Free-Standing Carbon Nanotube Composite Sensing Skin for Distributed Strain Sensing in Structures

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ABSTRACT

The technical challenges of managing the health of critical infrastructure systems necessitate greater structural sensing capabilities. Among these needs is the ability for quantitative, spatial damage detection on critical structural components. Advances in material science have now opened the door for novel and cost-effective spatial sensing solutions specially tailored for damage detection in structures. However, challenges remain before spatial damage detection can be realized. Some of the technical challenges include sensor installations and extensive signal processing requirements. This work addresses these challenges by developing a patterned carbon nanotube composite thin film sensor whose pattern has been optimized for measuring the spatial distribution of strain. The carbon nanotube-polymer nanocomposite sensing material is fabricated on a flexible polyimide substrate using a layer-by-layer deposition process. The thin film sensors are then patterned into sensing elements using optical lithography processes common to microelectromechanical systems (MEMS) technologies. The sensor array is designed as a series of sensing elements with varying width to provide insight on the limitations of such patterning and implications of pattern geometry on sensing signals. Once fabrication is complete, the substrate and attached sensor are epoxy bonded to a poly vinyl composite (PVC) bar that is then tested with a uniaxial, cyclic load pattern and mechanical response is characterized. The fabrication processes are then utilized on a larger-scale to develop and instrument a component-specific sensing skin in order to observe the strain distribution on the web of a steel beam. The instrumented beam is part of a larger steel beam-column connection with a concrete slab in composite action. The beam-column subassembly is laterally loaded and strain trends in the web are observed using the carbon nanotube composite sensing skin. The results are discussed in the context of understanding the properties of the thin film sensor and how it may be advanced toward structural sensing applications.

Keywords: carbon nanotube composite; optical lithography; spatial damage detection; MEMS; nanotechnology

1. INTRODUCTION

The challenges facing America’s aging network of infrastructure systems require engineers to more accurately quantify the performance and health of their structures. The need for improved health assessment is growing given the vast portion of infrastructure identified as requiring repair or replacement by a recent American Society of Civil Engineers (ASCE) report that designated American infrastructure with a “D+” grade point average [1]. Over the coming decades, demand for accurate structural health characterization is predicted to grow due to the need to adopt more cost-efficient repair and replacement strategies within severe budget constraints. The ramifications of inadequate approaches to infrastructure management can lead to catastrophes on scale with the recent the I-35W Minneapolis bridge collapse (2007) [2].

Contemporary sensing technologies fail to meet all of the needs of the structural management field. Structural integrity assessment in most industries is still based on visual inspection. Visual inspection is subjective, labor intensive, and may not be feasible in areas that are difficult to reach. An alternative approach is to install a structural monitoring system consisting of point sensors (i.e., sensors making a discrete, localized measurement). Point sensing provides valuable quantitative information as is needed for informed decision making; however measurements at a single location or finite set of locations is generally insufficient for accurate damage identification. Furthermore, densification of the sensor network remains prohibitive in terms of both cost and installation complexity. A potentially more effective approach may be to seek spatial measurements over an area or volume of a structure. This view has motivated researchers to aspire to develop spatial structural sensing technologies at the component level that can directly detect damage in the structure.
The potential for advanced sensing technologies is bolstered by advances in material science and nanotechnology. For instance, the discovery of carbon nanotubes [3] has generated significant interest in many sensing communities as they have displayed a range of exceptional physical and electrical properties. These properties include impressive strength, stiffness, aspect ratio, and a range of intriguing electrical characteristics [4]–[6]. The properties of carbon nanotubes can be extended to larger applications by embedding nanotubes in a polymer matrix to form nanocomposites [7]. This allows for the nanoengineering of thin film properties including an electrical impedance response to mechanical strain [8]. One established method for the uniform fabrication of such nanocomposites is a layer-by-layer (LbL) deposition process which utilizes oppositely charged polyelectrolytic solutions for surface adhesion to sequentially build films [7]. Through this process, films have been fabricated with capabilities for spatial strain sensing [9]. However, these methods are yet to take hold as an optimal sensing solution due to the challenges of instrumenting such sensors on an actual structure. For example, prior work in LbL assembled thin films sensors deposited the film on the structure itself; this is not a scalable approach and rules out retrofit application on existing structures. Another challenge identified is the approach of creating spatial mappings of film readings. While electrical impedance tomography (EIT) has been shown to be capable of deriving film resistivity mappings from a finite set of boundary measurements, the method is computationally expensive.

