Development of Fully Printed and Flexible Strain, Pressure and Electrochemical Sensors

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DEVELOPMENT OF FULLY PRINTED AND FLEXIBLE STRAIN, PRESSURE AND ELECTROCHEMICAL SENSORS

by

Dinesh Maddipatla

A thesis submitted to the Graduate College in partial fulfillment of the requirements for the degree of Master of Science in Engineering Electrical and Computer Engineering Western Michigan University December 2016

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In recent years, considerable research has been put into the development of printed electronics (PE) as well as the best ways to effectively and efficiently print electronic devices on flexible substrates. This thesis focuses majorly on the design and fabrication of novel flexible sensors using conventional printing processes.

Initially, a flexible printed strain gauge was fabricated successfully on a flexible paper substrate using flexography printing process. Silver (Ag) ink was printed on the paper substrate as metallization layer. The performance of the printed device was investigated by subjecting the strain gauge to a 3-point bend test, with a displacement of 1 mm, 2 mm and 3 mm at 3 Hz operating frequency. The electro-mechanical response of the fabricated strain gauge, as a function of electrical resistance revealed that the sensor has a longer functional life for smaller displacements which is especially advantageous for structural asset monitoring applications.

Then, a fully flexible carbon nanotube (CNT) capacitive pressure sensor was developed for the detection of varying applied pressures. The sensor was successfully fabricated using the screen printing technique. Polydimethylsiloxane (PDMS) was used as a dielectric layer and it was prepared using a PDMS pre-polymer and a curing agent mixed in a 10:1 ratio. The electrode design was directly screen printed using conductive CNT ink onto the PDMS dielectric layer. The capacitive response of the printed sensor for varying applied pressures demonstrated the feasibility
of employing CNT based electrodes and PDMS dielectric layer for the development of an efficient, flexible and cost effective pressure sensors in sports, military, automotive and biomedical applications.

Finally, a novel printed impedance based electrochemical sensor has been successfully developed on a paper substrate for the detection of various bio/chemicals. This flexible analytical device was fabricated by screen printing a two electrode sensor configuration on a wax-printed chromatography paper substrate. Ag ink was used for screen printing the working and circular electrodes. The electrical impedance spectroscopy (EIS) based response of the fabricated electrochemical sensor revealed the capability of the sensor to distinguish among varying levels (pico, nano, micro and milli) of potassium chloride (KCl) and glucose (C$_6$H$_{12}$O$_6$) concentrations as well as the potential of employing paper substrate based electrochemical sensors for bio/chemical sensing applications.
ACKNOWLEDGMENTS

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CHAPTER 1

INTRODUCTION

1.1 Background

In recent years, considerable research has been put into the development of printed electronics (PE) as well as the best ways to effectively and efficiently print electronic devices on flexible substrates [1-5]. PE uses traditional additive processes such as screen [6, 7], inkjet [8, 9], flexography [10, 11], aerosol [12, 13] and gravure printing [14, 15] for the fabrication of electronic sensing devices. These additive processes offer numerous advantages such as roll-to-roll (R2R) fabrication, low operating temperatures, mechanical flexibility, light weight, low cost, thinness, high throughput, less complex manufacturing steps and reducing wastage of material during fabrication [16-20]. Several electronic devices such as organic thin film transistors [21], RFID tags [22], electrochemical sensors [23], solar cells [24], and strain sensors [25] have been fabricated using these printing processes in R&D labs. Some printed electronic sensors such as temperature sensors, photodetectors or gas sensors with flexible capabilities are transitioning from R&D labs to industrial scale mass production. According to IDTechEX “Printed and Flexible Sensors 2015-2025: Technologies, Players, Forecasts” report by Dr. Guillaume Chansin, the worth of fully printed sensors market will be more than 8 billion dollars by 2025 (26).

From many years among mechanical sensors, strain gauges have been receiving growing interest for various applications such as shape monitoring, strain, force and pressure measurements in biomedical, robotic, automotive fields. Strain gauges are also used to record the age of aircraft through structural health monitoring [27-31]. Strain gauges are typically employed for monitoring the civil infrastructural integrity, which is important for high standard safety requirement in the mechanical and electrical engineering industries [32]. Hence strain gauges that are flexible, have
compact structures and quick signal response, with high strain tolerance and reliability for the precise results on the state of the device, are of profound interest. Strain gauges that have been fabricated using conventional methods are not flexible, rigid and are separated from the substance or medium that is being monitored [33, 34]. These devices tend to get damaged or broken which would result in incorrect or unreliable results. However, to overcome these limitations with conventional strain sensors, the printed strain gauges on flexible platforms are considered promising solutions [35]. These printable and flexible based strain gauges offer high sensitivity, mechanical flexibility, cost-efficient, smaller dimensions, and high strain tolerance without breaking [36]. This has led to the research of using printing technology on flexible platforms for the fabrication of strain gauges.

In recent years, a major focus is vested towards the development of highly sensitive and flexible pressure sensors in the field of automotive, military, sports, robotic and biomedical applications. With the rapid evolution of the pressure sensing field, a wide range of devices has been developed to help monitor pressure [37-39]. Typically pressure sensors that have been manufactured using conventional methods mostly lack the flexibility and conformability required for sensing applications [40, 41]. Moreover, most of these sensors must be developed on a rigid substrate and utilize either hanging structures or cavity based designs [42]. Additionally, these sensors are expensive to produce and don’t offer the flexibility, and stability required for many fields. Therefore, to overcome this problem, the author was interested in integrating and exploring the advantages of printing technology with the fabrication of pressure sensor on a flexible substrate using low-cost conductive carbon nanotube (CNT) ink.

A major focus is vested towards the development of cost effective and simple diagnostic tools [43]. Modern day health care systems need accurate, simple, compact and easy to use medical
diagnostic devices. Typically, in order to analyze a sample, laboratories often require multiple instruments, materials, and involve many complex steps and thus the analysis is a time-consuming process \[43, 44\]. Development of miniaturized laboratories is an effective solution that reduces the cost and time required to do these analyses \[45\]. Thus, the complex steps involved in the conventional laboratories can be converted to a single analytical platform \[46\]. Generally, this type of miniaturized analysis system is referred as a “lab-on-a-chip” (LOC) device. However, the fabrication of LOCs involves many integrated operations and is often constructed using glass, quartz or polymer-based platforms with many construction steps \[45\]. This has led to the research of exploring alternate promising techniques such as printing technology for the fabrication of LOCs on different cost-effective platforms such as paper.

1.2 Author’s Contributions


1.3 Organization of the Thesis

The author presents a brief overview of sensors along with their classification and characteristics in chapter 2. A brief introduction to printed electronics and various printing methods are discussed in chapter 3. The author discusses the three research projects in detail that have resulted from his master’s education in the next 3 chapters. The first project related to the
development of novel flexographic printed strain gauge on the paper platform is discussed in chapter 4. It includes the details about fabrication, various configurations, and test results. The second project is related to the development of CNT-based capacitive pressure sensor and the details about the design, fabrication steps, and test results are discussed in chapter 5. Chapter 6 presents the third project related to the development of a printed impedance based electrochemical sensor on a paper substrate and it includes the design details, fabrication steps, characterizations and test results. Finally, the conclusion for these projects and also necessary suggestions and improvements for future work is presented in chapter 7.

1.4 References


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CHAPTER II
INTRODUCTION TO SENSORS

2.1. Introduction

A sensor can be defined as an electrical component that senses or measures a physical parameter such as strain, pressure, temperature etc., and converts it to a suitable stimulus or processable signal such as electrical, mechanical, optical etc. The concept of sensing is very old as life itself. The revolution of sensor technology mostly started from the 19th century with the advancements in the fields of material sciences and engineering for numerous applications [1 - 7]. For example, the gyroscope was invented by jean Bernard Leon Foucault in 1852 [8]. Alexander Graham bell invented the first crude metal detector in 1881 [9] and Warren S. Johnson invented first electric thermostat in 1883 [10]. However, with the development of semiconductor technology and silicon technology in the 20th century, a new set of miniaturized sensing devices came into existence with advanced features, more accuracy, less response time, increased reliability and portability [11-21].

