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Design and Reinforcement: Vertically Aligned Carbon Nanotube-Based Sandwich Composites

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ABSTRACT Carbon nanotube (CNT) reinforcement of polymer composites has not yielded optimum results in that the composite properties are typically compromised by poor dispersion and random orientation of CNTs in polymers. Given the short lengths available for nanotubes, opportunities lie in incorporating CNTs with other structural reinforcements such as carbon fibers (CFs) to achieve improvement over existing composite designs. Growth of vertically aligned CNTs (VACNTs) offers new avenues for designing high-performance composites by integrating CFs and nanotubes into layered 3D architectures. To obtain composites with high rigidity and damping, we have designed and fabricated VACNT-based sandwich composites from simply stacking the freestanding VACNTs and CF fabrics and infiltrating with epoxy matrix. Comparing with the CF/epoxy laminates, the VACNT-based sandwich composites exhibit higher flexural rigidity and damping, which is achieved due to the effective integration of the VACNTs as an interfacial layer between the CF stacks. Furthermore, the lighter weight of these VACNT-based sandwich composites offers advantages in aerospace and transportation applications.

KEYWORDS: mechanical reinforcement · structural design · vertical alignment · carbon nanotubes · carbon fibers · sandwich composites

Carbon nanotubes have been regarded as ultimate short fibers on account of their high ratio of length to diameter and extremely high theoretical strength,[1] and they have been anticipated to eventually replace conventional reinforcements such as carbon fibers (CFs), Kevlar, and glass fibers. Although the mechanical properties of CNT-reinforced polymer composites have been studied for more than 10 years,[2,3] the reinforcing effect of CNTs is still far below what has been expected, even much less than that of CFs; this disappointing result is generally attributed to the poor dispersion and random orientation of CNTs in the polymer matrix[4] and has greatly hindered large-scale commercial applications of CNTs. With respect to this situation, the complete replacement of CFs with CNTs seems unrealistic in the short term,[5] while a feasible approach is to combine CFs with CNTs by taking full advantage of both components in unique ways for a synergistic result. It has been shown that adding CNTs into a CF/epoxy (CF/Ep) laminate composite can considerably improve interlaminar shear strength and impact toughness due to the remarkable CNT reinforcement and rich interfaces of the CNT-based composites,[6−8] while controlling the dispersion and alignment of CNTs in the interlaminar resin is still a great challenge for practical applications.

Fortunately, growth of vertically aligned CNTs (VACNTs) offers new avenues for designing high-performance composites by integrating CFs and nanotubes into layered 3D architectures. The mechanical enhancements of the interlaminar shear strength and fracture toughness have been achieved by introducing VACNTs into laminate composites *via* directly growing,[9] transfer-printing,[10,11] and bonding techniques,[12] but the manufacturing procedures of these composites are extremely complicated. On the other hand, high flexural rigidity and high vibration damping are two critical requirements in aerospace and automotive industries for ensuring a large load-bearing capacity and a long fatigue life of composites.[13] Theoretical and experimental efforts on improving these two attributes have been intensively carried out for several decades,[14] and theoretical analyses predicted as early as in 1985 that the composites would exhibit the maximum damping once short fibers were oriented along the through-thickness direction (*z*-direction) of laminate composites;[15] however, it has been extremely difficult to precisely control the alignment of short fibers, especially in the *z*-direction of the laminate composites. Now, the availability of freestanding VACNTs opens a door for simply creating such a proposed design to obtain

