

Inductively Coupled Multifunctional Carbon Nanotube-Based Nanocomposite Sensors

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Abstract—In this study, a high-performance inductively coupled thin film multifunctional sensor for structural monitoring applications is presented. A versatile layer-by-layer self-assembly sensor fabrication technique is employed to encode different sensing mechanisms (*e.g.* strain and corrosion/pH) within one homogeneous thin film structure. Judicious selection of various polyelectrolyte species, along with the incorporation of carbon nanotubes, permits multifunctional sensing. Upon deposition of these thin films on miniature planar coil antennas to form a resonant circuit, a passive wireless sensor is realized. Unlike traditional cabled or wireless sensing systems, the proposed passive sensor does not require a constant power source, thereby ideally suited for embedment within structural components.

Keywords—carbon nanotube composites, layer-by-layer, pH, RFID, strain, structural monitoring, thin film, wireless

I. INTRODUCTION

Over the past several decades, deteriorating civil infrastructures around the world have warranted the design and implementation of autonomous sensing systems for structural health monitoring (SHM). While tethered monitoring systems provide reliable sensor data and communication between sensors and a centralized data server, its high installation and maintenance costs have prevented their widespread adoption. In addition, only a few sensing channels can be installed per structure, thereby only permitting global-based damage detection (*i.e.* it can only identify critical structural damage).

As a result, the field of SHM has shifted from a global to a local and densely-distributed damage identification paradigm to identify and characterize progressive structural damage. The introduction of wireless sensors has dramatically reduced costs while simultaneously allowing dense sensor instrumentation layouts and providing reliable data communications between the sensing nodes and server [1]. Unfortunately, wireless sensors depend on a constant power supply such as batteries; engineers are required to replace these batteries every few years. Furthermore, for both cabled and wireless sensors, their form factor is usually too large to be embedded within structural components. Since damage (*e.g.* composite delamination, corrosion, cracking, among others) typically occurs beneath the structural surface, embedded sensors are crucial for accurate damage identification.

Unlike traditional wireless sensors, radio frequency identification (RFID)-based sensors offer the best of both worlds (*i.e.* it has a small form factor and does not require batteries) [2]. Rather, a portable reader generating a variable magnetic field inductively couples with the passive sensor to achieve wireless communications. It should be noted that the concept of RFID-based sensors is not new. In the past, many researchers have developed RFID sensor prototypes for measuring strain [3-5] and corrosion of steel in reinforced concrete [6]. For instance, [3, 4] have designed a capacitive peak strain sensor based on two concentric conductive pipes separated by a dielectric. Upon induced strains, the relative displacement between the two pipes induces a capacitance change which changes the resonant frequency of the RFID sensor. Similarly in [6], an exposed steel wire embedded in concrete acts as a switch; when corrosion attacks the exposed wire and eventually breaks the wire, a dramatic shift in resonant frequency is observed. The proposed sensor can be used to detect different threshold levels of corrosion by simply adjusting the diameter of the exposed steel wire.

In this study, a multifunctional carbon nanotube-polyelectrolyte-based nanocomposite passive wireless sensor for SHM applications is presented [7]. Using a layer-by-layer (LbL) thin film fabrication technique, different sensing mechanisms (*e.g.* strain and corrosion/pH) are encoded within a thin film by simply controlling the polyelectrolyte species deposited [7, 8]. Upon deposition of these thin films onto planar coil antennas (from Texas Instruments, Inc.), passive RFID sensors are formed [2]. Given an applied stimulus, the multifunctional RFID sensor responds by a change in its inherent properties, which is then wirelessly detected, via a portable reader, as a shift in its resonant frequency or system bandwidth. This paper begins with a brief discussion on RFID theory, followed by a description of the LbL sensor fabrication methodology. Preliminary wireless strain and pH sensing results are presented, along with discussions, and future work.