The usage of sensors has become a part of almost every activity in present-day human life. As examples, to track and monitor activity levels of a person such as movements, workouts and location throughout the day, a smartphone has to be equipped with a range of sensors such as gyro, accelerometer, proximity, GPS, compass, Wi-Fi, Bluetooth etc. According to Rajaram, the global market for sensors generated $101.9 billion in revenue in 2015 [22]. This market is estimated to generate a revenue of $113.2 billion by 2016 and $190.6 billion b 2021, with a compound annual growth rate (CAGR) of 11% over the six-year period 2016 – 2021. According to Madakasira, over the past decade (2006 – 2016) under the internet of Things (IoT), almost $3.4 billion was invested
towards innovative sensor processing related technologies, which is a significantly large amount compared to packaging, processing and energy harvesting technologies as shown in Fig. 2.1 [23]. Of these figures, it is clearly evident that both the need and usage of sensors in day to day modern life is increasing along with the time. Various automobile, mining, manufacturing, and utilities based industries have been automated with the integration of sensor technologies to achieve better product quality, increased throughput, and cost effective production.

2.2. Sensor Systems

![Figure 2.1. Funding for the sensor module technologies over the past decade (2006 – 2016).](image)

A simple sensor system can be described as a device that can generate an electrical or optical signal in relation to the physical, chemical or biological quantity measured. In other simple ways, the sensor system can be defined as an energy converter. Regardless of what we try to measure, we always deal with energy transfer between the sensor and object of measurement. In
present days, sensors can be divided into natural sensors and man-made sensors [24, 26]. The best examples for natural sensing systems are living organisms. They consist of various real-time controlled sensing systems with different sensors. The major sensors in living organisms are related to vision, smell, taste, touch, and sound [25]. The natural sensors in living organisms generally respond and transmit the signals with the electrochemical character through the ion transport. For example, the nerve fibers in the optical nerve which are surrounded by the fluid reservoir present in the central nervous system are responsible for the sensing and transmission of visual information such as color and brightness perception, contrast etc., from the retina to the brain. These nerve fibers sense and respond to the signal (visual information) in the electrochemical form and transmit this signal through the transfer of ions to the brain. In man-made sensors, the sensing system responds, processes and transmits the electrical signal through the transfer/displacement of electrons but not the ions. Usually, the output signal of manmade sensors will be electrical quantities such as charge, current, and voltage. Some of the examples for manmade sensors are accelerometer, strain gauge, infrared detector, optical sensor etc., [27]. A basic manmade smart/intelligent sensor system is shown in Fig. 2.2. The general architecture of a smart/intelligent sensor system will have sensor and transducer elements, interfacing modules with amplifiers and signal conditioners, signal conversion and processing components, communication interfaces, and programming devices [28, 29]. The primary sensing element of the smart/intelligent sensor system is the sensor and transducer element. It measures the measurand, which can be in any form such as temperature, pressure, flow etc. The electrical signal (obtained from the conversion of physical/chemical/biological parameters) from sensor and transducer element will be amplified if necessary. If the amplified electrical signal is difficult to read, it will be conditioned (using filtering, and or amplification) by the signal conditioner and converted to an easily readable
format for further processing. The conditioned analog signal is converted to digital values using analog to digital converters and processed, if necessary, by signal processors (using different techniques such as mathematical modeling, algorithms etc.) to be able to understand the digital numeric information in the signal by communication interface or computer and provide the corresponding measurement to the input measurand [30].

**Smart Sensor System**

![Smart Sensor System Diagram](image)

**Figure 2.2.** Smart/intelligent sensor system (man-made).
2.2.1 Types of Sensors

Sensors sense external stimuli through various detection modes. Depending upon the different detection modes, various types of sensors can be termed as thermal, mechanical, acoustic, optical, chemical and biological sensors.

2.2.1.1 Thermal Sensors

The measurement of changes in the kinetic energy/internal energy of a system i.e., molecules of a gas, solid, or liquid required by thermodynamics is called temperature. A thermal sensor is an instrument that specifically detects and quantifies temperature [24, 26]. Thermal sensors are mostly used to quantitatively determine the state of a substance or an object and the surrounding environment that is influencing the object’s state. The measured temperature of the thermal sensor is proportional to the input stimulus. The concept behind the working of most thermal sensors can be explained by using a fundamental or basic bimetallic thermostat. Bimetallic thermostats consist two metallic strips as shown in Fig. 2.3 (a). Both the metals have different coefficients of expansion and are attached together at a specific temperature using various techniques, such as bolting, riveting, and fastening. One side of the bimetallic strip is supported by a fixed end and the other end is left free. Let one metal strip be Metal A with a coefficient of expansion (α_A) and other metal strip be Metal B with a coefficient of expansion (α_B). The basic principle behind the bimetallic thermostat is that the different metals behave (expands or contracts) differently at varying temperatures. So, when there is a change in temperature, either one of metal A or metal B will expand or contract more than the other based on their coefficient of expansions α_A and α_B resulting in a bending or curvature change at the free end of the bimetallic strip in the thermostat as shown in Fig. 2.3 (b).
Figure 2.3. Schematic of bimetallic thermostat (a) Structure of bimetallic strip at normal condition and (b) Structure of bimetallic strip while temperature changes.

The radius of the curvature or bend is given by $\rho$,

$$\rho = \frac{2t}{3 (\alpha_A - \alpha_B)(T_2 - T_1)} \quad (1)$$

where, $t$ is defined as the overall thickness of the strip and $(T_2 - T_1)$ is the change in temperature.

When the temperature changes, the indicator or pointer attached to the bimetallic strip will move over the scale in proportion to the circular bending of the bimetallic strip. These types of thermal
sensors are used in various devices such as refrigerators, air conditioners, thermostat switches to monitor the temperature and in electrical heating devices as circuit breakers [31, 32].

### 2.2.1.2 Acoustic Sensors

Acoustic sensors typically detect the propagation of a mechanical, or an acoustic wave through or on the surface of a material. The amplitude and/or the velocity of the propagated wave changes with respect to the characteristics of its surrounding medium (propagation path of the wave) and these changes can be measured using phase and frequency characteristics of the acoustic sensor, which will be in correlation with the input quantity (such as pressure, humidity, magnetic fields, viscosity, temperature, biological matter, chemical vapors, mass) being measured [24, 26]. The concept of the acoustic sensor can be explained using a basic acoustic wave gas sensor (Fig. 2.4). The important components of the sensor are a piezoelectric substrate, interdigital transducers,
a gas sensitive layer/film deposited on the detector surface and a periphery circuit. The sensitive layer should be highly selective to a particular gas, even in the mixture of gasses. When the sensitive film absorbs the gas molecules from the environment, the velocity and/or the amplitude of the output wave will be attenuated. The details of the gas content can be determined with great accuracy using electronic systems such as oscillators by measuring changes in the phase and frequency characteristics of the amplified wave. Acoustic sensors have many applications in various fields and can be used as a pressure sensor, humidity sensor, temperature sensor, RFID sensor and much more [33-36].

2.2.1.3 Mechanical Sensors

Mechanical sensors detect the mechanical deformation due to mechanical forces in an object or a material and convert it to an electrical signal. Pressure, strain, stress, acceleration, position, and angle are some of the important properties/parameters that can be measured using mechanical sensors [24, 26]. The concept behind the working of most mechanical sensors can be explained by through a strain gauge. When an object or a material is subjected to an external force, there will be mechanical deformation in the object’s shape due to the strain experienced by it. A strain gauge is a device that measures the strain created on an object due to an external force. The strain gauge contains a conductive material (metal) in a zigzag pattern of parallel lines with contact pads (terminals) at both the ends to facilitate electrical connection as shown in Fig. 2.5. When an object is physically deformed (becoming wider or narrower) within its elasticity limits, the zigzag pattern of parallel lines in the strain gauge attached to the object will also get enlarged or compressed resulting in the change of electrical resistance in proportion to the strain experienced by the object. Strain gauges are mainly used to measure the movement of buildings or any other structures, fatigue life of various components in aircraft and automobiles [37, 38].
2.2.1.4 Optical Sensors

Optical sensors typically measure either the intensity change or a phase change or a reflectance change of a light beam and convert it into an electrical signal in proportion to the input stimulus [24, 26]. Optical sensors mainly work on techniques such as light scattering, spectral transmission changes, radioactive loses or micro bending. Optical sensors have many advantages such as electrical passiveness, multiplexing capabilities, wide dynamic range, and high sensitivity. The concept of optical sensors can be explained using the through-beam photoelectric sensor (Fig. 2.6). This sensing system has two components i.e., emitter and receiver in separate housings. The emitter has a light beam source (infrared or UV or visible) and it transmits the light aiming directly to the receiver and is a one-way noncontact transmission. If any object or an obstacle comes in the way of transmission, the receiver may pick up a weak transmitted light beam or may not detect anything. This changes the output state of the receiver. The through-beam sensor system is mainly used in production monitoring, package detection, motion detection, skewed lid detection, tool verification, and parts counting [39-43].
Figure 2.6. Schematic of through-beam photoelectric sensor (Optical sensor).

