Inkjet Printing of Transducers

by

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Abstract

In the past few years, inkjet printing has been emerging as a cost effective, environment friendly, net-shape microfabrication technique. This non-contact deposition technique facilitated the deposition of metallic and polymeric inks, biological proteins, and cells. The present work investigates the inkjet printing of microtransducers, with a focus on stress-sensing and movable microstructures. Piezoresistive and interdigitated capacitor based strain gauges were printed and tested. The inexpensive conductive polymer, poly(3,4-ethylenedioxythiophene) oxidized with poly(styrenesulfonate) (PEDOT:PSS), was used as base material. We have performed measurements on several test structures to show that PEDOT:PSS does preserve its piezoresistive properties after printing. As we were relying further on PEDOT:PSS as a base material for printed transducers, the mechanical and electrical properties of this commercially available ink were comprehensively investigated. A dedicated experimental setup, which was used for the mechanical and electrical characterization of test structures, and micro-topography measurements were combined in order to extract the parameters of the PEDOT:PSS thin film: a zero-stress electrical conductivity of $G=201 \text{ S/cm}$ and gauge factor of 3.63. The longitudinal and transversal piezoresistive coefficients were estimated to be, $\pi_L = -5.110 \times 10^{-10} \pm 13.7\% \text{ Pa}^{-1}$ and $\pi_T = 3.45 \times 10^{-10} \pm 4.34\% \text{ Pa}^{-1}$ respectively, which denote a piezoresistive material in a similar range of piezoresistivity as n-doped silicon and conventionally fabricated PEDOT:PSS.

A second explored direction was using inkjet microprinting technology for the fabrication of movable microstructures. An inkjet-printed CMUT (capacitive micromachined ultrasound transducer) was the target device, using ZnO as sacrificial layer and PEDOT:PSS as structural layer. The printed ZnO sacrificial layer was too rough, non-uniform and with a high porosity, so
that printing a conductive membrane on top of it was unsuccessful. An alternative solution approach used kapton tape, a polyimide, as movable membrane; experimental characterization has shown that the structure is not properly clamped along its rim, yielding a vibration at a frequency of 1.1 KHz, when actuated, compared to a resonant frequency of 9.3 KHz achieved by finite element analysis of the CMUT structure. The approach shows enough promise for further investigations, along directions suggested at the end of the thesis.
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Dedication

To My Parents:

Ali & Zahra
Chapter 1

Introduction

The field of inkjet printing of electronics and sensors has been emerging for the past decade where its market is expected to exceed $300 billion over the next 20 years [1]. Inkjet printing, a non-contact direct patterning technique, is the process of disposing liquid ink onto a substrate. Even though it is a 2D deposition technique, 3D films and devices can be fabricated by printing materials on top of each other. A large variety of inkjet compatible solutions can be used for printing, from living cells to nanotubes and nanowires or various polymer compounds and metallic nanoparticle solutions. Inkjet printing of polymers and materials is a fairly new microfabrication technique that has the potential of replacing other common and conventional techniques, as it is a cost-effective and an environment-friendly technique. The present work focuses on inkjet printing of organic polymers based microsensors by printing different material onto different rigid and flexible substrates. In microprinting, drop-on-demand inkjet printing is used to deposit “ink”, which can be a polymer, a solvent, a metal, and even a protein, from thermally actuated or piezoelectrically actuated nozzles. Drop-on-demand (DOD) technique is the approach used in the present work, due to its advantages over other alternatives, which are discussed below. On the other hand, there are limitations and drawbacks that have to be taken in consideration when dealing with this method.

Silver nanoparticles, for instance, have been used to print silver tracks, and even spiral inductors [2]-[3]. We have printed resistors using this silver ink on glass substrates and their conductivity varied according to sintering time. Nevertheless, we have preferred a cheaper alternative to the
expensive silver ink, based on the conductive polymer poly (3,4-ethylenedioxythiophene) oxidized with polystyrene sulfonated acid (PEDOT:PSS) [4]-[6]. The devices reported here use the conductive polymer PEDOT mixed with some PSS as base material. PEDOT:PSS mechanical and electrical properties and its printing technology have been previously discussed in literature [4],[10]. Besides investigating the use for printing droplets or simple tracks, functional devices have been reported, from transistors, capacitors, and basic piezoresistors, to RC filters and strain gauges [5]-[10]. Also, in a previous work of ours, polymer based piezoresistors and interdigitated capacitors were printed on flexible substrates [11]. The printed interdigitated capacitor we fabricated was the first of its kind to be presented in literature (in February 2009) [11]. In addition, we have been able to print zinc oxide, a piezoelectric material, through the use of zinc acetate solution.

Inkjet printing technique was used in many applications and fields. After a comprehensive literature review, here are some of the devices that exploited this technique. Printing was used to fabricate electronic luggage tags [7], gas sensors [7], fully reconfigurable chipless RFID tags [12], carbon nanotube sensor devices [13], polymer thin film transistors (where the material used in fabricating source, drain, and the gate was PEDOT:PSS) [14], convex microlenses by printing the viscous photoresist SU-8 [15], polymer films for detecting organic vapours [16], OLEDs and multicolor OLEDs [17]-[18], RF MEMS [19], microsensors [20], high speed thin-film transistor on flexible substrates [21], and polymer electronic structures [22].

Inkjet printing has already made an impact in the field of biomaterials and bioscience fields where it has been previously utilized for biosensor development, biochips, and DNA-synthesis, to name a few [23]. In many applications (e.g. tissue engineering) the compatibility constraints favor the microprinting techniques over the classic microfabrication processes. Cells, proteins,
scaffolds, etc. can be easily inkjet printed [23]-[30]. Printing technique was utilized to accurately place various cell types into soft scaffolds with the help of computer-aided design templates [31]. Not only that, other groups used inkjet printing techniques to fabricate the scaffolds themselves by indirect 3D fabrication, achieved by printing multiple layers of materials on top of each other [30],[32]-[33]. In the biological field, printing was used in the separation of peptides for mass spectrometer analysis and in fabricating reproducible optical fibre imaging sensors (material contains a PH sensitive indicator) on mass scale without the need to calibrate each sensor individually [34]-[35].

