Conformable Single-Walled Carbon Nanotube Thin Film Strain Sensors for Structural Monitoring
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ABSTRACT

The need for monitoring the condition of large-scale infrastructure systems has motivated a recent interest in novel sensor technologies for structural health monitoring. To provide the structural health monitoring system with data that captures local structural behavior, the measurement of component-level strain is valuable. Although current foil-based strain gauges are capable of measuring strain deformations, their low sensitivity and drift properties render them poorly suited for long-term field installation. In this study, a novel strain sensor is presented for the purpose of structural monitoring. The presented strain sensor is based on a layer-by-layer chemical film deposition technique to produce a nanocomposite consisting of single-walled carbon nanotubes (SWNT) and polyelectrolytes (PE). Engineered at the molecular-level, the performance of the thin film strain sensor can be optimized for structural monitoring applications, including attainment of high gauge factors. To validate the sensitivity and precision of the conformable SWNT-PE thin film strain sensor, this study undertakes a series of laboratory experiments where thin films are loaded under monotonic and cyclic load patterns.

INTRODUCTION

Over the past decade, excitement has surrounded structural monitoring technologies because of their importance in the management of aging infrastructure inventories. When automated damage detection is included within the design of a structural monitoring system, a structural health monitoring system emerges. Historically, the high costs associated with structural monitoring systems have limited the total number of sensors installed. However, a recent emergence of low-cost sensor technologies, including wireless sensors, encourages the use of even greater numbers of sensors in a single monitoring system. An advantage associated with using a large number of sensors is the ability to investigate structures at the component level. With structural damage inherently a local phenomena, local-based monitoring strategies will enhance current structural health monitoring systems’ resolutions. Identification of localized structural damage, including cracks and plastic deformation, can be identified from time history measurements of component strains and deformations. For this purpose, metal foil strain gauges are widely used to measure component strains both in the laboratory and in the field.
Metal foil strain gauges consist of a thin metal wire folded upon a polymer substrate; when the gauge undergoes strain, a corresponding change in the electrical resistance of the metal wire is observed. While metal foil strain gauges can serve as low-cost and accurate strain sensors, they do suffer from some performance limitations. For example, strain gauges are often of low resistance that require abundant amounts of electrical energy to operate. Furthermore, low gauge factors limit their measurement resolution; this can make accurate measurement of low levels of strain difficult. Last, metal foil strain gauges deployed in the field exhibit drifts due to temperature sensitivity and to long-term changes in metal foil conductivity properties. In response to these limitations, alternative transducer solutions for measuring strain have been offered. For example, fiber optic strain gauges that measure strain in concrete structures have been growing in popularity. Another set of alternative strain sensors include those fabricated upon silicon substrates using the fabrication tools offered by the field of microelectromechanical systems (MEMS).

MEMS fabrication techniques have been widely used to design novel sensing transducers for structural monitoring, including accelerometers, ultrasonic transducers and strain sensors. Conceptually, the MEMS design process begins with a mechanical sensor design which is then miniaturized to micrometer length-scales. This design process is one of scaling down sensor mechanisms from the macro-scale to the micro-scale. An opposite approach offered by the nascent field of nanotechnology is to design sensors from the opposite direction; beginning at the nano-scale, deliberate molecular manipulation can be used to design novel sensors. In this study, the impressive mechanical, chemical, and electrical properties of single-walled carbon nanotubes (SWNT) will be utilized to design a nanocomposite that can be used for strain sensing. The use of nanotechnology is important because it allows for the fine-tuning and tailoring of sensor properties so as to obtain strain sensors whose performance is superior to that offered by current metal foil strain gauges.

In this paper, we present a first step towards a methodical fabrication process by which thin film strain sensors can be created to rectify the performance deficiencies of metal foil strain gauges. Particularly, a thin film strain sensor is fabricated using single-walled carbon nanotubes (SWNT) and various polyelectrolytes (PE) combined in a layer-by-layer (LBL) assembly process. Various laboratory validation experiments will be performed with conformable SWNT-PE thin films mounted to structural specimens that are then loaded under monolithic tension and four-point bending.