2.2.1.5 Chemical and Biological Sensors

A chemical or biosensor sensor is a device that contains a transducer and a chemically or biologically coated sensitive film and produces typically a voltage or current signal when reacted with the analyte [24, 26]. The generated signal provides the information related to the identification and quantification of a chemical or a biological material in a given sample and also this signal would be in proportion to the concentration of the analyte. The concept of the chemical and biological sensor can be easily explained through a commercially available interdigitated biochemical sensor. The schematic of a typical biochemical sensor is shown in Fig. 2.7. It contains a chemically or biological sensitive film coated on the interdigitated electrode design (IDE). If an analyte is drop cast onto the sensing layer, a chemical or biological reaction occurs and it causes a change in impedance or capacitance across the IDEs in proportion to the concentrations of analyte. Chemical and biological sensors have many applications in biomedical, military, food, beverage and environmental industries [44].
2.2.2 Sensor Characteristics

The important measure to be considered while choosing a sensor is its performance. The performance of any sensor is defined by its characteristics. Some of the important sensor characteristics are sensitivity, reproducibility, resolution, non-linearity, accuracy, calibration, power consumption, cost, range.

2.2.2.1 Sensitivity

Sensitivity describes the relation between the input signal (physical) and the output signal (electrical) [45]. It provides the ratio of smallest change in the output signal to the smallest change in the input signal (Fig. 2.8). A sensor can have high sensitivity if the smallest change in the input can produce the biggest change in the output. Mathematically it can be written as:

\[
S = \frac{Y_2 - Y_1}{X_2 - X_1}
\]
2.2.2.2 Non-Linearity

The maximum deviation of the sensor response from its linear transfer function over the specified dynamic range is called nonlinearity [45]. It is typically measured by comparing the actual transfer function with a straight line that lies between 2 parallel lines that can encompass the whole transfer function, over the specified dynamic range.

2.2.2.3 Accuracy

The accuracy of the sensor points to its inaccuracy and it can be quantified by measuring the deviation of the output result from the expected or ideal value [45]. For example, assume that a resistive strain sensor has to generate 100 Ω per 20 mm elongation in the ideal case with a sensitivity of 5 Ω/mm. Let’s say that it the output obtained shows a resistance change of 80 Ω per 20 mm elongation with sensitivity 4 Ω/mm. This shows that the output of the sensor is inaccurate by 1 Ω/mm.
2.2.2.4 Resolution

The minimum value of measurand that is detectable by a sensor is called as resolution [45]. For example, if the least displacement that can be measured by a proximity sensor is 0.1 mm, then the resolution of this particular sensor is 0.1 mm. The resolution of a sensor is typically affected by the noise in the system and this effect can be reduced by employing narrow bandwidth filters, which increase the signal to noise ratio.

2.2.2.5 Reproducibility

Reproducibility is defined as the capability of the sensor to generate the same or similar results for a particular input over a period of time [45]. Reproducibility of the sensor determines its life span. Practically, most of the sensor devices will not produce similar results over a long period of time and their performance degrades because of many reasons such as accumulation of impurities, degradation or oxidation of sensing metals, aging of materials used in the sensor.

2.2.2.6 Noise

Noise is defined as an undesirable fluctuation that is generated along with the desired output signal [45]. It is typically distributed all over the frequency spectrum and limits the performance of the sensor system. The main causes of noises can be the internal components, wiring, electromagnetic signals (generated by transceiver or receiver circuits), mechanical vibrations, power supplies and any changes in temperature. Some types of noises that can be seen are white noise, shot noise, electronic noise, generation-recombination noise and pink noise.

2.2.2.7 Calibration

Calibration is defined as a process where the output results of the sensing system are compared with the known measurand to make sure that the system is working well [45]. Any sensor system has to be calibrated periodically to obtain the best output (most accurate) results.
2.2.2.8 Saturation

Every sensing system has its own operating boundaries or limits in which the output results will have a linear relationship with the input measurand (Fig. 2.9) [45]. Sensors become nonresponsive beyond its operating limits for whatever the amplitude of input signal is applied (above some threshold input value). This is called as saturation region and there will be no output of the sensor in this region.

![Diagram showing linear and saturation region](image)

**Figure 2.9. Sensor output showing linear and saturation region.**

2.3. Summary

This chapter provided a comprehensive overview of sensors, starting with brief history of sensors and its usage in today’s life along with the market forecasts for the next 10 years. This discussion was continued with the explanation of basic concept behind the workings of sensors, classification of sensors, where each type is explained with an example followed by some of the important sensor characteristics. The next chapter gives a brief introduction to printed electronics, its market and the types of printing methods that can be employed in fabrication of sensors.
2.4. References


[20]. A. Caballero, R. Martínez, V. Lloveras, I. Ratera, J.V. Gancedo, K. Wurst, A. Tarraga, P. Molina and J. Veciana, “Highly selective chromogenic and redox or fluorescent sensors of
Hg\textsuperscript{2+} in aqueous environment based on 1, 4- disubstituted azines”, Journal of the American Chemical Society, vol. 127, pp. 15666-15667, 2005.


CHAPTER III

INTRODUCTION TO PRINTING TECHNIQUES

3.1 Introduction

Printed electronics (PE) is an emerging and potentially significant manufacturing technology in the field of sensors. PE uses traditional additive processes for the fabrication of electronic sensing devices [1]. These additive processes offer numerous advantages such as roll-to-roll (R2R) fabrication, low cost, low operating temperatures, high throughput, less complex manufacturing steps and low wastage of material [2-4] during fabrication in comparison to the conventional silicon-based fabrication process, which involves high-temperature deposition and requires high-vacuum along with sophisticated photolithographic techniques [5-9]. Using these printing processes, several electronic devices such as strain sensors, humidity sensors, organic thin film transistors, electrochemical sensors, gas sensors, RFID tags, and solar cells have been fabricated [10-23].

The fully printed sensors on plastic substrates offer mechanical flexibility, lightweight, and thinness. According to IDTechEx “Printed and Flexible Sensors 2015-2025: Technologies, Players, Forecasts” reported by Dr. Guillaume Chansin, by 2025, the market of fully printed sensors (capacitive, piezoresistive, piezoelectric, photodetector, temperature, humidity, gas, biosensors excluding glucose) will be worth more than $8 billion (Fig. 3.1) [24]. Currently, the printed self-diagnosable glucose sensors, which can be disposable, are alone generating $6 billion of revenue per annum.
3.2 Types of Printing Techniques

The traditional printing techniques are classified into impact printing and non-impact printing. In impact medium, the information is created on the surface of the substrate by the partial transfer of ink through the information carrying medium, the printing plate (a fixed image carrier). On the other hand, non-impact printing techniques are based on digital control of information (design or image) and do not require any stable information carrying medium such as a printing plate.

The main printing techniques that are employed in printed electronics are screen printing, inkjet printing, flexographic printing, and gravure printing. Screen printing is used for creating thick film layers, inkjet for thin film layers, whereas flexography and gravure have been mainly used for the roll to roll printing. The specifications to choose a printing process for electrical applications is shown in Table 3.1. For example, in a particular application, if the desired thickness
of the ink film is 10 µm and the viscosity of the ink available is 15 Pa.s, then screen printing is opted for that application. The print quality mainly depends on the following parameters associated with the inks and substrates used [25].