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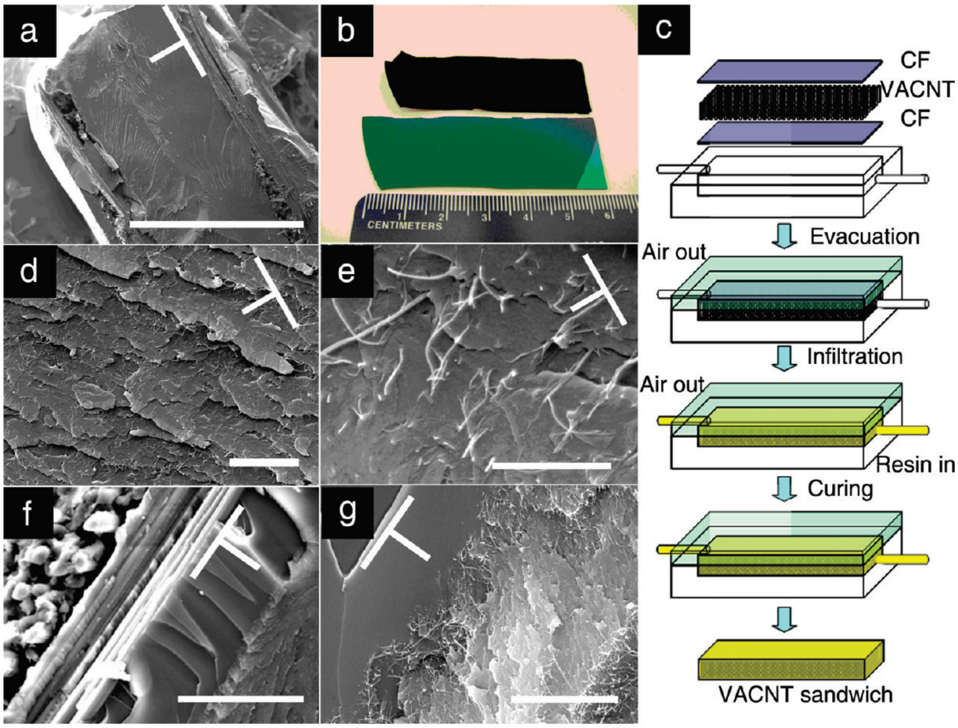


Figure 1. Structures and fabrication of the VACNT sandwich. (a) SEM image of the VACNT sandwich at a low magnification (scale bar, 1 mm). (b) The freestanding VACNTs and their substrate are about 50 mm long and 20 mm wide. (c) The VACNT sandwich was fabricated by using a VARTM technique, which consists of stacking a CF/VACNT/CF sandwich structure, evacuating air, infiltrating resin, and curing procedures. (d) The VACNT sandwich shows a good infiltration of VACNTs with epoxy (scale bar, 5 μm). (e) The VACNT sandwich retains a good alignment of CNTs along the through-thickness direction (scale bar, 1 μm). (f) Rough fracture surfaces in CNT-rich regions imply a tough fracture of the composites compared with smooth fracture surfaces of neat epoxy (scale bar, 50 μm). (g) The VACNT sandwich exhibits a good infiltration of VACNTs with epoxy and a good alignment of the CNTs (scale bar, 5 μm).

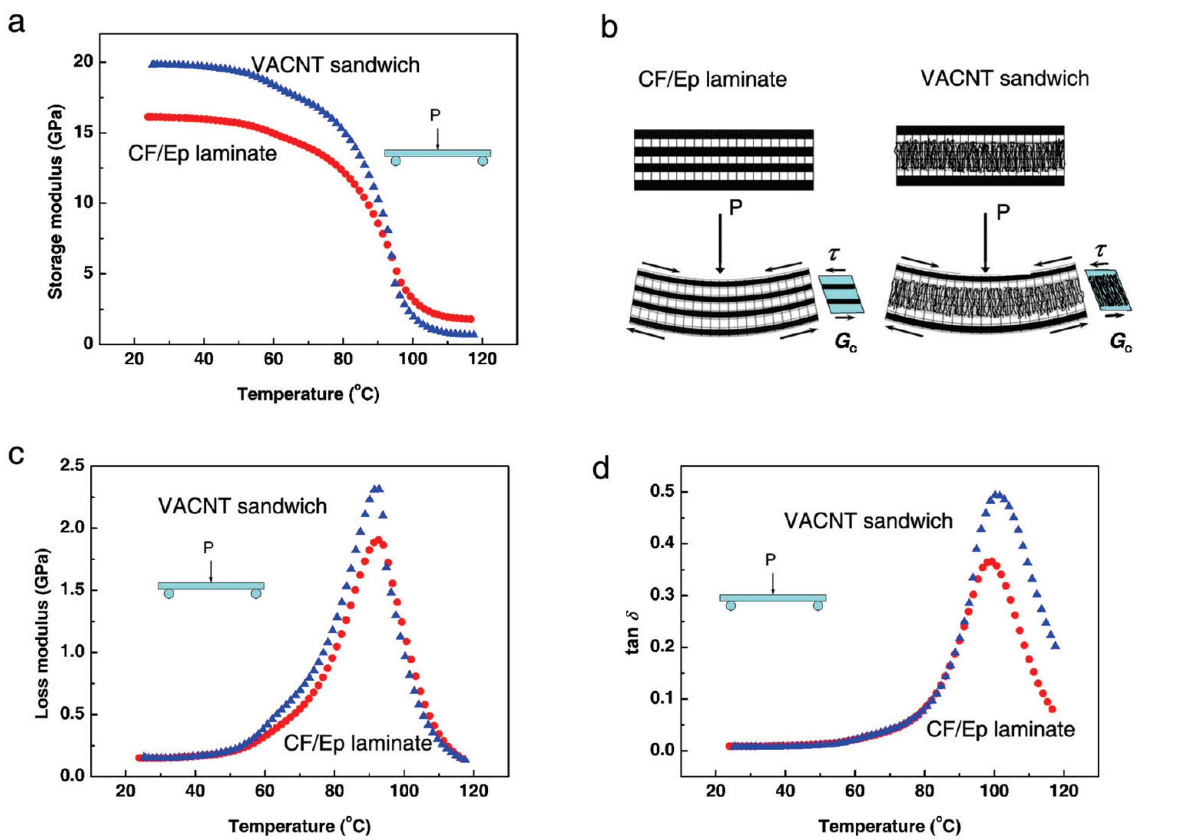
high flexural rigidity and damping, which has not been reported in literature so far.