1.1 Advantages of Inkjet Printing

Many features have attracted researchers to explore the field of inkjet printing. The capability of in-house fabrication, savings in materials and maintenance costs, being a more environment friendly technique, and cutting down on fabrication time significantly are certainly major attractions. It avoids extreme and costly photolithography techniques and lengthy fabrication processes (taking several weeks from designing the layout mask to getting to the final pattern), requiring expensive equipment, and special processing conditions (cleanroom and yellow-room) [36]. Inkjet printing has the potential of replacing other common and conventional techniques such as deposition (sputtering, LPCVD, PECVD, and evaporation), lithography, and even etching.
Figure 1. Process flow of fabricating some pattern using 1) conventional fabrication techniques which includes a) preparing the substrate, b) deposition of material on the whole substrate, c) deposition of photoresist on top, d) patterning the photoresist by photolithography, e) etching excess material, f) etching photoresist left; and 2) by inkjet printing by which it only requires depositing specific amount of material at a specific location.

Unlike conventional microfabrication techniques, which are considered subtractive processes, inkjet printing is an additive process. Normally, in a subtractive process, the process flow consists of deposition on wafers by one of the deposition techniques (oxidation, sputtering, evaporation, CVD, sol-gel, epitaxy, electrodeposition, spin casting), followed by a patterning process. Photolithographic patterning includes depositing photoresist on top of the layers already deposited, transferring the desired pattern from the designed optical masks to the photoresist, selectively removing the photoresist, and then etching away excess material by wet etching.
(isotropic or anisotropic where etching selectivity has to be taken in consideration) or dry etching
(plasma, DRIE) (figure 1 – 1). As noted in figure 1 – 2, inkjet printing is a non-contact direct
patterning technique where desired amount of solutions can be ejected and dispensed at a
specific and predetermined point on a substrate. It is a direct net shape process, without the
lengthy succession of the usual steps needed for the fabrication of micro and nano devices in
microelectronics. It is an environment-friendly technology as well, as it avoids the generation in
the process of large quantities of chemical waste [38]. The non-contact patterning is a simpler
and more cost-effective technique as large amounts of raw material can be saved. For example, if
a thin layer of some material is required to be deposited on a small portion of the substrate,
printing material only where it is required to be deposited is trivial using this technique. The
technology is relatively simple to set up, maintain and operate outside conventional cleanrooms,
saving expensive operational costs. This combines well with the possibility of transitioning from
rapid prototyping in the research phase to large-scale production, with a good control in
repeatability and fabrication yield. The non-contact patterning characteristics avoids potential
problems like cross sensitivity and chemical compatibility when different chemicals are to be
used in fabricating devices, e.g. for the case of multianalyte sensors [35].
Although inkjet printing is a 2D deposition technique, it can be used to build 3D devices and
systems by simply additively printing multiple layers of the same or different materials on top of
each other, after taking into consideration the changes in the characteristics of printed films due
to post processing and drying of the deposited films. Depending on the resources available for
the users, the printing technique can be exploited even further by using concurrent multiple
nozzles – containing same or different types of ink – to simultaneously deposit ink(s), improving
upon the speed of the fabrication process and increasing the functional area. This feature depends
on the printers themselves and the controllability users have on them. We were able to fabricate a movable-based structure (discussed later in chapter 4 in more details), by printing PEDOT:PSS “walls” to hold a suspended membrane, deposited over a zinc oxide sacrificial layer. The goal is to fabricate movable membranes, capacitively actuated, by etching away the sacrificial layer. Not many groups have gone into the printing of 3D systems yet, but some examples do exist in literature. In Fuller et al., etch released mechanical structures were introduced [36]. Another application has focused on printing 3D metal-insulator-metal crossovers, where the metal is silver nanoparticles and the insulator is SU-8, were sequentially printed to generate complex 3D structures [5].

Inkjet printing is less limited by substrate composition and characteristics and can accommodate a greater number of layers and range of materials than conventional microfabrication techniques, which makes it a more desirable mean of fabricating devices [36]. An example is its use for bioactive fluids, which cannot tolerate conventional techniques, like exposure to photolithography and etching chemicals; they could be, instead, accurately and easily deposited by printing [34]. This advantage also applies to temperature sensitive substrates, such as polymer films, which cannot tolerate multi-hundred degrees atmospheres required in deposition in conventional techniques [38].

Other advantages include the avoidance of costly cleanrooms (as they consume great amounts of energy) and minimizing the set of hazardous material (as printing reduces amount of excess raw material, mainly chemical, involved). In addition, it offers cheap manufacturing costs not only for mass production, but also for small-volume products [39].
1.2 Limitations and Drawbacks of the Printing Technique

On the other hand, and like other microfabrication techniques, inkjet printing has some drawbacks and limitations that have to be taken in consideration during the design phase. Some of these disadvantages might be irrelevant for a particular fabrication process, or their influence could be minimized through attentive measures (e.g. the choice of proper solvents). Even though printing allows deposition of wide variation of materials, it has a lower resolution than conventional deposition techniques. Printing-compatible inks have to be used, post-processing (e.g. thermal annealing) might be required, and the shape and characteristics of the printed layer/film typically change after post-processing. The equipment used in the present work is a MicroDrop printer, with a printing resolution of 5µm (whereas a resolution at the level of nanometers is typical for most deposition techniques in state-of-the-art cleanrooms).

