Materials Research Express

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PAPER

Stretchable multifunctional dielectric nanocomposites based on polydimethylsiloxane mixed with metal nanoparticles

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Keywords: stretchable dielectrics, silver and nickel nanoparticles, polydimethylsiloxane, capacitor, strain sensor

Supplementary material for this article is available online

Abstract

Next generation wearable electronics require stretchable dielectrics. There has been significant effort to characterize and improve the components of dielectric composites for use in these devices. In this work, a new stretchable dielectric material, composed by silver nanoparticles (Ag NPs), nickel nanoparticles (Ni NPs), and polydimethylsiloxane (PDMS), is prepared and characterized. The alternating arrays of Ag NPs groups and Ni NPs groups in the three-dimensional matrix of PDMS function as micro capacitors and prevent current percolation. Compared with PDMS alone, the alternating arrays exhibit a dielectric constant (k) that is increased by 1146% and can reach 35.13, with dielectric loss as low as 0.009. Slightly lower k and larger dielectric loss appear at high frequencies. The material exhibits negative temperature dependence, and the composition ratio affects the dielectric properties. The strain at break is 139.68% and the elastic modulus is as low as 3.57 kPa. By controlling the type, size and dispersion of metal nanoparticles in PDMS matrix, a parallel-plate capacitor with constant capacitance is achieved, demonstrating the dependence of the dielectric constant on the applied strain. Moreover, by replacing the parallel plates with cylindrical fibers, a capacitive strain sensor was demonstrated. After hundreds of stretching-releasing cycles, the dielectrics work normally. The excellent properties of this material suggest its significant potential for use in wearable electronics.

1. Introduction

There is growing application of flexible and stretchable dielectrics for use in artificial intelligence and wearable electronics, such as matrix for electroluminescent e-skin [1, 2], triboelectric nanogenerators [3], soft generators [4], dielectric elastomer transducers [5] and actuators [6], transistors for nonvolatile memory and biomedical e-skin [7–9], and two-dimensional semiconductors [10]. Flexible ferroelectric polymers, including P(VDF-TrFE) and its blends with ceramics, have been well developed due to their high permittivity [11, 12]. For the achievement of stretchable performances, significant efforts have been focused on the elastomer as a matrix of flexible nanodielectric materials [13].

Stretchable dielectrics require excellent mechanical properties and high dielectric constant. However, elastomers exhibit very low permittivity and excellent stretchable properties, such as polyurethane (PU) [14], poly-Styrene-co-Ethylene-co-Butylene-co-Styrene (SEBS) [15] and dicarboxy-terminated poly(acrylonitrile-co-butadiene) with cross-linking 1,6-bis(trichlorosilyl)hexane [16]. One effective method to improve dielectric properties is to compound nanomaterials into elastomers. Liquid metal can be dropped into polydimethylsiloxane (PDMS) by complex processes [17, 18]. Gate dielectric layers of intrinsically stretchable thin-film transistors have been prepared with P(VDF-TrFE):PVP blends [19], PU-co-PEG [20], ionic gels composed by ionic liquids (polyelectrolyte), and gel [21, 22], and were characterized by charging technique [23]. All these polymers have insufficient dielectric constant. The interfacial treatment of TiO2 did not significantly improve the dielectric constant after blending with SEBS [24]. Highly stretchable PDMS has been
optimized by using Fe\(^{2+}\) as cross-linking sites, where the modulus is larger than 0.2 MPa \(^{25}\). Ionic liquids, ceramics, and carbon materials (nanotubes and graphite) are blended together with elastomers \(^{26–28}\), but the amounts of added conductive materials cannot be increased infinitely to enhance the dielectric constants due to the limit of the percolation threshold \(^{29}\).