Here, our objective is to design and fabricate a VACNT-based sandwich composite (VACNT sandwich) by simply stacking the freestanding VACNTs with CF fabrics to obtain a significant reinforcement of the flexural rigidity and damping. We fabricated the VACNT sandwich by using a vacuum-assisted resin transfer mold (VARTM) technique and measured the flexural rigidity and damping by means of dynamic mechanical analysis. We found that the VACNT sandwich exhibits higher flexural rigidity and higher damping than the conventional CF/Ep laminate due to the vertical alignment of CNTs and the rich interfaces of the CNT-based composites; furthermore, the VACNT sandwich shows lighter weight and lower density on account of the effective replacement of CFs with VACNTs and the optimal structural design.

RESULTS AND DISCUSSION

We fabricated the VACNT-based sandwich composites (Figure 1a) by simply stacking the freestanding VACNTs (Figure 1b) and CF fabrics, forming a CF/VACNT/CF sandwich structure and then infiltrating epoxy resin *via* the VARTM technique (Figure 1c). There are some advantages of using the freestanding VACNTs and designing such a sandwich structure: First, the large-size freestanding VACNTs are much easier to handle during the manufacturing process than those VACNTs anchored to their substrates,[9] avoiding the complicated transfer-printing and wafer-removal procedures.[10,12,16] Second, the freestanding VACNTs exhibit good alignment and high porosity of 95 vol % (see Figure S1 in Supporting Information),[17] which is beneficial to maintain uniform orientation of CNTs and ensure efficient infiltration of resin. Third, the sandwich structure is helpful to effectively retain the vertical alignment of CNTs and to prevent the VACNTs from being distorted by capillary forces during the resin infiltration.[18,19] As a result, the effective infiltration of VACNTs with epoxy and the uniform alignment of CNTs in the *z*-direction can be clearly observed (Figure 1d−g). In addition, rough fracture surfaces in CNT-rich regions (Figure 1f,g), in comparison to brittle fracture surfaces in the neat epoxy, reveals that the VACNTs can considerably improve the fracture toughness of epoxy by dissipating much energy during fracturing.[10,20]

We utilized a dynamic mechanical analysis (DMA) technique to investigate the flexural rigidity and damping of the VACNT sandwich in terms of storage modulus (E', flexural rigidity in relation to stored energy) and loss modulus (E'', representative of dissipated energy) or damping loss factor (tan δ, a ratio of E'' to E'); changes in these viscoelastic parameters with elevated temper-



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Comparison between the CF/Ep laminate and the VACNT sandwich

Composites	layers of CF	Layer of VACNT	Mass fraction of CF	Mass fraction of VACNT	Density (g/cm^3)	Specific stiffness (GPa·cm^3/g)
CF/Ep laminate	4	-	0.291	-	1.28	12.58
VACNT sandwich	2	1	0.154	0.005	1.21	16.36