Issues such as compatibility of the fluid ink and its physical characteristics have to be studied prior to printing, which makes the formulation of printing-compatible inks a challenge; these aspects can influence the quality of the printed films and stability of droplets dispensed.

When choosing (or making) ink, many of its physical characteristics have to be taken into account such as viscosity, surface tension, density, evaporation rate, particle size, solid content, solvent, and shelf life [40]. Ignoring or not properly choosing suitable ink will lead to clogging the nozzle. These terms are listed and defined in Table 1.
One way to make nozzles less susceptible to clogging is by diluting the ink with an appropriate solvent. For example, we diluted the PEDOT:PSS solution with deionized water and the silver nanoparticles ink with ethylene glycol. This will reduce the solid content in the ink, and, allow users to print films continuously for longer periods of time; on the other side, the quality of the printed film will also be reduced, in terms of various parameters (e.g. its conductivity). Degradation in the quality of printed patterns and films can be (partially) compensated by

### Table 1. Physical characteristics of ink solutions [40]

<table>
<thead>
<tr>
<th>Physical Characteristic</th>
<th>Definition and Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity</td>
<td>Defines the form and the volume of the droplet; if too low, satellite droplets will be generated; the higher it is, the more difficult it becomes to control and it might lead to nozzle clogging.</td>
</tr>
<tr>
<td>Surface tension</td>
<td>Defines the velocity of the droplet tail and the breaking of the droplet; the higher the surface tension, the longer the pulse is required. It might lead to clogging the nozzle as well.</td>
</tr>
<tr>
<td>Density</td>
<td>The density of the whole ink; the higher the density, the longer the pulse is required.</td>
</tr>
<tr>
<td>Evaporation rate</td>
<td>How fast solvents in the ink evaporate; limits printing for long periods of time.</td>
</tr>
<tr>
<td>Particle size</td>
<td>Size in polymers and size of metallic nanoparticles; increase the agglomeration risk if solutions were left for long periods of time; the larger the particle the higher the possibility of clogging the nozzle.</td>
</tr>
<tr>
<td>Solid content</td>
<td>Defines conductivity or dielectric properties and the thinness of the film; also, possibility of clogging the nozzle in case the ink was not properly filtered.</td>
</tr>
<tr>
<td>Solvent</td>
<td>Fluid that carries the solid crystals and it is usually deionized water, ethanol, or ethylene glycol; prevents nozzle clogging in the long run and enables ink spreading on the substrate.</td>
</tr>
<tr>
<td>Shelf life</td>
<td>Inks' characteristics alter with time that leads to printed films with lower conductivity and mechanical performance.</td>
</tr>
</tbody>
</table>
repeating the printing procedures over and over again; i.e. instead of printing one layer of original PEDOT:PSS solution, printing 4-5 layers of diluted PEDOT:PSS can be an alternative.

In order to reduce the possibility of nozzle clogging even further, filtering the ink prior to printing is highly recommended, especially if the ink is susceptible to agglomeration. Prior to printing, we used to filter inks with 5μm nylon filters to make sure that all its contents can actually make it out of the nozzle as, sometimes, larger particles are formed in the ink due to dryness and agglomeration. In case the ink has high surface tension, adding the surfactant Triton x-100 was proven reliable by our colleagues and enhanced the quality of the printed layers.

In one of the studies (see Table 2), an example of adding solvents to original PEDOT:PSS solution (as received) was presented in a publication by Lopez et al. where the viscosity was measured with different ink-solvent combinations and ratios [4]. The anticipated viscosity depends on the nozzle itself. In our case, nozzles had a limit of 20 mPa.s, so 70%Pedot:PSS + 30% DI water and then 5% DMSO added to the solution, was within the nozzle’s limits.

**Table 2. Viscosity of different PEDOT:PSS diluted solutions [4]**

<table>
<thead>
<tr>
<th>Solution</th>
<th>Viscosity [mPa.s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEDOT:PSS out of the bottle</td>
<td>52</td>
</tr>
<tr>
<td>75% PEDOT:PSS + 25% DI Water</td>
<td>22.4</td>
</tr>
<tr>
<td>60% PEDOT:PSS+ 40% DI Water</td>
<td>14</td>
</tr>
<tr>
<td>50% PEDOT:PSS+ 50% DI Water (or 1:1)</td>
<td>9.8</td>
</tr>
<tr>
<td>1:1 +5% DMSO</td>
<td>11.3</td>
</tr>
<tr>
<td>1:1 +5% DMSO +1% Triton</td>
<td>12</td>
</tr>
<tr>
<td>1:1 +5% Ethylene Glycol +1% Triton</td>
<td>13.5</td>
</tr>
</tbody>
</table>
Another problem encountered in printing is what is called the *coffee-ring effect*, which is a puddle-like pattern formed around the printed tracks after solvents and other ink components have evaporated. This phenomenon relies heavily on the type of ink, and to a lesser extent on the substrate characteristics. Its effects can be reduced by choosing a solvent with higher boiling point and a lower surface tension, as this induces a surface tension gradient inward, Marangoni flow, which can compensate for the coffee-ring effect (which, by itself, creates an outward convective flow). For printing silver nanoparticles, for instance, ethylene glycol was used to enhance the film and made it more uniform [38], [41]. Another way to tackle this is by the help of surface-energy patterns that cause printed ink to be repelled and dewetted from pre-specified locations on the substrate [42]. This gives the user more ability to control the flow and the spread of the ink on the substrate.

As noted in Table 1, if ink was not chosen with appropriate physical characteristics, clogging the nozzle is highly probable. This is a disadvantage as it requires long period of cleaning time, and sometimes, the nozzle would be permanently damaged and it should be replaced. MicroDrop original nozzles are, unfortunately, very expensive, so a mechanical adapter and buffer circuit (to compensate for the impedance mismatch) have been designed in order to be possible to use MicroFab piezoelectric nozzles, which are much cheaper, with the MicroDrop inkjet microprinter.

