied from ~235 nm in the case of low aspect ratio to ~43 nm in high AR (Table 1). The NWs representing the intermediate AR had a mean

Table 1. Measured Nanowire Diameter and Length for Different Aspect Ratios

nanowire morphology	average diameter (nm)	standard deviation (nm)	average length (nm)
low aspect ratio	235.43	72.96	684.63
intermediate aspect ratio	83.64	13.73	840.63
high aspect ratio	43.87	6.30	737.63

diameter of ~84 nm and an average length of 840 nm. The diameter distribution of the ZnO NWs and the normal Gaussian distribution fitted to the data are illustrated in Figure 2 with the corresponding SEM images of nanowires.

Four different composites using modified carbon fabrics with three distinct nanowire morphologies and one bare carbon fabric as reference were prepared. The same fabrication process was used for hybridized and traditional composites. The cross sectional micrographs of these composites are shown in Figure 3. Figure 3a shows the traditional carbon fiber composite with a

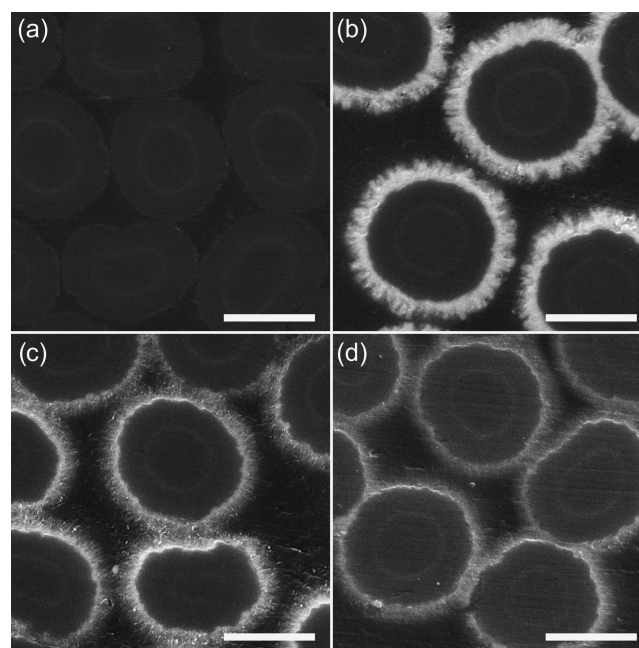


Figure 3. SEM cross-sectional images of (a) bare carbon fiber and the three different morphologies (b) low aspect ratio ZnO NWs (c) intermediate aspect ratio ZnO NWs (d) high aspect ratio ZnO NWs (scale bars are 5 μm).

discrete interface between the fiber and epoxy matrix. The cross-sectional view of the hybrid composites shows that the nanowires are properly wetted by the epoxy matrix in all cases. Defect-free interphases indicate the effectiveness of this fabrication procedure which overcomes the aforementioned challenges in nanofiller dispersion to assemble multiscale composites.

The varying diameters of the nanowires grown on the surface of the carbon fibers resulted in three different interphases between carbon fiber as the reinforcement phase and epoxy as the matrix phase of the hybrid composite. Due to the diameter of the nanowires, a nanoscale material gradient along the radial direction of the carbon fiber was created. This functionally graded variation begins with highest volume of the ZnO phase at the surface of the carbon fiber and gradually reduces up to zero at the matrix phase. Different diameters of nanowires lead to different profiles for the ZnO volume fraction across the interphases. Finally, each ZnO gradient profile results in a unique load transfer mechanism between reinforcement and matrix phases. For instance, the nanowire interphase in the low aspect ratio case contains a lower volume fraction of the epoxy due to the large diameter of NWs in this region as illustrated in Figure 3b. In contrast, the interphase with high aspect ratio is mainly occupied with epoxy rather than the ZnO nanowires