SWNT-PE THIN FILM FABRICATION

Since the discovery of the single-walled carbon nanotube in 1991 by Iijima [1], researchers have come to understand their unique electrical, chemical and mechanical properties. With optimal properties in mechanical strength, electrical conductivity, and mass density, carbon nanotubes exceed the performance of many current engineering materials. Individually, each carbon nanotube is a graphene sheet rolled to form a cylindrical structure whose diameter can span from 0.7 to 10.0 nm and whose length-to-diameter aspect ratio ranges from $10^3$ – $10^5$ [2, 3]. At such length scales, the rolled hexagonal/honeycomb graphene structure of each SWNT is highly
one-dimensional resulting in many of its impressive material properties [4]. For example, a one-dimensional structure allows electrons to travel greater distances since electrons are restricted from scattering, thereby greatly increasing its electrical conductivity. Moreover, the graphene honeycomb cylindrical structure provides for the great mechanical strength of SWNT. Many researchers have attempted to determine the mechanical properties of carbon nanotubes; the general consensus is that SWNT out-performs steel in terms of elasticity and ultimate strength [5].

While SWNT have outstanding mechanical and electrical properties that are of extreme interest to engineers, using SWNT alone is difficult from a fabrication standpoint. For example, researchers have utilized individual carbon nanotubes placed upon a MEMS-fabricate silicon substrate to act as mechanical sensors [6]. In the study described by [6], individual nanotubes are manipulated using atomic force microscopy (AFM) tips to obtain specific locations and orientations on the substrate. Because of the need for AFM, this process is time consuming and necessitates the use of expensive equipment. Alternatively, researchers can take full advantage of SWNTs by including them within composite materials. In this study, a SWNT composite, with desired bulk material properties, will be designed and fabricated for strain sensing. The resulting SWNT composite will exhibit linear changes in conductivity (resistivity) as a result of strain. This piezoresistive behavior will serve as the primary read-out mechanism for the strain sensor. The fabrication of the SWNT-based thin film strain sensor is accomplished using a layer-by-layer assembly process to create a thin film whose molecular structure consists of alternating layers of SWNTs and polyelectrolytes of high molecular weight [7]. Pursuit of an LBL process is largely motivated by the simplicity of the approach and the ability to attain well-controlled bulk material properties (mechanical and electrical).

Use of carbon nanotubes for the fabrication of sensors has been previously explored by other researchers. Dharap et al. [8] have proposed the design of strain sensors using a homogenous assembly of SWNT known as “buckypaper”. Their work has illustrated the potential for SWNT in the design of strain sensors defined by high degrees of linearity. Others have utilized the LBL methodology with multi-walled carbon nanotubes (MWNT) to fabricate mechanical sensors [9]. Although experimental results from those studies indicate a linear mechanical-electric behavior of the MWNT thin film, pullout of the concentric graphene tubes is witnessed at high strains resulting in linearity degradation [10].

Layer-by-Layer Assembly Method

A sufficiently thick film is necessary to allow the SWNT-PE strain sensor to be easily handled during fabrication and installation upon structural surfaces. In general, the process of building-up thicker LBL films entails the deposition of multiple layers of SWNT and PE materials upon a glass or silicon substrate. With alternating electric charge deposited on each layer, each film monolayer is adsorbed by the layer below through electrostatic and van der Waals forces [6, 9]. The specific steps taken in the LBL fabrication process are fully described herein.

Initially, a bare glass substrate is dropped into a polyelectrolyte bath to create the initial charged layer of the LBL assembly process. In this study, the polyelectrolyte selected for the thin-film strain sensor is polyvinyl alcohol (PVA). After dipping the
substrate in the PVA solution for a set period of time, the substrate is removed and placed in a rinsing bath of deionized water. To deposit a monolayer of SWNT, the substrate is next dipped in a solution of SWNT that is fully dispersed. To ensure the SWNT are uniformly distributed, the SWNT dipping solution is achieved through the use of a second polyelectrolyte bounded to the surface of individual nanotubes. An additional benefit of employing a polyelectrolyte to facilitate dispersion is they provide the SWNT monolayer with a charge opposite to that of the PVA monolayer resulting in greater cross-linking between layers. After dipping the substrate in the dispersed SWNT solution, the substrate is again placed in a rinsing solution of deionized water. This process completes the deposition of the first SWNT-PE bi-layer; the process is repeated until a desired number of SWNT-PE bi-layers are achieved. By controlling the substrate dipping time in each solution, constant-thick bi-layers can be fabricated to form SWNT-PE thin films of desirable total thickness.

