

DIELECTRIC AND FATIGUE LIFE ENHANCEMENT IN BaTiO₃/Epoxy RESIN BASED COMPOSITES USED AS PIEZOELECTRIC NANOGENERATOR

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Abstract

This study investigates the enhancement of dielectric properties and fatigue life in BaTiO₃/epoxy resin composites utilized as the active material in piezoelectric nanogenerators. Through a systematic approach, various fabrication techniques and composite formulations are explored to optimize the dielectric constants, energy density, and piezoelectricity while mitigating fatigue-related degradation. The physical character, phase formation, and chemical properties of these composites are identified via the optical camera, XRD, and FTIR methods, respectively. The frequency dependence of dielectric properties for all samples is measured by an LCR meter. The hysteresis P-E loops are investigated in order to calculate the energy density and energy loss density of materials. The piezoelectric properties of these composites are performed by studying the generated output voltage and current after applying mechanical force to the samples. Moreover, MWCNT nanomaterials have also been incorporated into these composites in order to improve their dielectric value and fatigue life. The results show that the dielectric constant (ϵ_r) and dielectric loss ($\tan\delta$) of these composites are independent of frequency. After loading BaTiO₃ into the epoxy resin matrix, the ϵ_r and $\tan\delta$ significantly increased with the increasing BaTiO₃ amount. The energy density and energy loss density of all composites were calculated from these P-E loops, and it is seen that pure epoxy resin shows the lowest energy density and energy loss density values. After loading BaTiO₃ into the epoxy resin matrix, both the energy density and the energy loss density of the composites significantly increased. Moreover, after adding 20 percent by volume of BaTiO₃ to the system, the energy density increases by 160% compared with pure epoxy resin. For the effect of MWCNT filler, it is seen that the ϵ_r , $\tan\delta$, energy density and energy loss density are significantly improved after adding 1.5 vol% of MWCNT into the system. The output current generated by applying mechanical force to the sample increased 27 times after adding MWCNT to the BT-filled epoxy resin composite. Finally, it can be concluded that all experimental results demonstrate significant enhancements in dielectric properties, energy density and electric output current, paving the way for the development of robust and efficient piezoelectric nanogenerators for diverse energy harvesting applications.

Keywords: Epoxy Resin; Barium Titanate; MWCNT; Composites; Piezoelectric Nanogenerato

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Introduction

Nowadays, piezoelectric nanogenerators have been recognized for their significance in various fields such as energy harvesting, wearable electronics, environmental monitoring, and healthcare applications, and their importance may have continued to grow in the intervening time. As technology continues to advance, the importance of piezoelectric nanogenerators is likely to increase, driven by the need for efficient and sustainable energy solutions in various applications. Barium titanate (BaTiO₃; BT) -based ceramics are the most suitable material for use as piezoelectric nanogenerators. However, the low breakdown strength of this BT ceramic results in a very low energy density, which hinders its applicability. The urge for the combination of the high dielectric constant of ceramics with the high breakdown strength and processability of polymers results in the development of polymer composite materials. Therefore, combining a BT piezoelectric filler with a polymer matrix can contribute a great deal to the energy harvesting application, which helps generate power when subjected to mechanical vibration from an ambient environment. However, in the case of lightweight and flexible structures, specific mechanical properties are more important, therefore investigating the strength of the reinforced epoxy polymers under different operating conditions has been the subject of several recent studies. Fatigue failure, as one of the most common reasons for breakdown, has been investigated as a vital property in designing epoxy composites subjected to cyclic loads. In this work, nanocomposites between epoxy resin and barium titanate are prepared in order to be used as a piezoelectric nanogenerator. By combining in the right ratio, these piezoelectric ceramic-polymer composites can achieve an excellently higher energy density and dielectric breakdown strength than either of the constituents alone. These composite materials not only enhance mechanical properties but also uniformity, processability, adhesion, and tunability, ultimately improving the overall performance and reliability of the device.

