Characterization of the spatial elastoresistivity of inkjet-printed carbon nanotube thin films

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Abstract

Next-generation lightweight-designed structures shall be able to perform self-state assessment via integrated health monitoring systems. In this article a carbon nanotube-embedded polymeric thin film is applied via inkjet-printing to perform spatial strain sensing in conjunction with using electrical impedance tomography. To gain an advanced understanding of the thin film’s spatial strain sensitivity, the elastoresistivity matrix, a fourth-order tensor correlating the strain state of a conductor into its normalized change in resistivity state, is characterized. The Montgomery method is adopted to derive the planar resistivity coefficients of the thin film, and a digital image correlation system is used to measure the planar strains. A validation test suggests that the calculated determinant of the correlated change in anisotropic resistivity shows a fairly similar result to the measured isotropic EIT reconstruction results.

Keywords: carbon nanotubes, inkjet-print, elastoresistivity, nanocomposite, electrical impedance tomography, spatial strain sensing, structural health monitoring

(Some figures may appear in colour only in the online journal)

1. Introduction

The concept of lightweight design is to minimize the weight of a structure without compromising its loading-capacity. Airplanes, space shuttles, motor vehicles, and prosthetic devices consist of lightweight parts that are designed through both material redistribution and adopting lightweight materials. Topological optimization allows the development of more efficient structure geometries for less amount of material usage. Bridge girders, airplane spoilers, and Flex-Foot Cheetah [1] are examples of structural components with optimized loading-capacity-to-weight ratio. Using materials with light self-weight such as aluminum/titanium alloys may also reduce the overall mass of a load-bearing structure. Composite materials including fiber reinforced polymers (FRPs) and polyurethane/honeycomb sandwich panels are popular for airplanes and vehicles due to their high load-to-weight ratio. These components can effectively reduce the overall operational vibration and noise levels to attain higher aerodynamic stability, offering passengers a more comfortable riding experience. Moreover, higher fuel efficiency can be accomplished for industries pursuing higher economical profit. However, improved strength-to-weight ratio entails high complexity in either structural geometry or material composition or both, bringing challenges to accurately modeling or analyzing structural performance. Hence to accompany the fast development of the lightweight design industry, a reliable, lightweight, and in situ structural health monitoring system shall be simultaneously employed to monitor the real-time structural behavior.
In-practice monitoring devices such as foil strain gages and fiber Bragg grating optical sensors can be applied as a tethered network to monitor localized strains of a structure in-service [2–4]. They are single-channeled transducers taking measurements merely over their applied points or path, hence the sensing resolution is inherently influenced by the density of sensor installation [4]. Accelerometers can assess the global behavior of a structure via collecting its vibrational data; similarly, piezoelectric transducers may be used under different guided-wave frequencies to identify both local- and global-damage [5]. Adopted by many civil infrastructures and aerospace structures, their heavy self-weight under desired sensing resolution have nevertheless limited their application to lightweight-designed structures [6]. Recent researches attempt to develop weightless sensing materials that can be easily integrated into the structure itself, such as a layer of self-sensing paint [7–9]. These thin film materials are usually assembled via bottom-up techniques to encapsulate desired functionality, which may come from novel nanomaterials such as carbon nanotubes (CNTs). Possessing excellent mechanical strength and electrical properties, CNTs can be cast into thin sheets alone or to be embedded within polymeric matrices to sense strain [10, 11], pH change [11, 12], pressure [13], or heat [14, 15]. Buckypaper is a thin sheet of randomly distributed CNTs whose bulk electrical property is sensitive to strains [10], implying its potential application as a strain gage. To facilitate large-strain sensing (i.e., ≥50%), recent studies have reported adding CNTs as nanofillers into polymeric matrices to assemble nanocomposites. Vacuum filtration [16] and compressed-air spray [7, 8] can assemble CNT-embedded sheets rapidly with a limitation on precision control of the thickness deposition. Spin-coating and layer-by-layer (LbL) assembly are effective in fabricating thin films with CNTs being evenly distributed throughout the bulk nanocomposites, while these techniques fail to produce thin films with complex layouts [11]. Techniques enabling high-precision control such as micropatterning involve usage of expensive equipment that can be challenging in producing large-area specimen. Therefore a fast, cheap, and reliable thin film fabrication method is sought to assemble CNT-embedded strain sensors.

Inkjet-printing is a cost-effective method offering precise patterning of micro- or nano-scaled materials such as gold nanoparticles, functional polymers, graphene, and CNTs [17]. It emerges as a more versatile substitution to traditional silicon wafer fabrication in the demand of fabricating flexible electronics. Miniaturized RFID tags [18], flexible conductive wires [19], organic light-emitting diodes [17], and DNA patterning [20] benefit from inkjet-printing for its non-contact, micrometer-resolution manufacturing scheme. Multiple studies have successfully inkjet-printed CNTs to demonstrate its potential application as electrodes or conductive traces using commercially available drop-on-demand (DoD) printers [21–23]. Reported sheet resistance can reach as low as 78 Ω/sq for single-walled CNTs and 760 Ω/sq for multi-walled CNTs (MWNTs) [23]. Kordas et al. [22] printed a MWNT conductive thin sheet that was demonstrated to be sensitive to high vapor pressure. Michalis et al. [24] inkjet-printed MWNTs over ethylene tetrafluoroethylene sheets as strain sensors; the reported gage factor is 0.9 ± 0.14 with hysteresis-free repeatability. Li et al. [25] aerosol jet-printed aligned MWNT sheets that can reach a strain sensitivity up to 80. Beyond being utilized as point-based strain gages, CNT thin films can be also applied as a spatial damage sensor while paired with the algorithm of electrical impedance tomography (EIT). EIT reconstructs the conductivity distribution within a given boundary from electrical responses collected at the boundary. Due to CNT’s piezoresistive nature, a localized conductivity change can be correlated to a localized strain change or permanent material deterioration at the spot. Loh et al [26] has demonstrated the possibility of applying an LbL-fabricated CNT-polyelectrolyte thin film as an impact damage sensor over an aluminum plate. Loyola et al [7] further extended its application to glass-FRP composites. Researchers such as Loyola et al [27] attempt to apply EIT-coupled CNT thin films as spatial strain sensors to monitor the strain distribution of a given area under loading. Therefore, it is important to correlate the reconstruction result to the corresponding strain state, and the aforementioned 1D characterization of CNT thin film’s gage factor hence is not sufficient.