One of the main applications of stretchable dielectrics is as the dielectric layer for capacitors. According to various requirements of electronic components for wearable devices, capacitors should show stable capacitance or monotonically increasing capacitance (capacitive sensors) during stretching \(^{30}\). Carbon nanotubes have been well utilized as electrodes of parallel-plate capacitors, which could show a change rate of 300% when subjected to large tensile strains \(^{31–33}\). Gold electrodes with microstructures could also be used as the electrodes of capacitive strain sensors \(^{34, 35}\). There are two main considerations for these capacitive sensors: the mismatch between the electrode and the dielectric layer caused by different materials, and the contradiction between excellent sensitivity and stable capacitance. As electrodes of the capacitors, Gold and CNTs require microstructure to accommodate mechanical strain. It is complicated to connect these robust electrodes to the soft polymer dielectric layer because the robust electrodes have much higher elastic modulus values and a much narrower range of elastic deformation. When stretched, the deformation of the microstructure is no longer synchronized with the tensile strain of the polymer and the joint between the electrode and the dielectric layer may be damaged, resulting in a mismatch \(^{36}\). This issue can be addressed by similar elastic modulus values between the electrode and dielectric layer or by even using the same materials. Therefore, stretchable nanocomposites’ composition, excellent dielectric performances, and matched modulus to that of electrodes are key features of capacitors that enable constant and sensitive capacitances at tensile strains.

Here, we report a new stretchable multifunctional dielectric material composited by Ag NPs, nickel nanoparticles (Ni NPs), and polydimethylsiloxane (PDMS). The alternating arrays of Ag NPs groups and Ni NPs groups in the three-dimensional matrix of PDMS act as micro capacitors, preventing current percolation and thus achieving high dielectric constant and dielectric loss as low as 0.009. The effects of composition, frequency, temperature, and mechanical stretching on the dielectric properties of these novel materials are studied. By changing the form of the structure, the capacitors with these composites as the dielectric layers not only exhibit stable capacitance, but also act as a strain sensor.

2. Experimental

2.1. Materials

Ag NPs and Ni NPs were purchased from Aladdin (see the particle size in figure S1 is available online at stacks.iop.org/MRX/7/015007/mmedia), and PDMS was purchased from Dow Corning (Sylgard 184). All reagents were used as received.

2.2. Synthesis of the composites

The elastomer is made of polydimethylsiloxane and prepared by mixing the liquid base monomer and the curing agent at 10:1 (w : w). This liquid mixture is then poured into a very shallow container and the air voids are removed by application of vacuum. Ag NPs were composited with liquid PDMS, followed by mechanic stirring for 5 min with ultrasonic assisted dispersion. After blending, Ni NPs were introduced to the obtained well-dispersed composite fluids and mixed by mechanic stirring for 10 min with ultrasonic-assisted dispersion. After the compositing process, the well-dispersed composites were transferred into cylindrical containers, followed by heat treatment. Preparation of the cylindrical samples with size of 8 mm diameter and 1 mm thickness was completed after dwelling at 60 °C for 23 h and sintering at 120 °C for 5 h and 40 min.

2.3. Highly stretchable composites

After blending the Ag NPs, Ni NPs and liquid PDMS, the liquid composites are sintered at 120 °C for 2 h. For the sample that is used to test the stress-strain curves, the liquid composites are transferred into a square mold before the heat treatment.

2.4. Stretchable parallel-plate capacitor

A dielectric layer is sandwiched between two parallel plates electrodes, as shown in the inset of figure 4(a). This dielectric layer is prepared with the above stretchable dielectric composites, of which the size is 18.5(L) × 4.5(W) × 0.9(t) mm\(^3\). The parallel plates electrodes are made of conductive adhesives (CAs) composited by PDMS and laboratory-prepared silver nanoparticles (W(Ag NPs) = 42%). Ag NPs were prepared by reducing silver nitrate with hydrazine hydrate, and Polyvinyl Pyrrolidone was used as dispersant. Please refer to the following report for specific methods: Feng et al Nanotechnology 30(2019) 185 501. The intrinsically stretchable
2.5. Stretchable fiber capacitor

First, a size Length = 35 mm and Diameter = 0.5 mm stretchable fiber made from PDMS is used as the core substrate for the capacitor. Second, the CAs described above cover the surface of the core substrate as the inner electrode (diameter = 1 mm and length = 4.7 mm). Then the liquid dielectric composites are coated onto the inner electrode (diameter = 2 mm), followed by storing in a drying box for 48 h at 26 °C. Finally, the CAs are coated onto the surface of the dielectric layer to serve as the outer electrode (diameter = 3 mm), and storing in a drying box for 9 days at 26 °C.