Figure 2. Dynamic mechanical analyses of the CF/Ep laminate and the VACNT sandwich in a three-point bending mode, reflecting responses of molecular motions to cyclic loads at different temperatures. (a) The VACNT sandwich (blue triangle) exhibits higher storage modulus than the CF/Ep laminate (red circle) due to the vertical alignment of CNTs and the resulting remarkable reinforcement of shear modulus. (b) The deformation of specimens in the three-point bending mode involves a bending deformation and a shearing deformation. (c) The higher loss modulus of the VACNT sandwich than that of the CF/Ep laminate implies high dissipation of energy during cyclic deformations. (d) The VACNT sandwich shows higher damping (a higher value of tan δ) than the CF/Ep laminate. (e) The VACNT sandwich exhibits a lower mass fraction of fillers, lower density, and higher specific stiffness than the CF/Ep laminate.

atures reflect responses of molecular motions to cyclic loads in different conditions (Figure 2a−d). First of all, the VACNT sandwich exhibits a high storage modulus (E') of 19.8 GPa, increasing by 3.7 GPa (23.1%) in comparison with E' (16.1 GPa) of the four-layer CF/Ep laminate (Figure 2a); the high flexural rigidity and load-bearing capacity are mainly attributed to the vertical alignment of CNTs and the resulting improvement in shear modulus. In a three-point bending test, deformation (Δ) of a beam consists of pure bending deformation ($\Delta_{bending}$) and shearing deformation ($\Delta_{shearing}$),[21] and the storage modulus of a sandwich can be experimentally measured and theoretically calculated as follows:[21,22]

$$E' = \frac{P}{\Delta} \times \mathrm{GF} \quad (1)$$

$$\Delta = \Delta_{bending} + \Delta_{shearing} = \frac{PL^3}{48(\mathrm{EI})_{eq}} + \frac{PL}{4(\mathrm{AG})_{eq}} \quad (2)$$

$$(\mathrm{EI})_{eq} = \frac{E_f bt^3}{6} + \frac{E_c bc^3}{12} + \frac{E_f td^2}{2} \quad (3)$$

$$(\mathrm{AG})_{eq} = \frac{G_c bd^2}{c} \quad (4)$$

where P is applied central load, GF is a geometry factor related with specimen sizes, and L and b are span length and specimen width, respectively; $(EI)_{eq}$ and $(AG)_{eq}$ are equivalent flexural rigidity and equivalent shear rigidity; t, c, and d are thicknesses of the face sheet, core, and sandwich, respectively; E_f and E_c are elastic modulus of the face sheet and that of the core, and G_c is shear modulus of the core. For the convenience of comparison, the CF/Ep laminate investigated can be considered as a CF/Ep-core sandwich, which has the same CF/Ep face sheets (E_f) and geometric sizes (b, t, c, and d) as the VACNT sandwich; therefore, the difference in storage modulus (E') between the CF/Ep laminate and the VACNT sandwich only depends on the values of E_c and G_c. It is worth mentioning that the VACNT/Ep core generally has a lower elastic modulus (E_c) than the CF/Ep core due to the waviness of CNTs and low CNT content (less than 0.5 wt %),[5,17] but the VACNT/Ep core exhibits a much higher shear modulus (G_c) than the CF/Ep core due to the vertical alignment of CNTs. According to mechanics of composites, the value of the shear modulus strongly depends on the fiber orientation; the composites with the fiber orientation in the z-direction have much higher shear modulus than those in the in-plane direction.[23,24] In the case of the VACNT/Ep core, the fiber orientation is nearly vertical to the direction of shear stress (τ; Figure 2b), while the case is just opposite for the CF/Ep core; as a result, the VACNT/Ep core exhibits much higher value of G_c than the CF/Ep core. In our work, the VACNTs play an important role in integrating and anchoring two CF/Ep layers, significantly increasing the shear modulus, effectively transferring shear stress in the through-thickness direction, and resulting in a high flexural rigidity, which is consistent with the CNT reinforcement of interlaminar shear strength of laminate composites.[12,25] Moreover, it is notable that the high flexural rigidity for the VACNT sandwich generally implies a high resistance to delamination failure which plagues conventional laminate composites.

