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POLYMER/CARBON NANOTUBE NANOCOMPOSITES FOR STRAIN SENSING APPLICATIONS

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The integration of nanomaterials, particularly carbon nanotubes (CNTs), into polymer matrices has enabled the development of advanced functional materials. The research investigates the potential of high-density polyethylene (HDPE) reinforced with multi-walled carbon nanotubes (MWCNTs) as a strain-sensing material. MWCNT loadings of 0.1 wt%, 0.5 wt%, and 1.0 wt% are investigated. The study examines the correlation between applied strain and the resulting change in electrical conductivity of the nanocomposite, encompassing material preparation, characterisation, and performance evaluation. The results demonstrate the feasibility of MWCNT/HDPE nanocomposites as sensitive and reproducible strain sensors, exhibiting a clear piezoresistive response.

Keywords: *Carbon nanotube, degree of electrical recovery, electrical conductivity, HDPE, nanocomposite, piezoresistivity, polymer, relaxation time, strain sensing.*

1. INTRODUCTION

Strain sensors are essential components in a wide range of applications, from structural health monitoring to biomedical devices. Conventional strain sensors, while effective, often require external power and signal acquisition systems, limiting their utility in embedded or remote sensing scenarios [1]. Optical fibre sensors, capable of embedded measurements, are often cost-prohibitive [2]. Nanomaterials, especially carbon nanotubes (CNTs), present a promising alternative for developing next-generation strain sensors due to their unique electromechanical properties [3], [5]–[7]. CNTs exhibit a change in electrical conductivity upon mechanical deformation, a phenomenon known as piezo resistivity, which can be exploited for strain sensing. This study investigates the strain sensing capabilities of MWCNT/HDPE nanocomposites, focusing on the relationship between applied strain and electrical conductivity, reproducibility,

and electrical recovery behaviour. A clear piezoresistive response in the fabricated nanocomposites was demonstrated, suggesting their potential for strain sensing applications.

2. EXPERIMENTAL DETAILS

2.1. Materials

MWCNTs (95 % purity, 20–50 μm length, 10–20 nm diameter) were purchased from the Chengdu Institute of Organic Chemistry, Chinese Academy of Sciences. HDPE (0.98 g/cm³ density, 4.0 g/min melt flow index (MFI)) was obtained from ExxonMobil Corporation, UK. Materials were used as received.

2.2. Nanocomposite Preparation

MWCNTs were dispersed within the HDPE matrix using a patented two-step method [8], [9]. This technique coats individual HDPE powder particles with MWCNTs, ensuring uniform dispersion and minimising agglomeration without altering the polymer's morphology. In the first step, MWCNTs were dispersed in deionized water using sonication to form a stable suspension. This suspension was then mixed with HDPE powder. In the second step, the mixture was heated to 80 °C, softening the HDPE particle surfaces and allowing the MWCNTs to adhere. This method promotes uniform MWCNT distribution by anchoring them to the HDPE particles before melt processing, thereby minimising agglomeration and maximising interfacial interaction. Nanocomposites with 0.1 wt%, 0.5 wt%, and 1.0 wt% MWCNT loadings were prepared. The mixtures were then hot-pressed at 135 °C and 18 tonnes of pressure to form rectangular sheets (100 mm x 15 mm x 5 mm).

2.3. Morphological Characterisation

The morphology and dispersion of MWCNTs within the HDPE matrix were analysed using field emission gun scanning electron microscopy (FEGSEM) (LEO 1530VP) at 5 kV. Samples were fractured to expose the internal structure and minimise deformation artefacts during sample preparation.

2.4. Electrical Conductivity Measurements

Electrical resistance measurements for (100 mm x 15 mm x 5 mm) samples were performed at room temperature using a three-point bending rig (W.E.S. Plastics Pty Ltd.). Controlled forces (9.8N, 19.6N, 29.4N, and 39.2N) were applied, and the corresponding deformation and resistance were recorded. Loading and unloading

cycles were repeated to assess reproducibility. Electrical recovery was investigated by monitoring resistance after unloading for extended periods (2 days) to determine the degree of recovery at various relaxation times.