In this study, highly purified single-walled carbon nanotubes have been obtained from Carbon Nanotechnologies, Inc. (CNI). The selection of CNI SWNTs is primarily based on their ultra low impurity content so that an optimal balance between the resulting SWNT-PE thin films’ mechanical and electrical properties can be systematically achieved in the laboratory. However, to be able to process these carbon nanotubes for the LBL assembly of SWNT-PE thin films, the SWNTs must be suspended in solution form. Ultra-pure SWNT have a tendency to quickly precipitate out of solution since mechanical perturbations to the solution will cause clumping of the dispersed SWNTs. Fabrication of high quality thin films requires good dispersion of SWNT. A series of experiments are carried out to evaluate the dispersion quality of different polymer solutions mixed with deionized water. Various polymer solutions of different concentration have been experimented with in order to achieve an optimal dispersion of the SWNT-polymer solution.

From a series of experiments, it is found that CNI carbon nanotubes can be easily dispersed in 0.5% to 1.0% poly(styrene 4-sulfonate) (PSS) solution with the assistance of a two-hour sonication bath. Here, PSS is used as the chemical agent that brings about good dispersion quality of the carbon nanotube solution. In addition, the dispersion of SWNT in a PSS solution will allow for the PSS to wrap around the SWNT cylindrical graphene structure to give them an overall negative surface charge [7]. Using 1% PVA as the polyelectrolyte layer for LBL assembly, initial hand-dipping shows excellent SWNT deposition and adsorption to form constant-thickness SWNT-PVA thin films. To confirm these findings, images of the thin films are obtained using scanning electron microscopy (SEM). As shown in Figure 1(a) and (b), the SEM images verify the distribution, load, and dispersion qualities of the SWNT solution.

Using the described assembly process, SWNT-PVA thin films are successfully fabricated with a different number of bi-layers, namely 35, 50, 100, 150, and 200 layers. After the films are assembled, films are removed from the glass substrate using a razor blade. The films are relatively ductile allowing them to be removed without tearing. Alternatively, a hydrofluoric (HF) solution can also be used to etch the SWNT-PE films from the substrate.
MONOTONIC TENSILE TESTING OF STRAIN SENSOR

In order to validate the performance of the free-standing SWNT-PVA films produced, including assessment of sensor sensitivities and noise properties, monotonic tensile tests are performed. The conformable SWNT-PE thin films are cut into small rectangular shapes (approximately 4.0 x 8.0 mm strips) that are then mounted to the sides of 1.9 cm diameter aluminum rods using standard strain gauge mounting procedures, including the use of post-yield epoxy, as shown in Figure 2(a). This unique epoxy, combined with the selection of a highly ductile material (such as aluminum), is suitable for assessing the capability of the SWNT-PE strain sensors at high strains.

Once the SWNT-PE thin films are mounted to the test sample surface, single-strand electrical wire is attached the two ends of the thin film using silver paste. The test samples are stored in a cool place allowing the silver paste and epoxy to dry for more than 12 hours before testing. This long storage time ensures that the silver paste dries and establishes a firm adhesion to both the test sample and SWNT-PE thin film, so as to prevent the lead wires from being pulled-out during loading. The entire test sample is then loaded into a standard laboratory load frame which is used to apply monotonic tensile loading on the aluminum specimen. The change in conductivity of the SWNT-PE thin film resulting from tensile loading is obtained by employing an Agilent 34401A digital multimeter. The conductivity of the film is measured in real-time using a 1 Hz sample rate; conductivity measurements are stored in a standard laboratory data acquisition system. To accurately measure the strain of the aluminum rods during tensile loading, an MTS extensimeter is simultaneously attached to the aluminum specimens during testing. Using the same data acquisition system as that used to measure thin film conductivity, deformation of the aluminum rods are measured by the MTS extensimeter at a sample rate of 10 Hz.

