Strain sensing using a multiwalled carbon nanotube film

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Abstract: The effectiveness of multiwalled carbon nanotubes (MWCNTs) as strain sensors is investigated. The key contribution of this paper is the study of real-time strain response at the macroscale of MWCNT film under tensile load. In addition, real-time voltage change as a function of temperature is examined. MWCNT films attached to a brass specimen by epoxy using vacuum bonding have been studied. The brass specimen is subjected to tensile loading, and voltage output from the MWCNT film is obtained using a four-point probe and a sensitive voltage measurement device. Experimental results show that there is a linear change in voltage across the film when subjected to tension, and the MWCNT film both fully recovers its unstressed state upon unloading and exhibits stable electromechanical properties. The effect of temperature on the voltage output of the nanotube film under no load condition is investigated. From the results obtained it is evident that MWCNT films exhibit a stable and predictable voltage response as a function of temperature. An increase in temperature leads to an increase in conductivity of the nanotube film. The study of MWCNT film for real-time strain sensing at the macroscale is very promising, and the effect of temperature on MWCNT film (with no load) can be reliably predicted.

Keywords: multiwalled carbon nanotube film, strain sensing, sensors, health monitoring, temperature, conductivity, voltage change

1 INTRODUCTION

Strain sensors are widely used in various fields of engineering for monitoring as well as for damage detection of critical infrastructure. Conventional strain gauges are limited by the requirements of the strain gauge to be able to measure strain only in designated directions and locations. The demand for multifunctional and multidirectional sensors is on the rise, and the use of carbon-nanotube-based sensors holds the promise of fulfilling these objectives owing to their superior mechanical and electrical properties [**1**, **2**].

The discovery of carbon nanotubes by Iijima [**3**] led to an ever-increasing interest in carbon nanostructures and their applications. This is largely due to the combination of their expected structural perfection, small size, low density, high stiffness, high strength, and excellent electronic properties [**1**, **2**]. As a result, carbon nanotubes have found applicability in a wide range of fields [**1**, **2**].

To date, various studies have been done on the reversible electromechanical characteristics of carbon nanotubes. Schadler *et al.* [**4**] observed a shift in the Raman peak when subjected to tension and compression while investigating load transfer in carbon nanotube epoxy composites. Tombler *et al.* [**5**] observed reversible electromechanical characteristics in an individual single-walled carbon nanotube deformed using an AFM tip. Semet *et al.* [**6**] demonstrated the reversible electromechanical characteristics of an individual multiwalled carbon nanotube (MWCNT) subjected to longitudinal bending. Recent experiments conducted by Jang *et al.* [**7**] on an individual MWCNT revealed that the electrical resistance increased when the MWCNT was elongated and that the change corresponded to the strain applied to the nanotube. The change in electrical resistance was recoverable to its preloading value when the MWCNT was strained below its elastic

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limit and released, but changed significantly when the nanotube reached its elastic limit and failed. All the above-mentioned studies relate the electromechanical properties at the nanoscale and not at the macroscopic scale.