Another disadvantage is that the volume and the final pattern of the printed film changes after processing (annealing, sintering, etc.) [37], depending on the annealing temperature and time in addition to the evaporation of solvents. These attributes add an uncertainty in the final thickness of thin films and printed layers [6]. In addition, post processing (sintering and annealing or even just air drying) is required in order to strengthen the cross-linking between metallic nanoparticles.
and organic molecules themselves, as printing leads to large discontinuities in films that may lead to a huge reduction in conductivity. According to the manufacturer, the longer the printed PEDOT:PSS films are baked, the more conductive it becomes [43]. In addition, the contamination brought by printers, the interaction between more than one kind of inks, and uncontrollable drying patterns (due to atmosphere, ambient temperatures and humidity) will adversely influence the devices’ characteristics and lead to non-uniform film thickness [32]. Another problem encountered when printing layers on top of each other is the difficulty of adhering droplets to previously solidified layers [36]. This leads to short term stability, but still they can be used to fabricate low-cost, disposable devices.

1.3 Organic Polymers as Ink

In our work, we intended to use mainly organic conductive polymers, such as PEDOT:PSS, in fabricating devices; a choice motivated by their inexpensive cost compared to metallic inks. In a presentation by VTT information technology, it was noted that conductive polymer ink costs around 400 Euros/kg versus 5000-10,000 Euros/kg for metal particles ink - polymer: 0.4 Euros/m² versus 5-10 Euros/m² [44]. Printing of organic polymers, and mainly PEDOT:PSS, are attracting people in industry and academia toward extensive studies and investigations into their properties and characteristics after printing; organic polymers are simple to fabricate using inkjet, compatible with various substrates, have relatively high conductivity, thermal and electrochemical stability, transparency to visible light, reproducibility, and, most importantly, commercially available in aqueous solutions [4]. All polymer and polymer light emitted diode (PLED) were fabricated [7]; it was used as the electrode in parallel plate capacitor and RC filter [6], [7]. Nonetheless, polymers suffer from low mobilities, and, hence, can only be used in
applications were high speeds are not essential, such as low cost memory devices [6]. Polymer thin film devices are reproducible with carefully designed processing techniques, yielding a potential of even lower cost fabrication process.

1.4 Goals

Initially, our goal was to design and fabricate, using inkjet printing technique, strain gauges inspired by the work presented in Ryosuke et al., proposing a cheaper alternative process flow [55]. During the progress of the project, the inexpensive PEDOT:PSS has replaced other metallic nanoparticles such as silver. Silver nanoparticles based inks have been used at the beginning for printing interconnection tracks and simple resistors but, needless to say, fabricating devices based on silver nanoparticles is more expensive than devices fabricated by photolithography, for example. The increased use of PEDOT:PSS inks generated the need for a more comprehensive characterization of the printed thin films, especially with respect to their electrical and mechanical properties. The success in fabricating 2D devices suggested the next step, the move towards (movable) 3D structures. Movable microstructures is a novel topic in inkjet printing technology, and it can open large doors for completely new applications, like 3D ultrasound imaging with 2D CMUT arrays on a flexible substrate.
Chapter 2

Technology and Process Flow of Printing Devices

The first thing coming to mind when printers are mentioned is desktop printers, widely used in offices, schools, and homes. Even such printers can be still used to deposit simple tracks and very simple devices, by simply emptying the ink reservoirs and filling them with appropriate conductive inks, then printing the desired pattern (prepared by CAD software). Nevertheless, here, we refer to a different kind of printers, with a more flexible control, dedicated to microprinting. Such specialized printers cost tens of thousands of dollars and require training to operate and maintain. Printers, in general, fall under two categories: continuous inkjet printers (CIJ) and drop on demand printers (DOD). In CIJ, which is typically used in packaging industries, a continuous stream of ink is forced out from a reservoir and the droplets are then formed according to “Rayleigh’s break,” where charged droplets pass through a guiding electrostatic field, which directs them to specific locations on the substrate or for re-circulation into the system. On the other hand, in DOD systems, drops are created “on-demand” or only when necessary through piezoelectric or thermal actuation. In CIJ, printing takes place at much higher velocities than in DOD, but recirculation of unused droplets in this method leads to waste in ink. This is not a suitable method of printing when dealing with expensive metallic ink. Also, DOD systems have higher printing resolutions than the CIJ printers as in DOD, the size of the
dispensed droplet has almost the same size of the orifice; whereas it is double the size of the orifice in CIJ printers [34].

2.1 Printer and Ink Used

A Microdrop Inkjet System (Microdrop Technologies GmbH, Germany) has been used to print the sensors (Figure 2). This printer has a horizontal resolution of 5µm (and a repeatability resolution of 1µm), and operates in the drop-on-demand mode, where users have controllability over the size, the velocity, and the shape of each droplet to be dispensed. The printing heads contain piezoelectric nozzles; a driving high voltage pulse induces a deformation into the piezoelectric material and generates a pressure pulse into the nozzle connected to the ink reservoir. A minute droplet of ink is ejected, depending on the balance of the existing dynamical forces acting upon the liquid. A tuning mode is usually necessary before printing a desired pattern, where various parameters (input voltage, pulse duration, frequency at the tip, and velocity of each droplet) are optimized. In this way, the size and the shape of individual droplets can be properly adjusted.