Nanocomposite sensing skins can be further developed through processes commonly used in microelectromechanical systems (MEMS). Patterning CNT based composites has been investigated by a number of researchers [10]–[13]. Of the various patterning methods attempted, optical lithography provides an effective approach for patterning polymer materials by leveraging mature cleanroom tools and processes. In using optical lithography, polymer-based thin films can be patterned with exceptional geometric control, thereby opening the door for a diverse set of applications [11]. Additionally, the use of widely established patterning tools would allow for a relatively low cost and repeatable means to the design and production of thin films sensors. Patterning thin film sensors broadens the options available to engineers in designing thin film electronics and, more specifically, impedance-based thin film sensors.

Here, a patterned nanocomposite sensing film is developed as the basis for future component-specific strain sensing skins. The potential for this patterning is first explored through the fabrication and testing of five linear sensing elements of varying width. These sensing elements are patterned on a flexible polyimide substrate using conventional optical lithography. The nanocomposite sensing material is deposited using the LbL fabrication process. These sensors are designed with varying sensor widths so that the limits of the fabrication process can be assessed in the context of strain sensing. The mechanical-electrical behavior of the sensing element is tested using uniaxial cyclic testing on standard tensile coupon specimens. The patterned sensing skin technology is further validated using a large sensing skin designed specifically for monitoring the strain profile of a flexural structural member. In this proof-of-concept study, two sensing skins are designed, fabricated and instrumented on the web of a steel I-beam in close vicinity to a welded beam-column connection exposed to cyclic lateral loading. The paper concludes with a summary of the key results and a discussion of the future work required to further advance the patterned sensing skin technology.

2. FABRICATION PROCESS

2.1 Materials

PI-2525 polyimide and VM-651 polyimide adhesion promoter were obtained from HD Microsystems. Poly(vinyl alcohol) (PVA) and poly(sodium 4-styrenesulfonate) (PSS) were obtained from Sigma-Aldrich. HiPCO Single walled carbon nanotubes (SWNT) were obtained from Unidym, Inc. SPR 220-3.0 photoresist from Rohm and Haas Co. and AZ 726 developer from Clariant Corporation were used as supplied by the Lurie Nanofabrication Facility at the University of Michigan. Silver paste and copper tape were obtained from Ted Pella, Inc. All solutions were made using 18 MΩ cm deionized (DI) water.

2.2 Optical Lithography

Fabrication is completed by layering thin films on a glass slide followed by detachment of the fabricated materials from the slide to yield a free-standing thin film. This process begins by spin coating adhesion promoter and then polyimide on
The nanocomposite film is then deposited by spinning a polyimide that is not covered with PR at 2000 rpm resulting in a thickness of around 12 microns for each polyimide layer. Four polyimide layers are deposited to produce a polyimide film with a thickness of around 48 microns (Fig 1a). The polyimide is cured on a hot plate by ramping the temperature from 25 °C to 300 °C at 5 °C/minute and then holding the temperature at 300 °C for one hour. Next, photoresist (PR) is patterned on the substrate using optical lithography. This begins with drying and priming the polyimide for PR using an image reversal oven to apply a hexamethyldisilazane (HMDS) layer. Next, a PR layer is spun over the sample at 2000 rpm and soft baked for 90 seconds at 115 °C. The PR is then exposed under a mask. A post exposure bake is completed for 90 seconds at 115 °C and then the PR is developed with AZ 726 developer for two rounds lasting 60 seconds each.

After PR is patterned on the polyimide substrate, the process transitions to the fabrication of the CNT thin film. The polyimide that is not covered with PR is treated with poly-L-lysine by soaking for five minutes to promote surface adhesion with the polyelectrolytes that will comprise the nanocomposite film. The nanocomposite film is then deposited over the polyimide and PR by the LbL directed-assembly method (Fig. 1b). When this is complete, the CNT composite film is lifted off of the PR covered areas by placing the samples in an acetone bath for 10 minutes and then placing this bath in a bath sonicator to apply surface energy to aid in tearing of the CNT composite film. During bath sonication, energy is applied for 30 seconds and then the samples sit still for one minute. This process continues for approximately 10 minutes until the PR film has fully lifted from the surface.

The specimens fabricated by robotic LbL assembly on glass slides (termed herein as small-scale sensing skins) are patterned as 5 parallel strips roughly 2 cm long with varying thicknesses: 10 um, 250 um, 500 um, 1000 um and 1500 um (Fig. 1c). At the end of the strips are square pads where silver past can be applied to create a wired electrical connection to a data acquisition system that is used to measure film resistances.