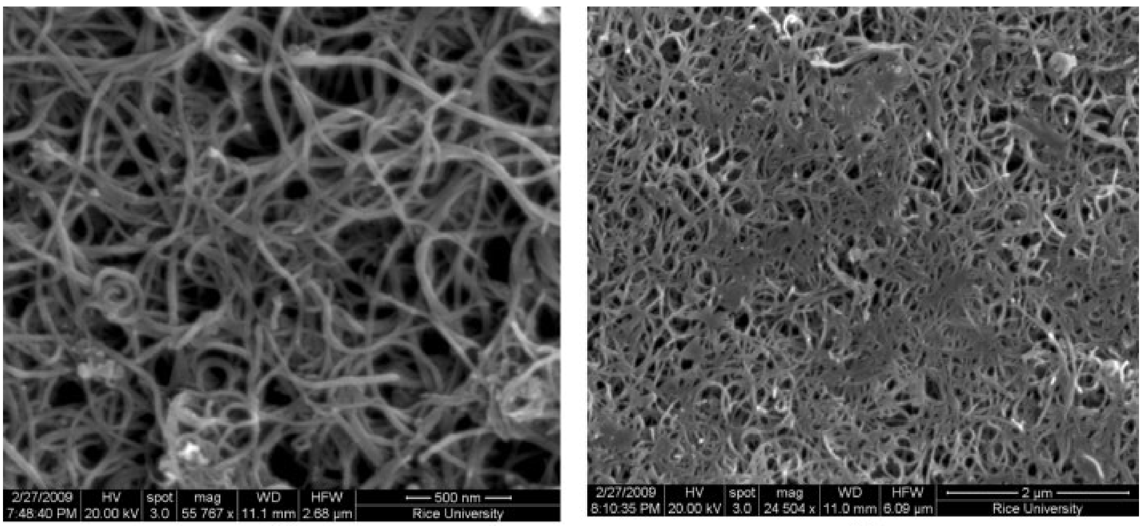
Strain sensing using carbon nanotube thin films (SWCNT film or buckypaper) was studied by Dharap *et al.* [**8**] and Li *et al.* [**9**], who were among the first to investigate and observe linear change in voltage across a film subjected to tension or compression [**8**]. Pushparaj *et al.* [**10**] discovered the fully recoverable electrical conductivity and compressive strain relationship by conducting compressive tests on a macroscopic MWCNT block. Zhang *et al.* [**11**] studied the strain-sensing capabilities of MWCNT/polycarbonate composites. Loh *et al.* [**12**] used a layer-by-layer fabrication method to prepare carbon nanotube–polyelectrolyte thin film for strain and corrosion sensing. Park *et al.* [**13**] investigated the strain-dependent electrical resistance characteristics of MWCNT/PEO composite films using an experimental set-up simultaneously to measure electrical resistance and strain, and found repeatable relations in resistance versus strain. Li *et al.* [**14**] conducted an experimental study on MWCNT films and investigated their potential use as strain sensors. Their study [**14**] mainly focuses on the response of MWCNT film to static loading and on resistance change to temperature, and reports the Fourier transform of the electrical response of samples to dynamic loading. Kang *et al.* [**15**] make use of a MWCNT/PMMA composite continuous strain sensor for structural health monitoring applications. Another study on the behaviour of MWCNT/PMMA composite films subjected to tensile strains was carried out by Pham *et al.* [**16**].

The use of single-walled carbon nanotubes is restricted by limited control of the purity, chirality, and electrical properties of single-walled carbon nanotubes produced by the available synthesis process. Multiwalled carbon nanotubes, on the other hand, are more economical and can be grown at relatively high purity. MWCNTs are also not subject to chirality-related restrictions on electrical properties and can be subjected to aggressive processing [**17**].

In the present study, MWCNT film (buckypaper) is used for strain sensing, and voltage measurements are performed to prove the use of MWCNT film for strain sensing at the macroscopic scale.

2 EXPERIMENT

The MWCNT film was ordered from NanoLab (NanoLab Inc., www.nano-lab.com, Newton, MA) and used 'as is'. The preparation of the MWCNT film is as follows. The MWCNTs are suspended in water using a surfactant Nanosperse AQ (prepared by NanoLab Inc.). The suspended nanotubes are then dispersed using a Branson Bath sonicator and sonicated for 30 min. Sonication results in a stable nanotube suspension which is then filtered using a porous filter under pressure. The freestanding nanotube film is then peeled from the filter after drying. The MWCNT film obtained has a relative density of about 50 per cent and a thickness of about 30–50 µm. The SEM image of the MWCNT film (Fig. 1) shows a densely packed mass of randomly



(a) (b)