Manika and Psarras (2019) studied the composite systems of epoxy resin and barium titanate nanoparticles by varying the content of the reinforcing phase. Their work reported that dielectric permittivity increased with filler content in the whole frequency range. Energy stored or harvested increases with the applied dc field and the coefficient of energy efficiency increased

dramatically with filler content reaching a value of 58.2% for the 7 phr BaTiO₃ nanocomposite at 200 V charging voltage and 10 s charge/discharge time. Furthermore, Wang *et al.* (2020) reviewed the design strategy of barium titanate/polyvinylidene fluoride-based nanocomposite films for high-energy storage. They have explained the mechanisms of the increase in energy density of BT/PVDF-based nanocomposites and provided a new idea for the development of BT/PVDF-based nanocomposites with higher energy density. Their report showed that researchers have tried various strategies to improve the energy density of BT/PVDF flexible nanocomposite films which can be summarized into six possible mechanisms: (1) improving compatibility and dispersibility; (2) increasing polarization area; (3) improving electric field distribution; (4) suppressing carrier migration; (5) microcapacitors; and (6) other mechanisms. So, the idea of introducing conductive fillers into the mixing composites is an interesting and easy way to improve dielectric and piezoelectric characteristics. Park *et al.* (2008) studied the effects of CNT/BaTiO₃ composite particles prepared by mechanical processes on the dielectric properties of epoxy hybrid films. Their result showed that the hybrid film fabricated by incorporating these composite fillers in an epoxy matrix had a high dielectric constant and similar dielectric loss as compared to the composite which contained neat BT particles. They also reported that the dielectric properties of these hybrid films were found to be dependent on both the content of MWNTs and mechanical processing time. Poh *et al.* (2014) used MWCNT and BaTiO₃ as fillers in epoxy thin film composites to investigate the dielectric properties of these thin film composites for embedded capacitor applications due to their high dielectric constant. They found that MWCNT filler provided a high dielectric constant value compared to BaTiO₃ because MWCNT was more conductive than BaTiO₃. Moreover, Ibaa and Alithari (2023) recently reported that the fatigue life of polymer materials like epoxy can be improved by using stiffeners such as carbon fiber and/or adding multiwall carbon nanotubes (MWCNTs). Their experimental results of the fatigue test with fully reversed bending stress showed a maximum increase of 788% in fatigue life when adding 1wt% MWCNTs to epoxy, while the maximum improvement ratio reached 2,500% when adding 1 wt% MWCNTs to composite. Therefore, based on

this literature, introducing carbon nanotubes into the mix with barium titanate and epoxy resin can actually further enhance the properties of the composite material, and it is believed that this offers additional benefits in the context of a piezoelectric nanogenerator. Moreover, the synergistic effects achieved by incorporating these three materials can lead to improved performance and functionality in energy-harvesting applications.

In this work, composites between epoxy resin and barium titanate are prepared in order to be used as a piezoelectric nanogenerator. Physical character, phase formation and chemical properties of these composites are identified via optical camera, XRD and FTIR methods, respectively. The frequency dependence of dielectric properties for all samples is measured by an LCR meter. The hysteresis P-E loops are investigated in order to calculate the energy density and energy loss density of materials. The piezoelectric properties of these composites are performed by studying the generated output voltage and current after applying mechanical force to the samples. Moreover, MWCNT nanomaterials have also been incorporated with these composites in order to improve their dielectric value and fatigue life.

Experimental procedure

Epoxy resin (bisphenol A-epichlorohydrin polymer) and hardener (poly[oxy (methyl-1,2-ethanediol)], alpha.-hydro.-omega.-(2-aminomethylethoxy)-, ether with 2-ethyl-2hydroxymethyl)-1,3-propanediol (3:1)) supplied by Buehler (EPOXICURE™ 2) were used in this study. The epoxy resin and hardener were mixed using a stoichiometric ratio of 100:23 (w/w) by the conventional casting method. The mixture was cured at room temperature for 6 hours. Barium titanate (BaTiO_3 or BT) nanopowder, with 99.95% purity and 50 nm of average particle size purchased from Inframat® Advanced Materials™, was selected to be used as a piezoelectric filler. A multi-walled carbon nanotube (MWCNT) with 99.95% purity and an average diameter of 12 nm purchased from Nano Generation Co., Ltd., was selected to be used as a conductive filler. The loading of BaTiO_3 in the epoxy resin varied from 20 vol% to 30 vol% while the MWCNT loading was fixed at 1.5 vol% for all samples. The BaTiO_3 and MWCNT fillers were homogeneously dispersed in the epoxy resin matrix by stirring on a magnetic stirrer for 30 min at room temperature. The hardener was put into the mixture and continually stirred for 2 min. The mixture was then vacuum degassed to a pressure of