The VACNT sandwich shows higher values of loss modulus (E'') and tan δ than the CF/Ep laminate (Figure 2c,d); the high damping is closely associated with the vertical alignment of CNTs, rich interfacial areas of the nanocomposites, and high thermal conductivity of the VACNTs. First, the VACNT/Ep core exhibits high anisotropy due to the vertical alignment of CNTs, and its elastic modulus in the in-plane direction is much lower than that in the z-direction.[17] Correspondingly, the interfacial slip in the in-plane direction is rather easily triggered under compressive and tensile loads during the three-point bending deformation (Figure 2b) and then results in much higher dissipation of energy.[15,26] Second, the VACNT sandwich has much more interfacial areas than the CF/Ep laminate on account of the small diameter and large specific surface area of the CNTs, and consequently, much interfacial slip occurs during cyclic deformations and leads to the high energy dissipation and loss modulus;[27−29] such a phenomenon can also be observed by comparing the CF/Ep laminate with the neat epoxy (see Figure S2 in Supporting Information). Third, the high thermal conductivity of the VACNTs can also accelerate the dissipation of energy as heat and cause a damping improvement.[16,30,31] Therefore, the high dissipation of energy and the high damping of the VACNT sandwich is mainly attributed to the vertical alignment of CNTs, the rich interfacial areas of the CNT-based composites, and the high thermal conductivity of CNTs.

Besides the high flexural rigidity and damping, the VACNT sandwich exhibits a lower density and higher specific stiffness (a ratio of flexural rigidity to density) than the CF/Ep laminate (Figure 2e). It is worth mentioning that the VACNT sandwich has only half the mass fraction of fillers of the CF/Ep laminate, but it exhibits much higher flexural rigidity and damping, implying that the VACNTs can not only partially replace CF fabrics and reduce weight, but also further improve the dynamic mechanical properties as a result of the optimal design of composite structures. Owing to low density and high stiffness and damping, the VACNT sandwiches can be utilized as competitive candidates for high-performance composites in aerospace and transportation fields.

We further investigated reinforcing effects of VACNTs on static compressive and tensile modulus. Both the VACNT sandwich and the CF/Ep laminate exhibit remarkable anisotropy (Figure 3a), and their compressive modulus in the in-plane direction is much higher than that in the z-direction on account of considerable CF reinforcement in the in-plane direction (Figure 3b). Comparing with the CF/Ep laminate, the VACNT sandwich exhibits much lower compressive and tensile modulus in the in-plane direction and even in the z-direction (Figure 3a−d), indicating that the VACNTs cannot significantly improve the elastic modulus as much as two CF fabrics. Furthermore, the VACNT sandwich even has almost the same low compressive modulus as the neat epoxy (Figure 3b), totally different from the significant VACNT reinforcement in VACNT/PDMS composites reported in literature.[17] Such a weak VACNT reinforcement is mainly attributed to the low CNT content (a mass fraction as low as 0.005), waviness of the CNTs, and high stiffness of epoxy matrix.[32−34] In spite of its low elastic modulus, the VACNT sandwich still exhibits higher flexural rigidity than the CF/Ep laminate (Figure 2a) due to the vertical alignment of CNTs and the resulting reinforcement of the shear modulus. Hence, we can clearly understand that the continuous CFs and the VACNTs play totally different roles in the mechanical reinforcement; the former mainly dominates the load-bearing in the in-plane direction, while the latter effectively anchors two CF/Ep layers in the z-direction and significantly improves the shear-deformation resistance. Such a remarkable reinforcement in the VACNT sandwich cannot be achieved by us-