II. RADIO FREQUENCY IDENTIFICATION BACKGROUND

Any inductively coupled or radio frequency identification system consists of two main parts: a reader (which is simply a coil antenna), coupled with an alternating current (AC) source, and a passive sensor tag or transponder. When an AC current at a frequency (f) passes through the reader coil antenna, a

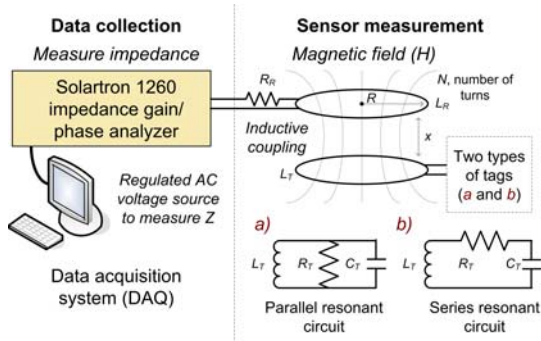


Figure 1. A schematic illustrating RFID wireless interrogation of a (a) parallel or (b) series resonant tag and data acquisition setup.

corresponding magnetic field is generated in its vicinity. A sensor tag, also equipped with a coil antenna, when placed in close proximity of the reader, induces a voltage drop and current due to inductive coupling (Faraday's Law) [2]. Typically, the induced voltage and current is used to power onboard tag electronics and to wirelessly transmit data back to the reader. However, no tag electronics are used in this study; all sensing data are detected by the reader via changes in the inductive coupling properties of the reader-tag system.

A. The Reader

In its simplest form, the RFID reader consists of a loop antenna (with inductance L_R and some inherent series resistance R_R) coupled with an AC sinusoidal source (where the subscript R denotes the reader). In this study, a Ti (Texas Instruments, Inc.) planar inductive coil is employed as the reader antenna; for the AC sinusoidal source, a Solartron 1260 impedance gain/phase analyzer (an automated frequency response analyzer) is utilized. As the Solartron 1260 impedance gain/phase analyzer passes a regulated AC current of a particular frequency through the antenna, a magnetic field (H) is generated near the vicinity of the coil [2]. As mentioned earlier, the magnetic field can be used to power any remote RFID tag. Moreover, as the impedance analyzer sweeps through a range of frequencies, it also measures the impedance of the reader coil along with any induced changes as communicated from the passive sensor tag.

B. The Tag or Transponder

In general, an RFID sensor tag with no onboard electronics consists of only three discrete circuit elements, namely a coil antenna with inductance L_T and its inherent series resistance R_S , a resistor (R_T), and a tuning capacitor (C_T) (where the subscript T denotes the tag), configured in a series or parallel circuit fashion (Fig. 1). In either case, the RFID tag can be characterized by its resonant frequency (f_n) and its system bandwidth (B) [2]. The tag resonant frequency is independent of the circuit configuration as shown in (1) [9]:

$$f_n = 1/2\pi\sqrt{L_T C_T} \quad (1)$$

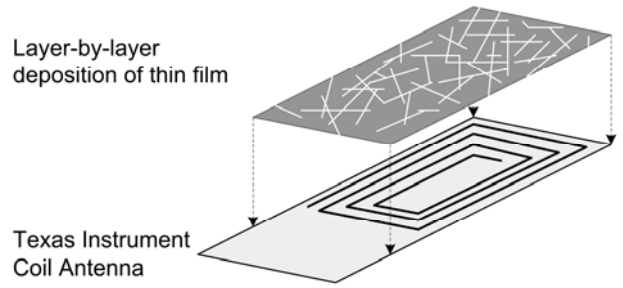


Figure 2. A schematic illustrating the layer-by-layer deposition of multifunctional thin film sensors onto planar coil antenna substrates.

