Effect of ZnO nanoparticles on mechanical properties of polyacrylate composites

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Abstract: Dielectric elastomer (DE) is a class of electro-driven flexible smart polymer materials, often using polyacrylate as an elastomer material. Unmodified acrylates typically have higher viscoelasticity and poor elasticity, resulting in slower response and less deformation during the elastomer process, resulting in higher driving voltages and less electrical strain. In this study, ZnO nanoparticles were added to polyacrylate (CN 9021NS) to study the effect of nanoparticles on the mechanical properties of composites, and the mechanism of ZnO nanoparticles on the improvement of composite properties was analyzed and elaborated. The results showed that the addition of ZnO nanoparticles could effectively improve the mechanical properties of elastic bodies. When the nano-ZnO particle content is 5 wt%, it has a lower Young's modulus (1.38 MPa), the highest elongation at break (115.72%) and a higher tensile strength (1.165 MPa).

Keywords: Dielectric Elastomers; Polyacrylates; Nanoparticles; Mechanical Properties

1. Introduction

Dielectric elastomer (DE), as a kind of electroactive polymer, is a kind of electro-driven flexible intelligent polymer material, which has the advantages of high electromechanical conversion efficiency, light weight, low price, flexible movement, easy forming and not easy to fatigue damage. Dielectric elastomer is a capacitor that converts electrical energy into mechanical energy by coating a flexible electrode on the surface^[1].

In terms of materials, nonlinear and viscoelastic homogeneous materials are suitable for most dielectric elastomers, such as existing soft materials including natural rubber, synthetic rubber, elastomers, polymer composites, and nanoarticulated polymer composites. In recent years, elastomers such as carboxylated nitrile butadiene rubber (XNBR), styrene-butadiene-styrene block copolymer (SBS) and hydrogenated nitrile butadiene rubber (HNBR) have also received some attention^[2-5]. As a kind of dielectric elastomer, acrylate has the advantages of high electro-induced strain, good temperature adaptability and excellent biocompatibility that other dielectric elastomer materials cannot achieve.

Acrylic elastomers are made from a mixture of aliphatic acrylates and are currently the most widely used dielectric elastomer materials. Its elasticity mainly depends on the mild cross-linking of the aliphatic groups of the flexible branched chains and the acrylic polymer chains. It has a relatively high dielectric constant and elastic modulus, high energy density, large deformation, and low mass density. Polyacrylate elastomers can achieve deformations of up to more than 380% under high-strength pre-stretch conditions. Because of its good performance in driving deformation, it is one of the most widely used dielectric elastomers. Among them, the VHB series tape produced by 3M company is the most representative, and its theoretical energy density can reach 3.4 MJ/m^[6]. It has different thickness and length types, good compatibility, and is often used in the processing of a variety of drive equipment, generators, etc^[7]. However, unmodified acrylic elastomers have large viscoelasticity, resulting in a slower response time, a slow deformation response, and long-term relaxation that seriously affects their working life. In this paper, the addition of nano-ZnO particles to polyacrylate (CN 9021NS) was mainly studied, the mechanism of nano-ZnO particles on the improvement of elastomer properties was explored, and the composite materials with excellent mechanical properties were prepared, so as to improve the elasticity of the composites, so that the elastomers can have higher deformation during work, and realize the large strain of the elastomers, thereby improving their work efficiency.

2. Experiment

2.1 Experimental materials

The main experimental materials are: Acrylate resin oligomer (CN 9021NS), Isodecyl acrylate (IDA), 1,6-hexanediol diacrylate (HDDA), Isobornyl acrylate (IBOA), Trimethylolpropane triacrylate (TMPTA), Ethyl (2,4,6-trimethylbenzoyl) phenylphosphinate (TPO-L), ZnO nanoparticles (<100nm).

2.2 Preparation of composite materials

During the design of the mixed solution formula, a variety of ultraviolet curing agents were added to the prepolymer solution as photoinitiators, so light avoidance treatment must be carried out during the mixing process of the solution and subsequent steps. Here we use opaque, acid and alkali resistant, and organic solvent resistant high-density polyethylene seal cans (150 ml) with LDPE material inner sealing caps for the mixing and storage of solutions.