This paper proposes to characterize the elastoresistivity of an inkjet-printed CNT thin film using sensor analysis to correlate its electrical property change to the undergoing strain state. An elastoresistive coefficient correlates the resistivity change of a conductive body due to an elastic strain; it is a component of a fourth-rank tensor which is the linear transformation between the second-rank tensors of resistivity and strain [28]. To characterize each coefficient, a square-shaped CNT thin film is inkjet-printed over a polyethylene coupon, which is tensile-loaded. During the loading test, the Montgomery method is adopted to simultaneously measure the resistance change in both the longitudinal direction and the transverse direction of the thin film. The characterized elastoresistivity tensor is then applied to correlate the strain state distribution map developed over a CNT thin film into a conductivity map, which is compared with the spatial conductivity map reconstructed by EIT to verify the results. Previous works have been dedicated to verifying CNT thin film’s spatial strain sensing capabilities, such as under uniaxial loads [29] and bending loads [30]. In this study, a coupon with a center crack is tensile-loaded to create a mixed damage situation with both severe (i.e., crack opening) and mild strain deformation. To obtain the spatial strain state, a digital image correlation (DIC) system is employed during the experiment by using the printed thin film itself as the speckle pattern. The work attempts to interpret EIT results in terms of the strain state of a loaded body, gaining knowledge of the spatial electromechanical behavior of the inkjet-printed CNT thin films. Future research are motivated to enable spatial strain state sensing of the thin film via implementation of the characterization results.
2. Mathematical background

2.1. Elastoresistivity

The elastoresistivity of an object is defined as the change of the material’s resistivity due to applied strain [28]. Because CNTs are randomly distributed within the printing ink, it is reasonable to assume that each printed thin film in its pristine state has an isotropic resistivity, \( \rho \). When a strain state is applied, the thin film’s inherent resistivity changes due to the tunneling effect and/or CNT self-distortions, and this change was observed to be orientation-dependent [31, 32]. The piezoresistivity is defined as the change of its resistivity due to applied stress, and the constitutonal law can be described as:

\[
\frac{\Delta \rho}{\rho} = \pi \cdot \tau,
\]

respectively

\[
\frac{\Delta \rho_k}{\rho} = \sum_{i=1}^{3} \sum_{m=1}^{3} \tau_{km} \gamma_{im},
\]

where \( \Delta \rho \) is the tensor representing the change in resistivity, and \( \tau \) is the tensor of applied stress. The subscripts of the normalized change of resistivity \( \Delta \rho_k/\rho \) indicate that the voltage potential is measured in the direction of \( i \) while current being applied in the direction of \( k \). In a similar trend, \( \tau_{km} \) is the stress component in the direction of \( m \) acting on the plane normal to the direction of \( l \). Hence \( \pi \) is a fourth-order tensor representing the material’s piezoresistivity coefficients which can be listed in terms of a \( 6 \times 6 \) matrix. Assuming the CNT thin film possesses material properties with cubic symmetry, equation (1) under the plane-stress situation can be rewritten in the matrix form as

\[
\frac{1}{\rho} \begin{pmatrix}
\Delta \rho_1 \\
\Delta \rho_2 \\
\Delta \rho_3
\end{pmatrix} = \begin{pmatrix}
\pi_{11} & \pi_{12} & 0 \\
\pi_{12} & \pi_{11} & 0 \\
0 & 0 & \pi_{44}
\end{pmatrix} \begin{pmatrix}
\tau_x \\
\tau_y \\
\tau_{xy}
\end{pmatrix},
\]

where \( \pi_{11} \) correlates the normal stress to the change of resistivity in the same direction, and \( \pi_{12} \) correlates stresses to the changes in resistivity in the transverse direction. \( \pi_{44} \) is called the shear piezoresistivity, which characterizes the voltage change with a current flown in its transverse direction due to the applied shear stress [33].

It is known that the Hooke’s law states that for an isotropic material under plane-stress condition,

\[
\begin{pmatrix}
\tau_x \\
\tau_y \\
\tau_{xy}
\end{pmatrix} = \begin{pmatrix}
\frac{E}{1-\nu^2} & \frac{E\nu}{1-\nu^2} & 0 \\
\frac{E\nu}{1-\nu^2} & \frac{E}{1-\nu^2} & 0 \\
0 & 0 & \frac{E}{2(1+\nu)}
\end{pmatrix} \begin{pmatrix}
\varepsilon_x \\
\varepsilon_y \\
\varepsilon_{xy}
\end{pmatrix},
\]

where \( E \) is the elastic modulus of the material, \( \nu \) is the Poisson’s ratio, and \( \varepsilon_x, \varepsilon_y, \varepsilon_{xy} \) are the components of the strain tensor. Combining equations (2) and (3) it follows that

\[
\frac{1}{\rho} \begin{pmatrix}
\Delta \rho_1 \\
\Delta \rho_2 \\
\Delta \rho_3
\end{pmatrix} = E \begin{pmatrix}
\pi_{11} + \pi_{12}\nu & \pi_{12} & 0 \\
\pi_{12} & \pi_{11} + \pi_{12}\nu & 0 \\
0 & 0 & \pi_{44}(1 - \nu^2)
\end{pmatrix} \begin{pmatrix}
\varepsilon_x \\
\varepsilon_y \\
\varepsilon_{xy}
\end{pmatrix}.
\]