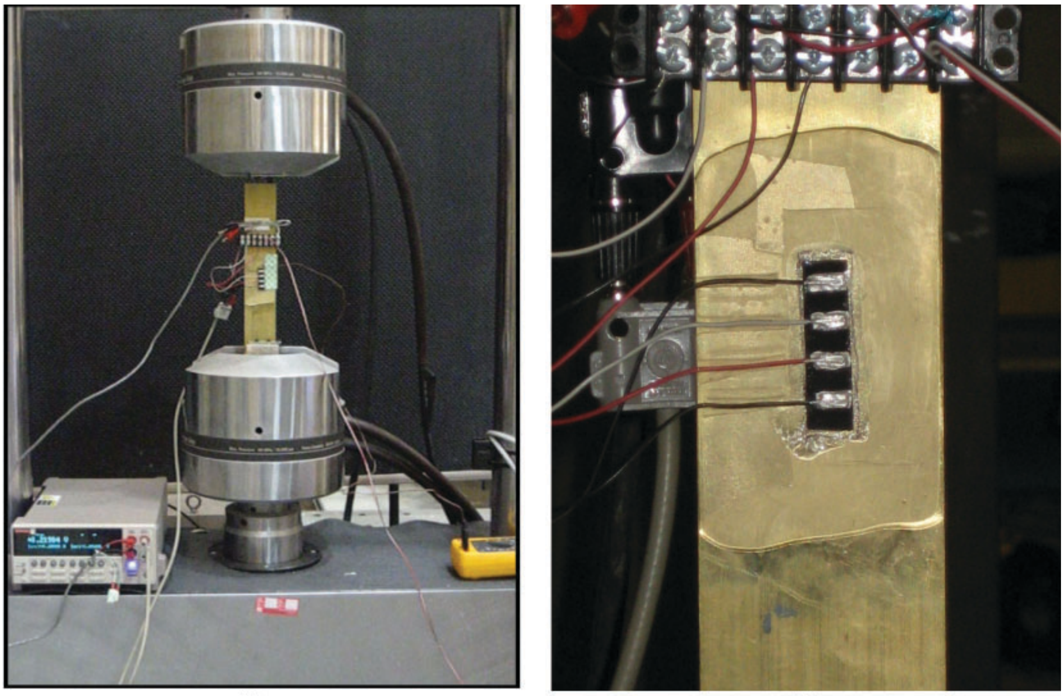
Fig. 1 SEM image of MWCNT film at different resolutions: (a) 500 nm; (b) 2 µm oriented MWCNTs, and this orientation gives rise to its isotropic properties [**18**].

The carbon nanotube film is attached to a $12 \times 2.5 \times 0.25$ inch brass specimen with a Young's modulus of 166 GPa, as shown in Fig. 2(a). An epoxy coating is applied between the carbon nanotube film and the conducting brass specimen, for perfect strain transfer and for insulation. The carbon nanotube sample is bonded to the specimen over the epoxy coat by a 24 h vacuum bonding method to ensure that the bond between the carbon nanotube and the specimen is firm and complete, and that no slippage occurs between the brass specimen, epoxy coating, and carbon nanotube film. A conventional strain gauge is attached on the other side of the brass specimen for strain measurements and comparison. The brass specimen is mounted in a universal testing machine, and an axial tensile load is applied to it.

A four-point probe is attached to the carbon nanotube film by using silver epoxy. The brass specimen is subjected to uniaxial tension in a servo hydraulic test frame, and current is passed through the outer two probes. By applying a constant current across the outer probes, the change in voltage across the two inner contacts due to the tensile load is measured and recorded. Voltage is measured using a Keithley 2400 instrument. The Keithley 2400 has high-precision voltage/current sourcing/measurement capabilities, as low as $\pm 1\,\mu$V to ± 210 V. The data are acquired at a rate of 50 samples/s. Load is applied in increments of 1 klbf at a rate of 10 klbf/min and held constant for several seconds until a stable reading is obtained. The load applied to the specimen is in the elastic range. The strain from the conventional foil strain gauge attached on the opposite side of the brass specimen is obtained using a VISHAY 2120B strain gauge conditioner/amplifier. The data are presented as obtained; no analogue or digital filters have been implemented.

The MWCNT film had an initial resistance of 2.20225 Ω, measured by a four-point probe (by passing a current of 100 mA). As the four-point probe is attached using silver epoxy leads, the contact resistances need not be taken into account for calculation purposes. The high accuracy of the Keithley 2400 voltage measurement device allowed measurements with μV precision.

For temperature-based experiments, the brass bar is heated using a heater, and the temperature is measured using a thermocouple. The change in voltage as a function of temperature is measured with the specimen clamped in a universal testing machine with no applied load.



(a) (b)

Fig. 2 Experimental set-up: (a) brass specimen with MWCNT film in universal testing machine; (b) close-up view of MWCNT bonded to brass specimen with silver epoxy leads

3 RESULTS

3.1 Voltage stability under zero load and current passed through outer probes

The samples were first tested to observe their stability under zero load conditions. The plot of voltage output from the inner probes on the nanotube film under zero load conditions is shown in Fig. 3. The input current applied to the MWCNT sample under zero load was 100 mA. The nanotube film took a very short time to stabilize, and the voltage reading was stable to the order of 0.1 mV.