2.6. Dielectric properties

Electric conductor silver fluid (FY20, Shanghai Julong Ltd), was used as electrode material and was coated onto the two bases of the cylindrical sample, followed by drying at 60 °C for 60 min. The dielectric constant and loss were tested using a HP Agilent 4284 A precision LCR meter, with frequency that ranged from 10² to 10⁶ Hz and a temperature range of 25 °C to 150 °C.

2.7. Characterization

The x-ray diffraction (XRD curves) were obtained by x-ray diffraction equipment, with current of 40 mA, voltage of 200 kV, speed of 5° min⁻¹, and range of 10° ~ 80°. Scanning electron microscopy (SEM) images and energy dispersive x-ray (EDX) maps were analyzed by field emission scan electron microscope, with voltage of 15 kV and scanning backscattered electrons. Samples with cuboid shape of 20 × 5 × 1 mm³ were fixed on the leadscrew rail to withstand stretching-releasing cycles and acquire the stress-strain (σ−ε) curves. The stretchable parallel plate capacitor and fiber capacitor were characterized by Hitachi LCR meter.

3. Results and discussion

3.1. Fabrication of the composites

The fabrication process of the dielectric composites is shown in figure 1. First, Ag NPs are mixed into liquid PDMS, followed by introducing Ni NPs into the mixture. Liquid composites are obtained after mechanically stirring and ultrasonic assistance. The composites are incubated in a drying cabinet and sintered in oven to prepare the solid dielectric composites.

The schematic distribution of metal nanoparticles in the PDMS matrix is shown in the image that appears like a crystal cell (figure 1(f)). In this picture, the balls and PDMS are real, but all the solid and dashed lines with yellow, red, blue, and brown color are phantom lines. A small cell gathered by several Ag NPs is located in the center of the large cell, while the small cells gathered by several Ni NPs are located in the 8 vertices of this large cell. The Ag groups and Ni groups were placed alternatively in the matrix, in a strategy to prevent current percolation.

3.2. Microscope characterization

Figure 2(a) shows the XRD maps of the composites, which exhibited peaks corresponding to silver and nickel. According to the element index analysis, no miscellaneous peaks appear. Figure 2(b) shows the Raman maps. Due to the small number of nickel peaks and the weak strength, the peaks of the composites mainly coincide with the positions of the silver peaks.

The SEM images of the composites prepared with w(Ag) = 30% and w(Ni) = 30%, are shown in figures 2(c) and (d), respectively. Small amounts of nanoparticles are grouped together, and then Ag groups and Ni groups are alternately distributed inside the PDMS matrix. In figure 2(d), inside the yellow circle is the silver group, and inside the red box is the nickel group. The Ag NPs are shown in the inset SEM image. All nanoparticles are covered with organic matter and with the inherent magnetic effect of nickel, it is difficult to clearly see the particles. However, almost all the nanoparticles in these SEM images can be seen and counted individually, so the dispersion state can be confirmed.

For the composites prepared with w(Ag) = 30% and w(Ni) = 30%, the SEM image and EDX maps are shown in figure 2(e). Similarly, inside the yellow circle is the silver group, and inside the red box is the nickel group. Although ultrasonically-assisted mechanical agitation did not result in the uniform distribution of silver nanoparticles and nickel nanoparticles, nickel was introduced into the matrix after adding and blending Ag NPs. Thus silver group and nickel group with a size of about 1 micron were formed. In the SEM image, the brightness of Ag groups is higher than that of Ni groups, and the PDMS is mainly located in the dark region. The EDX maps reveal distribution of PDMS (represented by Si) throughout the sample, and the Ag groups and Ni groups are...
placed alternatively. For the sample with composition of $w(\text{Ag}) = 25\%$ and $w(\text{Ni}) = 35\%$, the SEM image and EDX maps are shown in figure 2. This law of distribution for these components is same as for the above composite. The higher magnification EDS image shows the alternating distribution and interface of the Ag group and the Ni group, as shown in figure S2. The distribution of silver elements is represented by red pixels, and nickel is represented by green.