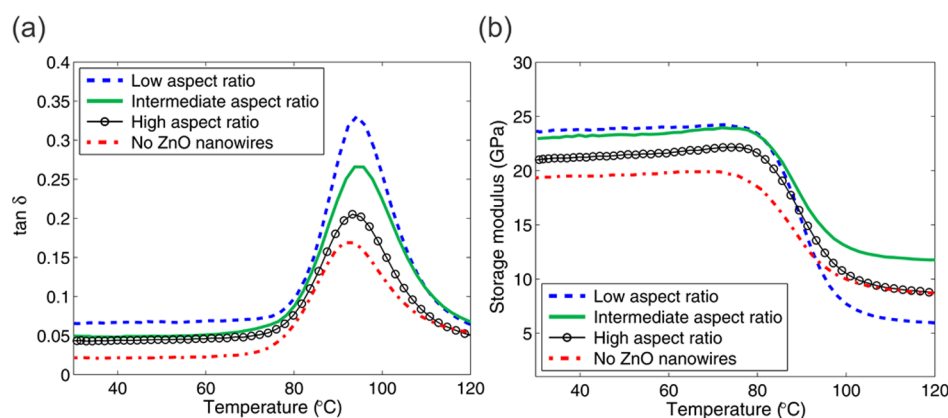


Figure 4. Dynamic mechanical analysis of hybrid composites (a) loss tangent and (b) storage modulus.

because of their small diameter. The difference between interphases due to the altered NW diameters is clearly observable in the micrographs of the hybrid composites in Figure 3. To determine the overall volume fraction of the ZnO phase in the composites thermogravimetric analysis (TGA) was conducted. As anticipated, the composites with low AR nanowires had the highest volume fraction of the ZnO phase ($\sim 2.94\%$) compared to intermediate AR and high AR cases having $\sim 1.48\%$ and $\sim 1.05\%$, respectively.

Although the intrinsic damping behavior of the composites comes from the viscoelastic behavior of the matrix phase, the nature of the interphase between reinforcement and matrix phases can significantly influence the dynamic response of the composites. The interfacial behavior and influence of the microstructures on the stiffness of composites are often characterized using dynamic mechanical analysis (DMA).^{27–30} DMA has also been used to study interfacial damping as well as the quality of the interface in composite materials.^{31,32} DMA testing provides a measure of viscoelastic properties such as the storage (E') and loss (E'') moduli at elevated temperatures. The E' represents the flexural rigidity related to the amount of stored energy whereas the E'' represents the amount of dissipated energy in the materials. The damping loss factor ($\tan \delta$, the ratio of storage modulus to loss modulus) can be determined at a range of temperatures. Thus, the contribution of the nano/micro structures and the adhesion quality between different phases in a composite material can be revealed as a function of temperature using DMA. To study the damping capacity of carbon fiber reinforced composites with the intention of interphase characterization, DMA has been used as an accurate measurement technique.^{1,31,32} Moreover, this technique has also been used to detect the influence of the fiber surface treatment on damping properties of the fiber reinforced composites.^{33,34} Here, the DMA data is utilized to indirectly show the effect of nanowire morphology and the interphase property of the developed multiscale composites. Dynamic mechanical thermal measurements were performed on a minimum of four composite specimens for each case study. The variation of loss factor and storage modulus in the composite samples with temperature in three-point bending mode is shown in Figure 4. At room temperature, the damping ratio of the composites without ZnO nanowires is ~ 0.02 . The composite samples with ZnO nanowires exhibit higher energy dissipation as well as higher rigidity compared to the composites with bare carbon fabric.