3.2 Voltage change as a function of applied static load

The brass specimen was subjected to tensile load of 0–8 klbf, and an input current of 100 mA was applied to the MWCNT film. The change in voltage was measured using the four-point probe. The change in voltage as a function of strain measured using the conventional strain gauge is shown in Fig. 4. A linear relationship exists between the change in voltage obtained from the nanotube film and strain gauge readings. The load was applied in increments of 1 klbf, and, at each load step, sufficient time was allowed for the voltage reading to stabilize (about 30–60 s). The gauge factor G of the MWCNT is calculated by means of the formula

$$G = \frac{\Delta R}{R\varepsilon} = \frac{\Delta V}{V\varepsilon} \tag{1}$$

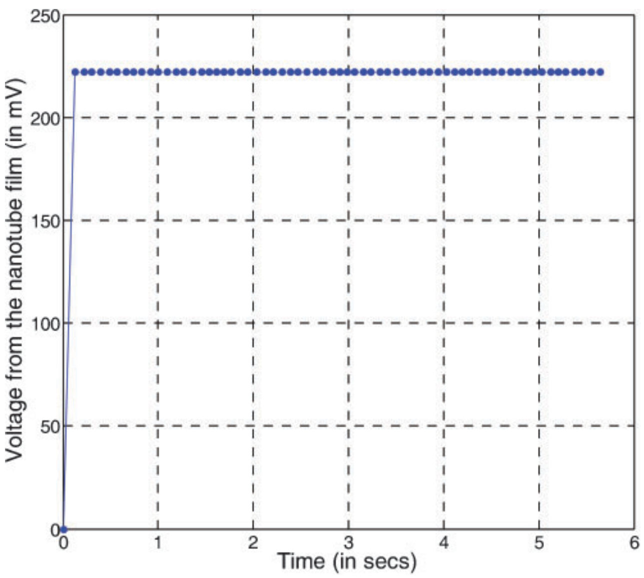


Fig. 3 Voltage stability under zero load

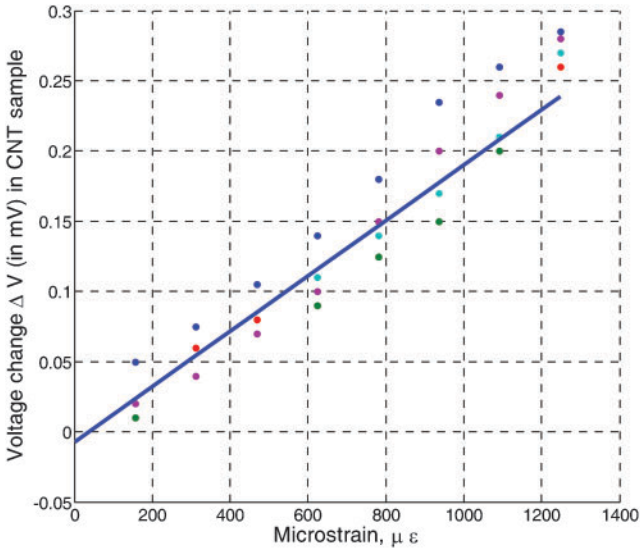


Fig. 4 Specimen subjected to tensile loading; change in voltage in MWCNT film as a function of strain in the brass specimen

The calculated gauge factor of MWCNT film according to the above formula is 0.3482, which is low, and its improvement requires further investigation.

As can be seen in Fig. 4, the change in voltage as a function of strain remains linear until 1000 microstrain ($\mu\varepsilon$); it becomes non-linear beyond 1000 $\mu\varepsilon$.

The change in voltage across the inner two probes is partly due to change in the dimension of the MWCNT film (change in length along the direction of loading) but mainly due to change in the resistivity of the film. The film (sheet) resistivity measured using the four-point probe is given by [**19**]

$$\rho_s = \frac{V}{I} C\left(\frac{a}{d}, \frac{d}{s}\right) \tag{2}$$

where V is the voltage across the inner two probes, I is the input current across the outer two probes, and $C(a/d, d/s)$ is a factor that depends upon the dimension of the film (a is the length of the film, d is the width of the film, and s is the distance between two probes in the four-point probe).