**Table 3.1. Specifications of printing techniques employed in printed electronics**

<table>
<thead>
<tr>
<th></th>
<th>Gravure</th>
<th>Flexography</th>
<th>Inkjet</th>
<th>Screen</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Image Carrier</strong></td>
<td>Engraved cylinder</td>
<td>Plate</td>
<td>Digital</td>
<td>Stencil</td>
</tr>
<tr>
<td><strong>Ink Film Thickness (µm)</strong></td>
<td>0.02-12</td>
<td>0.17-8</td>
<td>0.01-0.5</td>
<td>3-30</td>
</tr>
<tr>
<td><strong>Printing Speed (m/min)</strong></td>
<td>8-100</td>
<td>5-180</td>
<td>0.02-5</td>
<td>0.6-100</td>
</tr>
<tr>
<td><strong>Line Width (µm)</strong></td>
<td>15</td>
<td>15</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td><strong>Viscosity (Pa.s)</strong></td>
<td>0.01-1.1</td>
<td>0.01-0.5</td>
<td>0.001-0.1</td>
<td>0.5-50</td>
</tr>
</tbody>
</table>

- The wetting properties of the substrate,
- The surface properties such as surface energy, thickness, roughness and surface tension of the materials involved,
- The viscosity of the ink being printed,
- Curing time and temperature,
- The location and environmental conditions around the printer such as vibrations, humidity, and temperature,
- The printing speed and
- The printing pressure.
3.2.1 Screen Printing

Screen printing is a technique in which a paste-like material (ink) is transferred onto the substrate. Screen printing is typically done either by hand, or semi or fully automated. The screen printer mainly consists of the squeegee, stencil and a fabric screen with the design on it. Typically, screen fibers are made of plastic, natural silk or metal fibers and the squeeze is made of rubber. A section in the screen will be imposed by the design of the desired print and the ink is allowed to pass through it and the desired design is created on the substrate being used. The ink is transferred either by pushing or forcing the ink by squeeze through the fabric screen and thus, this printing process is also referred as push through the process (Fig. 3.2). The print quality mainly depends on mesh count, wire diameter, and deflection angle of the screen. The screen printing technique has capabilities to print electronic devices at a low cost, with very little or no material wastage and provides an end product that can be extremely flexible. With rotary screen printing capabilities with roll to roll, processing at ambient conditions, it is possible to fabricate a large number of printed devices in a relatively short period of time [25].

![Figure 3.2. Schematic of screen printing process.](image-url)
3.2.2 Inkjet Printing

Inkjet printing is a non-impact printing method (NIP) and it uses a digital image signal to print the design by propelling ink droplets onto the substrate instead of any physical image carriers. Typically, in inkjet printing, the inks will be in a liquid state with low viscosities and can be directly printed onto the substrate. Inkjet printing is classified into continuous inkjet printing and drop on demand inkjet printing. In continuous Inkjet printing, a continuous flow of ink droplets is controlled electronically by a voltage source. Some of the ink droplets are subjected to electrostatic charge and are deflected to get a negative print by electrostatic deflectors while the uncharged drops are used to print the desired image onto the substrate. In a drop on demand inkjet printing, the ink droplets are created only if it is required by the image signal to get the desired print. The droplets are formed either by using thermal or piezoelectric techniques [25] as shown in Figure 3.3. In thermal inkjet printers, the ink droplets are forced or impelled out of the cartridge nozzle by a vapor bubble formed because of the vaporization of ink liquid. In piezoelectric inkjet printers, the volume of the cartridge nozzle is mechanically deformed by the piezoelectric material based on the image signal and the ink droplets are released from the nozzle. Inkjet printing has advantages such as non-mask fabrication, high print quality, cost efficiency, scalability from tabletop devices to big press units. There are also few drawbacks associated with inkjet printing such as nozzle clogging due to inactivity and it requires frequent cleaning cycles [25].
3.2.3 Flexographic Printing

Flexographic printing is a well-established R2R high throughput rotational printing method. It is an indirect impact based printing technique that can provide a wide range of ink thickness with the same resolution. The main parts of the flexographic printer are anilox roller, an impression cylinder, doctor blade, and the ink reservoir. Anilox roller (a steel cylinder), that has finely engraved cells on its surface made of chromium or ceramics, collects the specific amount of ink from the ink reservoir. Then, the collected ink is transferred onto the elevated structures of the printing plate. With the help of the plate cylinder, the ink on the printing plate is printed on the substrate as shown in Figure 3.4. It is capable of addressing problems such as contact finger geometry as well as production line output [25].
3.2.4 Gravure Printing

Gravure printing is an impact based printing technique that provides economical production with fast outputs along with high-quality printing. It uses low viscosity inks and is known for its robustness. The image carrier cylinder (gravure cylinder), doctor blade, ink reservoir, and the impression cylinder are some of the main components of the gravure printer as shown in Figure 3.5. Typically the impression cylinder is made of rubber, the gravure cylinder is made of copper coated steel and the doctor blade is manufactured with steel. In gravure printing, the image of the desired design is engraved on the surface of the gravure cylinder. The entire gravure cylinder is flooded by ink from the ink reservoir and the extra ink is wiped off from the gravure cylinder by the doctor blade before printing. The ink is transferred onto the substrate from gravure cylinder with high pressures. The substrate movement is controlled by the impression cylinder [25].
3.3 Summary

This chapter provided an overview of printed electronics, starting with a brief introduction to printing technologies and its usage in manufacturing electronic devices along with the market forecasts for the next 10 years. This discussion was continued with the classification of printing technologies along with the explanation of basic concepts such as working principles, and important parts of the printers. The next chapter discusses a project that involved the development of a strain sensors on a paper platform using flexography printing technology. It includes the details about fabrication, experimental setup, and test results.

3.4 References


CHAPTER IV

A NOVEL FLEXOGRAPHIC PRINTED STRAIN SENSOR ON A PAPER PLATFORM

4.1 Introduction

Strain sensors have been receiving growing interest for monitoring shape, stress, strain, force and pressure in the biomedical [1], robotic [2] and automotive [3] application areas. Strain sensors are also being used to record the aging of aircraft through structural health monitoring [4]. In addition, strain sensors are employed for monitoring the civil infrastructural integrity, which is important for the high standard safety requirements in the mechanical and electrical engineering industries [5]. However, strain sensors that have been fabricated using conventional methods are often not flexible enough, are rigid and are separated from the substance or medium that is being monitored [6,7]. Hence, these devices tend to get damaged or broken, which would result in incorrect or unreliable results. Thus, flexible strain sensors fabricated using printing processes is envisioned as a promising solution to overcoming the limitations associated with conventional strain sensors. Printed and flexible strain sensors offer mechanical flexibility and are relatively cost efficient [8,9]. This has led to the research of using printing technology on flexible platforms for the fabrication of strain sensors.

Printed electronics (PE) is an emerging and potentially significant manufacturing technology in the field of sensors. [10-12] PE uses traditional additive processes such as a screen, inkjet, flexography, aerosol and gravure printing for the fabrication of electronic sensing devices. These additive processes offer numerous advantages such as roll-to-roll (R2R) fabrication, low operating temperatures, low cost, high throughput, less complex manufacturing steps and low wastage of material during fabrication [13-16]. Using these printing processes, several electronic
devices such as organic thin film transistors [17], RFID tags [18], electrochemical sensors [19], solar cells [20], strain sensors [21] have been fabricated. Even though, there are reports on the fabrication of strain sensors on glass substrates [22], printed circuit boards [23] and polyethylene terephthalate substrates [24] using screen, inkjet and aerosol printing processes, there are no reports on the use of R2R flexographic printing for the development of strain sensors on paper substrates. Paper, which is known as an eco-friendly substrate, was used because it is typically more flexible, biodegradable, easily recyclable and more cost efficient than conventionally reported silicon, plastic and glass substrates for the manufacture of flexible electronics [25]. Flexography, an indirect printing process, is known for its capability in depositing a wide range of thicknesses with the same resolution [26,27]. A typical flexography system comprises an impression cylinder, printing plate, anilox roller, doctor blade and the ink reservoir.