On the other hand, the tag bandwidth varies between a series and parallel resonant circuit as given in (2) [9]:

$$B_{series} = R_T / 2\pi L_T \text{ and } B_{parallel} = 1/2\pi R_T C_T \quad (2)$$

C. Coupled Reader-Tag System

As mentioned earlier, the impedance analyzer measures the complex impedance response of the reader coil. When no sensor tag is in the vicinity of the reader, the measured impedance (over a given frequency range) is expressed as:

$$Z = R_R + j\omega L_R \quad (3)$$

where ω is the natural cyclic frequency ($\omega = 2\pi f$) [2]. On the other hand, when a transponder enters the detectable range of the reader (as governed by the generated reader magnetic field), the sensor response is superimposed onto the measured impedance (Fig. 1). For a parallel resonant tag circuitry, the overall reader-tag impedance response is expressed as (4),

$$Z = R_R + j\omega L_R + \frac{k^2 \omega^2 L_R L_T}{1/(R_S + j\omega L_T) + 1/R_T + j\omega C_T} \quad (4)$$

where k (between 0 and 1), the coupling factor, qualitatively describes the mutual inductance between the reader and tag [2]. The first two terms in (4) are contributions from the reader coil antenna and its inherent series resistance, while the last term is due to inductive coupling between the reader and tag. A detailed derivation of the reader impedance can be found in [2].

III. LAYER-BY-LAYER SENSOR FABRICATION

A layer-by-layer self-assembly thin film fabrication technique is employed to fabricate multifunctional carbon nanotube-based nanocomposite sensors [7, 8, 10]. In short, a charged substrate such as a planar coil antenna printed on poly(ethylene terephthalate) (PET) is sequentially immersed in alternating-charged solutions to deposit polyelectrolyte and nanoparticle species one monolayer at a time, where adsorption of each additional monolayer is based on electrostatic and van

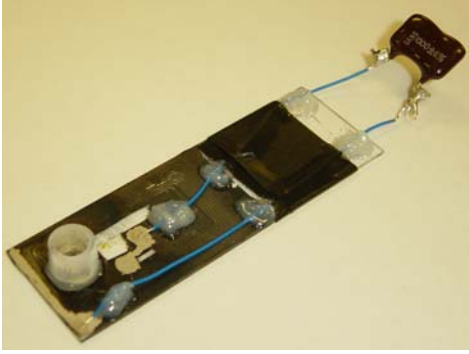


Figure 3. A (SWNT-PSS/PANI)₅₀ thin film is deposited directly onto a Ti planar coil antenna to form a parallel resonant sensor tag ($f_n = 3.28$ MHz using a 1,000 pF tuning capacitor). A plastic well is mounted on top of the thin film to hold different pH buffer solutions.

der Waals force interactions [10] (Fig. 2). Here, single-walled carbon nanotubes (SWNT) are ultrasonically dispersed in 1.0 wt. % poly(sodium 4-styrene sulfonate) (PSS) to form the polyanionic solution. In addition, 1.0 wt. % poly(vinyl alcohol) (PVA) or poly(aniline) (PANI) emeraldine base solutions are used as the polycationic LbL counterpart. The LbL process begins by dipping the substrate in the PVA or PANI solution for 5 min which is then followed by 3 min of rinsing in deionized water and 10 min of drying. Then, the substrate, along with the adsorbed monolayer, is then immersed in the SWNT-PSS solution for 5 min. Upon rinsing and drying, one bilayer is formed on the substrate, where repetition of the aforementioned process yields multilayer thin films. Given any two LbL constituents A and B , $(A/B)_n$ denotes an n -bilayer thin film (typically $n = 50$ or 100).

Unlike other thin film fabrication techniques (*e.g.* molding, spin coating, among others), the LbL methodology permits the incorporation of a variety of polymer and nanoparticle species within a homogeneous yet multilayer thin film structure [7, 8, 10]. In addition, specific electromechanical and electrochemical sensing transduction mechanisms can be embedded within the film by controlling the type of polyelectrolyte species adsorbed [7, 8]. In this study, three different carbon nanotube-based thin film sensors are presented, namely 1) pH-sensitive thin films based on PANI, 2) strain-sensitive films based on PVA, and 3) conductive films based on gold nanoparticles dispersed in PVA for inductive coupling and wireless communications.