In this article, we characterize the elastoresistive coefficients by directly mapping strains to the change in resistivity of the thin film. Hence equation (4) can be rewritten as

\[
\frac{\Delta \rho}{\rho} = m \cdot \varepsilon,
\]

respectively

\[
\frac{1}{\rho} \begin{pmatrix}
\Delta \rho_1 \\
\Delta \rho_2 \\
\Delta \rho_3
\end{pmatrix} = \begin{pmatrix}
\pi_{11} & \pi_{12} & 0 \\
\pi_{12} & \pi_{11} & 0 \\
0 & 0 & \pi_{44}
\end{pmatrix} \begin{pmatrix}
\varepsilon_x \\
\varepsilon_y \\
\varepsilon_{xy}
\end{pmatrix},
\]

where \( m \) denotes the fourth-order elastoresistivity tensor to be characterized.

2.2. Electrical impedance tomography

EIT was initially developed as a non-invasive technique to visualize the interior of human chest via electrical observations from epidermal sensors. The existence of tumor or focus can trigger a localized conductivity change within the human body that can be identified. For a conductive body \( \Omega \), upon which a defined electrical field is applied, a unique boundary voltage potential is observed if there exists no internal current source. Consequently, the summation of current density within \( \Omega \) is zero, or,

\[
\nabla \cdot (\sigma \nabla u) = 0,
\]

where \( u \) is the scalar voltage potential, and \( \nabla u \) is hence the electrical field within \( \Omega \). We restrict the following discussion regarding EIT to a body with isotropic conductivity, i.e., where \( \sigma \) is the scalar conductivity. The voltage distribution \( \nabla u \) can therefore be directly solved, and the boundary voltage potential \( V \) can be evaluated as

\[
V = \nabla u \cdot n,
\]

where \( n \) is the unit vector normal to \( \partial \Omega \), the boundary of \( \Omega \). Direct measurements of discrete \( V \) can be realized by installing multiple equal-space electrodes on \( \partial \Omega \). However, an EIT problem evaluates the conductivity distribution through the observed boundary voltage potential, thus solving equation (6) becomes an inverse problem. To take into account the influence of electrode contact resistance, the complete electrode model is often adopted [34], or,

\[
\int_{\partial \Omega} \sigma \frac{\partial u}{\partial n} \, dn = I_i, \hspace{1cm} (8a)
\]

\[
\int_{\partial \Omega} \sigma \frac{\partial u}{\partial n} \, dn = V_i, \hspace{1cm} (8b)
\]

where \( I_i \) denotes the electrode a current being injected in to
create $\nabla u$ across the medium, and $z_l$ is the electrode contact resistance. Equations (6) and (8) constitute the EIT equations to be evaluated via finite element (FE) analysis. Voltage responses from a pre-defined current injection pattern are collected to evaluate $\sigma$ via least-squares estimations. Conventional EIT solvers employ iterative calculation to estimate the absolute value of $\sigma$. In this study, a one-step inverse method developed by Alder and Guardo [35] to estimate the change in conductivity of the given area is adopted. The algorithm is based on maximum a posteriori (MAP) method to estimate the linearized solution. In the end, the change in conductivity, $\Delta \sigma$, is calculated as

$$\Delta \sigma = (H^TW + \lambda R)^{-1}H^TW\left(\frac{\Delta V}{V_0}\right),$$  

(9)

where $V_0$ is the boundary voltage measured when $\Omega$ is under pristine state, and $\Delta V$ is the change in voltage signal after damage. The sensitivity matrix $H$ linearly transforms the unit change in conductivity to a boundary voltage response, and the transformation is stabilized by the weighing matrix $W$, the regularization matrix $R$, and the hyperparameter $\lambda$ which controls how much influence $R$ posts to the equation. The weighing matrix $W$ takes into account the Gaussian white noise introduced by voltage measurements, and the regularization matrix $R$ smooths out the calculation to overcome its ill-posed nature. In this study, the NOSER regularization method is selected to perform the inverse calculation [36]. To select an appropriate hyperparameter $\lambda$, a wide range of $\lambda$ values are tried, and the one of which the signal-to-noise ratio of the conductivity reconstruction is equal to that of the voltage measurements is chosen [35, 37].

In the preceding discussion, the conductivity and its change was considered isotropic. However, an anisotropic strain state changes the conductivity accordingly in an anisotropic way, i.e., $\sigma$ is a tensor. According to Hamilton et al [38] the anisotropic EIT cannot be solved uniquely. In the current article we therefore continue to use the standard EIT and expect that the calculated isotropic conductivity is an equivalent measure of the actual anisotropic one. The EIT evaluation was performed using an open-source, Matlab algorithm EIDORS that calculates $H$ based on assuming that the conductivity remains isotropic on $\Omega$ [37]. Hence the evaluated conductivity value is considered as the resultant of the true anisotropic state. More details on this equivalency are discussed in section 4.2.