Assume that resistivity ρ_s of the carbon nanotube film remains constant as strain changes from 0 to 0.109 per cent

$$\rho_s = \frac{V_1}{I} C_1\left(\frac{a}{d}, \frac{d}{s}\right) \quad \text{at 0.0 per cent strain} \tag{3}$$

$$\rho_s = \frac{V_2}{I} C_2\left(\frac{a}{d}, \frac{d}{s}\right) \quad \text{at 0.109 per cent strain} \tag{4}$$

$$\Delta V = \rho_s I \left(\frac{1}{C_2} - \frac{1}{C_1} \right) = 55 \ \mu\text{V} \tag{5}$$

Using formula (5), the change in voltage due to change in dimension of the MWCNT film from 0 to 0.109 per cent strain is calculated as 55 μV. The corresponding constants are $C_1 = 2.0202$ at 0 per cent strain and $C_2 = 2.0197$ at 0.109 per cent strain.

For the results presented in Fig. 4, the change in voltage at a strain of 0.109 per cent is about 224 μV. It can be seen that the change in voltage due to the change in dimension of the carbon nanotube film is small (~24 per cent), and the remainder of the change in voltage is due to change in resistivity (~76 per cent). Evidently, it can be concluded that the change in voltage is mainly due to the change in resistivity of the film. The contribution of Poisson's effect to change in resistivity is also important; resistivity and dimensions are partly coupled when cracks/defects exist [**20**, **21**]. This requires further investigation.

3.3 Real-time voltage response to quasi-static tensile loading/unloading

Another set of tests was conducted to observe the real-time behaviour of nanotube film to loading and unloading cycles, and the voltage results are plotted in Figs 5(a) and (b). The linear change in voltage across the film obtained upon loading was fully reversible, and it was observed that, when the brass specimen was unloaded, the voltage reading immediately returned nearly to zero-load voltage conditions. The loading and unloading test was conducted repeatedly, and fully reversible electro-mechanical characteristics of nanotube film were observed. The strain response in Fig. 5(a) illustrates