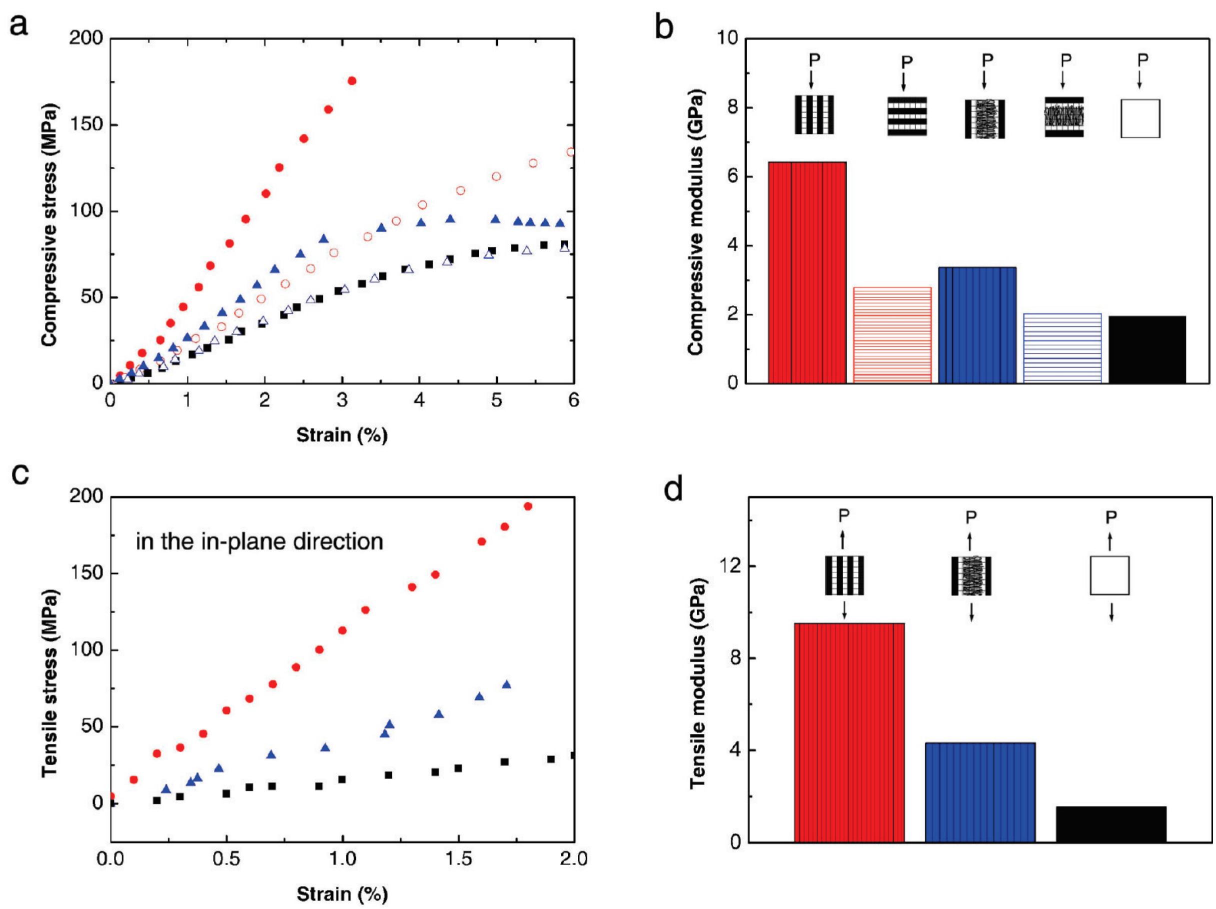

Figure 3. Static mechanical properties of the CF/Ep laminate, the VACNT sandwich, and the neat epoxy. (a) The CF/Ep laminate (red circle) and the VACNT sandwich (blue triangle) exhibit high compressive strength in the in-plane direction (solid pattern) than that in the *z*-direction (hollow pattern), indicating high anisotropy of these composites. (b) The VACNT sandwich exhibits nearly same compressive modulus as neat epoxy (black square), much lower than the CF/Ep laminate in the in-plane direction and even in the *z*-direction, implying a weak VACNT-reinforcement. (c) The tensile stress−strain curves and (d) the tensile modulus of composites in the in-plane direction also show that the reinforcing effect of the VACNTs is less than that of the continuous CF fabrics.

ing conventional CF fabrics and random CNTs separately. Therefore, combining CF fabrics with VACNTs *via* an optimal design is an effective method for taking full advantages of both two components to obtain high-performance composites.

To elucidate the damping dependence on deformation modes, we measured the dynamic mechanical properties by using a single cantilever mode (Figure 4a−c), in which specimens undergo simultaneous torsion and tension deformations (Figure 4d)[35] and exhibit different storage modulus from that in the three-point bending mode (Figure 4a). Both the VACNT sandwich and the CF/Ep laminate exhibit much higher values of E'' (~0.5 GPa) and tan δ (~0.04) at room temperature in the single cantilever mode (Figure 4b) than those in the three-point bending mode (Figure 2c); the reason for the high loss modulus and damping is that much more energy was consumed to overcome restrictions of cantilever fixtures during the cyclic deformation, and thus, resulted in high dissipation of energy.[35] Therefore, the damping of composites strongly depends on not only the intrinsic viscoelasticity of the specimens but also the deformation conditions (see Figure S3 in Supporting Information). In our work, the vertical alignment of the CNTs in the *z*-direction definitely plays an important role in improving the flexural rigidity and damping during the bending deformation. It is notable that the design of optimal structures is extremely important for obtaining high-performance composites with high flexural rigidity and damping in practical applications by fully considering CNT alignments, geometric sizes of composites, compatibility between components, deformation modes of composites, and so on.