3.3. Dielectric properties

The dielectric properties of the composite can be affected by the composition, frequency, and temperature, and the dielectric measurements are shown in figure 3. At room temperature (25 °C), the dielectric constant ($k$) of cured PDMS is 2.82 at frequency of 1 kHz, as shown in figure 3(a). At the same temperature and frequency, the composites of $w(\text{Ag}) = 30\%$ and $w(\text{Ni}) = 30\%$ exhibit $k$ of 35.13, a value that is 1146% higher than that of PDMS alone. When the frequency increased to 1 MHz, the $k$ was 33.47, only 4.7% lower, indicating slight frequency dependence. The dielectric composites of $w(\text{Ag}) = 25\%$ and $w(\text{Ni}) = 35\%$ were determined as $k$ of 26.85 at 1 kHz and 25.57 at 1 MHz. The amount of silver present has an important effect on the $k$ value.

Figure 3(b) shows the dielectric loss of the composites. At low frequency range, less than 1 kHz, the composites exhibit significant lower loss than PDMS. The dielectric loss of the composites is only 0.01 at high frequency.

Next, the temperature dependence was investigated, as shown in figures 3(c)–(f). For the composition of $w(\text{Ag}) = 30\%$ and $w(\text{Ni}) = 30\%$, the $k$ decreased from 35.10 to 28.23 at 1 kHz and from 33.46 to 27.27 at 1 MHz as the temperature increased from 25 °C to 150 °C, as shown in figure 3(c). The dielectric loss is 0.020 ~ 0.046 for the full temperature range, as shown in figure 3(d). For the composition of $w(\text{Ag}) = 25\%$ and $w(\text{Ni}) = 35\%$, the $k$ decreased from 26.84 to 21.60 at 1 kHz and from 25.56 to 21.04 at 1 MHz as the temperature increased from 25 °C to 150 °C, as shown in figure 3(e). The dielectric loss was 0.019 ~ 0.047 over the full temperature range, as shown in figure 3(f). The composites exhibited a negative temperature coefficient. For the temperature range of 25 °C to 150 °C, the dielectric constant was always slightly higher at low frequency than at high frequency, while the dielectric loss showed the opposite trend. In isothermal mode, the temperature dependence in the whole frequency band was the same as the above rule, as shown in figure S3. In general, the dielectric constant of ceramic materials increases with increasing temperature, and the dielectric constant of non-polar polymers decreases with increasing temperature [37]. The matrix (PDMS) in this work is a non-polar polymer, and its dielectric properties exhibit a negative temperature coefficient. The ability of atoms to move in nanocomposites affects the number of dipole moments and thus reduces the relative permittivity. The flexibility
of PDMS deteriorates with increasing temperature \cite{38, 39}. At temperatures higher than 90 °C, PDMS is not a very soft polymer. This will inevitably affect the polarization of the Ag group and the Ni group to further reduce the dielectric constant.

### 3.4. The parallel-plate capacitor

An intrinsically stretchable parallel-plate capacitor with almost constant capacitance was fabricated, in which the dielectric composites (w(\text{Ag}) = 30\% and w(\text{Ni}) = 30\%) were utilized as the middle dielectric layer. The stretchability of the capacitor was achieved not only by the electrode layers prepared with the stretchable and conductive CAs, but also by using the same PDMS matrix for the electrode layers and the middle layer.