In this work, the conventional flexographic printing technique was employed for fabrication of strain sensors with different trace lengths and widths on a flexible paper substrate. Silver (Ag) ink was printed on the paper substrates as a metallization layer. The capability of the sensor was demonstrated by investigating the electro-mechanical response obtained from a 3-point bend test for displacements of 1 mm, 2 mm, and 3 mm at 3 Hz operating frequency. The responses of the fabricated strain sensors as a function of electrical resistance is analyzed in this chapter.

4.2 Experimental

4.2.1 Device Fabrication

The strain sensors were fabricated at the Mac Arthur Corporation using a flexographic printer (Webtron 750). Ag ink (Suntronic Aqueous Silver Flexo Ink 3) from Sun Chemical was used as metallization layer and paper (10 Pt. C1S Carolina®, Spec# 19236) from Fasson® was used as the substrate. The Ag lines were printed on the substrate using 250 CPI line
screen/10.0 BCM anilox (CPI = cells per inch/ BCM = billion cubic microns per square inch) at a press speed of 30 FPM (feet per minute). A total of 50 strain sensors have been fabricated with different meander trace lengths and widths. For experimental convenience, six sensors (Sensor #1, Sensor #3, Sensor #18, Sensor #42, Sensor #44 and Sensor #45) were chosen and the photographs of the six sensors with different meander trace lengths and widths (Table 1) are shown in Figure 4.1. No analysis was done on the sensor #18 since it had a break in the grid traces (Fig. 4.2). The RMS roughness and average thickness values of the strain sensors were measured using a Bruker Contour GT-K profilometer. Figure 4.3 (a) and Figure 4.3 (b) shows the 3D profilometry images of the strain sensors. Table 2 shows the measured RMS roughness and average thickness values of the strain sensors.

Figure 4.1. Flexographically printed strain sensors.

Table 4.1. Total trace lengths and trace widths of the sensors

<table>
<thead>
<tr>
<th>Sensor No.</th>
<th>Trace Width (mm)</th>
<th>Total Trace Length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.20</td>
<td>292.50</td>
</tr>
<tr>
<td>3</td>
<td>0.30</td>
<td>259.60</td>
</tr>
<tr>
<td>18</td>
<td>0.30</td>
<td>193.40</td>
</tr>
<tr>
<td>42</td>
<td>0.25</td>
<td>160.70</td>
</tr>
<tr>
<td>44</td>
<td>0.35</td>
<td>143.90</td>
</tr>
<tr>
<td>45</td>
<td>0.40</td>
<td>126.00</td>
</tr>
</tbody>
</table>
4.2.2 Experiment Setup

To evaluate the performance of the printed devices, the strain sensors were subjected to a 3-point cyclic bend test. The experimental setup is shown in Fig. 4.4. The strain sensors were placed on the supports of a 3-point bend test fixture (Mark-10 ESM 301 motorized test stand with a vertically movable platform) (Fig. 4.5). The movable platform, capable of moving upwards and downwards was used to apply different displacements (1 mm, 2 mm, and 3 mm) on the meander trace of the strain sensors in the Y-direction. A Ag conductive epoxy paste (Circuit works CW2400) was used to bond the connecting wires to the contact pads of printed sensors. The devices were then connected to an Agilent E4980A precision LCR meter using alligator clips. When the strain sensors were subjected to a 3 point bend test at 3 Hz frequency, the displacement causes the grid traces in the strain sensor to deform, thus varying its resistance. This change in resistance is measured by the LCR meter, which is in turn connected to a PC with a custom built LabVIEW program for data acquisition and post-processing of the data.
Table 4.2. RMS roughness and Average thickness values of the sensors

<table>
<thead>
<tr>
<th>Sensor No.</th>
<th>Roughness (µm)</th>
<th>Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.38±0.48</td>
<td>2.17±0.35</td>
</tr>
<tr>
<td>3</td>
<td>4.20±0.48</td>
<td>2.70±0.35</td>
</tr>
<tr>
<td>42</td>
<td>4.04±0.48</td>
<td>2.95±0.35</td>
</tr>
<tr>
<td>44</td>
<td>3.66±0.48</td>
<td>2.87±0.35</td>
</tr>
<tr>
<td>45</td>
<td>4.97±0.48</td>
<td>3.09±0.35</td>
</tr>
</tbody>
</table>

4.3 Results and Discussion

The purpose of subjecting the strain sensors to a 3-point bend fatigue test, which induces maximum bending moment at the middle of the printed trace, was to investigate the behavior of the strain sensor and its fatigue life to varying displacements. An optimum strain sensor is expected to display a consistent change in resistance over its functional life cycle. Figure 4.6 to Figure 4.9 shows the response of the strain sensors (Sensor #1, Sensor #3, Sensor #42, and Sensor #44) towards a 3 mm displacement. It was observed that the resistance changed inconsistently throughout the entire test. These results demonstrated that the paper substrate could not withstand a displacement of 3 mm. Hence, sensor #45 was subjected to a smaller displacement of 1 mm, for investigating its fatigue life (Fig. 4.10). This sensor was subject to 10,000 cycles of displacement. It was observed that a change of 1.3±0.3% in average resistance was obtained, when compared to the base resistance during the first 3500 cycles. After 3500 cycles, the resistance change was inconsistent throughout the test. Then, another sample of sensor #45 was subjected to a 2 mm displacement (Fig. 4.11). The results demonstrated a change of 10.3±0.7% in average resistance, when compared to the base resistance during the first 50 cycles. After this, the resistance change was inconsistent throughout the test.

These results show that the paper based strain sensor has a longer functional lifecycle.
Figure 4.3 (a). RMS roughness using vertical scanning interferometry 3D topography.
Figure 4.3 (b). Average thickness using vertical scanning interferometry 3D topography.
Figure 4.4. Experiment setup of 3-point cyclic bend test.

for smaller displacements. This feature/property is especially advantageous for structural asset monitoring applications where the displacements are minimal. Moreover, the measurement of these minimal displacements are crucial for the integrity of structures such as buildings and bridges.

Figure 4.5. Printed strain sensor placed on the supports of Mark-10 equipment for 3-point cyclic bend test (a) Strain sensor at relaxed position and (b) Strain sensor at bent position.
Figure 4.6. The response of the sensor #1 towards 3 mm displacement.

Figure 4.7. The response of the sensor #3 towards 3 mm displacement.

Figure 4.8. The response of the sensor #42 towards 3 mm displacement.

Figure 4.9. The response of the sensor #43 towards 3 mm displacement.
In this work, the fabricated flexible strain sensors using Flexographic printing with Ag ink as metallization on a paper substrate has been analyzed. The capability of the sensor to detect changes in its resistance, caused by the displacement of the sensor, due to an external force was successfully demonstrated. A cyclic 3-point bend fatigue test, produced only 1.3±0.3% change in bend resistance with respect to base resistance value for 3500 cycles for 1 mm displacement thus showing a long enough functional life cycle for the strain sensor, when compared to 2 mm and 3 mm displacements. Due to the continuous application of displacement, the change in resistance with respect to base resistance was inconsistent. This is because the paper substrate of the strain sensor loses its self-restorative/reflexive nature (coming back to its original position) after a few cycles or with large displacements. This low cost, easily disposable strain sensor can be employed in structural monitoring applications where the detection of small displacements is important.

4.4 Summary

In this work, the fabricated flexible strain sensors using Flexographic printing with Ag ink as metallization on a paper substrate has been analyzed. The capability of the sensor to detect changes in its resistance, caused by the displacement of the sensor, due to an external force was successfully demonstrated. A cyclic 3-point bend fatigue test, produced only 1.3±0.3% change in bend resistance with respect to base resistance value for 3500 cycles for 1 mm displacement thus showing a long enough functional life cycle for the strain sensor, when compared to 2 mm and 3 mm displacements. Due to the continuous application of displacement, the change in resistance with respect to base resistance was inconsistent. This is because the paper substrate of the strain sensor loses its self-restorative/reflexive nature (coming back to its original position) after a few cycles or with large displacements. This low cost, easily disposable strain sensor can be employed in structural monitoring applications where the detection of small displacements is important.
In the next chapter, the author discusses a project that involved the development of a carbon nanotube based capacitive pressure sensor fabricated using the screen printing method. It includes the details about fabrication, experiment setup, and test results.