700 mmHg and cured at room temperature overnight to settle.

The physical, chemical and dielectric properties such as dielectric constant and dielectric loss were investigated for all samples. The optical camera was used to investigate the physical properties of these composites. The chemical properties of all composites were investigated using Fourier transform infrared (FT-IR) spectroscopy (Spectrum GX spectrometer, Perkin Elmer). The phase formation of all samples was examined by room-temperature X-ray diffraction (XRD; Rigaku SmartLab®) using $\text{Cu K}\alpha$ radiation. For the electrical measurement, the silver paste was painted on both sides of the samples. The dielectric properties of all composites were studied as a function of frequency at room temperature. The capacitance was measured by an LCR meter (HP-4284, Hewlett-Packard Inc.) controlled by a computer system. The dielectric constant (ϵ_r) of all samples was then calculated using the geometric area and thickness of the samples. The polarization hysteresis loops were investigated in order to calculate the energy density and energy loss density of materials. The piezoelectric properties of these composites were also performed by studying the generated output voltage and current after applying mechanical force to the samples.

Results and discussion

Epoxy resin (bisphenol A-epichlorohydrin polymer) was used as a matrix combined with 20, 25 and 30 percent by volume of BaTiO_3 (BT) nanopowder. The phase formation of all composites including their starting members is revealed by XRD as shown in Figure 1(a). It is seen that only the BaTiO_3 phase is noticeable in every composite since the epoxy resin phase is non-crystalline, so it cannot be observed by the XRD technique. FT-IR technique is then used to investigate the phase formation of these composites which is revealed in Figure 1(b). The FT-IR spectrum of pure epoxy resin is represented by the green line. The main transmittance bands are located at 2920 cm^{-1} , 1605 cm^{-1} , 1508 cm^{-1} , 1240 cm^{-1} , 1032 cm^{-1} , 828 cm^{-1} and 554 cm^{-1} . The first peak at 2920 cm^{-1} corresponds to asymmetrical C-H stretch of $-\text{CH}_2$ group. The second set of peaks at 1605 and 1508 cm^{-1} corresponds to the C-C stretching vibration in aromatic. The peaks at 1240 and 1032 cm^{-1} correspond to asymmetrical aromatic C-O stretch and symmetrical aromatic C-O stretch, respectively. The peak at 828 cm^{-1} corresponds to -CH out of plane deformation in aromatic and the

last peak centered at 554 cm⁻¹ corresponds to the characteristic frequency of C₆H₄X₂-para (X represents any functional group). These bands are the characteristic bands of epoxy resin (Maity *et al.*, 2008). After loading BT into the epoxy resin matrix, the spectra are almost similar to those of pure epoxy resin except for the peak at around 512 cm⁻¹ peak. The broad peak at around 450-500 cm⁻¹ is the most important peak for barium titanate, where the wave number of 500 cm⁻¹ corresponds to the vibration of the bond between titanium and oxygen (Ti-O bond) and the wave number of 450 cm⁻¹ corresponds to the vibrations between metal atoms and oxygen (Ti-O and Ba-O bonds) (Asimakopoulos *et al.*, 2014). This FT-IR spectra confirmed the existence of both end members, which are BaTiO₃ and epoxy resin phases in all composites with no evidence of the formation of new bonds or foreign bands.