The x, y and z coordinates of the starting point, the number of droplets in each direction, and spacing between two consecutive droplets have to be specified for each desired pattern. Even though MicroDrop offers a wide variety of nozzles, MicroFab nozzles (MicroFab technologies, USA) have been adapted to the system, for they have a lower cost and proven to be more reliable in printing PEDOT:PSS. An external impedance adapter circuit was used to interface MicroFab nozzles (80µm and 40µm nozzles were used in our studies) with the MicroDrop printing system.
Figure 2. Controlling the shape and the size of the droplet to be deposited (left) and the Micro Drop printing system used to fabricate the devices (right)

PEDOT:PSS, Clevios PH 500 (H.C. Starck, Germany) was chosen as ink. Even though we have successfully printed silver nanoparticles (Cabot Printing Electronics and Displays, Albuquerque, USA) before, PEDOT:PSS was chosen for the present study, as a much cheaper alternative to silver. In addition, printing PEDOT:PSS is easier and faster than printing silver, with deionized water used as a cleaning solvent for the nozzle. Cleaning after the use of silver-based inks requires both a stronger solvent (e.g. ethanol) and ultrasound cleaning of the nozzle tip in a sonication bath. Sonicating the tip regularly may lead to degradation of the nozzle in a really short period. Silver printing may still be required when high conductivity traces are desirable. In
order to get a highly conductive solution, manufacturer recommends adding 5% dimethyl sulfoxide, DMSO, to the PEDOT:PSS solution.

The PEDOT:PSS solution used (PEDOT:PSS with a ratio of 1:2.5 by weight) contains solid content of 1.0-1.3% and has a viscosity of 8 -25 mPa.s. This ink has an advantage of having good photo and thermal stability and the capability of staying stable for 6 months if stored at 20 degrees in sealed containers [45]. Its resistivity depends on baking temperature: the higher the baking temperature, the higher the conductivity, but the material might suffer degradation in conditions of high temperatures (higher than 225 degrees), UV-light, and oxygen [43].

By itself, the cationic charged PEDOT is insoluble and thus non-processable; As a result, another ionomer, PSS, was added. This generates a water-soluble complex where oligomeric PEDOT segments are attached to the long chains of the PSS.

The polyimide substrate used is a DuPont (Ohi, US) Kapton HN polymide film (general purpose film) with a thickness of 25 µm [46].

2.2 Design and Printing

According to supplier’s recommendation, 5% DMSO has to be added to the PEDOT:PSS solution to ensure maximum conductivity. The solution was printed on flexible substrates, such as paper, transparency films, and polyimide. The sensors studied in this paper were printed on paper in the first phase of experiments and then on the more reliable and versatile polyimide films. Printing devices requires balancing many factors. Even though an 80µm nozzle was firstly used for printing, this does not mean that the size of each droplet printed droplet will be exactly 80µm, due to droplet dispersion. The thickness of the printed thin film is determined in part by
the overlapping of the adjacent droplets, so the minimum spacing between two adjacent printed droplets has to be taken into account. Lopez et al. [4] have investigated the relation between the spacing and the PEDOT:PSS film thickness. It turned out that with drop separations of 20µm, 25µm, and 30µm, the mean film thickness was 439.9 nm, 305 nm, and 221.2 nm respectively [4]. An 80µm nozzle was used in the first experiments to print the interdigitated electrodes and the piezoresistors’ tracks, with a drop spacing of about 50µm. The thickness of the printed film, only one layer of PEDOT:PSS was printed, was estimated to be 100-150nm. A thicker film can be obtained by repeating the printing over the same pattern in successive layers. Depending on the nature of the substrate, PEDOT:PSS may diffuse into it (the same way any other ink would diffuse into paper). This will, ultimately, affect the spread out of the droplet and the film thickness. Another source of variability, not investigated before, is the speed of the moving stage where the substrate is placed (in this kind of printers, the nozzle is fixed, while the stage holding the substrate is movable). Figure 3 shows two printed piezoresistors using the same pattern and printing parameters, with the exception of the value of the relative moving speed. The higher the moving speed of the stage, the lower the quality of tracks, but the spacing between the parallel tracks is preserved. On the other hand, if the stage is moving at a really slow speed, the quality and the thickness of the film is much better; but the design will have to have wider gaps between parallel lines in order to prevent interconnections and short circuiting between adjacent lines. Identical printing parameters and procedures were used to print piezoresistors, capacitors, and in a second research phase, the supporting walls of the movable structures. The procedure consisted in specifying the dimensions, write the associated command lines, and wait for about 2 minutes for each device to be printed. A simplified job command language is used to control the position of the nozzle with respect to the printer’s stage (i.e. where the substrate is located). The macro-
language set the starting point, number of droplets to be dispensed, and the spacing between two adjacent droplets. For this case, the spacing was set to 50µm. The input voltage was set to 52 V amplitude and a pulse width of 200 µs. The stage’s speed was set to 78mm/sec (Figure 4,7,8). If these sensors were to be fabricated using conventional fabrication techniques, the lift-off process would have been used [8], [47]. After printing, it is recommended to anneal the devices for 90 min at a temperature of 80°C for the solvents to evaporate.

Figure 3. Pattern printed with a stage speed of 10mm/sec (left) and the same pattern printed with a stage speed of 78mm/sec (right)
2.2.1 Printing Piezoresistor on Paper

For the piezoresistor (Figure. 4), the gap between the two parallel tracks is 500 µm, the length of each track is 12mm, and each track’s width is estimated to be 150-200 µm. Nine tracks were printed. For this resistor, no topographical measurements or 3D simulations were done. As only one layer of PEDOT:PSS was printed, the thickness is estimated to be around 500nm, considering that the ink diffuses into the paper substrate.