3. RESULTS AND DISCUSSION

3.1. Morphology

SEM images (Figs. 1–3) revealed the morphology of HDPE and MWCNT powders and the dispersion of MWCNTs within the HDPE matrix. The patented dispersion process resulted in a uniform distribution of MWCNTs on the HDPE surface, minimising agglomeration. Increasing MWCNT content led to denser network formation, which is crucial for electrical conductivity. While some agglomerates were observed, they potentially contributed to the formation of conductive networks during hot-pressing [12].

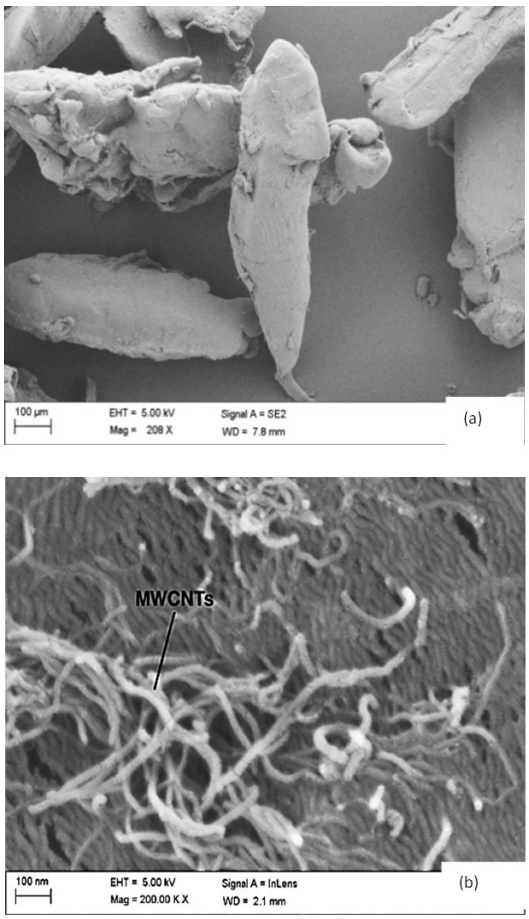

Fig. 1. SEM images of HDPE and MWCNT powder micrographs [10].