The improved damping behavior of hybrid composites is mainly affiliated with the vertically aligned ZnO nanowires at the interface, which creates additional reinforcement in the out-of-plane direction of the laminated composites and a nanoscale graded interphase. It has been shown that oriented fillers in the thickness direction of laminate composites leads to higher energy dissipation.¹³ Besides adding the third dimension to the composite architecture, a high mechanical interlocking between carbon fiber and matrix has been created through the nanowires resulting in larger contact area and consequently higher interfacial slippage as one of the possible energy dissipating mechanisms. On the other hand, the nanoscale material gradient between two phases with dissimilar elastic modulus provides a smooth strain transition from compliant matrix into the rigid carbon fibers. The gradient interphase improves the damping performance in two different aspects. First, the gradient absorbs a large amount of the induced energy before the strain transfers from reinforcement phase into matrix phase and vice versa. Second, the large mismatch in thermal conductivity of the epoxy matrix and carbon fibers is reduced by this material gradient. Better thermal conductivity in this region helps release generated thermal energy at the interface and keeps the high performance across the entire elevated temperature without interface debonding due to the thermal effect (Figure 4a). It is worth mentioning that the discussed damping mechanisms are possible parameters which might contribute in dynamic behavior of these composite. However, still it is not clear which parameters are crucial and have the highest impact on energy absorption of multiscale composites at the nanoscale level.³⁵

The composites with ZnO nanowires exhibit not only higher loss factor but also improved elastic properties over the composites with bare carbon fabric (Figure 4b). The enhanced bulk elastic property of the composites is in agreement with a previous analytical study on overall mechanical properties of carbon fibers coated with ZnO nanowires.²⁶ The total deflection of the midpoint of the composite beam in a three-point bending test consists of the pure bending of each laminate and shearing deformation between lamina. Higher storage modulus of the composites indicates that the superior damping behavior due to the nanowire interphase does not compromise the elastic properties. However, the improved storage modulus, measured in a three-point bending test, might imply an enhancement in the bonding of laminates suggesting that the developed composites have higher resistance to the shear deformation.

The DMA results show that the energy absorption capacity and storage modulus of the developed hybrid composites can be nanoengineered by controlling the morphology of nanowires at the interface (Table 2). Composites with the lowest

Table 2. Variation of Composites Viscoelastic Properties with Aspect Ratio of the ZnO Nanowires

composite	nanowire AR	variation in $\tan \delta$ (%)	variation in storage modulus (%)
low AR case	2.91	209.5	21.9
intermediate AR case	10.05	133.3	18.55
high AR case	16.8	104.7	8.5

nanowire AR exhibit a 209.5% increase (3 times) in $\tan \delta$ whereas composites with the largest AR nanowires showed a 104.7% increase (2 times). For all three different morphological cases, the loss factor increases over the entire temperature range with respect to the composites with bare carbon fabrics. Decreasing the aspect ratio of the nanowires increases the loss factor significantly, indicating the crucial role of the NW morphology on the bulk properties of the hybrid composites.

The observed trend of damping enhancement for composites with different nanowire morphologies does not bear resemblance to that in the stiffness variation. For instance, at room temperature, the storage modulus enhancement of the intermediate AR case is more than twice that of the storage modulus improvement of the high AR case. The large increase that appeared in energy dissipation capacity of the low AR case compared to intermediate AR case cannot be seen in the variation of the storage modulus. These results imply that the changes in dynamic behavior of the composites can be a combination of several consequences due to the existence of the ZnO nanowire at the interface. The nanowires directly contribute to improved overall material properties as the third phase of composites, while they indirectly enhance the mechanical response through improving load transfer and adhesion quality at the interphase region. Another feature that might improve the damping properties without affecting the elastic modulus is the influence of the piezoelectric properties of ZnO nanowires. The piezoelectric phase of the composite converts the induced cyclic stress into electrical charges. These charges are then dissipated as heat through the composite

volume as a resistor. This phenomenon is known as piezoelectric damping and has been used to increase the energy dissipation capacity of CFRP laminates in a similar manner by dispersing piezoelectric particles at interlayers.^{36,37} Thus, several likely features are contributing to the damping improvement of the developed composites and correlating the damping enhancement with storage modulus variation might not be feasible.