4.5 References


CHAPTER V

DEVELOPMENT OF A NOVEL CARBON NANOTUBE BASED PRINTED AND FLEXIBLE PRESSURE SENSOR

5.1 Introduction

In recent years, a major focus is vested towards the development of highly sensitive and flexible pressure sensors in the field of automotive, military, sports, robotic and biomedical applications. With the rapid evolution of the pressure sensing field, a wide range of devices has been developed to help monitor pressure [1-6]. Typically, pressure-sensing mechanisms are classified into three types; piezo resistive, piezoelectric and capacitive type mechanisms. Among these various types, capacitive pressure sensors are widely employed due to its advantages such as low power consumption, less sensitive to humidity and temperature when compared to the other two pressure sensing mechanisms [7-10]. These pressure sensors are typically manufactured using conventional methods and mostly lacks the flexibility and conformability required for the sensing applications [11-13]. Moreover, most of these sensors must be developed on a rigid substrate and utilize either hanging structures or cavity based designs [13-15]. Additionally, these sensors are expensive to produce and don’t offer the flexibility, conformability and stability required for the applications in various fields. Therefore, to overcome these problems, the researcher was interested in exploring alternate techniques for the fabrication of pressure sensors using flexible materials.

Over the last couple of years, considerable research has been put into the development of printed electronics as well as the best ways to effectively and efficiently print electronic devices [16, 17]. One innovative option is the use of screen printing process in developing printed electronics. The screen printing process allows for various electronics to be printed at a low cost, with very little material wastage and an end result that is extremely flexible. With screen printing,
it is also possible to manufacture a lot of prints in a relatively short period of time. Screen printing is typically done by hand or semi or fully automated. The screen printer mainly consists of the squeegee and a screen with the design on it. The screen itself is made of metal and has a section made of the screen in the design of the desired print to allow the ink to pass through and create the desired design on whatever substrate [18-20]. The researcher in this study considered the use of a screen printed CNT capacitive pressure sensor and investigated its performance based on the capacitive response for the first time.

In this work, a CNT pressure sensor was tested and compared as a viable option for pressure monitoring. Capacitive sensors use the capacitance measured between two opposing electrodes to determine how much pressure is applied to a surface. Conventional screen printing processes were used to fabricate the fully flexible capacitive pressure sensor. The non-conductive layer, or dielectric layer, was made using PDMS. PDMS was chosen for this experiment due to its nonconductive properties and its high flexibility. The practical use of the sensor as pressure sensor was then demonstrated by investigating its capacitive response under varying applied pressures.

5.2 Experimental

5.2.1 Chemicals, Materials, and Sample Preparation

A CNT ink, with an average particle size of 105-113 nm, was used as the metallization layer (Inktec). The ink was kept in a cool place and sealed until needed, to prevent the ink from drying out. Delrin® Acetal Resin block, which serves as the base for mold was acquired from McMaster-Carr®. PDMS, a soft polymer, was purchased as a two-part heat curable silicone elastomer kit Sylgard® 184 from Dow Corning. The Sylgard® 184 prepolymer was mixed in a 10:1 (w/w) ratio with the included curing agent and stirred vigorously until well mixed. The mixing introduces bubbles that were removed by setting aside the mixture at room temperature for
30 minutes. The prepared PDMS was poured into molds with the thickness (1.15 mm). Once, the remaining bubbles in the molds were either allowed to degas or manually poked out, the molds were placed in an oven for 40 minutes at 100-115 °C and then allowed to cool. A small outline integrated chip (SOIC) test clip (Panoma Electronics 6108) was used as a means to connect the pressure sensor to an Agilent E4980A precision LCR meter. The copper pins, on the SOIC test clip, were bent to create a better contact with the metallization layer.

5.2.2 Sensor Fabrication

A screen printing press (AMI MSP 485) from Affiliated Manufacturers Inc. was first calibrated and the screen printing template was installed. Then, CNT ink was used to print samples of the top and bottom electrode directly onto PDMS due to its better adhesion properties. The samples were then cured in the oven at 100 °C for 20 minutes and then allowed to cool. The screen printing press and screen were then thoroughly cleaned using ethylene glycol di-acetate. Special care was taken when cleaning the squeegee and screen as to prevent damage and prolong their use. The schematic of the sensor design and picture of the printed sensor with top, bottom, and dielectric layers are shown in Figure 5.1 (a) and 1 (b), respectively. The dimensions of various spots used in the design of top electrode are shown in Table 5.1. An average thickness of 8.9 µm and average roughness of 0.8 µm was measured for the printed CNT layer on PDMS using a Bruker Contour GT-K profilometer (Fig. 5.2).

5.2.3 Experiment Setup

The experiment setup is shown in Fig. 5 3. A sensor was placed between a force gauge (Mark-10 model M5-200) and vertically moveable platform (Mark-10 ESM 301 motorized test stand) connected to the LCR meter. The sensor can be subjected to various pressures by varying
the compressive forces that can be applied due to vertical motion of the movable platform. The change in capacitance was measured using a custom-built LabVIEW™ program on a PC connected to the LCR meter via a USB cable. A stable platform was also created to help support the sensors and keep it flat to optimize the results.

A test was run on spot B of the top electrode to measure the capacitive response of the CNT-based sensor. Initially, the capacitance of the pressure sensor was recorded for one minute, with no force applied to set a base capacitance. Then, the sensor was subjected to the minimum detectable pressure for one minute, after which the compressive force was released. The response
of the sensor was again recorded for another one minute. This cycle was continued for different increasing compressive forces up to the maximum detectable compressive force. It was observed that both sensors were rendered reversible, after each compressive force was released, due to the fact that the capacitance always attained its base capacitance value.

5.3 Results and Discussion

Various compressive forces were applied to the pressure sensor configuration and the capacitive response was recorded to investigate the performance. When the sensor was subjected to different displacements, the capacitances varied accordingly. Figure 5.4 shows the capacitive response of the sensor with the CNT ink, over increasingly varying compressive forces. The base capacitance was measured to be 6.49 pF. It was observed that the capacitance increased up to 7.02 pF as subsequent compressive forces were applied to a maximum detectable compressive force of 337 kPa. This resulted in an 8.2% increase from the base capacitance to the maximum capacitance. Figure 5.5 shows a normalized change in capacitive response for the CNT pressure sensor at increasing pressures. A linear relationship was observed between the average percentage change in capacitance and pressure. A 0.021 percentage change in capacitance per kPa and a correlation coefficient of 0.9971 was obtained for the CNT pressure sensor. The results show the feasibility of employing a CNT-based pressure sensor configuration as an efficient and cost-

Table 5.1. Dimensions of the spots present in the top electrode

<table>
<thead>
<tr>
<th>Design (Top Electrode)</th>
<th>Length (cm)</th>
<th>Width (cm)</th>
<th>Diameter (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>4</td>
<td>1</td>
<td>Not Applicable</td>
</tr>
<tr>
<td>B</td>
<td>3</td>
<td>1</td>
<td>Not Applicable</td>
</tr>
<tr>
<td>C</td>
<td>1.5</td>
<td>0.5</td>
<td>Not Applicable</td>
</tr>
<tr>
<td>D</td>
<td>Not Applicable</td>
<td>Not Applicable</td>
<td>1</td>
</tr>
<tr>
<td>E</td>
<td>Not Applicable</td>
<td>Not Applicable</td>
<td>0.5</td>
</tr>
</tbody>
</table>
Figure 5.2. 3D output of vertical scanning interferometry of printed CNT on PDMS illustrating an (a) average thickness (ΔZ) of 8.9 µm and (b) average roughness of 0.8 µm.
Figure 5.3. Experiment setup.

Figure 5.4. The graph displays the differences in percent increase with respect to the different pressures applied on CNT based pressure sensor.
Figure 5.5. This graph shows a normalized change in capacitive response for CNT pressure sensor at increasing pressures.

- effective way to monitor pressure in various applications related to aerospace, automobile, sports and biomedical industries.