IV. EXPERIMENTAL RESULTS AND DISCUSSION

A. Wireless pH Sensor Validation

The thin film (SWNT-PSS/PANI) _{n} pH wireless sensor is realized by depositing the multilayer film directly onto Texas Instruments, Inc. (Ti) coil antennas (printed on PET) during the LbL process. To ensure that the thin film and coil antenna are electrically isolated, a thin coating of Ted Pella Aerodag G (graphite aerosol) is sprayed onto the surface of the coil prior to LbL fabrication. Realization of a parallel resonant tag circuitry is accomplished by drying colloidal silver paste (Ted Pella)

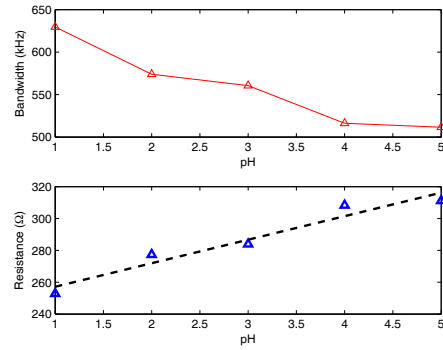


Figure 4. (a) Upon wireless sensor interrogation via the RFID reader, the system bandwidth is extracted and shown as different pH buffer solutions are pipetted into the plastic well. (b) The film resistance is back-calculated using (2) to validate its near-linear increase in resistance with increasing pH.

over two ends of the (SWNT-PSS/PANI)₅₀ antenna and connecting it to the inductor in parallel (Fig. 3). In addition, a 1,000 pF tuning capacitor is included in the sensor design to set the sensor resonant frequency (f_n) at approximately 3.28 MHz.

Since the pH sensor is required to be exposed to a variety of pH solutions, a small plastic well is mounted on the film surface using high-vacuum grease (Dow Corning) as shown in Fig. 3. The small well is capable of containing approximately 1 mL of liquid while holding its contents without leakage onto other areas of the sensor. Validation of the pH sensor begins by pipetting various pH buffer solutions (pH 1 to 5) one at a time into the plastic well. The solutions are kept in the well for 5 min to allow the resistance of the (SWNT-PSS/PANI)₁₀₀ thin film to stabilize before the Solartron 1260 impedance gain/phase analyzer and its coil antenna is employed to wirelessly interrogate the remote sensor.

From Fig. 4(a), it can be seen that the passive wireless pH sensor's bandwidth decreases nonlinearly with increasing pH. This inherent nonlinearity is expected, since, from (2), it is apparent that RFID system bandwidth is inversely proportional to the (SWNT-PSS/PANI)₅₀ thin film resistance. However, based on a previous study as conducted by [7], it has already been shown that thin film resistance should increase linearly in tandem with increasing pH. Upon back-calculating thin film resistance using (2), it can be observed from Fig 4(b) that film resistance indeed varies near-linearly with pH.

B. Wireless Strain Sensor Validation

As mentioned in Section III and [7, 8], piezoresistive sensitivity is encoded in SWNT-based LbL thin films using poly(vinyl alcohol) as the LbL counterpart. The resulting (SWNT-PSS/PVA) _{n} thin films exhibit an increase in resistance in tandem with increasing applied strain (to $\pm 10,000 \mu\text{m}\cdot\text{m}^{-1}$) (results not shown here) [7, 8]. Here, (SWNT-PSS/PVA)₅₀ thin films are deposited onto Ti coil antennas as shown in Fig. 2. To realize a parallel resonant wireless sensor tag, the thin film is electrically connected in parallel to the antenna similar with the thin film pH sensor. Here, a 1,500 pF capacitor is employed as the tuning capacitor.