1) First, a certain mass of CN 9021NS is taken for vacuum pumping treatment to prevent internal bubbles from affecting the performance of the material. Then CN 9021NS, IDA, HDDA, IBOA, TMPTA, TPO-L are mixed according to the ratio in Table 1.

Material and Reagents	Content (wt%)
Acrylate resin oligomer (CN 9021NS)	70
Isodecyl acrylate (IDA)	17
1,6-hexanediol diacrylate (HDDA)	5
Isobornyl acrylate (IBOA)	5
Trimethylolpropane triacrylate (TMPTA)	1
Ethyl (2,4,6-trimethylbenzoyl) phenylphosphinate (TPO-L)	2

Table 1: Formula of mixed solution

2) The obtained mixed solution is stirred with a planetary solder paste mixer (ZB 500S) for 90 minutes to fully mix the solution.

3) Nano ZnO particles are taken according to the mass fractions of 0, 2.5 wt%, 5 wt%, 7.5 wt%, and 10 wt% respectively, and added to the CN 9201NS prepolymer solution, which is then sealed in a high-density polyethylene seal can.

4) The planetary solder paste mixer is used again to stir for 90 minutes, allowing the nanoparticles to fully mix with the mixed solution.

5) The fully stirred ZnO and CN 9021NS composite material is again subjected to light-avoiding vacuum degassing for 30 minutes, fully excluding internal bubbles, to obtain a solution that can be used for the next step.

3. Material characterization

3.1 Microscopic morphology

In order to characterize the morphology of ZnO particles in the composite material and the distribution state of nanoparticles in the material, the prepared ZnO and CN 9021NS composite material was analyzed by the Supra 55 scanning electron microscope produced by the Japanese Zeiss company.

3.2 Mechanical property test

In order to test the mechanical properties of the composite material, it is necessary to prepare tensile specimens for the experiment. The tensile specimens are designed as dumbbell-shaped tensile specimens, with detailed dimensions as shown in Figure 1. The mixed solution is cast into the designed injection mold, then UV cured, and after demolding, the tensile specimens are obtained.



Figure 1: Dumbbell shaped tensile specimen

Mechanical performance testing mainly measures physical quantities such as stress, strain, and elongation. The ZQ-990LA electric universal tensile testing machine from Zhi Qu Precision Instruments Co., Ltd. is used to perform mechanical tensile tests on the tensile specimens. The specimens are stretched at a speed of 200 mm/min until they break. Three tensile specimens are prepared for each material, and three tensile tests are conducted. The average value is taken as the final result, and a stress-strain diagram is obtained.

4. Results and discussion

4.1 Distribution of nanoparticles



Figure 2: SEM image of the original CN 9021NS composite

As shown in Figure 2, it is the SEM scanning electron microscope image of the original CN 9021NS composite material without adding any nanoparticles. The SEM scanning electron microscope image of the original CN 9021NS composite material shows that it contains a small amount of bubbles and impurities inside.



Figure 3: SEM images of composites with different nanometer ZnO particle contents. (a) 2.5 wt% ZnO; (b) 5 wt% ZnO; (c) 7.5 wt% ZnO; (d) 10 wt% ZnO

As shown in Figure 3, with the increase in the content of added nano-ZnO particles, the distribution of nanoparticles in the material becomes more and more dense. When the content of nano-ZnO particles is 2.5 wt%, there are a few nanoparticles distributed, but they are unevenly dispersed, and a small number of agglomerates are dispersed in the matrix; When the content of nano-ZnO particles is 5 wt%, nanoparticles are dispersed in the composite material without a specific state, the nanoparticles show unsaturated dispersion, the distance between nanoparticles is far, and they are wrapped by the composite material; When the content of nano-ZnO particles is close, there is no obvious agglomeration phenomenon, and when the content of nanoparticles reaches 7.5 wt%, it has good compatibility with the material; When the content of nano-ZnO particles is 10 wt%, nanoparticles are dispersed in phenomenon, excessive nanoparticles are concentrated together to form a large number of agglomerates and stick together, and cannot be dispersed into the composite material.