Figure 4. A printed piezoresistor. Solder paste is used to connect the pads to the wires
2.2.2 Printing Interdigitated Capacitor on Paper

A simplified schematic of the printed interdigitated capacitor is shown in Figure. 5. For this case, the overlap length, \( a \), is 10\,mm, electrode length, \( c \), is 17\,mm, both finger width and gap area (\( b \) and \( g \)) have 500\,\mu m and the total length of the device, \( l \), is of about 25\,mm (see figure 3). The number of finger pairs, \( n \), is 11. The theoretical initial capacitance, \( C_0 \), is:

\[
C_0 = C_{S0} + C_p = (2n-1)p \frac{ah}{g} + C_p
\]

where \( C_p \) is the equivalent parasitic capacitance, \( n \) is the number of pairs of electrodes, \( h \) is the thickness of the dielectric material, \( p \) is the permittivity, \( a \) is the common length between two adjacent electrodes, and \( g \) is the space gap between two adjacent electrodes. As explained in Matsuzaki et al., the electric field passing from one electrode to the adjacent ones through the substrate is much larger and more dominant than the electric field crossing between fingers through air gaps [20]. This is mainly due to the substrate’s electric permittivity being larger than air’s, especially that the substrate is almost 300 times thicker than the PEDOT/PSS film. If the electric field lines would only pass through air, the initial capacitance of the printed capacitor, will be \( C_{S0} = 0.5578\, {\text{fF}} \) (\( n=11 \) pairs, \( a=10\,\text{mm} \), \( h, 500\,\text{nm} \) approx., and \( g=500\, \mu m \)). In reality most of the electric field is passing through the substrate, which has a relative dielectric constant \( \varepsilon_r=3.5 \), resulting in a static capacitance estimated to be 1.12\, {\text{pF}} (same geometric dimensions as before, but \( p \) is considered the dielectric constant of the substrate (paper) and \( h \) the thickness of the substrate). Finite element simulations using Comsol Multiphysics (Comsol AB) have confirmed that the concentration of charges is localized much more in the conductive part diffused into the substrate than in the part above, in the air (Figure. 6). The associated electric
flux density, $D$, is therefore concentrated in the substrate as well, especially around the bottom corners.

**Figure 5.** Simplified schematic of the printed interdigitated capacitor

**Figure 6.** Distribution of surface electric field displacement, $D$ (C/m$^2$). $D$ is, also, known as the flux density
**Figure 7.** A printed interdigitated capacitor. Solder paste is used to attach the connecting wires to the sensor

**Figure 8.** Organic based polymer printed interdigitated capacitor
2.2.3 Printing of Piezoresistor on Polyimide

Similar procedures were followed to print devices on polymide, using a nozzle with 40 microns diameter. For this study, two different samples were printed. The drop spacing was set to 50µm for this case, as we were aiming for a smoother and better quality film. We relied on printing multiple layers in order to achieve a thicker film. We were anticipating a track width of 300µm, length of 13.125mm, and spacing of 0.6 mm between two adjacent parallel tracks (see Figures 9 and 10). The procedure was repeated 10 times so that we achieve much thicker tracks. Thickness of tracks, after the 10\textsuperscript{th} print, was about 3.5 microns as seen in topographical graphs as seen in figures 11, 12, and 13. A thicker film can be obtained by repeating the printing procedure over the same pattern in successive layers. Depending on the nature of the substrate, PEDOT:PSS may diffuse into it; unlike in our previous work, we did not face this problem in this experiment as the polyimide, PI, is originally hydrophobic and we had to make it more hydrophilic by plasma treatment. This will, ultimately, affect the spread out of the droplet and the thickness of the printed film. For this experiment, placing the substrate in the plasma cleaner for 3 minutes at high power was sufficient for the printed ink solution to preserve its pattern and ensure a strong adhesion with the polyimide substrate. Another source of variability, which has been investigated before, is the speed of the stage (the nozzle is fixed and the stage is moving). The speed was set to 24mm/sec as we were aiming for better quality [11].

The inkjet parameters for this process are: 40µm diameter nozzle, driven by rectangular pulses of 42V amplitude and 200µs pulse width, with a stage speed set to 24mm/sec. If these sensors were to be fabricated using conventional fabrication techniques, the lift-off process would have been used [8], [47]. After printing, it is recommended to anneal the devices for 90 min at a temperature of 80°C for the solvents to evaporate, so that the polymer molecules join and form
stronger bond, which leads to a higher conductive tracks. Intuitively, Annealing the pattern after each print would have made the printed ink more conductive and the cross-sectional area to be more uniform; in our case, PEDOT:PSS was printed on top of a wet layer which led to wider tracks and more parabolic cross-sections (Figures 11,13,14).

**Polyimide film** is a hydrophobic material and, in order to enhance the adhesion between the ink and the film, the substrate was treated in a plasma chamber. The polyimide substrate used in this experiment was 4cm x 0.8cm and had a thickness of 500µm.

For the piezoresistor (figure 9 and 10), we anticipated a gap between the two parallel tracks of 600 µm, the length of each track of 13.125mm, and each track width to be 300 µm. Nevertheless, experimental measurements of the surface topography using white light interferometry (Wyko equipment) (figures 11,12,13,14) made at 8 different positions on the printed pattern have shown a deviation from the theoretical predictions. The average track length has 13mm.125, the average track width has 450 µm, with a thickness of 3.56 µm (for 10 consecutive PEDOT printed layers), while the average measured gap was of 480 µm (slightly exceeding 500 µm at some locations). We notice that the cross-sectional area of the PEDOT tracks are actually parabolic. This is due to the fact the 10 layers of PEDOT were printed after each other, without any annealing process between two consecutive prints. The annealing phase (80°C for 30min) took place after the last (10th print). The width of each track is not completely uniform. The width and thickness of the tracks had been smoother and more uniform if the printed structure would have been annealed after every print; this requires moving the substrate from its place after each printed layer, and risks to destroy the proper alignment between consecutive prints.

Nevertheless, results obtained were not satisfactory and another sample had to be prepared. All procedures followed to print the previous sample were followed in here except that it was
repeated 3 times only and the post-processing procedures constituted of annealing the piezoresistor at $200^\circ$ C for 10 minutes. As seen from the topographic image in figure 14, the width of the track was about 650 mm and the thickness was around 2.47 $\mu$m. the length of each track was still 13.125 mm.