### 2.3 Layer-by-Layer Film Fabrication

The nanocomposite sensing skins are fabricated with an established layer-by-layer fabrication process. This process utilizes oppositely charged polyelectrolyte solutions to attract thin layers of each solution to the substrate surface. The substrate is sequentially dipped in two solutions of opposite charge in order to build up a well-controlled, uniform thin film. The positively charged solution used here is 1.0 wt. % PVA and the negatively charged solution is 1.0 wt. % PSS. Single wall carbon nanotubes (0.1 wt. %) are non-covalently dispersed in the PSS solution using deep tip (3.178 mm tip, 150 W, 22 kHz, 90 minutes) and bath (135W, 42 kHz, 360 minutes) sonication. The deposition sequence for one layer of one polyelectrolyte solution consists of dipping in the solution (PSS or PVA) for four minutes followed by submersing in DI water twice for two minutes each. These steps are then followed by low pressure air drying for seven minutes and high pressure air drying for one minute. This results in the application of a single monolayer (PSS or PVA).

Once this process is completed, the process is repeated using the oppositely charged polyelectrolyte solution to yield a single thin film bi-layer. The bilinear process is repeated until a film with a thickness of 50 bilayers is fabricated. The fabrication process is automated with a LbL robotic fabrication setup. Further explanation of the layer-by-layer process is available in previous publications [8]. Once the nanocomposite film is deposited and patterned by the lift-off process,
the film is annealed for 20 minutes at 180 °C on a hot plate. Finally, the polyimide substrate is removed from the substrate by etching away a layer of glass using buffered hydrofluoric acid.

2.4 Larger-Scale Development

The fabrication methodology is scaled up for the development of component-specific strain sensors over larger areas. The larger-scale fabrication is completed on four inch (10.2 cm) diameter glass wafers. The process is the same as previously summarized with the exception of certain aspects of the optical lithography and nanocomposite deposition. The HDMS priming, PR spinning, and developer processes are automated with an automated optical lithography cluster tool when fabricating on a four-inch glass wafer. LbL deposition is performed by hand to accommodate the larger fabrication area. The wafer specimens were hand dipped in solution (PVA and PSS) for five minutes, rinsed with DI water, and then dried completely to fabricate each monolayer. Fabrication is otherwise completed as with the smaller glass slides. The larger-scale sensing skins are patterned in the center of the four inch glass wafers in 8 parallel strips roughly 3.5 cm long, 1.5 mm thick, and spaced 4.5 mm apart.

3. EXPERIMENTAL PROCEDURE

3.1 Instrumentation

Fabricated small-scale sensing skins are epoxy bonded to PVC composite specimens for uniaxial tensile testing. This requires the addition of electrodes to the patterned sensing skins. Colloidal silver paste is used to glue copper wires to the film surface that serve as electrodes. As previously mentioned, bond pads are designated in the film design to allow for increased area over which colloidal paste can be applied. A digital multimeter is used to probe the attached electrodes and to determine film resistance once electrodes are dry. The multimeter is used to measure resistance and these values are collected throughout uniaxial cyclic testing. A picture of the instrumented PVC coupon is shown in Fig. 1d.

3.2 Uniaxial Cyclic Testing

The mechanical-electrical response of the sensing skins is determined using uniaxial cyclic testing on a hydraulic load frame. In this process the films are loaded for three cycles of tension-compression. The strain in the structural member is also monitored using a traditional 120 Ω metal foil strain gage opposite the patterned thin film sensor. The ambient behavior of the small-scale sensing skins on the structural members is observed for five minutes prior to each test. This allows for the observation of a drift that is commonly displayed by this type of sensing film. The specimen is then loaded at a rate of 0.5 mm/min and strain data is collected throughout the loading at a 1 Hz sample rate.

3.3 Scaled Proof-of-Concept

The large-scale sensing skins (Fig. 2b) are instrumented on a realistic structural system to illustrate the potential impact of patterned sensing skin technologies. These thin film sensors are epoxy bonded directly onto the surface of steel I-beam members. Once bonded to the surface of a steel beam element, copper tape and colloidal silver paste are used to create electrodes for resistance measurements. Sensing elements are connected to a Wheatstone bridge with a matching resistor to produce a voltage that is low-pass filtered and amplified with a gain of 50. These boards are then connected to a wireless sensing unit that supplies a source voltage (5V) to the sensors and allows for data acquisition. Data is collected from the sensor as quasi-static loading is applied to the structural system.

For this proof-of-concept test, the sensing skins are placed on the web of a steel beam connected to a traditional welded beam-column connection (Fig. 2a). The beam-column assembly supports a concrete deck that is constructed to act in composite action with the beam. The beam-column assembly is loaded laterally at the top of the column with a hydraulic jack. The specimen is also instrumented with conventional strain gages for comparison purposes.
4. RESULTS

4.1 Pattern Fabrication

A macroscopic view of the patterned sensing elements is presented in Fig. 1c. To see more clearly the quality of the patterned films, the films are imaged under a traditional optical microscope; microscope images (20 times magnification) of the edges of the nanocomposite films can be seen in Fig. 3. All sensor geometries attempted were patterned successfully using standard optical lithography processes. There was no noticeable deterioration of pattern quality with decreasing feature size, indicating that the lower limit on feature sizes was near that of conventional lithography materials (i.e., approximately 2 um). There was difficulty at times in achieving nanocomposite film tearing during lift-off resulting in an incomplete lift-off process but when lift-off is achieved, film geometries were defect free.