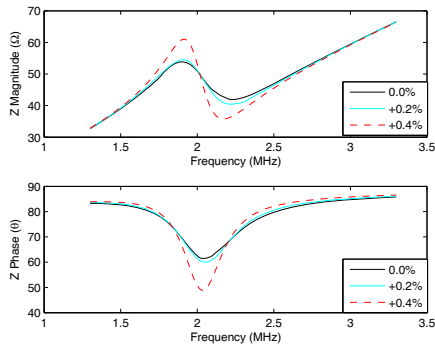


Figure 5. The corresponding impedance (a) magnitude and (b) phase response as measured by the RFID reader when (SWNT-PSS/PVA)₅₀ thin films are strained (only showing tensile strain responses for clarity).

In order to effectively induce strain on the coil antenna and the (SWNT-PSS/PVA)_n thin film, the passive tag is epoxy (CN-Y post-yield epoxy) mounted to a PVC coupon for strain testing. Upon sufficient drying after six hours, the PVC coupon and sensor tag is mounted on an MTS-810 load frame. The load frame is then commanded to execute an increasing monotonic strain ($\pm 4,000 \mu\text{m}\cdot\text{m}^{-1}$ at $2,000 \mu\text{m}\cdot\text{m}^{-1}$ intervals) to the specimens; at each interval, the load frame holds its displacement and load to allow time for the RFID reader to interrogate the strain sensor tag.

From Fig. 5, it can be seen that the impedance magnitude and phase response, as determined wirelessly by the RFID reader, varies depending on the level of induced strains. The piezoresistive thin film in the parallel resonant circuit causes system bandwidth changes due to applied strain, as governed by (2) and shown in Fig. 6(a). Upon back-calculating the thin film resistance (Fig. 6(b)), once again the thin film resistance increases linearly in tandem with applied strain.

C. Carbon Nanotube-Gold Nanocomposite Coil Antenna

In an effort to develop a complete SWNT-based nanocomposite passive wireless sensor, the current work focuses on enhancing thin film conductivity and patterning them into coil antennas for wireless communications. It has been shown experimentally that thin film conductivity dramatically increases upon the incorporation of gold nanoparticles within the SWNT-based LbL structure. Preliminary impedance measurements on the complete SWNT thin film sensor suggest inherent series resonant behavior. Work is underway to further enhance film conductivity to achieve passive wireless communication capabilities.

V. CONCLUSIONS

In conclusion, a carbon nanotube-based thin film passive wireless strain and pH sensor is presented. By controlling the type of polyelectrolyte species deposited during LbL assembly, different sensing transduction mechanisms can be encoded within the film. Upon deposition of these thin films onto Ti coil antennas and connecting them in parallel to a tuning capacitor, passive wireless communication is realized. Experimental

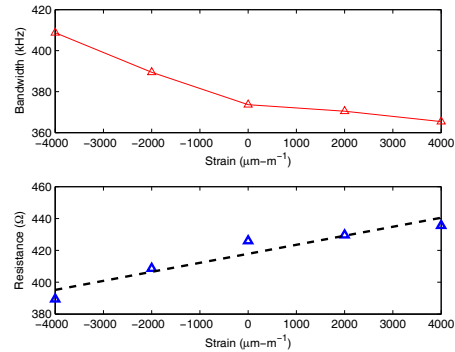


Figure 6. (a) System bandwidth of the strain sensor tag decreases with increasing applied strain. (b) The film resistance is back-calculated using (2) to validate its near-linear increase in resistance with increasing strain.

results from both strain sensing and pH detection suggest nonlinear changes in RFID system bandwidth as a function of the applied external stimuli; however, upon back-calculation of thin film resistance, both films exhibit near-linear changes in resistance in tandem with strain or pH. A complete carbon nanotube-based thin film sensing system is currently underway.

ACKNOWLEDGMENT

This research is supported by the National Science Foundation (Grant Number CMS-0528867).

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