**Figure 9.** Printed PEDOT:PSS based piezoresistors on polyimide and rubber substrates
Figure 10. Schematic model of piezoresistor printed

Figure 11. Topographical image showing two parallel tracks with the gap in between them
Figure 12. Topographical image showing the width of a track. Coffee ring effect is noted in this image.

Figure 13. Plot showing the dimensions of one of the tracks from sample 1.
2.3 3D Movable Structures

In an attempt to fabricate the very first fully printed 3D movable structure, we intended to print a capacitive micromachined ultrasound transducer (CMUT), which is used in medical imaging and normally fabricated in silicon using conventional microfabrication techniques. We were not able to fully print the planned device as the sacrificial layer used, zinc oxide, was porous and not smooth, making it difficult to print PEDOT:PSS on to top of it. We had to use instead the solid, but stiff, kapton tape (a polyimide film) as the membrane. Despite the non-uniformity of printed
ZnO layer, the layer was sufficient to keep the kapton membrane from going down into the gap between the PEDOT:PSS walls.

The fabrication process is illustrated in figure 15. First, a 30µm wide back-etching hole is drilled through a 100µm thick steel substrate. The purpose of this hole is to allow the etchant to access and attack the sacrificial layer later in the process. As an experiment, two similar devices were fabricated, with and without a hole in the substrate. In the device with a hole, the sacrificial layer was etched away in a matter of seconds, as expected; the sacrificial layer of the second device (no substrate hole) was not removed, even though the two devices were placed in the same container containing the appropriate etchant overnight. After drilling the hole(s), droplets of PEDOT:PSS were printed around the hole(s) forming the structure holding the membrane. As printing horizontally and vertically is much easier and more feasible than printing circular structures, a rectangular structure for a CMUT cell was chosen, with 400µm length and 330µm width. We have used the 40µm nozzle and follow the exact same printing procedure used previously to print the devices in section 2.2; 100µm spaced insulating PEDOT:PSS droplets were printed to generate non-overlapping dots, which facilitates etching the sacrificial layer by allowing the etchant to attack the sacrificial layer from all sides and through the PEDOT:PSS walls (see figures 16 and 17). The PEDOT:PSS pattern was printed 10 consecutive times, to generate a layer thickness of almost 5µm (figure 20). The different substrate explains the thickness difference compared to piezoresistor printing. In section 2.2.3, the printing was also repeated 10 times, but the film’s thickness was around 3.5µm.
After the PEDOT:PSS printing, a zinc oxide layer was indirectly deposited to fill the gap between the printed walls, through inkjet printing of Zn(O\textsubscript{2}CCH\textsubscript{3})\textsubscript{2}. 0.5M zinc acetate, an aqueous solution, was printed. This solution, when baked at 200°C for only 10 minutes, transforms into zinc oxide solid crystals. The transportation of the substrate from the printer’s...
stage to the heater may cause a slightly shift in the printed solution; thus, the resulting ZnO polycrystalline layer was not as uniform and smooth as the typical printed films (figures 18 and 19). Larger variations in thickness were observed. Even though only one layer of zinc acetate was printed, the thickness of the sacrificial layer exceeded the thickness of the 10 layer PEDOT:PSS walls. After printing the sacrificial layer and sticking the kapton tape on top of the substrate – covering the whole printed structure – one layer of conductive PEDOT:PSS was printed on the top of the insulating membrane; the steel substrate and the top layer PEDOT:PSS are the CMUT cell electrodes, connected to the voltage supply by gold wires. After the last inkjet printing phase, the substrate was placed in a beaker containing 5% sulfuric acid solution. The membrane is typically released in a matter of seconds.

We encountered a problem when we tried to measure the thickness and other topography characteristics the CMUT cell; the high reflectivity of the steel substrate have impeded the optical measurements using white light interferometry (Wyco equipment). We had to use the more complicated Filmetrix F20, a thin film measurement system, (San Diego, CA, USA), which gives less 2D and 3D information than the Wyco system.
Figure 16. PEDOT:PSS walls around μEDMed holes

Figure 17. Illustration of PEDOT:PSS walls printed
**Figure 18.** Cross-sectional view of a printed zinc oxide layer

**Figure 19.** Another cross-sectional view of a printed zinc oxide layer
Figure 20. A cross-sectional view of a PEDOT:PSS wall
Chapter 3

Stress Sensing Structures

The general mechanical and electrical characteristics of printed stress sensing structures were studied. We can divide the experiments conducted into two major parts: structures printed on paper and structures printed on polyimide. The separation is not dictated by the nature of the substrate, but rather by the orientation of the applied forces on the two sets of sensors. The piezoresistor and interdigitated capacitors printed on paper were subject to compression forces by placing the initially flat surface on different cylindrical surfaces, to induce in this manner bending moments through the curvature of the substrate. The second set of sensors was subject to tensile forces applied to them longitudinally and transversally. A Bose strain-stress equipment was used to stretch the substrate and provide well-controlled calibrated strain and stress measurements. It was difficult to have similar measurement setups for the two cases. The sensors printed on paper were tested using simpler and less precise procedures compared to the polyimide-substrate sensors. Nevertheless, in these experiments, we were able to study the characteristics of the substrate, the printed pattern, and performance of the whole device. The second set of experiments yielded more accurate and quantitative results, which allowed us to investigate and study further the electrical and mechanical characteristics of PEDOT:PSS.
3.1 Sensors Printed on Paper

After printing silver and polymeric tracks to study the potential of the printers we have in the lab, we started printing devices on paper as a first attempt. We were able to print piezoresistors on flexible substrates. The inkjet printed interdigitated capacitor for stress sensing, made in our lab, was the first of its kind reported in the scientific literature [11].