Figure 2(a) exhibits the capacitances at different tensile strains, and the inset is a schematic of the sample’s structure and the test method. Two copper cylinders were used to connect the electrodes of this capacitor and
the LCR meter. The parts of the middle layer that were not covered by electrodes were sandwiched tightly between two clamps of the lead screw to apply tensile stress to the capacitor. During stretching, the capacitances at 100 Hz were 2.81 pF and 2.73 pF for tensile strains of 0% and 100%, respectively. No significant fluctuations of this capacitor’s performance occurred with the applied strains. This property was contributed by a continuous decrease of the relative dielectric constant \( \varepsilon_r \) during stretching. The capacitance of parallel-plate capacitors is given by

\[
C = \varepsilon_0 \varepsilon_r \frac{S}{t}
\]  

(1)

where \( C \) is the capacitance, \( \varepsilon_0 \) is the permittivity of vacuum, \( S \) is the area of the electrode, and \( t \) is the thickness of the middle layer. With increased strain, the area increased gradually and the thickness continued to decrease. The areas (S) at various strains are given in figure 4(b). The inset shows the direction of the tensile force (F) and the sizes of this capacitor including the electrode length (L), width (W), and the middle layer’s thickness (t). The S was given by

\[
S = L \times W
\]  

(2)

and monotonically grew from 18.25 mm² at 0% strain to 25.81 mm² at 100% strain. As the strain grew, the length improved from 4.45 mm to 8.90 mm, the width decreased monotonically from 4.10 mm to 2.90 mm, and the thickness slowly reduced monotonically from 0.9 mm to 0.39 mm, as shown in figure 4(c). Using formulas (1) and (2), the k at 100 Hz of the composites under different strains was calculated, as shown in figure 4(d). The values dropped from 15.66 to 4.67 with increasing strain.

Figure 4(e) presents a schematic of dispersion in the cross section of this capacitor. The colors are the same as those in figure 1. Large-diameter Ag NPs are distributed in the upper and lower parts of the PDMS matrix, where electrons are hopping on a large scale and thus current percolation occurs because the gaps between the edges of the particles are less than 10 nm [36]. However, as the result of this large number of electron hoppings, these large-sized silver nanoparticles prepared in our laboratory cannot be used as fillers for dielectric layers. Commercial silver nanoparticles were used with the above two-step heat treatment process. In contrast, Ni NPs and small-diameter Ag NPs alternately locate in the middle parts of the PDMS matrix, where the occurrence of percolation is nearly impossible because the gaps between the edges of most particles are much larger than 10 nm. However, the dielectric constant of the middle region is increased due to heterogeneous interfaces and polarizations. Finally, an intrinsically stretchable capacitor is prepared since the top and bottom parts are conductors and the middle contains dielectrics. Use of the same matrix of electrodes and dielectrics prevents mismatch. However, the increase of the particle gaps during the stretching process leads to deformation of the
interfaces and weakening of the polarization, and thus the dielectric constant gradually decreases to realize constant capacitance.

3.5. The cylindrical fiber capacitor
Wearables and medical tools are inseparable from electronic fibers [40, 41]. Fiber materials require electrical sensors, but also mechanical flexibility, stretchability, and scalability. Therefore, industrialized electrical fibers must be multimaterial and capable of scalable production to achieve the above comprehensive performance [42]. The multimaterial property means that scalable stretchable fiber prepared by thermal draw method is used as a carrier, and then the electrical and dielectric materials are packaged or encapsulated [43, 44]. However, the PDMS fibers in this work were not prepared by the thermal drawing process, so it was difficult to prepare ultralong fibers for scalability. Instead, a short fiber was used as a substrate for a multimaterial cylindrical capacitive strain sensor.