3. Experimental details

3.1. Inkjet-printed CNT thin films

To inkjet-print MWNT thin films, a MWNT-containing ink shall be prepared. MWNTs tend to agglomerate into bundles due to the high van der Waals force between individual tubes; therefore they shall be separated from each other in aqueous state and stabilized to maintain the separation. In this study Pluronic F127 (Sigma Aldrich) was used as the stabilizer for its prolonged stabilizing ability for nanoparticles; it is a triblock copolymer exhibiting amphiphilic properties. A 2 wt% Pluronic solution was made by adding Pluronic powder into deionized (DI) water and constantly stirred at $65^\circ$C until no polymer particles can be seen. MWNT powder (Sigma Aldrich, $\geq 98\%$ carbon basis) was added to the cooled-down Pluronic solution at a concentration of 3 mg ml$^{-1}$. In order to improve the adhesion between MWNTs and polymer chains, 1-Methyl-2-pyrrolidinone (NMP) (Sigma Aldrich, $\geq 99.0\%$) at an amount of approximately 2 wt% was added into the mixture, which was subjected to a 3 s on, 120 s off tip sonication for 1 hr (50% power, Bandelin Sonopuls HD3200, 20 kHz), followed by another hour of tip sonication on a 3 s on, 3 s off cycle. The sonicated solution was then centrifuged at 3000 rpm for 30 min; the supernatant was collected and centrifuged again at 3000 rpm for another 30 min. To further eliminate large agglomerates that could clog the printer, the centrifuged ink was filtered by PTFE membrane filters with pore size of 5 and 1.2 $\mu$m, successively. The filtered ink was eventually ready to be printed.

In this study a DoD inkjet printer with piezoelectric printer head from Epson (Epson XP322) was used. An electrical pulse forces the piezoelectric chamber to deform, pushing drops of ink out of the printer nozzle onto the substrate. It is preferred over thermal printers which may cause heat-induced agglomeration. The printing resolution of the printer used in this study is 40 $\mu$m. The layout of the thin films, as seen in figures 1 and 2(a), were developed using AutoCAD, a commercially available computer-aided design software. The open-source graphic editor Inkscape was used to tune the printing process. Each thin film for performing elastoresistivity characterization has a dimension of $40 \times 40$ mm$^2$, consisting of four printing layers overlapping each other to reach the desired conductivity. A thorough cleaning of the printer head is required after printing 4–7 samples due to CNT settlement, which clogs the printer nozzle and causes fading.
prints. Each specimen was probed with a multimeter (Keithley 2110) in both longitudinal and transverse directions to measure its pristine gage resistance. All the printed specimens showed similar readings, indicating that inkjet-printing is a highly repeatable thin film deposition method. Four electrodes were attached to the four corners of a thin film to wire the specimen to electrical instruments using a two-part silver epoxy (Ted Pella, Inc.). Each specimen was placed in an oven at 80°C for 3 h to cure the epoxy. Specimens used for EIT reconstruction were printed with a dimension of 25 × 25 mm² over polyethylene terephthalate (PET) sheets. A total of 16 electrodes with 4 per side were painted around its perimeter using conductive silver paint (Ted Pella, Inc.), as seen in figure 2(a). This electrode layout allows two 8-pin flexible flat cable connectors to be wired to the thin film. Figure 2(b) shows the scanning electron microscope image of a printed specimen, whose morphology shows uniform distribution of CNT nanoparticles. The percolated network of CNTs observed suggests that the fabricated thin film exhibits homogeneous electrical property. It is observed that the multilayered structure of the thin film may still form an entangled CNT network with no obvious layer separation, ensuring continuous electrical paths to be established among individual nanotubes.

3.2. Montgomery method

The Montgomery method was developed to characterize the electrical resistivity of anisotropic solids [39]. Equation (5) suggests that $m_{11}$, $m_{12}$, and $m_{44}$ are the three unknowns to be characterized experimentally. Figures 3(a)–(b) demonstrate the conventional testing scheme to characterize $m_{11}$ and $m_{12}$, respectively, via a uniaxial tensile test. A uniformly distributed tensile strain $\varepsilon_y$ was applied along a specimen’s gage length, across which the gage resistance was probed to calculate $\rho_y$. Probing the specimen in transverse direction as

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Figure 2. (a) A tensile testing coupon covered by a CNT thin film was prepared for EIT measurement. (b) The scanning electron microscope image of the inkjet-printed CNT thin film shows a percolated morphology.

Figure 3. The illustrations demonstrate the conventional tensile testing setup for characterizing the elasto-resistive coefficient of (a) $m_{11}$ and (b) $m_{12}$.
shown in figure 3(b) would provide information regarding \( \rho_x \) and therefore \( m_{11} \) and \( m_{12} \) can be derived according to equation (5). However, it is impossible to probe the thin film in both directions simultaneously due to the short circuit formed by the permanently installed electrodes along the perimeter, i.e., the electrode installation scheme in figures 3(a) and (b) would have to merge.

To solve the electrode issue, a four-point probing approach named the Montgomery method is adopted to measure one’s anisotropic resistivity by testing only one specimen [39]. Figure 4 illustrates the measurement scheme of the Montgomery method over a square-shaped specimen, where each of the four corners is attached with an electrode. The square-shaped specimen with anisotropic resistivity can be hypothetically transformed into a rectangular equivalent with an isotropic resistivity, i.e., \( L'_x = L'_y \), \( \rho'_x = \rho'_y \), and \( L_x = L_y \), \( \rho_x = \rho_y \). Voltage responses across a pair of adjacent electrodes are collected while current is injected through the other pair, either in the load-applied direction or the transverse direction. The measurement data can be used to estimate \( L_x / L_y \), or the dimensional aspect ratio of the rectangular equivalent, and eventually derive the actual resistivity through equations [39]

\[
\rho_x = \frac{\pi d L'_x L_x U_x}{8 L'_x L_y I_x} \sinh \left( \frac{\pi L'_x}{L_x} \right) \quad (10a)
\]
\[
\rho_y = \frac{\pi d L'_y L_y U_y}{8 L'_x L_y I_y} \sinh \left( \frac{\pi L'_y}{L_y} \right) \quad (10b)
\]

where \( d \) is the specimen’s thickness, \( U_x, U_y \) and \( I_x, I_y \) are voltage responses collected and currents injected in the loading direction and the transverse direction, respectively. Hence \( m_{11} \) and \( m_{12} \) can be derived using equation (5).