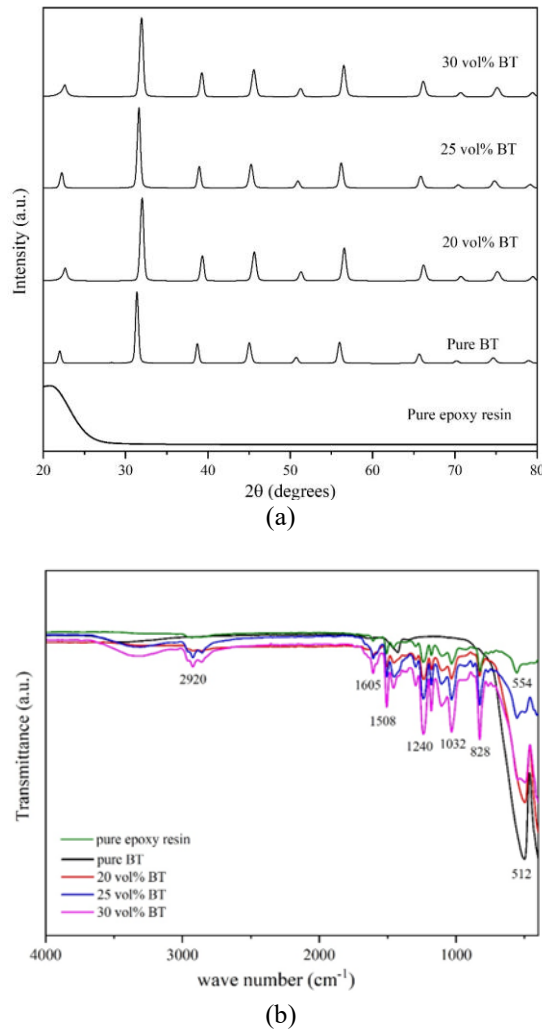


Figure 1. (a) XRD patterns and (b) FTIR spectra of the BT-filled epoxy resin composites in various contents

The frequency dependence of the dielectric constant and dielectric loss at room temperature for the BT-filled epoxy resin composites with various contents is shown in Figure 2(a). The graph revealed that the dielectric constant and loss of these composites are independent of frequency. After loading BT into the epoxy resin matrix, the dielectric constant (ϵ_r) and dielectric loss ($\tan\delta$) significantly increased with the increasing BT amount. This can easily be explained by the rise of the barium titanate phase in the sample, which has higher dielectric properties than neat epoxy resin. Typical P-E hysteresis loops of all BT-filled epoxy resin composites were measured at room temperature and also shown in Figure 2(b). The energy density and energy loss density of all samples were calculated from these P-E loops, as shown in a small frame in the figure and the calculated values are tabulated in Table 1. From the result, it is seen that pure epoxy resin shows the lowest energy density and energy loss density values. After loading BT into the epoxy resin matrix, both the energy density and the energy loss density of the composites significantly increased. It is clearly seen that when adding 20 percent by volume of BT to the system, the energy density increases by 160% compared with pure epoxy resin. This can be explained by the large dielectric permittivity of barium titanate (BT) presented in the composites. Generally, the expression for the maximum energy storage density of a capacitor is given by $U_{max} = \frac{1}{2} \epsilon_0 \epsilon_{eff} E_B^2$, where ϵ_{eff} is the effective permittivity of the composite and E_B is the breakdown field strength. To have a maximum energy density, the nanoparticle volume fraction in a composite should be rationally chosen in such a way that the combination of the effective permittivity and the breakdown strength gives the maximum value of energy density.