As can be seen in Figure 4b, the composites with low AR nanowires demonstrate a sharp drop in storage modulus above the glass transition temperature, whereas the ones with intermediate AR nanowires show a thermally stable behavior over the entire range of temperature change. This observation implies that the morphological differences at the interface might affect the thermal stability of composites above their glass transition temperature. Moreover, in some nanocomposites with polymer matrix, the interaction between nanostructures and matrix can result in a polymer interphase which can lead to enhanced properties compared to the bulk polymer.³⁸ In the case of CFRP composites with ZnO nanowires, the morphology and surface properties of the nanowire might affect and control the polymer structural properties (e.g., orientation, crystallinity and crystal size) of the interphase. Thus, unsatisfactory response of the polymer interphase above T_g can be considered as another possible explanation for the sharp drop in flexural rigidity of composites with low AR at this temperature. On the other hand, the composites with low AR nanowires possess the highest ZnO volume fraction, and according to the literature, similar trends in storage modulus variation of the composites with high concentration of nanofillers have been observed.^{39,40} Damaged interfaces can behave as defects in a composite and can cause a large reduction in its flexural rigidity. To detect any possible damage occurring during the DMA tests, micrographs of the tested composites with low AR were collected. Cross section SEM images from different points of the composite showed no trace of damage or nanowires failing due to the experiment (Figure S1 in the Supporting Information).

The composition of the ZnO phase and material gradient at the interphase region of the developed composites can be controlled by two parameters: length and diameter of the nanowires. The length of nanowires governs the thickness of the interphase region, which is the distance between carbon fiber and pure matrix phase, whereas the nanowire diameter

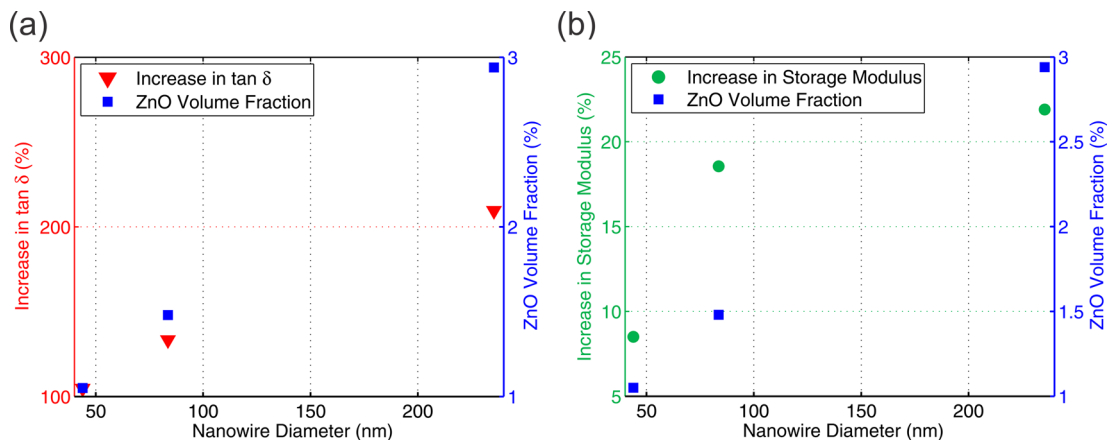


Figure 5. Role of nanowire morphology and ZnO volume fraction on the variation of (a) $\tan \delta$ and (b) storage modulus in the CFRP composites with ZnO nanowires.

dictates the volume occupied by the ZnO phase. However, it was preferred to keep the nanowire lengths similar to those that resulted in a large interfacial shear strength enhancement of CFRP composites through the ZnO nanowire interface.²³ The altered diameter of the nanowires changed the composition and gradient profile of the interphases at the nanoscale level. For instance, the volume fraction of the epoxy in the interphase region of the high AR case is much higher compared to that of the low AR case in which the interphase region is mostly occupied by thick ZnO NWs. Thus, the gradient of the ZnO and epoxy compositions at the interphase changes by altering the spacing between individual nanowires, which is controlled by their aspect ratio. The amount of the exposed nanowires in the cross-sectional micrographs presented in Figure 3 also indicates the differences in volume fractions and gradient of the ZnO phase in each case study. Figure 5a shows the damping performance of the composites as a function of the nanowire diameter and ZnO volume fraction in the developed composites. The loss factor of the composites with large nanowire diameter (~235 nm) and ZnO volume fraction (~3%) increased more than 3 times at room temperature. Figure 5b demonstrates the effect of the nanowires' morphology on variation of storage modulus and volume fraction of ZnO in the carbon fiber composites at room temperature. The flexural rigidity of the composites increases by decreasing the ZnO volume fraction or increasing the nanowires' aspect ratio, assuming that nanowires from different cases have similar lengths. This tailorable graded interphase enables the design of novel hybrid composites with desired energy absorption capacity and improved elastic properties compared to traditional composites with discrete interfaces.