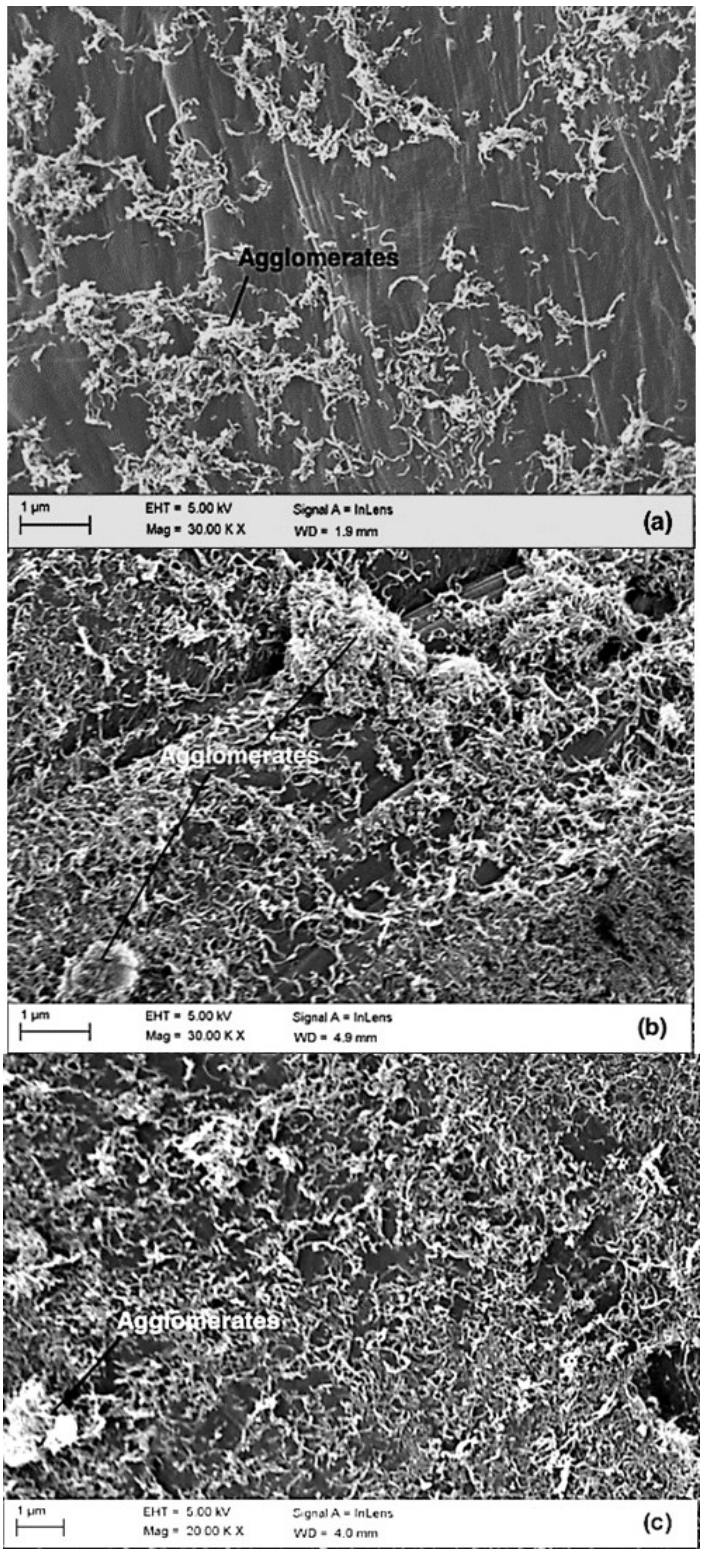

Fig. 2. SEM images of (a) 0.1 wt.% MWCNT/HDPE nanocomposite powders; (b) 0.5 wt.% MWCNT/HDPE nanocomposite powders; (c) 1.0 wt.% MWCNT/HDPE nanocomposite powders [10].