1.5 vol% of MWCNT was added to all BT/epoxy resin composites. Physical character and phase evolution were investigated and revealed in Figure 3. It can be seen that the pure epoxy resin is transparent. After loading BT nanopowder into the epoxy resin matrix, the sample becomes opaque. And after adding MWCNT to the system, the composite becomes a blackish color. For the XRD result, only the BaTiO₃ phase is seen in the composites since the epoxy resin phase is non-crystalline, so it cannot be observed by the XRD technique. However, after adding MWCNT to the BT/epoxy resin composite, all peaks shift to a lower 2θ angle, which indicates an expansion to a higher d-spacing as explained by Bragg's law ($2d \sin \theta = n\lambda$) (Cullity, 1956). Dielectric properties and hysteresis P-E loops were investigated for all

MWCNT/BT/epoxy resin composites and the data is also summarized in Table 1. From the results, it is seen that the dielectric constant (ϵ_r), dielectric loss ($\tan\delta$), energy density and energy loss density are significantly improved, where the largest dielectric constant and energy density are 14.79 at 10 kHz and 210 $\mu\text{J}/\text{cm}^3$ respectively when 1.5 vol% of MWCNT is added to a 30 vol% BT-filled epoxy resin composite. The presence of MWCNT distributed around the epoxy matrix was acting as a mini capacitor for the composite (Li *et al.*, 2008a). This is because when the electric field was applied, the charge accumulated on the nanotube due to MWCNT's electrical conductivity properties

(Li and Chou, 2008b). Moreover, the rise in dielectric loss and energy loss density can also be explained by the occurrence of an agglomeration of MWCNT. Generally, the highly conductive MWCNT particles make it easy to form conductive paths in the composites (Lu and Wong, 2008). When the composites become more conductive, the leakage current occurs which causes part of the electrical energy to transfer to thermal energy (Chang, 2012). As a result, the dielectric loss and energy loss density of all composites increase, which is caused by dissipation energy when the MWCNT loads into the system.

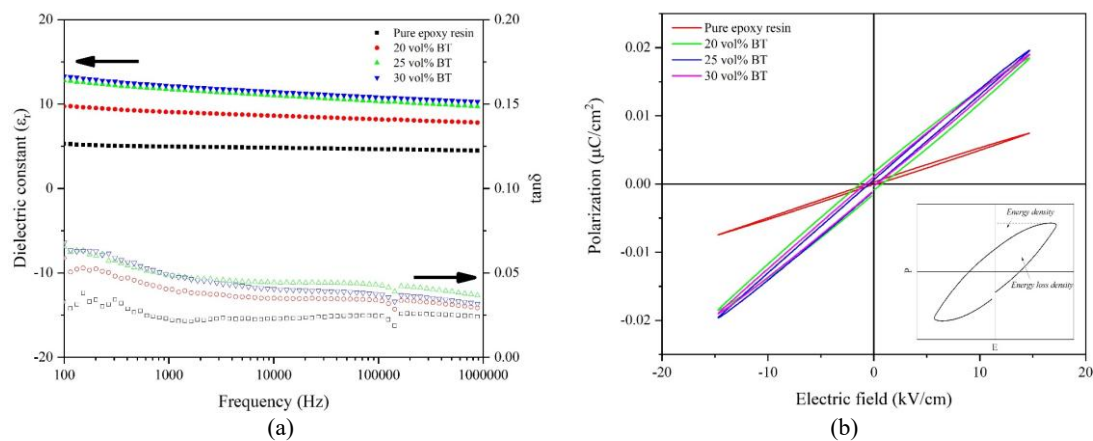


Figure 2. (a) Frequency dependence of dielectric constant and loss at room temperature and (b) Hysteresis loops for the BT-filled epoxy resin composites