CONCLUSION

Hybrid carbon fiber composites with a ZnO nanowire interphase have been developed, and the role of nanowire morphology on damping characteristics of these composites has been investigated. Direct growth of vertically aligned ZnO nanowires on the fabric rather than incorporation of other nanofillers (e.g., carbon nanotubes) simplified the fabrication process allowing laminated composites to be produced through VARTM. Remarkable enhancement in damping properties along with improvement in flexural rigidity of the composites was achieved due to the novel nanowire interphase between the reinforcement and matrix phase. The aspect ratio of the ZnO nanowires was controlled through the hydrothermal growth parameters by changing the diameter of nanowires while holding the nanowire length constant. Different diameters of ZnO nanowires altered the spacing between the nanowires and consequently the composition profile of the ZnO and epoxy phases at the interface region. The nanoscale graded interphase significantly affected the energy absorption capacity of the hybrid composites. The loss factor of the composites increased more than 200% (3 times) for low aspect ratio ZnO nanowires with respect to composites with bare carbon fabrics. The dynamic mechanical analysis showed that the nanowire interphase plays a crucial role in energy dissipation and mechanical reinforcement of the woven carbon fabric composites over the entire temperature range. Because the morphology of the ZnO nanowires can be accurately controlled, this result suggests a novel, effective method to tune the vibration damping characteristics of the bulk hybrid composites without losing primary flexural rigidity of the composites. Furthermore, the piezoelectricity and semiconduct-

ing effects of ZnO nanowires integrate multifunctional behavior to the composites.

EXPERIMENTAL SECTION

ZnO Nanowire Synthesis. Various methods such as hydrothermal,^{41,42} chemical vapor deposition (CVD)⁴³ and electro-deposition^{44,45} can be used to synthesize ZnO nanowires. Here, ZnO nanowires are grown on the surface of carbon fibers in a plain weave fabric configuration (FG-CARB5750 supplied by U.S. Composites, Inc.) via a two-step process consisting of nanoparticle deposition followed by hydrothermal growth.

In the nanoparticle deposition step, the fabrics are dip-coated in a colloidal suspension of ZnO nanoparticles to deposit layers of the nanoparticles on the fiber surface. These nanoparticles act as ZnO nucleation sites in the hydrothermal growth step. Two different sizes of ZnO nanoparticles in colloidal solution were synthesized following the procedure introduced by Hu et al.⁴⁶ The size of nanoparticles increases with increasing temperature and using solvents with longer chain length. For the synthesis of the smaller nanoparticle solution, separate solutions of 12.5 mM zinc acetate dehydrate (Alfa Aesar, 99.5%) and 20 mM NaOH (EMD Millipore, 99.5%) in ethanol (Decon Laboratories, 100%) were prepared. Then 40 mL of the zinc acetate solution and 40 mL of the NaOH solution were added to 320 and 100 mL of ethanol, respectively. The solutions were heated separately to 65 °C then mixed together under vigorous stirring and heated at 65 °C for 45 min. The larger nanoparticle suspension was prepared by using 2-propanol (Fisher Scientific, >95%) as the solvent, instead of ethanol. Separate solutions of 6.25 mM zinc acetate dehydrate and 10 mM NaOH in 2-propanol were heated at 90 °C for 60 min and cooled to room temperature. Then 80 mL of the zinc acetate solution and 80 mL of the NaOH solution were added to 280 and 60 mL of 2-propanol, respectively. The two diluted solutions were heated separately to 75 °C and then mixed together under vigorous stirring and heated at 75 °C for 90 min. After each dip-coating step, the fabrics were annealed at 150 °C for 10 min three times for proper adhesion between the nanoparticle seed layer and the carbon fabric surface. After the nanoparticle deposition step, a thin and conformal polycrystalline ZnO seed layer covered the surface of the carbon fabric.