CONCLUSION

In summary, we designed and fabricated the VACNT-based sandwich composites by simply stacking CFs and VACNTs and forming a three-dimensional sandwich structure. The vertical alignment of CNTs in the through-thickness direction plays an important role in improving not only the shear modulus and flexural rigidity of composites, but also the energy dissipation and damping properties. Comparing with the conventional CF/Ep laminates, the three-dimensional VACNT-based sandwich composites exhibit great advantages in

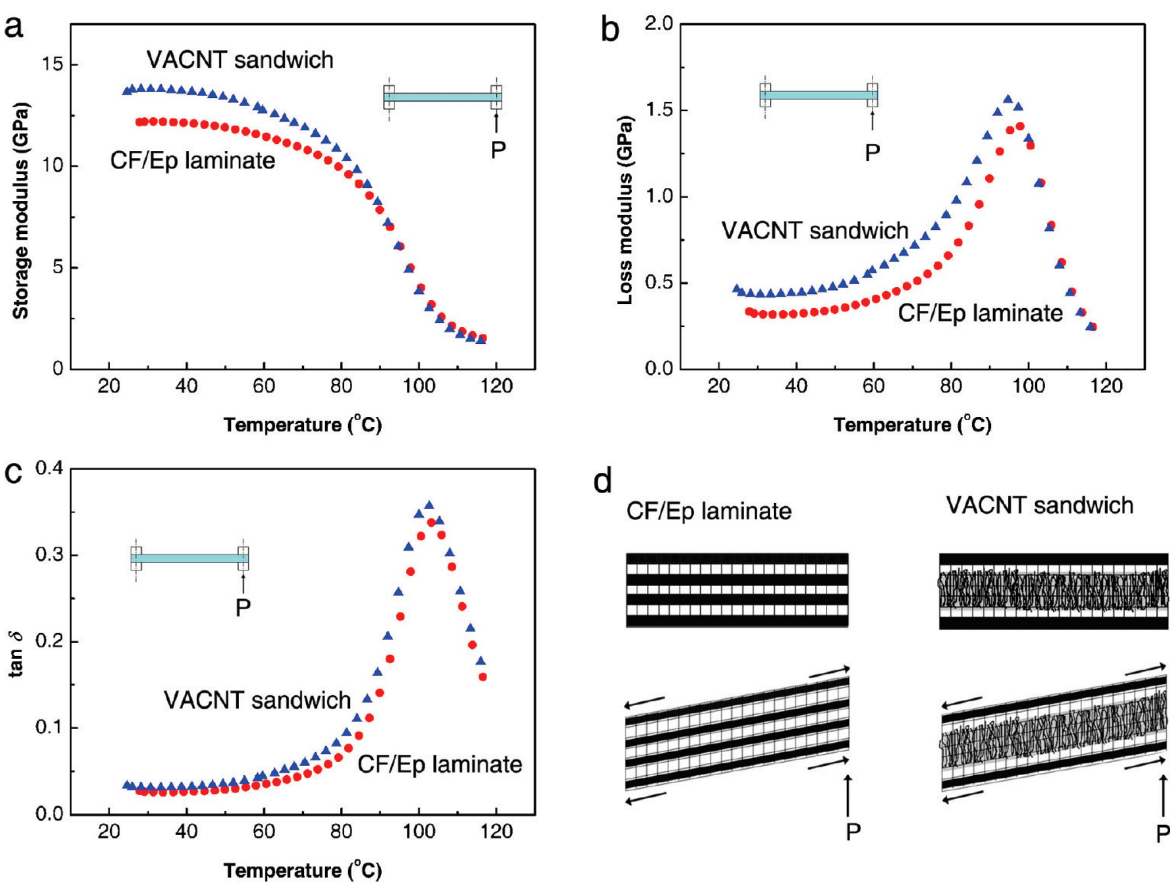

Figure 4. Dynamic mechanical analyses of the CF/Ep laminate and the VACNT sandwich in a single cantilever mode. (a) The VACNT sandwich (blue triangle) exhibits high storage modulus. (b) The higher loss modulus of the VACNT sandwich than that of the CF/Ep laminate (red circle) implies high dissipation of energy and high damping during the deformation. (c) The VACNT sandwich exhibits a higher value of tan δ than the CF/Ep laminate. (d) The deformation of specimens in a single cantilever mode is a combination of torsion and tension deformations.