It is nevertheless challenging to directly characterize the shear elastoresistivity, \( m_{44} \), via similar approaches. The loading scheme in figures 3(a)–(b) results in a strain distribution described as

\[
\varepsilon_x = -\nu\varepsilon_0, \quad (11a)
\]
\[
\varepsilon_y = \varepsilon_0, \quad (11b)
\]
\[
\varepsilon_{xy} = 0, \quad (11c)
\]

where \( \varepsilon_0 \) is the applied uniformly distributed strain. Equation (5) can therefore be rewritten as

\[
\frac{\Delta \rho_x}{\rho} = m_{11}(-\nu\varepsilon_0) + m_{12}\varepsilon_0, \quad (12a)
\]
\[
\frac{\Delta \rho_y}{\rho} = m_{12}(-\varepsilon_0) + m_{11}\varepsilon_0. \quad (12b)
\]

Suppose there exists a coordinate system \( x'y' \) that is \( 45^\circ \) rotated from the \( xy \) coordinates. The elastoresistivity matrix, \( m' \), established in \( x'y' \), can be transformed as: \( m' = \alpha m_0 \alpha^{-1} \), where \( \alpha \) is the coordinate transformation matrix defined in Smith et al [40]. Hence the new elastoresistive coefficients \( m'_{11} \) and \( m'_{12} \) in the \( x'y' \) coordinates can be derived as [40]:

\[
m'_{11} = \frac{1}{2}(m_{11} + m_{12} + 2m_{44}), \quad (13a)
\]
\[
m'_{12} = \frac{1}{2}(m_{11} + m_{12} - 2m_{44}). \quad (13b)
\]

This is equivalent to loading the same specimen with a thin film printed in \( 45^\circ \) rotated direction. However, under pristine condition where the thin film remains isotropic, the elastoresistive coefficients shall be independent of rotation, or \( m'_{11} = m_{11}, m'_{12} = m_{12} \). Equations (13a) and (13b) therefore yield the following conclusion:

\[
m_{44} = \frac{m_{11} - m_{12}}{2}. \quad (14)
\]

Therefore the shear elastoresistive coefficient \( m_{44} \) can be derived from \( m_{11} \) and \( m_{12} \), under the condition of remaining isotropic. It is noted that equation (14) is in analogy to the relation between the stiffness matrix shown in equation (3), where in this case the stiffness quantities \( E \) and \( \nu \) are related to the shear modulus. In this study all the elastoresistive
coefficients were characterized to correlate strains developed over the thin film from being pristine to just being loaded, hence it is reasonable to maintain the assumption that the thin film is isotropic. More details will be discussed in the Results and discussions section.

CNT thin films were inkjet-printed over a PET substrate, which were cut into tensile testing coupons in a dimension of \(60 \times 160 \text{ mm}^2\), as seen in figure 1. A uniaxial tensile load was applied over the specimen under a constant displacement rate of 25 \(\mu \text{m s}^{-1}\), until a total of 120 \(\mu \text{m}\) displacement was reached; this gives an approximate longitudinal strain of 1.2%. During testing, a CMOS-based multi-channel switch controlled by a microcontroller (Arduino Uno) was used to switch between the two pairs of electrodes (i.e., one in loading direction and other in transverse direction) to inject current and measure voltage response. Previous results showed that the CNT thin films exhibit different strain sensitivities under tensile strains and compressive strains; similar observations were reported in many literature \[31, 32\]. Therefore in this study two elastoresistivity matrices were characterized, namely \(m^+\) for tensile strains and \(m^-\) for compressive strains. Equation (5) is hence modified as:

\[
\frac{1}{\rho} \begin{pmatrix}
\Delta \rho_x \\
\Delta \rho_y \\
\Delta \rho_{xy}
\end{pmatrix} = \begin{pmatrix}
m^+_{11} & m^+_{12} & 0 \\
0 & m^+_{22} & 0 \\
0 & 0 & m^+_{44}
\end{pmatrix} \begin{pmatrix}
\varepsilon_x \\
\varepsilon_y \\
\varepsilon_{xy}
\end{pmatrix},
\]

where \(m^+_{ij} = \begin{cases} 
  m^+_{ij} & \text{for } \varepsilon_i > 0 \\
  -m^-_{ij} & \text{for } \varepsilon_i < 0
\end{cases}\) (15)

where \(\varepsilon_i\) is \(\varepsilon_x\), \(\varepsilon_y\), or \(\varepsilon_{xy}\). It should also be noted that the shear elastoresistivity is independent from strain directions, or,

\[
m^+_{44} = \frac{m^+_{11} - m^+_{12}}{2}, \quad (16a)
\]

\[
m^-_{44} = \frac{m^-_{11} - m^-_{12}}{2}, \quad (16b)
\]

\[
m^+_{44} = m^-_{44}. \quad (16c)
\]

Equation (15) expands the total number of unknowns from 2 to 4; testing additional specimens in different probing orientations can add more equations to solve the additional unknowns. Therefore specimens with CNT thin films rotated at 0° and 40° were tested, as demonstrated in figures 5(a) and (b).

To measure strains developed in both directions, a DIC system (Vic-3D System 5 Mpx @ 75 Hz, Correlated Solutions, Inc.) was employed to measure the 2D strain distribution over the thin film covered area. The printed thin film served as a speckle pattern for the DIC strain analysis. It was observed that the natural light reflection over CNT particles created a refined pattern perfectly matching the camera’s resolution, as seen in figure 6. The characterization results are presented and discussed in the Results and discussions section.

3.3. Tensile test on a center crack specimen

As mentioned in previous sections, an EIT verification test was carried out over a tensile testing coupon with a center crack introduced, as seen in figures 2(a) and 6. The PET specimen was tensile-loaded at a constant displacement rate of 100 \(\mu \text{m s}^{-1}\). The load paused at every 500 \(\mu \text{m}\) for taking a picture of the specimen for DIC analysis and making an EIT...
across the thin was injected between two electrodes that are facing each other. In this study, a DC current with a magnitude of 0.5 mA developed to enable as many voltage measurements as possible. Light exposure was set to 200 ms.