Once firmly mounted in the load frame, a monotonic tensile load pattern is applied to the aluminum coupon over a 60 second time period. During loading of the coupon, the electrical resistance of the SWNT-PE thin film is measured. Figure 3(a) overlays the SWNT-PE thin film electrical resistance time-history upon the time-history plot of the specimen strain. In this experiment, a 200 layer SWNT-PE thin film was employed. At the initiation of the loading, the resistance of the SWNT-PE thin film is 830 Ω. During monotonic loading, the film undergoes an increase in

Figure 1. (a) SEM image of a two bi-layer of SWNT-PVA film with SWNT dispersed in 0.5% PSS. (b) Highly magnified image of Figure 1(a) showing individual carbon nanotubes.
resistance in tandem with increasing coupon strain. As can be seen, the ductile specimen undergoes a maximum tensile strain of 7.3% before failure at which point the electrical resistance of the thin film is greater than 1.1 kΩ. More insight can be gained by plotting the change in resistance of the thin film as a function of the measured strain. In Figure 3(b), we see that the percentage change in the SWNT-PE thin film resistance is linearly proportional to the strain. By fitting a straight line to the resistance($R$)-strain($\varepsilon$) data, the gauge factor ($Gauge Factor = (\Delta R/R)/\varepsilon$) of the thin film is estimated as 4.52. Another observation is the well behaved resistance-strain linearity of the thin film; this behavior suggests the material has tremendous promise for strain sensing. A series of monotonic tensile tests are performed using SWNT-PE thin films of different thicknesses. In fact, further tensile testing shows the gauge factor can be up to 25 depending on the concentration of SWNT used when fabricating the multilayer thin films.

FOUR-POINT BENDING TEST OF SWNT-PE STRAIN SENSOR

While the monotonic tensile tests illustrate the ability for the SWNT-PE thin film strain sensor to capture continuous tensile strain, the experiment is not capable of assessing how SWNT-PE thin films perform under compression. To make such an assessment, a four-point bending test of a highly ductile fiber reinforced cementitious
plate element (13 mm by 76 mm by 305 mm) is used to test the compressive strain capacity of the SWNT-PE thin film sensor. With 100 layer SWNT-PE thin film sensors mounted to both sides of the cementitious plate, the plate is cyclically loaded. For this experiment, the reversibility of the SWNT-PE thin film’s resistance will also be assessed during cyclic loading. In order to evaluate the precision and accuracy of the SWNT-PE thin films for strain sensing, reference strain gauges (Texas Measurements YFLA-5-5L) are mounted alongside the SWNT-PE strain sensors. In this set of tests, both the resistance of the thin film sensor and the metal foil strain gauge are continuously measured using the Agilent 34401A digital multimeter. The setup of the instrumented cementitious plate element is shown in Figure 4(a).

Four-point bending of the cementitious plate is repeated for a total of four cycles, where for each cycle the maximum vertical deflection at the midpoint of the plate is set at 6 mm (0.24”). The cementitious sample is loaded and unloaded at a rate of 2 mm (0.08”) per minute. The time-histories of the resistance of both sensors can be plotted, as shown in Figure 4(b). As witnessed by the resistance time-histories, the behavior of the SWNT-PE thin film correlates well with the change in resistance of the reference gauge. However, it is observed that there appears to be a mild drift in the film resistance over time. This drift might be attributed to minor polarization of the SWNT-PE thin film, although more testing is needed to make this conclusion. The SWNT-PE strain sensor displays reversible behavior and accurately captures the tensile and compressive strain as experienced by the cementitious plate under four-point bending.

CONCLUSION

In this paper, a simple and low-cost methodology for the fabrication of single walled carbon nanotube-polyelectrolyte (SWNT-PE) composites using an LBL assembly process is described. The use of carbon nanotubes in the design of novel structural sensors is motivated by impressive mechanical and conductive electrical properties. LBL fabrication is capable of producing free-standing SWNT-PE thin films of desired thickness; in addition, bulk electrical and mechanical properties of the films can be tailored based on the concentration of SWNT and the type of polyelectrolyte selected. In a similar manner, the conductivity and piezoresistive
properties of the SWNT-PE strain sensor can be tailored to meet specific performance objectives including high gauge factors. The result is a conformable SWNT-PE strain sensor well suited for monitoring the strain deformation of structural elements. Monotonic tensile loading and four-point bending experiments are performed on structural elements with the SWNT-PE strain sensor mounted. With sensitivities higher than traditional strain gauges, SWNT-PE thin films potentially offer greater strain measurement resolution. In the near future, various studies will be performed to further enhance the properties of the SWNT-PE thin film strain sensors. In particular, the strain sensor will be fine-tuned using specific polymers and different SWNT to PE concentration to reduce signal to noise ratios and to eliminate observed drifts in resistance measurements. More elaborate laboratory and field validation experiments will be performed to further assess the performance of SWNT-PE strain sensors.

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REFERENCES