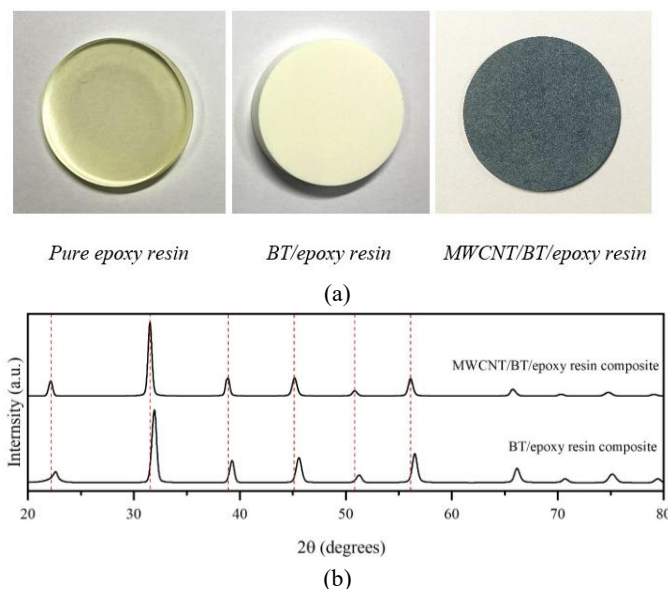
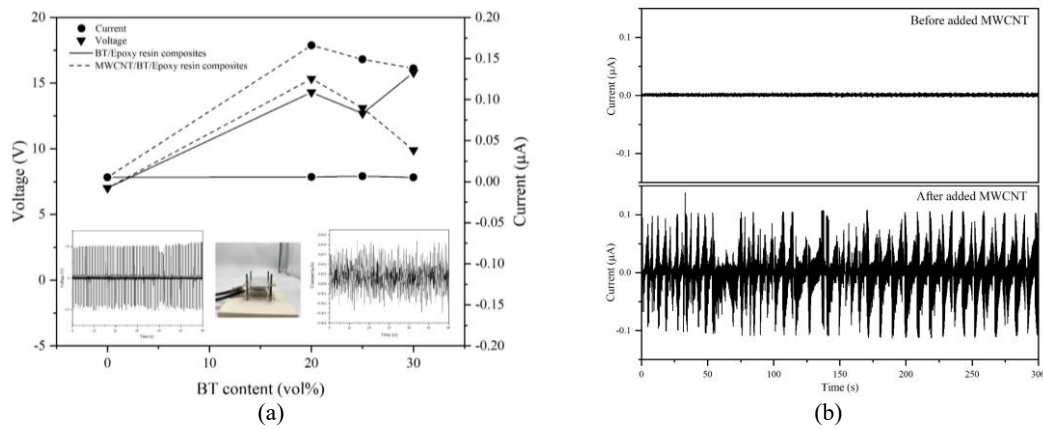


Figure 3. Physical characters and XRD patterns compared between epoxy resin-based composites before and after filled with fillers

Table 1. Electrical properties of BaTiO₃ and MWCNT -filled epoxy resin composites

Samples	Dielectric constant (ϵ_r) at 10 kHz	Dielectric loss ($\tan\delta$) at 10 kHz	Energy density ($\mu\text{J}/\text{cm}^3$)	Energy loss density ($\mu\text{J}/\text{cm}^3$)
Pure epoxy resin	4.81	0.023	56.7	5.64
20 vol% BT filled epoxy resin composite	8.62	0.035	147.6	31.9
20 vol% BT + MWCNT filled epoxy resin composite	11.73	0.041	161.6	27.3
25 vol% BT filled epoxy resin composite	11.01	0.044	148.4	12.6
25 vol% BT + MWCNT filled epoxy resin composite	12.52	0.043	173.7	26.0
30 vol% BT filled epoxy resin composite	11.45	0.040	145.3	17.6
30 vol% BT + MWCNT filled epoxy resin composite	14.79	0.047	210.0	61.8

**Figure 4. (a) The average electrical output signals of BT-filled epoxy resin composites under mechanical pressing compared with those after adding MWCNT into system and (b) mechanical fatigue test of 20 vol% BT-filled epoxy resin composites for 1000 cycles before and after adding MWCNT**