An additional light source was required to reduce the image noise level, so a lamp enclosed in a soft box was placed above the two camera lenses of the DIC system. The duration of light exposure was set to 200 ms.

Generally the more data available the easier to solve an inverse problem; therefore a current injection pattern was developed to enable as many voltage measurements as possible. In this study, a DC current with a magnitude of 0.5 mA was injected between two electrodes that are facing each other across the thin film, and the voltage difference between two adjacent electrodes were measured between all possible pairs. A total of 16 current injections were carried out for one complete EIT data acquisition term, resulting in a total of 256 voltage data. An Arduino (MEGA 2560)-controlled CMOS multi-channel switch was used to switch between current injection and voltage measurement programmed by Matlab Simulink. The whole data acquisition process took approximately 2.56 s.

4. Results and discussions

4.1. Characterization of elastoresistivity

In this study two sets of \( \mathbf{m}^* \) were evaluated: the first set takes the geometrical change of the thin film due to the tensile load into account, and the second set does not. To differentiate between them, a different notation, \( \mathbf{n}^* \), is used to represent the elastoresistivity matrix not considering the geometrical change. The constitutional equation with the alternative matrix \( \mathbf{n}^* \) is then

\[
\frac{1}{\rho} \begin{pmatrix} \Delta \rho_x \\ \Delta \rho_y \\ \Delta \rho_z \end{pmatrix} = \begin{pmatrix} n_{11}^* & n_{12}^* & 0 \\ n_{12}^* & n_{11}^* & 0 \\ 0 & 0 & n_{44}^* \end{pmatrix} \begin{pmatrix} \varepsilon_x \\ \varepsilon_y \\ \varepsilon_z \end{pmatrix},
\]

where \( n_{ij}^* = \begin{cases} n_{ij}^- & \text{for } \varepsilon_i > 0 \\ n_{ij}^+ & \text{for } \varepsilon_i < 0 \end{cases} \).

Analogously to \( \mathbf{m}^* \),

\[
n_{44}^+ = n_{44}^*.
\]

4.1.1. Characterizing \( \mathbf{m}^* \). As mentioned in section 3.2, the thin film’s geometrical change is accounted for evaluating its 2D resistivity via the Montgomery method; however, it should also be accounted for evaluating its strain sensitivity. Equation (10a) can be rearranged as

\[
\rho_x = R_x^* A L, \tag{19}
\]

where

\[
R_x^* = \frac{\pi}{8} \frac{L_x}{L_y} \sinh \left( \frac{\pi L_y}{L_x} \right), \tag{20a}
\]

\[
A = L_y^i d_i, \tag{20b}
\]

\[
L = L^i c_i. \tag{20c}
\]

It is analogous to calculating the bulk resistivity of a rectangular conductor with a gage length of \( L \) and a cross-sectional area of \( A \). The resistance response measured across \( L \) is hence \( R_x^* \). According to Ciureanu et al [28], the normalized gage resistivity change, \( \frac{\Delta R_x^*}{\rho_x} \), can be correlated to the strain state via

\[
\frac{\Delta \rho_x}{\rho} = \frac{\Delta R_x^*}{R_x^*} = \varepsilon_x + \varepsilon_y + \varepsilon_z, \tag{21}
\]

where \( \Delta R_x^* \) is the change in gage resistance before and after being loaded, \( R_x^* \) is the bulk resistance along \( L \) under pristine condition, and \( \varepsilon_z \) is the strain developed along the thickness direction. Due to the Poisson’s effect, \( \varepsilon_z \) for isotropic materials can also be derived as

\[
\varepsilon_z = -v(\varepsilon_x + \varepsilon_y), \quad \text{where } v = \frac{\nu}{1 - \nu}. \tag{22}
\]

Equation (22) is true for normal strains under the plane-stress condition, upon which equation (2) is based. Substituting equations (15) and (22) into (21) yields

\[
\frac{\Delta R_x^*}{R_x^*} = (m_{11}^* + 1 + v)\varepsilon_x + (m_{12}^* - 1 + v)\varepsilon_y. \tag{23}
\]

Keep in mind that there are in fact five unknowns, namely \( m_{11}^*, m_{12}^*, m_{22}^*, m_{33}^* \) and \( v \) (i.e., a function of \( \nu \)). It should be noticed from equation (22) that \( v \) is coupled with the rest of the unknowns; it infers that the thin film’s Poisson’s ratio, \( \nu \), cannot be derived. Instead of \( \mathbf{m}^* \), a new elastoresistive coefficient matrix including the material property of the
unknown Poisson’s effect, $m^{a0}$, is characterized in this study, and
\[
\frac{\Delta R^{y}}{R_{x,0}} = (m^{y0}_{11} + 1)\varepsilon_{x} + (m^{y0}_{12} - 1)\varepsilon_{y},
\]
where
\[
m^{y0}_{ij} = m^{y0}_{ij} - \nu, \quad \text{for } i, j = 1, 2.
\]

Hence two loading cases presented in figures 5(a)–(b) provided four independent equations to derive all the coefficients of $m^{a0}$.

Figure 7 plots the resistivity values derived via the Montgomery method with respect to the strain developed in the loading direction (i.e., $\rho_{x}$ is plotted against $\varepsilon_{x}$, and $\rho'_{y}$ is plotted against $\varepsilon'_{y}$ with respect to the x'y' coordinate). It can be observed that the difference between $\rho_{x,0}$ and $\rho_{y,0}$ is less than 0.1, and it is also true for that between $\rho'_{x,0}$ and $\rho'_{y,0}$. It suggests that printing alternatively in rotated directions could effectively reduce the thin film’s anisotropy in resistivity due to the printer head movement. Although the difference cannot be totally eliminated, it is relatively small (i.e., less 5% of the original signal) comparing to the actual resistivity values, which range from 1.62 to 1.82. The result shows that inkjet-printing is able to reproduce CNT thin films with nearly identical electrical properties, ensuring reliable and repeatable sensing capability. Moreover, a fairly linear trend is observed between each resistivity change and the strain in loading direction, $\varepsilon_{y}$, supporting the linearization assumption of the proposed characterization method.