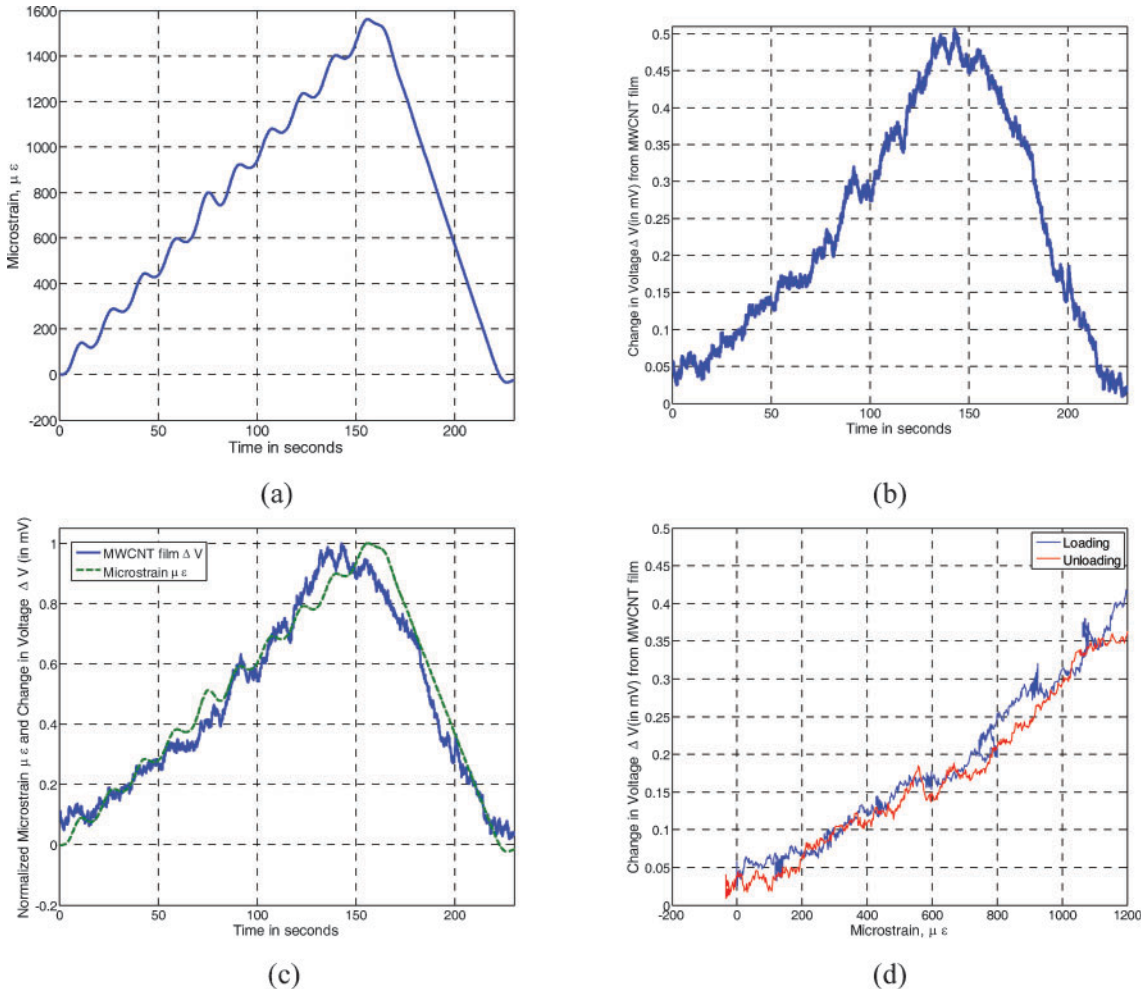


Fig. 5 (a) Strain input given to the brass specimen subjected to tensile load; (b) real-time voltage response of MWCNT film subjected to tensile load; (c) normalized real-time voltage and strain response; (d) voltage and strain response for loading and unloading

the kind of loading/unloading adopted. As shown by Fig. 5(b), the change in voltage follows the strain response closely, both during loading and unloading. Some minor deviations do occur, as can be seen in Fig. 5(b). Normalized plots of voltage versus time and strain versus time are shown in Fig. 5(c); it is