4.2 The impact on mechanical properties

4.2.1 The impact on stress-strain

The stress $\sigma_N(MPa)$ and strain $\varepsilon_N(\%)$ of the tensile specimen are calculated according to the following formula:

$$\sigma_{\rm N} = \frac{F(x)}{A_0} = \frac{F(x)}{bh} \tag{1}$$

$$\varepsilon_{\rm N} = \frac{l(x) - l_0}{L_0} \times 100 \tag{2}$$

Where F(x) is the tensile force loaded onto the tensile specimen (N); The F(x) data of the tensile test comes from the uniaxial tensile test; A_0 is the initial cross-sectional area of the tensile specimen in the unstretched state(mm2); The l(x) is the distance between the benchmark points corresponding to different stretching displacements (mm); The l(x) data of the tensile test comes from the uniaxial tensile test; The l_0 is the initial distance between the benchmark points of the gauge distance (mm).



Figure 4: Stress-strain diagram of tensile specimens with different amounts of nano-ZnO particles

From Figure 4, it can be seen that the slope of the stress-strain curve of the tensile sample is the lowest when no ZnO nanoparticles are added. When the content of ZnO nanoparticles added to CN 9021NS is between 0-7.5 wt%, the slope of the stress-strain curve of the tensile sample gradually increases as the content of ZnO nanoparticles added increases; When the content of ZnO nanoparticles added to CN 9021NS is between 7.5 wt%-10 wt%, the slope of the stress-strain curve of the tensile sample decreases as the content of ZnO nanoparticles added increases, but it is still higher than the original CN 9021NS tensile sample.

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When the slope of the stress-strain curve increases with the increase of the content of added nanoparticles, this is due to the strong interaction between the nanoparticles and the interface they contact within the elastomer after adding nanoparticles to CN 9021NS, and there is also friction between the nanoparticles, which increases the stiffness of the material, reduces the toughness, and makes the tensile sample harder. Therefore, as the content of added nanoparticles increases, the tensile sample has a smaller strain under the same stress.

When the slope of the stress-strain curve decreases with the increase of the content of added nanoparticles, this is due to the content of added nanoparticles exceeding the threshold, and too many nanoparticles in the elastomer agglomerate. These agglomerated nanoparticles destroy the cross-linked structure inside the elastomer, causing the stiffness of the tensile sample to decrease, the toughness to increase, and become softer. Therefore, as the content of nanoparticles is added, the tensile specimen has a greater strain under the same stress.

4.2.2 Impact on Young's modulus

The magnitude of Young's modulus represents the rigidity of the material. The larger the Young's modulus, the less likely the material is to deform. For elastic bodies, the smaller the Young's modulus, the better the elasticity of the material, the softer it is, and the same force will result in a greater deformation. The Young's modulus E (MPa) of the tensile specimen of the superelastic material can be calculated by the following formula:

$$E = \frac{\sigma_{\rm N}}{\varepsilon_{\rm N}} \tag{3}$$

Where σ_N is stress; ε_N is strain.



Figure 5: Young's modulus of tensile specimens with different amounts of nano-ZnO particles

As shown in Figure 5, it represents the Young's modulus at the initial stage of tensile testing for samples with different contents of nano-ZnO particles. The Young's modulus of tensile samples with different nano-ZnO particle contents is different. Compared with the CN 9021NS prepolymer without added nano-ZnO particles, as the content of added nano-ZnO particles increases, the Young's modulus of the tensile sample first increases and then decreases. When the content of added nano-ZnO particles increases from 0 to 7.5 wt%, the Young's modulus of the tensile sample gradually increases. When no nano-particles are added, the Young's modulus of the tensile sample is 0.858 MPa. When the content of added nano-ZnO particles increases from 0 to 7.5 wt%, the Young's modulus of the tensile sample reaches a maximum of 1.834 MPa. Compared with the tensile sample prepared from the original CN 9021NS prepolymer, the Young's modulus has increased by 114%. When the content of added nano-ZnO particles increases. When the content of added nano-ZnO particles increases from 7.5 wt% to 10 wt%, the Young's modulus of the tensile sample gradually decreases. When the content of nano-ZnO particles reaches 10 wt%, the Young's modulus of the tensile sample is 1.052 MPa. Compared with the content of added nano-ZnO particles at 7.5 wt%, the Young's modulus has decreased by 43%. However, it is still greater than the original CN 9021NS Young's modulus, and

compared with the tensile sample prepared from the original CN 9021NS prepolymer, the Young's modulus has still increased by 23%.