The piezoelectric capabilities of these composites were determined by applying mechanical force to the samples and the electrical output signals were collected. Figure 4 displays the output voltage and current generated by the epoxy resin-based composites with fillers when they were mechanically pressed. The positive and negative output signals were measured under a periodic vertical compressing and releasing process exerted by a simple mechanical pressure of 150 N at a frequency of 3 Hz. The epoxy resin-based composites with an effective area of a 1-inch-diameter disk harvested the open-circuit voltage and short-circuit current from mechanical deformation, and the average values of each sample are plotted in Figure 4(a). The graphs of positive and negative output signals of the 20 vol% BT-filled epoxy resin composite, which exhibited the highest performance, are also shown in the small pictures in this figure. The intensities of the open-circuit voltage and short-circuit current of this composite reached approximately 15 V and 0.006 μA , respectively. Figure 4(b) shows a comparison of the

short-circuit current generated by a 20 vol% BT-filled epoxy resin composite before and after loading MWCNT into the system. The output current generated by the BT-filled epoxy resin composite is very few microamperes ($\sim 0.006 \mu\text{A}$), whereas the MWCNT/BT -filled epoxy resin composite can generate an output current of 0.166 μA , which has increased 27 times. However, the output voltage did not appear to be significantly different after the addition of MWCNT. The large increase in the electric current can be explained by the fact that MWCNT's electrical conductivity properties create more conductive paths in the composites, resulting in a rise in the output current (Poh *et al.*, 2014). The durability test was also carried out by repeatedly pressing and releasing it for 1000 cycles in order to investigate the mechanical stability of this composite. The mechanical fatigue test of the 20 vol% BT-filled epoxy resin composite before and after loading MWCNT into the system is shown in Figure 4(b). It can be clearly seen that even though there is a slight deviation in the output signal, a stable current signal

is observed over a period of testing time for both samples.

Therefore, from these results, it can be concluded that the synergy between BaTiO₃ nanoparticles and the epoxy resin matrix facilitates improved dielectric and piezoelectric performance, enabling efficient energy conversion from mechanical to electrical energy. Furthermore, the incorporation of multi-wall carbon nanotubes (MWCNT) into the composite system further enhances its properties, leading to even greater improvements in dielectric constant and energy density. The experimental findings also suggest that the addition of MWCNT enhances the electrical conductivity of the composites by improving charge transport and polarization under mechanical stress. This results in a significant boost in the overall performance of the piezoelectric nanogenerator, including enhanced output current and voltage generation capabilities. Moreover, the presence of MWCNT can also contribute to the mechanical reinforcement of the composite, improving its durability and fatigue resistance. However, it should be noted that not only the piezoelectricity of the nanofiller is mainly dependent on the orientation of polarization within the crystal, but the agglomeration of these nanostructures (nanoparticles and nanotubes), content composition, and porosity of the samples also play an important role.

Conclusion

Barium titanate (BaTiO₃ or BT) is selected to be used as a piezoelectric filler for epoxy resin-based composites in order to be used as piezoelectric nanogenerators. The effect of this BT filler amount on the dielectric constant, dielectric loss, energy density, energy loss density, and electric output signals generated by mechanically pressing all composites is investigated. From the results, it was found that the dielectric constant and loss of the BT-filled epoxy resin composites are independent of frequency, and the ϵ_r and $\tan\delta$ values increase with increasing BT amounts. The hysteresis P-E loops were used to determine the energy density and energy loss density of each composite, and the results demonstrate that pure epoxy resin has the lowest energy density and energy loss density values. Both the energy density and the energy loss density of these composites dramatically increased upon introducing BT into the epoxy resin matrix. Furthermore, in comparison to pure epoxy resin, the energy density of the system increases by 160% with the addition of 20 percent by volume of BT. When 1.5 vol% of MWCNT filler is added to the

system, it is observed that the ϵ_r , $\tan\delta$, energy density, and energy loss density considerably improve for all BT-filled epoxy resin composites. Moreover, the output current generated by applying mechanical force to the sample increased 27 times after adding MWCNT to the BT-filled epoxy resin composite. Finally, for the mechanical fatigue test, it can be clearly seen that even though there is a slight deviation in the output signal, a stable current signal is observed over a period of testing time for both BT-filled epoxy resin composites before and after loading MWCNT into the system. Therefore, it can be summarized that the addition of MWCNT not only enhances the dielectric constant and electrical conductivity of the composites by improving charge transport and polarization under mechanical stress but can also contribute to the mechanical reinforcement of the composite, improving its durability and fatigue resistance.

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