5.4 Summary

In this project, the author has successfully fabricated a flexible CNT pressure sensor for impact and pressure detections in sports, military, automotive and biomedical applications using screen printing technique. Polydimethylsiloxane (PDMS) was used as a dielectric layer and it was prepared using a PDMS prepolymer and a curing agent in a 10:1 ratio. The electrode design was directly printed using CNT ink onto the PDMS. An average thickness of 9 µm and average roughness of 0.9 µm was measured for the printed CNT layer on PDMS. The capability of the sensor to distinguish between varying compressive forces was demonstrated. The capacitive response of the CNT pressure sensor showed a capacitance increase from 6.49 pF to 7.02 pF over a compressive force of 337 kPa. This resulted in an 8.2% increase from the base capacitance to the maximum capacitance. A 0.021 percentage change in capacitance per kPa and a correlation coefficient of 0.9971 was obtained for the pressure sensor. The results show the feasibility of the
CNT-based capacitive pressure sensor configuration as an efficient and cost-effective way to monitor pressure in various applications in automobile, biomedical industries.

In next chapter, the author discusses a project that involved the development of an electrochemical sensor on paper based substrate fabricated using screen printing process. It includes the details about fabrication, sensor characterization, experiment setup and test results.

5.5 References


CHAPTER VI

DEVELOPMENT OF A PRINTED IMPEDANCE BASED ELECTROCHEMICAL SENSOR ON PAPER SUBSTRATE

6.1 Introduction

In recent years, research and development have been directed towards the development of cost-effective and rapid diagnostic tools [1-3]. Modern day health care systems need accurate, simple, compact and easy-to-use medical diagnostic devices. Often, in order to analyze a sample, laboratories require multiple instruments and materials as well as the involvement of many complex steps in a time-consuming process [1, 4]. Development of miniaturized sensing systems is an effective solution that reduces the cost and time required to perform these analyses [5]. Thus, the complex steps involved in conventional laboratories can be converted to a single analytical platform [6]. Generally, these types of miniaturized analysis systems are referred as “lab-on-a-chip” (LOC) devices. However, the fabrication of LOCs typically involves many integrated operations and devices are often constructed of glass, quartz or polymer-based platforms with many construction steps [5-8]. This has led to the research of exploring alternate promising techniques, such as printing technology for the fabrication of LOCs on different cost-effective platforms such as paper.

Paper-based analytical devices have attracted much attention over the last decade in the bio-medical diagnostic field [9-11]. These devices have evolved as low-cost diagnostic platforms with many advantages such as ease-of-use, less complexity, fewer fabrication steps, rapid response, affordability, sustainability, as well as disposability for applications in the medical, environmental and food related industries [12-14]. Moreover, paper-based analytical devices fabricated with traditional printing methods offer very simple fabrication steps, less wastage of
materials, cost efficiency and require low manufacturing temperatures [15-17]. The major advantage of adopting printing techniques for paper-based analytical devices is the capability to be fabricated in miniaturized sizes, which enables the trace detection of sample volumes, in the milliliter (ml) to nanoliter (nl) scale. Therefore, the development of printed paper based lightweight and portable analytical devices is important.

In this work, an electrochemical sensor with a screen printed Ag electrode is fabricated on a wax-printed flexible paper-based substrate. The capability of the fully printed electrochemical sensor is demonstrated by investigating the EIS (electrical impedance spectroscopy) response of the device towards various concentrations of potassium chloride (KCl), which is a commonly used laboratory salt, and glucose (C$_6$H$_{12}$O$_6$), which is a common sugar, and the results are reported in this paper.

6.2 Experimental

6.2.1 Chemicals and Materials

Whatman grade No. 1 chromatography paper from VWR was used as the substrate. A conductive Ag flake ink (Acheson ELECTRODAG 479SS) from Henkel was used for metallizing the reference and circular electrodes. Potassium chloride (KCl) (in crystalline form) and D-(+)-Glucose (C$_6$H$_{12}$O$_6$) (in powder form) were purchased from Sigma–Aldrich. Deionized (DI) water was used to prepare 1 pM, 1 nM, 1 μM and 1 mM ionic solutions of KCl and C$_6$H$_{12}$O$_6$.

6.2.2 Wax Printing of Paper Substrate

The chromatography paper was fully wax-printed on one side using the Adobe® Illustrator® CS6 software and a Xerox ColorQube 8570 color printer. Then, the wax-printed paper was heated in an oven (from VWR) at 100 °C for 2 minutes to yield a hydrophobic region.
6.2.3 Screen Printing of the Electrochemical Sensor

A screen printing process was employed to metallize the electrochemical sensor with Ag based ink. The two electrode configuration was designed in CoventorWare® software. The sensor was designed with a working electrode of 1700 µm radius and a circular electrode with inner and outer radius of 2900 µm and 3900 µm, respectively as shown in Fig. 6.1(a). A stainless steel screen, from Microscreen®, with wire diameter, mesh count and the deflection angle of 28 µm, 325 and 22.5º, respectively was used. A photo pure polymer (MS-22) of 12.7 µm thickness was used as an emulsion to the screen. The Ag ink was deposited on the wax-printed substrate using an MSP-485 screen printer from Affiliated Manufacturers Inc. The screen printed sample was sintered using a photonic curing system (NovaCentrix Pulseforge® 1200) at 400 V for a duration of 500 ms for two passes. Figure 6.1(b) shows the fully fabricated electrochemical sensor on wax printed paper substrate.

![Diagram of the two-electrode electrochemical sensor configuration](a)

![Fully fabricated electrochemical sensor](b)

**Figure 6.1.** (a) Schematic of the two-electrode electrochemical sensor configuration and (b) Electrochemical sensor with screen printed Ag electrode on the wax-printed paper substrate.
The vertical scanning interferometry 3D topography of the printed electrodes (Fig. 6.2) was measured using a Bruker Contour GT-K profilometer (Bruker Biosciences Corporation, USA) installed with Bruker Vision software. An average thickness of 29.4 µm and average roughness of 2.8 µm was measured for the printed Ag electrode on the wax-printed paper substrate.

6.2.4 Experiment Setup

The experimental setup is shown in Fig. 6.3. EIS measurements were performed on the printed sensor using an Agilent E4980A precision LCR meter, at room temperature. Small outline integrated chip (SOIC) test clips from Pomona® Electronics were used to connect the LCR meter to the printed sensor. Initially, to establish a reference signal, 120 µl of DI water was drop-casted onto the sensor. Then, 120 µl of different concentrations (1 pM, 1 nM, 1 µM and 1 mM) of KCl and C₆H₁₂O₆ were drop casted onto the sensor to obtain the impedance response, at an operating frequency range of 20 Hz to 2 MHz with 1 mV applied voltage. A custom built LabVIEW program was used to observe and analyze the impedance response of the sensor. All measurements were performed at room temperature.

6.3 Results and Discussion

The impedance response of the fabricated sensor was initially tested towards different concentrations (1 pM, 1 nM, 1 µM and 1 mM) of KCl solution. Figure 6.4(a) shows the EIS response of the sensor towards DI, 1 pM, 1 nM, 1 µM and 1 mM solutions of KCl measured at a frequency range of 20 Hz to 2 MHz and applied voltage of 1 mV. A better signal-to-noise ratio was observed from 20 Hz to 7 kHz. For example, at 20 Hz, it was observed that the impedance decreased from 853 kΩ to 691 kΩ to 678 kΩ to 545 kΩ to 22 kΩ as the concentration of the KCl solution was increased from DI to 1 pM to 1 nM to 1 µM to 1 mM, respectively. At 7 kHz, it was observed that the impedance decreased from 717 kΩ to 566 kΩ to 541 kΩ to 430 kΩ to 9 kΩ as
Figure 6.2. 3D output of vertical scanning interferometry of printed Ag on paper substrate illustrating an (a) average thickness ($\Delta Z$) of 29.4 µm and (b) average roughness of 2.8 µm.
Figure 6.3. Experiment setup.

The concentration of the KCl solution was increased from DI to 1 pM to 1 nM to 1 µM to 1 mM, respectively.

Figure 6.4(b) shows the percentage change in the EIS response of the sensor towards 1 pM, 1 nM, 1 µM and 1 mM KCl solutions at operating frequencies of 20 Hz and 7 kHz when compared to the EIS response of DI water. It can be seen that the percentage change in impedance increased, with an increase in the concentration of KCl solution. As an example, for the measurements at 20 Hz, impedance percentage changes of 19%, 21%, 36% and 97% was obtained for the 1 pM, 1 nM, 1 µM and 1 mM KCl solutions, respectively when compared to that of the DI water.