mechanical and damping reinforcement, lower density, and lighter weight, and thus, there exists a great potential for these sandwich composites to be utilized in aerospace and transportation industries. Furthermore, the combination of CFs with VACNTs *via* optimal design opens a door for the fabrication of high-performance composites with excellent heat transfer, electrical conductivity, antishock, and energy storage.

EXPERIMENTAL SECTION

Growth of Freestanding VACNTs. The freestanding VACNTs were prepared by using a water-assisted chemical vapor deposition technique. A Si/SiO_2 wafer, on which a 10 nm aluminum layer and a 10 nm iron catalyst layer were deposited in advance, was put into an alumina tube and heated up to 750 °C in a buffer gas of Ar/H_2 mixture (15 vol % H_2); then ethylene gas (the carbon source) and water vapor was introduced into the alumina tube, and the VACNTs began to grow on the catalyst-containing substrate through a thermal decomposition of ethylene. The flow of ethylene was stopped after 30 min of the CNT growth, the flow of buffer gas and water vapor was continued for another 10 min to automatically release VACNTs from the substrate through a water-assisted oxidation reaction, and then the freestanding VACNTs, with an average height of 600 μm, were obtained. Microstructures of the freestanding VACNTs were characterized by using a scanning electron microscopy and a transmission electron microscopy.

Fabrication of VACNT-Based Sandwich Composites. The freestanding VACNTs were inserted into two layers of woven CF fabrics (plain weave ultralight CF fabrics, Fibre Glast Development Co., No. 2363) to form a CF/CNT/CF sandwich structure. Next, the sandwich structure was put into a mold, and a vacuum-assisted resin transfer molding (VARTM) technique was utilized to infiltrate the sandwich structure with resin. Epoxy resin (Fibre Glast Dev. Co., No. 2000), which had been mixed with curing agents (Fibre Glast Dev. Co., No. 2120) and degassed under vacuum at 60 °C for 30 min in advance, was infused slowly into the sealed mold to ensure an enough infiltration of the sandwich structure with resin. The infusion process was stopped once the mold was fully filled with the epoxy. The composites were cured at room temperature for 24 h and then taken out of the mold and postcured in an oven at 100 °C for 4 h. According to the same procedures, neat epoxy and a CF/Ep laminate with four layers of CF fabrics were obtained as well.

Mechanical Testing. Dynamic and static mechanical properties of the composites were measured by using a dynamic mechanical analyzer (DMA Q800, TA Instruments) and an electrodynamic test system (Electroplus 3000, Instron), respectively. Specimens for DMA measurements were 4 mm in width, 1.3 mm in thickness, and 30 mm in length. A three-point bending mode and a single cantilever mode were utilized for evaluating viscoelastic behaviors of the composites. The viscoelastic parameters such as storage modulus (E'), loss modulus (E''), and tan δ (damping loss factor, a ratio of E'' to E') were automatically collected at oscillation frequencies of 1 and 10 Hz when specimens

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were heated up from room temperature to 120 °C at a constant heating rate of 2 °C · min^{-1}. It is worth mentioning that the damping measurement by using DMA method here is somewhat different from that by using conventional mechanical impedance method, but analyses from both methods are consistent in describing the viscoelastic behaviors of the composites, that is, the response of molecular motions to cyclic loads in different conditions. Dumbbell-shaped and cubic specimens were prepared for tensile and compressive measurements, and the tensile and compressive rates were 1 and 0.5 mm · min^{-1}, respectively.

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Supporting Information Available: SEM and TEM images showing the microstructures of the freestanding VACNTs and viscoelasticity of composites in three-point bending and single cantilever modes. This material is available free of charge *via* the Internet at http://pubs.acs.org.

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