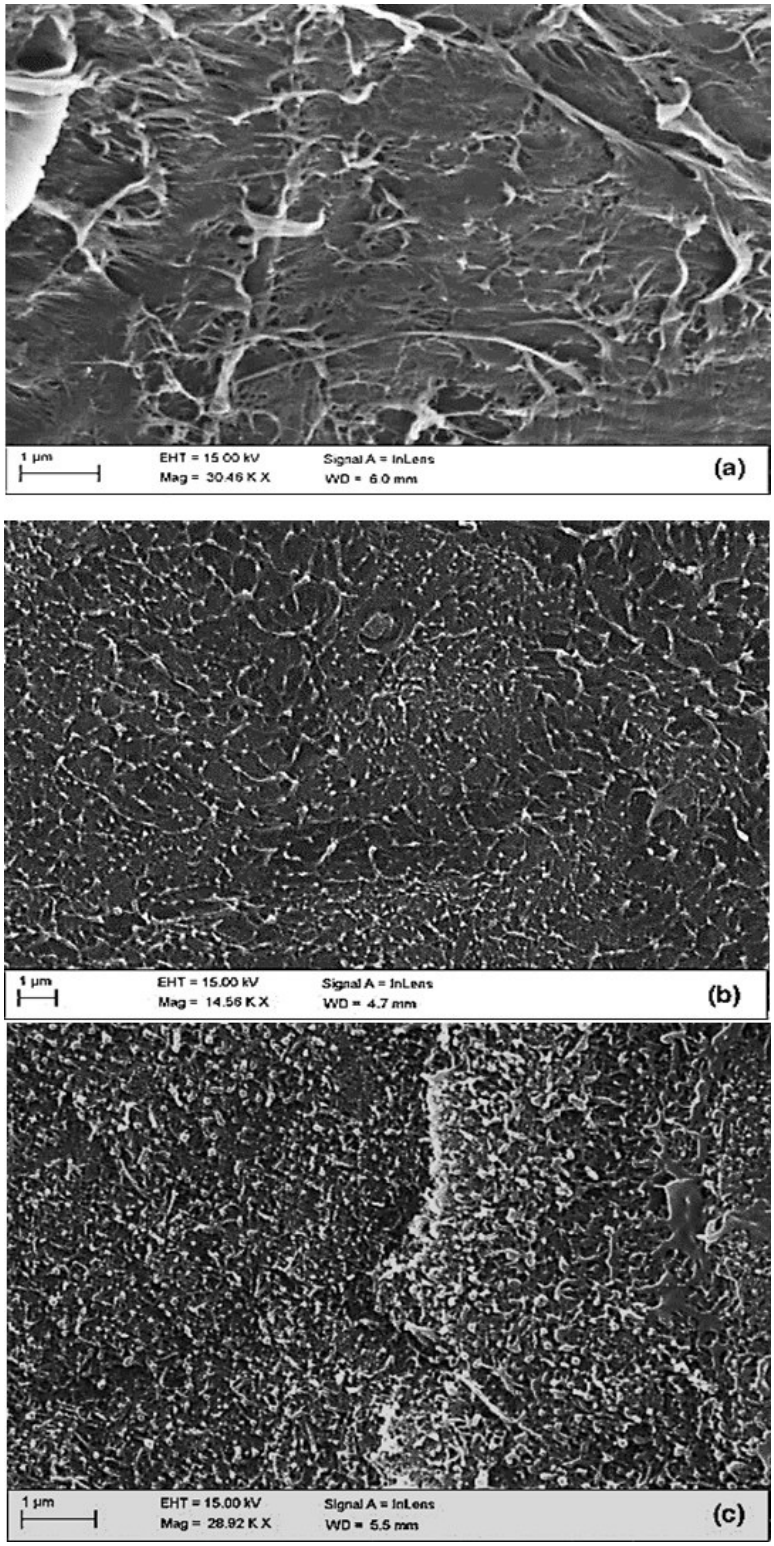

Fig. 3. SEM images of surfaces fracture of (a) 0.1 wt.% MWCNT/HDPE nanocomposite sheet; (b) 0.5 wt.% MWCNT/HDPE nanocomposite sheet; (c) 1.0 wt.% MWCNT/HDPE nanocomposite sheet [10].

3.2. Conductivity of MWCNT/HDPE Sheets

The initial electrical resistance decreased significantly with increasing MWCNT content (Table 1), indicating the formation of percolating conductive networks. The relationship between MWCNT content and electrical resistance is clearly shown in Table 1. While the precise percolation threshold was difficult to determine with the limited data points, the trend suggested that the 1.0 wt% MWCNT nanocomposite was closer to the percolation threshold, exhibiting the lowest resistance. This observation aligns with previous studies showing enhanced electrical stability near the percolation threshold [14].

Table 1. The Initial Electrical Resistances of MWCNT/HDPE Nanocomposites

Weight fraction of MWCNTs in each sample	Average electrical resistance (kΩ)
0.1 wt.%	1057
0.5 wt.%	149
1.0 wt.%	13.3

3.3. Deformation-Dependent Electrical Conductivity

The electrical resistance of the nanocomposites increased linearly with applied force and deformation (Fig. 4 and Table 2). This piezoresistive behaviour arises from the disruption of the conductive MWCNT network as the inter-tube distances increase under tensile strain [15], [16]. The 5 mm sample thickness might have limited the sensitivity to smaller deformations.

Table 2. Results of the Applied Forces, Deformations and Resistance for the 0.5 wt.% MWCNT/HDPE Nanocomposite

Applied force (N)	Deformation (mm)	Resistance (kΩ)
0	0	136.44
9.8	0.37	136.50
19.6	0.82	136.56
29.4	1.28	136.66
39.2	1.81	136.93

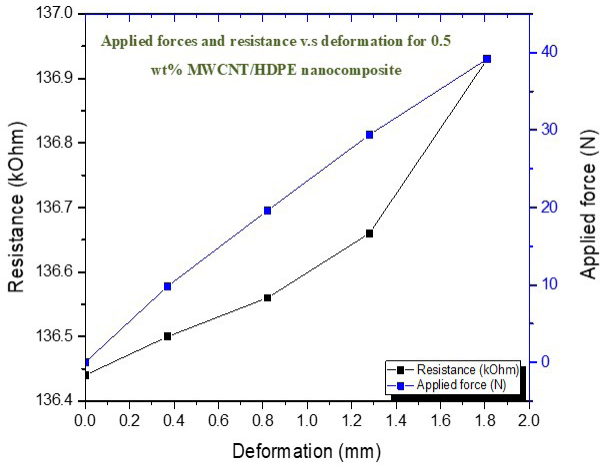

Fig. 4. Plot of applied force and resistance versus deformation for 0.5 wt.% MWCNT/HDPE.