Figure 8 plots the result of every elastoresistivity coefficient calculated at each strain state with respect to $\varepsilon_{y}$. The value of each coefficient is observed to be linearly dependent to the strain value. It can be explained by the tunneling effect of a CNT nanocomposite that contributes to its piezoresistivity. When the CNT nanocomposite is deformed by strain, the enclosed CNTs have been re-oriented, creating a new CNT network. Therefore its strain sensitivity is also different from its previous loading state, and it is dependent on its current strain state.

It is also observed that the thin film is much more sensitive to compressive strains than to tensile strains, as the initial value of $m_{11}^{+}$ is much higher than that of $m_{11}^{-}$, and the slope of $m_{11}^{+}$ is much steeper than that of $m_{11}^{-}$. Similar results can also be observed between $m_{12}^{+}$ and $m_{12}^{-}$. The difference of a CNT nanocomposite’s strain sensitivity toward compressive- and tensile strains has been reported by numerous studies in the past [31, 32, 41]; most of them found that the nanocomposite is more sensitive to compressive strains than to tensile strains, similar to the finding in this study. The reason of this phenomenon still remains unclear, but Schadler et al [31] discovered that the lower Raman peak of CNT nanocomposites under tension than compression may suggest a more insufficient load transfer between CNTs and the matrix under tensile load [31].

The shear elastoresistivities of $m_{31}^{+}$ and $m_{31}^{-}$ are evaluated based on equation (16a)–(16c). As seen in figure 8, they are fairly overlapping with each other with minor differences that could possibly due to environmental error. The result validates the independence of strain directions of shear elastoresistive coefficients. Equation (26a)–(26b) summarizes the final characterization result of $m^{a0}$. Due to its strain-dependency, each coefficient is derived by extrapolating the data shown in figure 8 to the point of $\varepsilon_{y} = 0$. This is achieved by performing least-squares quadratic fitting over the data and calculate the result at $\varepsilon_{y} = 0$, as shown in figure 8. Hence the result in equation (26a)–(26b) are the elastoresistivity matrices under zero strain state. Note that figure 8 is plotted solely with respect to the strain in the loading direction; it only demonstrates the approximate linearity between each coefficient and the longitudinal strain level applied in this study. Therefore calculating the slope of each curve may not be beneficial to other loading situations. A more sophisticated model with consideration of the material’s nonlinear elastoresistive behavior shall be established in future research to take into account the strain-dependency.

\[
m^{a0+} = \begin{pmatrix} 0.59 & 1.74 & 0 \\ 1.74 & 0.59 & 0 \\ 0 & 0 & 2.02 \end{pmatrix} \quad \text{for } \varepsilon_{y} > 0, \quad (26a)
\]
\[
m^{a0-} = \begin{pmatrix} 5.82 & -3.46 & 0 \\ -3.46 & 5.82 & 0 \\ 0 & 0 & 2.04 \end{pmatrix} \quad \text{for } \varepsilon_{y} < 0. \quad (26b)
\]

Figure 9 plots the variation of $m^{a}$ with respect to a Poisson’s
Figure 9. The variation of elastoresistive coefficients due to Poisson’s ratio is plotted. Except for shear strains, a larger Poisson’s ratio would result in lower strain sensitivity.

ratio varying from 0 to 0.5; the result is calculated based on equation (25). The elastoresistivity of the thin film decreases as its Poisson’s ratio increases, except the shear elastoresistive coefficients, which are independent of strain directions. It suggests that the thin film’s strain sensitivity can be impacted by a large Poisson’s ratio. However, such impact is minor if the Poisson’s ratio is lower than 0.3, which is the range of typical polycrystalline and amorphous materials [42].

4.1.2. Characterizing \( n^* \): As mentioned in earlier sections, EIT maps the local conductivity change induced by damages or strain-states over an FE model. The FE model is usually constructed based on the geometry of the structure under its pristine condition, i.e., assuming the boundary shape remains intact before and after the damage. Therefore it is reasonable to additionally characterize the thin film’s elastoresistivity without considering geometrical change, as denoted in equation (17), for EIT analysis. In this case, equation (21) is simplified as

\[
\frac{\Delta \rho_s}{\rho} = \frac{\Delta R^s_{xx}}{R^s_{xx,0}}.
\] (27)

Therefore,

\[
\frac{\Delta R^s_{xx}}{R^s_{xx,0}} = n_{11}^* \varepsilon_x + n_{12}^* \varepsilon_y.
\] (28)

Similarly, four unknowns including \( n_{11}^* \), \( n_{11}^* \), \( n_{12}^* \), and \( n_{22}^* \) are derived from the two loading cases shown in figures 5(a)–(b). Comparing equation (24) to (28) it can be concluded that,

\[
n_{11}^* = m_{11}^{\varepsilon_1} + 1, \tag{29a}
\]

\[
n_{12}^* = m_{12}^{\varepsilon_2} - 1, \tag{29b}
\]

\[
n_{22}^* = m_{22}^{\varepsilon_3} + 1. \tag{29c}
\]

Equations (30a)–(30b) summarize the characterization result of \( n^* \). Note that these are the elastoresistivity matrices used to correlate the strain values from DIC measurement and being compared with the EIT results, which are presented in the following section.