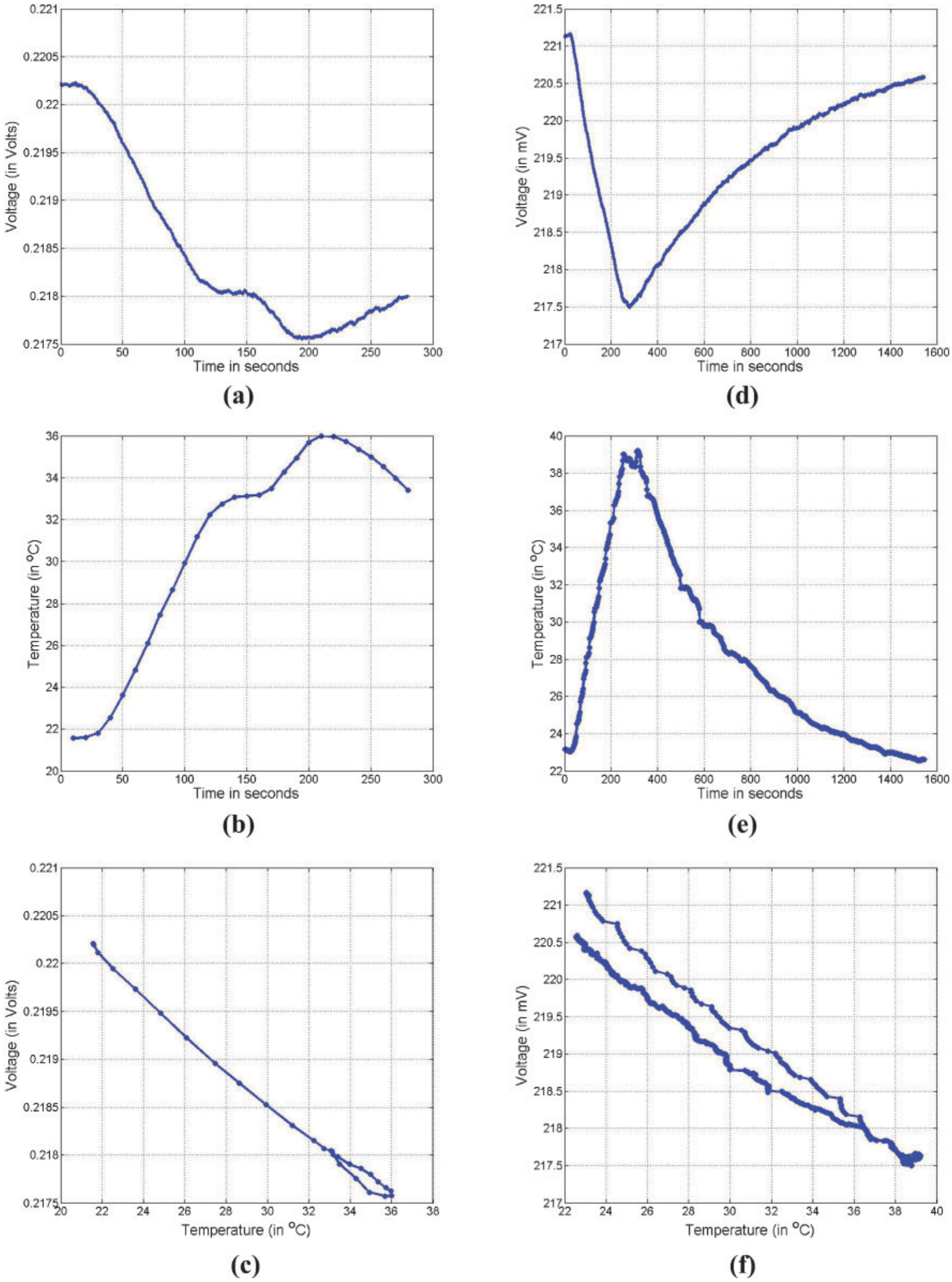


Fig. 6 (a) Voltage versus time, (b) temperature versus time, and (c) voltage versus temperature as the brass specimen is heated from 21.1 to 35 °C; (d) voltage versus time, (e) temperature versus time, and (f) voltage versus temperature as the brass specimen is heated from 21.1 to 37.78 °C and cooled

interesting to note how the change in voltage tracks the strain. A continuous change in voltage ΔV across MWCNT film as a function of strain (Fig. 5(d)) shows a linear relationship between them, and highlights the real-time response of the MWCNT film to load changes.

It can also be observed from the results in Fig. 5(d) that the electromechanical characteristics are fully reversible, and voltage across the nanotube film corresponds linearly to strain during both loading and unloading. Semet *et al.* [**6**] and Jang *et al.* [**7**] observe similar reversible electromechanical characteristics at the nanoscale using an individual MWCNT; the present results reinforce a similar characteristic trend observed at the macroscale.

The effect of temperature on MWCNT film was studied by heating the specimen and clamping both ends of the specimen in a universal testing machine with no load. It was observed that the resistance of the MWCNT film decreased upon heating. A plot of voltage versus temperature is shown for two different experiments in Figs 6(a) to (c) and Figs 6(d) to (f). A linear behaviour was observed in the voltage response of MWCNT film to temperature. However, it was noted that the resistance (calculated from the voltage response with an input current of 100 mA) changed by 0.0217 Ω only for a temperature difference of 13.9 °C (21.1–35 °C). Similar results were obtained by Koratkar *et al.* [**22**] for the temperature range 20–200 °C, where the authors observed only a small change in resistance with temperature. Although the study conducted by Koratkar *et al.* [**22**] was on vertically aligned MWCNTs, the results in the present study indicate a similar trend in the voltage response of randomly aligned MWCNT films.

4 CONCLUSION

The effectiveness of MWCNT films in measuring real-time strain at the macroscale has been investigated experimentally. It can be concluded from the results of this study that there is a linear relationship between the measured change in voltage in the MWCNT film and the measured strain in the brass specimen. It has also been observed that the MWCNT film responded very well to loading and unloading. Fully reversible electromechanical characteristics have been observed from the results; the MWCNT film demonstrated excellent recovery to preload conditions upon unloading. The relationship between temperature and voltage change has been studied in a limited temperature range. The testing needs to be performed over a wider range of temperatures before general conclusions can be drawn. However, the limited results presented do indicate excellent predictable voltage response to temperature changes. The results presented in this paper are very encouraging and indicate the potential of using MWCNT film as a strain sensor at the macroscale.

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