The piezoresistive behaviour during cyclic loading and unloading is shown in Fig. 5. Applying a 4 kg load to both 0.5 wt% and 1.0 wt% MWCNT/HDPE samples resulted in an increase in electrical resistance, corresponding to the deformation (strain) of the material. This is attributed to the disruption of the conductive MWCNT network as the material stretches, increasing the inter-tube distances. Upon unloading, the resistance decreased, but did not fully return to its initial value, indicating incomplete recovery of the conductive network. Furthermore, subsequent loading cycles exhibited a progressively higher initial resistance, suggesting cumulative and irreversible changes within the MWCNT network due to repeated loading. Figure 5 illustrates this behaviour for the 0.5 wt% MWCNT/HDPE nanocomposite. This partially irreversible change in resistance with deformation highlights the material's potential for strain sensing applications, although the hysteresis effect should be considered for accurate measurements. Further investigation into the microstructural changes occurring within the MWCNT network during these loading cycles could provide valuable insights into this behaviour.

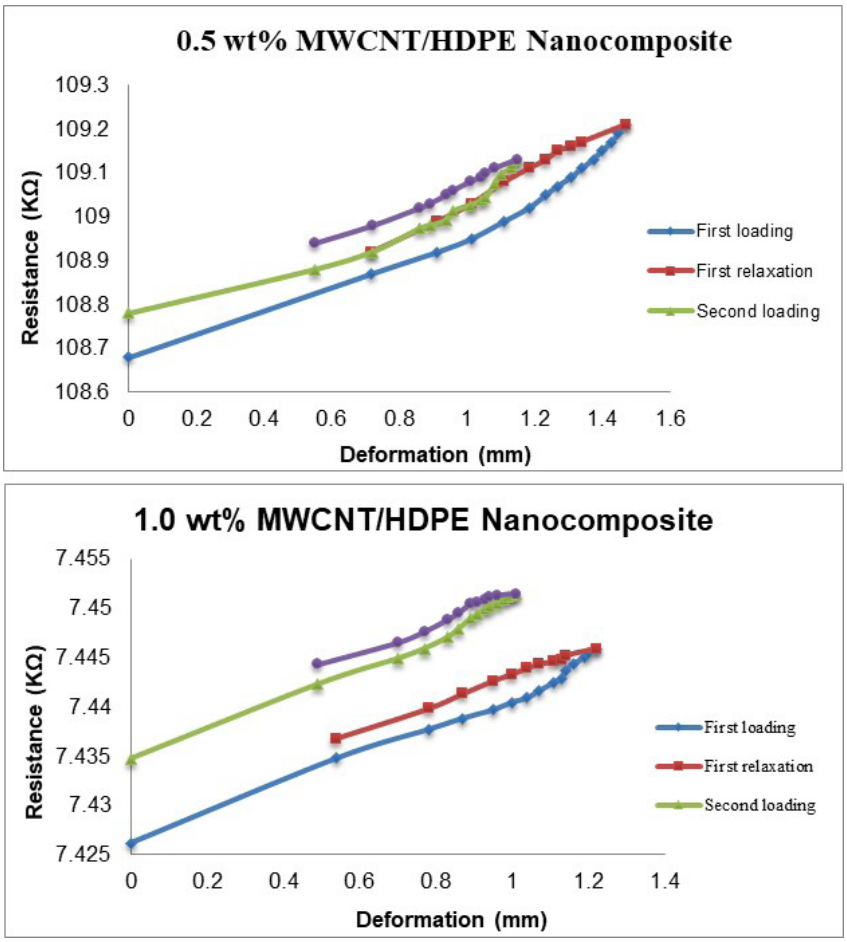

Fig. 5. Plot of resistance versus deformation for 0.5 wt.% and 1.0wt% MWCNT/HDPE nanocomposite.

3.4. Electrical Recovery Behaviour

3.41. Electrical Recovery and Viscoelasticity

Upon unloading, the electrical resistance of both nanocomposites did not fully return to its initial value after two days of relaxation (Fig. 5). This incomplete recovery is attributed to the viscoelastic nature of the HDPE matrix [17]. The polymer chains require time to relax back to their original configuration after deformation. This time-dependent recovery can be described using the Voigt-Kelvin model [17], which can be described using Eq. (1):

$$R(t) = R_m(\varepsilon) + R_d \exp(-t/\tau), \quad (1)$$

where R(t) is the resistance as a function of time, $R_m(\varepsilon)$ is the unrecoverable resistance at strain ε, R_d is the change in resistance due to deformation, t is time, and τ is a time constant at the given strain.