When the Young's modulus of the composite material increases with the increase of the content of added nano-particles, it is mainly due to the interaction between the nano-particles after the nano-particles are added to the elastomer, the influence of the nano-particles on the interface of the elastomer, and the result of the entanglement of the molecular chains inside the composite material. Because the nano-particles have a strong interface polarization^[8], and have good compatibility with the CN 9021NS prepolymer, they can be well dispersed in the elastomer. These nano-particles in the elastomer interact with the elastomer interface to form a filler network, greatly improving the rigidity of the elastomer, reducing toughness, and increasing the Young's modulus. As the content of nano-particles increases, the distance between the nano-particles becomes very close, and the interaction force between them becomes stronger, leading to a continuous increase in the Young's modulus of the tensile sample.

When the Young's modulus of the composite material decreases with the increase of the content of added nano-particles, it is mainly due to the saturation of the content of nano-particles, which causes the distance between the nano-particles to be very close, and they cannot be evenly dispersed into the material, causing agglomeration. There are voids inside these agglomerates, which can easily cause internal defects in the material and structural damage. This leads to a decrease in the rigidity of the material and a decrease in the Young's modulus. Although the excess nano-particles cause the Young's modulus to decrease, there are still some nano-particles with good dispersion inside the material. Therefore, when the content of nano-ZnO particles reaches 10 wt%, the Young's modulus of the tensile sample is still higher than that of the tensile sample without particles.



Figure 6: Changes in Young's modulus during stretching of tensile specimens with different amounts of nano-ZnO particles

As can be seen from Figure 6, the Young's modulus of the composite material is not a constant value during the stretching process, but is constantly changing. The Young's modulus of tensile samples with different nanoparticle contents first drops rapidly during the stretching process, then tends to stabilize, and finally rises slowly. That is, the Young's modulus-strain graph shows strong non-linearity.

In the initial stage of stretching, the Young's modulus of the material is very large. This is because when nanoparticles are added to the material, the nanoparticles will form a filler network with the contacted interface, and the addition of nanoparticles binds the mechanical properties of the composite material. The Young's modulus of the composite material is mainly composed of three parts: the entanglement between the molecular chains inside the composite material, the friction between the added nanoparticles, and the interaction between the nanoparticles and the contact interface. The interaction between the filler networks has the greatest impact on the Young's modulus.

As the sample is stretched and the strain is about 0-10%, these filler networks that improve the rigidity

of the sample deform under the action of external forces, become chaotic, and then cause damage, and can no longer increase the rigidity of the composite material. The Young's modulus of the material gradually decreases. This phenomenon is called the Payne effect^[9]. At the same time, as the sample continues to stretch, the nanoparticles slip on the interface, and the nanoparticle agglomerates inside are destroyed under the action of external forces, which causes a significant decrease in the Young's modulus of the composite material. But when the strain exceeds 10%., the Young's modulus of the composite material. The cross-linked network and nanoparticles that mainly provide the Young's modulus have failed, and the mechanical properties of the material tend to stabilize.

Finally, the Young's modulus of the material shows a slight increase. This is because the nanoparticles slip and change position with the stretching of the sample and the destruction of the internal structure, and the nanoparticles that change position come into contact with the elastomer interface again, the interaction force reappears, interface polarization occurs, and the material begins to have stress redistribution. Nanoparticles begin to provide a certain rigidity to the material again, and the Young's modulus increases slightly.

4.2.3 The impact on elongation at break

The elongation at break is an indicator of the softness and elasticity of the elastomer, reflecting the toughness and ductility of the composite material, and can to some extent reflect the deformation and driving ability of the dielectric elastomer during operation. The higher the elongation at break, the better the softness and elasticity of the elastic material. The higher the elongation at break, the greater the deformation potential of the dielectric elastomer.