Figure 5(a) shows the schematic of the cylindrical structure. The composite materials for the electrode and the dielectric layer were the same as those used for the above parallel-plate capacitor. The cylindrical PDMS fiber
and hollow cylindrical electrodes and dielectric layers were stacked in sequence. The dispersion of various particles was the same as that of the former parallel-plate capacitor. Figure 5(b) displays the performances of this sensor. The initial capacitance ($C_0$) was 226 pF. The change rate ($\Delta C/C_0$) was monotonically improved to 0.6 at 50% strain. The gauge factor (GF) was given by

$$GF = \frac{C - C_0}{\varepsilon C_0}$$

where $\varepsilon$ represents the strain and C represents the capacitance at the strain of $\varepsilon$. GF at 10% strain was 3 and dropped to 1.2 at 50% strain. Since there was no linearity, this capacitive sensor did not exhibit a constant GF.

3.6. Mechanical performance

The stretchable properties of the materials were determined and are shown in figure 6. For the composites of $w$(Ag) = 30% and $w$(Ni) = 30%, the stress ($\sigma$)-strain ($\varepsilon$) curve was obtained, as presented in figure 6(a). The maximum stress of 452.02 kPa occurred at a strain of 135.17%, the strain at break reached 139.68%, and the elastic modulus was only 3.57 kPa according to the fitted line. For the composites of $w$(Ag) = 25% and $w$(Ni) = 35%, the maximum stress of 572.58 kPa appeared at 120.90% strain, the strain at break was 126.16%, and the elastic modulus was 5.18 kPa.

Hundreds of stretching-releasing-restretching cycles under strain of 0% ~ 80% ~ 0% were applied to the composites. The dielectric properties for the composition of $w$(Ag) = 30% and $w$(Ni) = 30% are shown in figures 6(c) and (d). Each time the sample was subjected to a certain number of cycles, the conductive silver paste was coated onto the sample at 0% strain and then the dielectric properties were tested. The $k$ at 1 kHz was 21.67, 19.42, 19.13, and 18.22 after 0, 10, 100, and 1000 cycles, respectively, indicating a continuous and slow reduction (as indicated by the brown arrow). The dielectric loss at 1 kHz was 0.048 ~ 0.050 and exhibited no obvious change after 1000 cycles, showing weak cycle dependence. The dielectric performance of these materials demonstrated the strong frequency dependence. The insets shown in figure 6(d) are optical photos of the sample before and during stretching.
The dielectric properties for the composition of $w(\text{Ag}) = 25\%$ and $w(\text{Ni}) = 35\%$ were determined and are shown in figures 6(c) and (f). The test method was as described above. The $k$ at 1 kHz was 17.97, 14.75, 14.03, and 12.91 after 0, 10, 100, and 1000 cycles, respectively, indicating a continuous and slow reduction (as indicated by the brown arrow). The dielectric loss at 1 kHz was 0.049 ~ 0.057 and exhibited no obvious change after 1000 cycles, showing weak cycle dependence. The dielectric performance of these materials also demonstrated strong frequency dependence.

4. Conclusion

In summary, new stretchable dielectric composites were prepared based on alternating arrays of silver and nickel groups in a three-dimensional matrix of PDMS. Compared with PDMS alone, the dielectric constant of the composites ($w(\text{Ag}) = 30\%$ and $w(\text{Ni}) = 30\%$) increased by 1146% and was as high as 35.13. The dielectric loss was 0.009 at 25 °C and 1 MHz. Slightly lower $k$ value and larger dielectric loss were evident at low frequencies.
The composites exhibit a negative temperature dependence of $k$. The strain at break was 139.68% and the elastic modulus was only 3.57 kPa. Using these composites as dielectric layers and CAs as electrodes, parallel plate capacitors exhibit stable capacitance, while cylindrical fiber capacitors act as strain sensors. Nevertheless, these dielectrics could endure hundreds of stretching-releasing cycles. These novel dielectric composites exhibit significant potential for wearable electronic application.

Acknowledgments

This work was supported by Shenzhen Peacock Team Plan [KQTD20170809110344233], Shenzhen Science and Technology Innovation Commission [JCTJ20170811160129498] and Bureau of Industry and Information Technology of Shenzhen through the Graphene Manufacturing Innovation Center [201901161514].

Conflicts

None.

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