The initial resistance for the second loading cycle was lower than the final resistance of the first unloading cycle, indicating partial recovery during the two-day relaxation period.

3.42. Reproducibility and Degree of Electrical Recovery

Electrical resistance did not immediately return to its initial value, demonstrating a viscoelastic response of the polymer matrix [17], [18]. The degree of electrical recovery was calculated (Tables 3 and 4) using Eq. (2):

$$\text{The degree of electrical recovery (\%)} = \frac{R_{initial} - R_{final}}{R_{initial} - R_{loaded}} x100, \quad (2)$$

where $R_{initial}$ is the initial resistance of the material before any load or deformation is applied; R_{loaded} is the resistance of the material at the point of maximum deformation or load; R_{final} is resistance of the material after a specific relaxation time.

It was observed that the 1.0 wt% MWCNT nanocomposite exhibited better reproducibility during loading/unloading cycles. The incomplete recovery is attributed to the time-dependent relaxation of polymer chains [17]. The relaxation time significantly impacts the calculated degree of electrical recovery. Tables 3 and 4 present the degree of electrical recovery in two cycles.

Table 3. Degree of the Electrical Recovery at Each Deformation for the 0.5 wt.% MWCNT/HDPE Nanocomposite

First relaxation cycle		Second relaxation cycle	
Deformation (mm)	Degree of electrical recovery (%)	Deformation (mm)	Degree of electrical recovery (%)
0.718	99.95	0.550	99.95
0.913	99.94	0.720	99.95
1.013	99.93	0.860	99.96
1.111	99.92	0.890	99.95
1.183	99.92	0.940	99.95
1.233	99.93	0.960	99.96
1.268	99.93	1.010	99.95
1.308	99.94	1.040	99.95
1.338	99.95	1.055	99.95

Table 4. Degree of the Electrical Recovery at Each Deformation for the 1.0 wt.% MWCNT/HDPE Nanocomposite

First relaxation cycle		Second relaxation cycle	
Deformation (mm)	Degree of electrical recovery (%)	Deformation (mm)	Degree of electrical recovery (%)
0.540	99.97	0.490	99.97
0.780	99.97	0.700	99.98
0.870	99.96	0.770	99.98
0.950	99.96	0.830	99.98
1.000	99.96	0.860	99.98
1.040	99.96	0.890	99.98
1.070	99.96	0.910	99.98
1.110	99.97	0.930	99.99
1.130	99.97	0.940	99.99
1.140	99.98	0.960	99.99

After two loading/unloading cycles, the degree of electrical recovery for both 0.5 wt% and 1.0 wt% MWCNT nanocomposites was approximately 99.76 %. Tables 3 and 4 present the degree of electrical recovery at various deformation levels for both nanocomposites. The second relaxation period showed a higher degree of recovery at each deformation level compared to the first, suggesting improved reproducibility with subsequent cycles. This is consistent with the smaller resistance gaps observed between loading and unloading in the second cycle.

The 1.0 wt% MWCNT nanocomposite exhibited smaller deformations and higher degrees of electrical recovery at each deformation level compared to the 0.5 wt% nanocomposite. While the ultimate recovery after two days was similar for both, the 1.0 wt% nanocomposite demonstrated better reproducibility throughout the loading/unloading cycles. This enhanced performance is likely due to the denser conductive network formed by higher MWCNT concentration that facilitates more efficient recovery of conductive pathways upon unloading [18].

4. CONCLUSION

This study has demonstrated the potential of MWCNT/HDPE nanocomposites as strain sensors. The nanocomposites exhibit a clear and reproducible piezoresistive response, with electrical resistance increasing linearly with applied strain.

The patented dispersion process ensures relatively uniform MWCNT distribution, enhancing performance and stability. Future research should focus on optimising MWCNT concentration, sample geometry, and testing parameters to tailor sensor sensitivity and response time. Investigating long-term durability and performance under cyclic loading is crucial for practical applications. The degree of electrical recovery was found to be dependent on the relaxation time, highlighting the importance of carefully considering and reporting this parameter in strain sensing applications.

Further studies are needed to fully understand the viscoelastic behaviour and optimise the recovery characteristics of these nanocomposites for strain sensing applications.

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