In the hydrothermal growth step, the fabrics were placed into a hydrothermal growth solution and kept at 86 °C in a closed container for the desired growth time (2.5–12 h).⁴⁷ For the synthesis of ZnO nanowires having the highest aspect ratio, an aqueous solution of 67 mM $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (zinc nitrate hexahydrate) (Sigma-Aldrich, $\geq 99.0\%$), 34 mM HMTA (Sigma-Aldrich, $\geq 99.0\%$), 7.7 mM polyethylenimine (Sigma-Aldrich, average $M_w \sim 800$) and 0.49 M NH_4OH (Ricca, 28.0–30.0 wt %) was used. ZnO nanowires having an intermediate aspect ratio were synthesized from an aqueous solution of 48 mM $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 24 mM hexamethylenetetramine, 7.7 mM polyethylenimine and 0.49 M NH_4OH . For the synthesis of ZnO nanowires having the lowest aspect ratio, cadmium ion impurities ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, Fisher Scientific, $\geq 98.0\%$) were incorporated into the standard growth solution made without polyethylenimine. This modified growth solution used 4.8 mM or 9.6 mM of $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and 0.39 M of NH_4OH to achieve the desired nanowire morphology.

Fabrication of Multiscale Composites. To fabricate the hybrid composites, six layers of surface-modified carbon fabrics were stacked and infiltrated with epoxy resin using the VARTM technique. Epon 862 and Epikure 3230 with the ratio of 100:35 were mixed to prepare the epoxy matrix. The low viscosity of the polymer wetted the nanowires completely; however, the ZnO nanostructures slightly decreased the penetration rate of the epoxy resin through the carbon fibers compared to bare fabrics. Once the epoxy resin infused into the entire composite, the composite was subjected to a 100 psi (689 kPa) pressure by means of a hot press. After initial hardening of the epoxy matrix at room temperature for 10 h, the samples were cured at 80 °C for 2 h, followed by heating at 125 °C for 3 h. The samples were cooled to room temperature in the hot press. The mass fraction of the carbon fiber and ZnO nanowires were determined by combining the

data from thermogravimetric analysis (TGA; TA Instruments Q50) and ASTM D3171 (2011). A 10 °C min⁻¹ temperature ramp was used to 200 °C, followed by a 5 °C min⁻¹ up to 800 °C. Then, the estimated mass fractions of the components were converted to the volume fraction of the carbon fibers and ZnO nanowires. The estimated volume fraction of the carbon fibers varied between 60% and 65%.

Structural Characterization. The morphologies of the ZnO nanowires grown on the carbon fabric were examined by scanning electron microscopy (TESCAN VEGA3 LM) at an acceleration voltage of 10 kV. The obtained micrographs were utilized to determine the dimensions (length and diameter) of the ZnO nanowires.

Mechanical Characterization. To characterize the viscoelastic properties of the composites and make a comparison between different nanostructured interfaces, DMA (Seiko DMS 110) was conducted in the three point bending configuration with a 20 mm beam span. The specimens were tested under loading frequency of 50 Hz while the temperature was ramped from room temperature to 120 °C at a rate of 2 °C min⁻¹. The DMA specimens were machined from cured polymer composites to beams with a length of 60 mm, width of 10 mm and thickness of ~1 mm.

■ ASSOCIATED CONTENT

📄 Supporting Information

Postmechanical testing micrographs of the composites cross section. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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Notes

The authors declare no competing financial interest.

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