When l(x) is equal to the distance l_{max} between the gauge reference points at the time of fracture of the tensile specimen, the fracture elongation E_b of the tensile specimen can be calculated using the following formula:



Figure 7: Elongation at break of tensile specimens with different amounts of nano-ZnO particles

As shown in Figure 7, the elongation at break of tensile specimens with different contents of nano-ZnO particles is presented. From the figure, it can be concluded that adding nano-ZnO particles will increase the Young's modulus of the tensile specimens. Compared with the CN 9021NS tensile specimens without added nano-ZnO particles, as the content of added nano-ZnO particles increases, the elongation at break of the tensile specimens shows a trend of first increasing and then decreasing. When the content of added nano-ZnO particles increases from 0 to 5 wt%, the elongation at break of the tensile specimens continues to increase. When no nano-particles are added, the elongation at break of the tensile specimens is 75.25%. When the content of added nano-ZnO particles increases from 0 to 5 wt%, the elongation at break of the tensile specimens reaches a maximum of 115.72%, compared with the original CN 9021NS prepolymer prepared tensile specimens, the elongation at break increased by 53.8%. When the content of added nano-ZnO particles increases from 5 wt% to 10 wt%, the elongation at break of the tensile specimens shows a continuous decreasing trend. When the content of added nano-ZnO particles is 7.5 wt% and 10 wt%, the elongation at break of the tensile specimens is 94.35% and 87.56% respectively,

compared with the nano-ZnO particle content of 5 wt%, they decreased by 18.5% and 24.3% respectively, but are still higher than the elongation at break of the original CN 9021NS prepolymer prepared tensile specimens.

The reason for the increase in the elongation at break of the tensile specimens as the content of nanoparticles increases is that the nano-particles have a large specific surface area and high surface binding energy, and the nano-particles interact with the elastomer interface to entangle and crosslink together. When the tensile specimen is stretched by an external force, the elastomer between the nano-particles deforms, and at the same time, the internally dispersed nano-particles will also transfer, shift, and share the stress received, absorb and buffer the impact energy received by the tensile specimen, thereby increasing the toughness of the tensile specimen and improving the elongation at break.

The reason for the decrease in the elongation at break of the tensile specimens as the content of nanoparticles decreases is that after the nano-particles are added to the elastomer, the nano-particles will form a filler grid with the contacted interface. However, when the content of nano-particles exceeds the threshold, the excess nano-particles cannot be evenly dispersed, the distance is too close, and at the same time, these unevenly dispersed nano-particles in the material also restrict the mobility of the large molecular chains in the elastomer, which will increase the rigidity of the tensile specimens and decrease the toughness. At the same time, an excess of nano-particles will produce an agglomeration effect, clustering together and unable to be evenly dispersed into the matrix, these agglomerated nano-particles have voids, these voids become the source of force and destruction when subjected to external forces, which makes the tensile specimen internal defects appear, and the structure becomes unstable. During the stretching process, these agglomerated nano-particles will also produce stress concentration when subjected to external forces, causing defects to be destroyed in advance, and the micro-cracks caused by nano-particles under external forces develop into macroscopic damage and break, thereby causing the material to break prematurely and reduce the elongation at break of the tensile specimens. Although an excess of nano-particles reduces the elongation at break, there are still some well-dispersed nano-particles inside the material, so when the content of nano-ZnO particles reaches 10 wt%, the elongation at break of the tensile specimens is still higher than that of the tensile specimens without added nano-ZnO particles.

4.2.4 The impact on tensile

Strength Tensile strength (Rm), also known as tear strength, is a material characteristic value for assessing the strength performance of materials. Tensile strength is the maximum mechanical tensile stress that a tensile specimen can withstand. If the applied stress exceeds the tensile strength, the material will break and fail: the absorption of force decreases until the material specimen finally tears. However, before reaching the actual tensile strength value, the material will undergo plastic deformation after experiencing elastic deformation, and finally fail completely.

When F(x) is equal to the tensile force F_{max} at the time of fracture of the tensile specimen, the tensile strength T_s of the tensile specimen can be calculated using the following formula:



Figure 8: Tensile strength of tensile specimens with different amounts of nano-ZnO particles

As shown in Figure 8, the tensile strength of tensile specimens with different contents of nano-ZnO particles is different. As the content of nano-ZnO particles added to the composite material increases, the tensile strength of the tensile specimens shows a trend of first increasing and then decreasing. Compared with CN 902NS without added nanoparticles, when the content of added nano-ZnO particles is between 0-7.5 wt%, the tensile strength of the tensile specimens gradually increases, and the tensile strength of the nano-ZnO particles with a content of 5 wt% is slightly lower than that of 7.5 wt%, which is 1.165 MPa. The tensile strength of the tensile specimens prepared by CN 902NS without adding nanoparticles is 0.471 MPa. When the content of added nano-ZnO particles increases to 7.5 wt%, the tensile strength of the tensile specimens gradually decreases to 7.5 wt%, the tensile strength of the tensile specimens gradually decreases. When the content of added nano-ZnO particles is between 7.5 wt%, the tensile specimens gradually decreases. When the content of added nano-ZnO particles is between 7.5 wt%, the tensile strength of the tensile specimens gradually decreases. When the content of added nano-ZnO particles is between 7.5 wt%, the tensile strength of the tensile strength of the tensile specimens gradually decreases. When the content of added nano-ZnO particles increases to 10 wt%, the tensile strength of the tensile specimens with a nano-ZnO particle content of 7.5 wt%, but it is still higher than the tensile strength of the original CN 9021 tensile strength of the original CN 9021 tensile specimens.