4.2 Mechanical-Electrical Response of Small-Scale Sensing Skins

The resistance of each instrumented small-scale sensing skin was observed for five minutes prior to cyclic loading of the PVC bar. This observation displayed a rapid exponential signal decay that is commonly observed in such sensing skins [8]. The results for each sensor under cyclic loading can be seen in Fig. 4. Here it is readily apparent that each sensor was effective in tracking the strain of the specimen (note that Fig. 4f is the measured strain using the metal foil gage). It can further be observed that all sensors have similar signals with limited noise and fairly uniform sensitivities. The initial bulk resistance, sheet resistance, and gage factors of the five sensors of varying thickness can be seen in Fig. 4. The sensing elements of larger widths are more conductive as is expected for these nanocomposite films (Fig. 5a). The sheet resistance of all fabricated elements was very similar with the exception of the 10 micron thick sensing element (Fig. 5b). The results also indicate a fairly uniform gage factor across all sensor geometries despite the significant variance in sensor thicknesses (Fig. 5c).

Figure 3. Images of patterned sensors magnified at 20x showing some limited roughness along patterned film edges.

Figure 2. Sensing skin placement and instrumentation on a steel beam-column structural specimen: (a) location of the sensing skin on the structural system; (b) close-up view of two instrumented sensing skins on the web of the steel beam.
Figure 4. Change in resistance under cyclic loading sensing elements of thickness: (a) 10 microns; (b) 250 microns; (c) 500 microns; (d) 1000 microns; (e) 1500 microns; (f) measured specimen strain using a tradition strain gage.

Figure 5. Electrical properties of sensing elements: (a) Initial resistance; (b) sheet resistance; (c) gage factor.
4.3 Application Proof-of-Concept

A large-scale sensing skin (Fig. 6) is fabricated to observe strain in the web of a beam responding in flexure. The sensor was instrumented on a steel beam within a beam-column structural system with a composite slab as shown below in Fig. 2a. A plot observing resistance trends in the eight sensing elements on the beam during system loading can be seen in Fig. 7. These plots correspond to one cycle of lateral inter-story drift (1.5%) response. Elements 1, 2, 5, 6, 7 and 8 track the response of the beam well when compared to the waveform collected by traditional 120 Ω strain gages installed at the top, middle, and bottom of the beam on the opposite side of the web (Fig. 8). While the response is tracked well, there is some variation of the amplitude of the measured voltage signal suggesting variation in the gage factors of the films themselves. For example, elements 5 through 8 are clearly below the beam neutral axis and hence, these elements should have amplitudes increasing with depth since strain increases with depth. Discrepancy in the amplitudes may be attributed to variations in the films over their long lengths or due to local ripples in the polyimide skin when epoxy bonded to the beams. Elements 3 and 4 provided erroneous readings due to what is suspected to be faulty electrode connections. Element 3 trends properly compared to Element 1 but at 240 seconds the film response jumps; the film also outputs an unexplained spike at 300 seconds. Regardless, the results suggest the large-scale sensing skins are viable sensing platforms, but also indicate these platforms require additional investigation to improve their performance.

5. CONCLUSION

Nanocomposite sensing skins are patterned and tested for mechanical-electrical response. The sensor was fabricated on a flexible polyimide substrate using conventional optical lithography tools then epoxied to a PVC bar for testing. All geometries attempted were successfully patterned suggesting a limiting feature size near that of conventional lithography materials (2 um). Five small-scale sensing skin elements of varying width displayed similar sheet resistances and gage factors when instrumented and tested in uniaxial tension. The uniformity of gage factor with varying geometry was unexpected when considering the wide range of sensor geometries tested. These materials and processes were then extended to develop a component-specific sensor for monitoring the distribution of strain in a beam web. This sensor was instrumented on a beam-column structural system with a composite slab where it displayed the potential to measure the strain profile in a steel beam web. While the film elements trended well, additional work is needed to achieve a uniform gage factor of the elements. It is suspected the gage factor is varying due to the means of application of the film and not due to the film itself. For example, ripples in the film during epoxy bonding may enhance the gage factor is uncontrollable and non-repeatable ways. To remedy this potential issue, a thicker encasing layer is currently under investigation. None the less, the malleability of the patterning and fabrication processes utilized provides the platform for the development of component-specific structural sensors for components of a vast range of structural systems.
Figure 7. Voltage during the loading of the beam-column structural system: signal for thin film sensing elements located from the top (element 1) to the bottom (element 8) on the web.
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REFERENCES