\[
\begin{pmatrix}
1.59 & 0.74 & 0 \\
0.74 & 1.59 & 0 \\
0 & 0 & 3.02
\end{pmatrix}
\quad \text{for } \varepsilon_l > 0, \tag{30a}
\]

\[
\begin{pmatrix}
6.82 & -4.46 & 0 \\
-4.46 & 6.82 & 0 \\
0 & 0 & 3.04
\end{pmatrix}
\quad \text{for } \varepsilon_l < 0. \tag{30b}
\]

4.2. Reconstruction of the spatial strain distributions

Figures 10(a)–(c) plot the DIC measurement results of the tensile-loaded coupon with a center crack under a total displacement of 1500 \( \mu \text{m} \). Figure 10(a) plots the transverse direction strain, \( \varepsilon_y \), due to the Poisson effect. Note that the stripe-patterned positive contrast shown in the middle of the image came from the out-of-plane deformation of the coupon due to the existence of the center crack. Figure 10(b) plots \( \varepsilon_y \) the strain developed in loading direction. Clusters of large tensile strains are observed near both crack tips, indicating the formation of stress concentrations as known from e.g., linear elastic fracture mechanics [43]. Figure 10(c) plots the planar shear strain \( \varepsilon_{xy} \).

Figure 11(a) plots the normalized EIT reconstruction result of the tensile-loaded coupon. An FE model was constructed to include the presence of the center crack for EIT analysis, hence localized resistivity changes milder than the crack opening can be observed. Major negative contrasts are observed near both crack tips, indicating the existence of large tensile deformation. This is similar to the DIC result shown in figure 10(a), of which however, is still not a fair match to the EIT reconstruction in terms of the damage severity. This is because an EIT result reconstructs the resultant of a resistivity state, instead of a single vector of that. Hence to compare the results, the strain state data presented in figures 10(a)–(c) were first correlated to the normalized resistivity change via equation (17) by \( n^* \) presented in equations (30a)–(30b). Suggested by Hamilton et al. [38], the EIT result of an anisotropic medium reconstructs the determinant of its resistivity state. The square-root of the determinant of the normalized resistivity change was therefore calculated as

\[
\det \left( \frac{\Delta \rho}{\rho} \right) = \sqrt{\Delta \rho_x \Delta \rho_y - \Delta \rho_{xy}^2},
\] (31)

Then result is then normalized to a range of \([-1, 1]\), and then plotted in figure 11(b). Comparing to figures 10(a), 11(b) illustrates damage severity closer to the situation presented in figure 11(a), cohering with the idea from Hamilton et al. [38]. A possible explanation can be found in the determinant of the strain tensor, which is a measure of geometric distortion. In analogy the EIT result is a scalar measurement of localized resistivity change particularly caused by the tunneling effect of the distorted CNT network. Therefore, it is a reasonable hypothesis to relate the determinant of normalized resistivity change to the EIT reconstruction result. It should be noted that the resolution of the DIC data is \( 226 \times 226 \), which is nearly two magnitudes higher than that of the EIT data, which is \( 4 \times 4 \). It may explain the difference between figures 11(a)
and (b) in mapping the out-of-plane deformation. Moreover, the MAP method adopted in this study in solving EIT problems usually causes over-smoothing of the reconstructed area, which can be observed in figure 11(a), to be larger than it is in real life. Despite of these differences, it can be concluded that the characterized elastoresistive matrix can be used to correlate the overall strain state, and the resultant of the correlation is similar to its EIT reconstruction results.

Figure 10. The DIC strain measurement in (a) transverse direction ($\varepsilon_x$), (b) longitudinal direction ($\varepsilon_y$), and (c) shear direction ($\varepsilon_{xy}$) of the tensile-loaded coupon with a center crack are plotted.

Figure 11. (a) The EIT reconstruction identifies the location and severity of the stress concentrations near both tips of a center crack. (b) The square-root of the determinant of the resistivity values correlated from the DIC strain measurements is shown.
5. Conclusion

This paper presents the characterization result of the elastoresistivity matrix of an inkjet-printed CNT thin film for spatial damage sensing. Inkjet-printing is a cost-effective technique to rapidly pattern nanomaterials, assembling CNT thin films with uniform electrical properties. When coupled with EIT, the CNT thin film can be applied as a spatial damage sensor. To further explore its potential in spatial strain state sensing, its fourth-rank elastoresistivity matrix was characterized to derive its strain sensitivity with respect to strain directions. A linear correlation was established between the thin film’s normalized resistivity state and its strain state under the plane-stress condition. The Montgomery method for characterizing anisotropic resistivities was adopted to estimate $\rho_x$ and $\rho_y$ of a square-shaped CNT thin film under uniaxial tensile loading. The test result suggested that the CNT thin film is more sensitive to compressive strains than to tensile strains, hence two elastoresistivity matrices with four unknowns were characterized for both types of strains. The thin film’s Poisson’s ratio, however, was not able to be derived simultaneously, but its influence was found to be insignificant under the value of 0.3. Lastly, the characterized results were applied to correlate the strain state of a tensile-loaded coupon with a center crack. The correlated result of the CNT thin film represented its normalized change in resistivity state due to the tensile strain, and its determinant map was in fair agreement with the EIT reconstruction performed over the CNT thin film’s coverage. The characterized elastoresistivity matrix provides an alternative approach to understand the spatial strain sensitivity of a CNT thin film, and helps interpreting the tomographic reconstruction of a loaded body in terms of its strain state. However, the final target for damage detection is the identification of the strain field, which is influenced by the damage. Although it is still challenging to derive the complete strain field based solely on EIT reconstruction results, the determinant of the change of the resistivity field may still provide quantitative evaluations of the structure under assessment. This study helps advance inkjet-printed CNT thin film coupled with EIT toward its real-world application as a spatial damage monitoring device for lightweight-designed structures. Future research will consider assembling stratified nanocomposites via inkjet-printing to further improve the sensor’s strain sensitivity and simplify the data analysis process.

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