When the tensile strength of the tensile specimens increases with the increase of the nanoparticle content, this is due to the addition of nanoparticles. Due to the friction between the particles and the interaction between the particles and the interface, a crosslinked network is presented inside the composite material, which improves the mechanical properties of the elastomer material, greatly improves the rigidity of the material, the Young's modulus is greatly improved, and the tensile strength of the specimen also shows a gradually increasing trend with the increase of the nanoparticle content. The greater the tensile strength of the composite material with added nanoparticles, the stronger the ability to resist damage, and the greater the tensile force that can be withstood. However, the increase in tensile strength is synchronized with the increase in Young's modulus. Excessive rigidity reduces the softness and toughness of the composite material, which is not conducive to the rapid response of the elastomer and produces a large strain.

When the tensile strength of the tensile specimens decreases with the increase of the nanoparticle content, this is due to the excessive nanoparticles in the matrix that cannot be dispersed, resulting in agglomeration, and the presence of voids inside these agglomerates causes defects in the material. These agglomerates and voids will cause stress concentration and damage and fracture under force. At the same time, excessive nanoparticles will cause the filler network inside the material to be chaotic, the structural stability will decrease, and defects will be more easily induced under external force, causing the interaction force between the nanoparticles and the interface to fail. This leads to a decrease in tensile strength. But when the content of added nano-ZnO particles increases to 10 wt%, the tensile strength of the tensile specimens is still higher than the tensile strength of the original CN 9021NS tensile specimens. This is due to the fact that there is still a part of ZnO nanoparticles in the material that is well distributed, which improves the strength of the composite material to a certain extent, thereby improving the tensile strength of the sample.

5. Conclusions

(1) Saturation is reached when ZnO nanoparticles are added to the material with a content of 7.5 wt%. As the more nanoparticles are added, the more dense the distribution of particles in the composite, and when the saturation state is exceeded, the nanoparticles will be too dense and agglomerate.

(2) The addition of nanoparticles to the CN 9021NS improves the mechanical properties of the composite. It will increase the Young's modulus, elongation at break and tensile strength of the tensile specimen.

1) When ZnO nanoparticles were added to CN 9021NS, the Young's modulus of the composites would increase, and when the addition content was 0-7.5 wt%, the Young's modulus would increase with the increase of nanoparticle content, and when the nanoparticle content exceeded 7.5 wt%, the Young's modulus would decrease. However, it is still higher than the Young's modulus of CN 9021NS, and the excessively high Young's modulus also reduces the deformation properties of the elastomer.

2) When the nanoparticle content is 5 wt%, the nanoparticles have the best performance for improving the elongation at break of the tensile specimen, and the elongation at break of the tensile specimen with nano-ZnO particles reaches 115.72%. It is 1.54 times the elongation at break of the original CN 9021NS tensile specimen.

3) The tensile strength of the composites was increased by adding ZnO nanoparticles to the material,

and with the increase of the content of ZnO nanoparticles, the tensile strength first increased and then decreased, and when the content of nano-ZnO particles was 7.5 wt%, the tensile strength reached the maximum, which was 1.217 (MPa). It is 2.57 times the tensile strength of the original CN 9021NS tensile specimen. However, too high tensile strength means that the elastomer is not soft and not conducive to the deformation of the elastomer.

Therefore, from the perspective of mechanical properties, the addition of ZnO nanoparticles to CN 9021NS is excellent for the improvement of mechanical properties. When the content of nano-ZnO particles is 5 wt%, it has a lower Young's modulus (1.38 MPa), the highest elongation at break (115.72%) and a higher tensile strength (1.165 MPa).

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