Similar tests were performed for the C₆H₁₂O₆ solution and Fig. 6.5 shows the response of the printed sensor towards varying concentrations of C₆H₁₂O₆, at operating frequencies of 20 Hz and 7 kHz with an applied voltage of 1 mV. Due to application of low voltage, the dipole moment of C₆H₁₂O₆ will be very small at lower frequencies. It was observed that the percentage change in impedance increased, as the concentration of the C₆H₁₂O₆ solution was increased. For the measurements at 20 Hz, an impedance percentage change of 10%, 24%, 34% and 49% was obtained for the 1 pM, 1 nM, 1 µM and 1 mM concentrations of C₆H₁₂O₆ solutions, respectively. The change in the measured EIS response of the printed sensor can be attributed to the variations in ionic concentration at the interface of the sensor-analyte. The responses obtained revealed
Figure 6.4. (a) EIS response for a frequency range of 20 Hz to 2 MHz and (b) percentage change in impedance response at 20 Hz and 7 kHz of the printed sensor towards various concentrations of KCL at applied voltage 1 mV.
Figure 6.5. Percentage change in the impedance response of the printed sensor towards various concentrations of \( \text{C}_6\text{H}_{12}\text{O}_6 \), at 20 Hz and 7 kHz and applied voltage of 1 mV.

detection levels as low as 1 pM and also demonstrated the capability of the printed electrochemical sensor to distinguish among various levels (pico, nano, micro and milli) of sample concentrations.

### 6.3.1 Reason for the Decrease in Impedance Response at Higher Frequencies

It was observed that the reactance part of the impedance is capacitive. A model equivalent circuit for the EIS measurement is shown in Fig. 6.6. The equivalent circuit consists a resistor (R) and capacitor (C) in parallel. Theoretically, at low frequencies, capacitor behaves as an open circuit and real part dominates the reactance part. At higher frequencies, the capacitive reactance gradually decreases and dominates the real part. The decrease in the reactance part affects the total impedance by decreasing the impedance value of the sensor when the frequency crosses a certain
level [18]. The impedance \(Z\), angular frequency \(\omega\), real part of impedance and phase angle \(\phi\) can be calculated using equations 2 to 5.

\[
Z = R + \frac{1}{(j\omega C)} \quad (2)
\]
\[
\omega = 2\pi f \quad (3)
\]
\[
|Z| = \frac{1}{\sqrt{(R)^2 + (\omega C)^2}} \quad (4)
\]
\[
\phi = \tan^{-1}(\omega CR) \quad (5)
\]

6.4 Summary

In this chapter, a screen printed impedance based electrochemical sensor was successfully fabricated on the paper substrate. The printed sensor’s response towards KCl, displayed impedance percentage changes of 19\%, 21\%, 36\% and 97\% for the 1 pM, 1 nM, 1 µM and 1 mM solutions, respectively when compared to DI water at 20 Hz operating frequency and 1 mV applied potential. Similarly, the impedance response towards \(\text{C}_6\text{H}_{12}\text{O}_6\) displayed impedance percentage changes of 10\%, 24\%, 34\% and 49\% for the 1 pM, 1 nM, 1 µM and 1 mM solutions, respectively. The results obtained thus demonstrate the capability of the printed sensor to distinguish among various levels
(pico, nano, micro and milli) of sample concentrations as well as the potential of employing paper substrate based electrochemical sensors for bio/chemical sensing applications.

In the next chapter, the author concludes all three projects and provides some suggestions for the future work.

6.5 References


CHAPTER VII

CONCLUSION AND FUTURE WORK

7.1 Conclusion

In the course of this thesis work, the author successfully demonstrated the fabrication of a flexible strain gauge on the paper platform using traditional printing process i.e., flexography. A carbon nanotube (CNT) based capacitive pressure sensor was fabricated using screen printing process and its capability to monitor pressure has been demonstrated successfully. In addition, an electrochemical sensor was fabricated on wax printed chromatography paper substrate using screen printing technique has also been demonstrated. The performance results obtained through the research work of all the three projects are listed below:

In the first research project, the author developed a flexographically printed flexible strain sensor on paper based substrate, using silver (Ag) as a metallization layer. The capability of the printed strain sensor to detect changes in its resistance due to the displacement of the sensor was investigated by using 3-point bend test on a Mark-10 ESM301 test stand. The outcome of the electromechanical response of 1 mm displacement at 3 Hz operating frequency, showed only 1.3±0.3% change in bend resistance with respect to base resistance value for 3500 cycles for 1 mm displacement thus showing a long enough functional life cycle for the strain sensor, when compared to 2 mm and 3 mm displacements. This demonstrated the capability of the flexible paper-based printed device to be used as a strain sensor in sensing applications for safety measures mainly in industries such as aerospace, automobile, and construction of structural buildings.

In the second project, a flexible CNT-based capacitive pressure sensor was developed by employing the screen printing technique for the impact detections and pressure monitoring in
automotive, military, sports and biomedical applications. A Polydimethylsiloxane (PDMS) prepolymer and a curing agent was mixed in a 10:1 ratio, and the resulting hardened and transparent polymer was used as a dielectric layer. The top and bottom electrode designs were directly printed onto the PDMS using CNT ink. The capability of the sensor to distinguish between varying compressive forces were demonstrated using the Mark-10 ESM301 test stand. The capacitive response of the CNT pressure sensor showed a capacitance increase from 6.49 pF to 7.02 pF over a compressive force of 337 kPa. A 0.021 percentage change in capacitance per kPa and a correlation coefficient of 0.9971 was also measured from the capacitive response graph of the pressure sensor. The results show the feasibility of the CNT-based capacitive pressure sensor configuration as an efficient and cost-effective way to monitor pressure in various applications in automobile, aerospace, and biomedical industries.

In the third project, a novel printed impedance based electrochemical sensor has been successfully developed on a paper substrate for the detection of various bio/chemicals. This flexible analytical device was fabricated by screen printing a two electrode sensor configuration on a wax-printed chromatography paper substrate. Silver (Ag) ink was used for screen printing the working and circular electrodes. The capability of the fabricated electrochemical sensor for detecting trace concentrations of bio/chemicals such as potassium chloride (KCl) and glucose (C₆H₁₂O₆) was investigated. The electrical impedance spectroscopy (EIS) based response of the printed sensor towards KCl, displayed impedance percentage changes of 19%, 21%, 36% and 97% for the 1 pM, 1 nM, 1 µM and 1 mM solutions, respectively when compared to DI water at 20 Hz operating frequency and 1 mV applied potential. Similarly, the impedance response towards C₆H₁₂O₆ displayed impedance percentage changes of 10%, 24%, 34% and 49% for the 1 pM, 1 nM, 1 µM and 1 mM solutions, respectively. The EIS based response of the printed sensor revealed
detection levels as low as picomolar (pM) concentrations as well as the capability of employing paper-based electrochemical sensors for bio/chemical sensing applications.

7.2 Future Work

Based on the experience gained during the course of the thesis, the author believes that the current three projects can be improved by implementing some of the following suggestions.

A Novel Flexographic Printed Strain Sensor on Paper Platform

- Use stretchable inks such as silver flexo ink from Liquid X as metal layer to avoid the breakage of the conductive lines to withstand even higher displacements [1].
- Use DuPont Tyvek paper as a substrate which offers high flexibility and resists deformation and can withstand higher displacements [2, 3].

Development of a Novel Carbon Nanotube based Printed and Flexible Pressure Sensor

- Improve the adhesion between the CNT ink and PDMS by modifying the surface properties of the PDMS.
- Apply passivation layer onto both sides of electrodes before testing to avoid leaching of the CNT ink due to poor adhesion.

Development of a Printed Impedance Based Electrochemical Sensor on Paper Substrate

- Detect lead (Pb) and mercury (Hg), which are toxic heavy metals and environmental pollutants.
- Integrating the current prototype with an optical detection system thus allowing the dual-detection capability for reducing the possibility of false positives.
7.3 References