3.1.1 Experiment Setup

The general characteristics of the printed sensors have been firstly examined in zero-stress conditions, by measuring the variation of their resistive and reactive components with frequency (10Hz to 20kHz) using the NI ELVIS II board (National Instruments, Texas, USA). Then the printed sensors were placed on cylindrical pipes with different curvatures, to generate strain through bending moments. In order to measure the change in resistance (and capacitance for the printed capacitor), commercial strain gauges (OMEGA, UK) were used as reference when taking measurements under mechanical stress conditions. The purpose of using these strain gauges was to estimate the strain due to variation in substrate’s curvature by:

$$GF = \frac{\Delta R}{R \cdot \varepsilon}$$  

Eqn. 2

where $\Delta R$ is the change in resistance, $R$ is the initial resistance, $\varepsilon$ is the strain, and $GF$ is the gauge factor of the commercial sensor. According to the manufacturer, $GF$ is equal to 2.
R was measured to be 366.932 Ω. The change of resistance was calculated by measuring the resistance of the strain gauges (fixed on cylinder surface), by connecting it into a Wheatstone bridge (Fig. 22). R₁ and R₃ were chosen to be 1000 Ω, while R₂ has 360 Ω. The resistance of the strain sensor, Rₓ, was calculated by the following equation:

\[ V_{out} = \left( \frac{R_x}{R_x + R_3} - \frac{R_2}{R_1 + R_2} \right) V_{in} \]  

Eqn. 3

After estimating the strains induced and relating them to different pipes’ curvatures accordingly, piezoresistors and then interdigitated capacitors were placed on the same pipes and their resistance and capacitance, respectively, were measured.
The zero-strain impedance of the piezoresistor was measured (placed on flat surface). The zero-strain conditions corresponds to an R-C parallel circuit, with both equivalent resistance and capacitance having a frequency-dependent behavior (figure 24).

3.1.2 Piezoresistor

The zero-strain impedance of the piezoresistor was measured (placed on flat surface). The resistive and the reactive components of impedance are plotted in figure 23. The sensor can be considered as purely resistive up until a frequency of about 1kHz, but a refined model needs to be considered for higher frequencies operation. A simple physical model of the devices in zero-strain conditions corresponds to an R-C parallel circuit, with both equivalent resistance and capacitance having a frequency-dependent behavior (figure 24).

Figure 22. Wheatstone bridge configuration for strain measurement. $R_1 = R_3 = 1000 \Omega$, $R_2 = 360 \Omega$. and $R_x = 360 \Omega$ denotes the resistance of the sensor.
Figure 23. Resistive and reactive components of the impedance of the piezoelectric sensor vs. frequency

Figure 24. Schematic of the equivalent (frequency-dependent) RC circuit.
If we consider the parallel R-C configuration as equivalent circuit (Figure 24), then:

\[
Z_{\text{total}} = \frac{1}{\frac{1}{R} + \frac{1}{j\omega C}} = \frac{R}{1 + R^2 \omega^2 C^2} - j \frac{\omega CR^2}{1 + R^2 \omega^2 C^2}
\]  

Eqn. 4

where \( Z_{\text{total}} \) is the total impedance of the sensor, \( R \) and \( C \) are the resistance and the capacitance of the sensor, respectively, at a specific angular frequency, \( \omega \). In this configuration, a least mean square error fit provides the following relations:

\[
R_{[\text{M}\Omega]} = 18 - 0.000062 \cdot f_{[\text{Hz}]}
\]  

Eqn. 5

\[
C_{[\text{pF}]} = 3.3 - 0.000049 \cdot f_{[\text{Hz}]}
\]  

Eqn. 6

where \( R \) is the resistance, \( C \) is the capacitance, and \( f \) is driving frequency for the R-C circuit.

According to equations (5) and (6), \( R \) is approximately 18 M\( \Omega \) and \( C \) is approximately 3.3 pF.

**Figure 25.** Best-fit line of the resistor in the RC circuit vs. frequency.
A second experiment was conducted to detect the change in resistance with strain. As the strain increased, resistance decreased (for this particular orientation of the strain gauge). This is consistent as well with the measurements on the reference strain gauge from Omega (figure 27). The gauge factor for the inkjet resistor on paper is not constant with strain, as in the case of the reference strain gauge. For strain up to 0.015%, the gauge factor, GF, is about -81.04 and then it changes significantly to about -280. GF is expressed by:

$$ GF = \frac{\Delta R}{R} \cdot \frac{1}{\varepsilon} \quad \text{Eqn. 7} $$

where $\Delta R$ is the change in resistance, $R$ is the initial resistance, and $\varepsilon$ is the strain. Nevertheless, the high gauge factor cannot be attributed only to dimensional changes induced by the stress (as in the case of metallic strain gauges), but rather to a piezoresistive behavior of the PEDOT:PSS.
film. The specific anisotropic characteristics of piezoresistive behavior are analyzed later on in the present work.

![Graph](image)

**Figure 27.** Change in resistance over initial resistance vs. strain.

### 3.1.3 Interdigitated Capacitor

In the case of the capacitive sensing, under zero-strain conditions, the resistive component decreases with frequency, (similarly with the piezoresistor device), but the reactive component increases (figure 28). A similar RC circuit is used as a first-order behavioral model, with the capacitive behaviour dominant in the desired frequency range.
Figure 28. Frequency dependency of the resistive and reactive components of the impedance of the interdigitated electrode sensor vs. frequency.

By following the same steps as before, the RC model is plotted (figure 28). The capacitance is estimated to be around 8.5-8.6pF, for frequency lower than 1200Hz (figure 29). The frequency dependence of the zero-strain capacitance can be represented by a best-fit law:

$$C_{[pF]} = 8.6 - 0.000024 \cdot f_{[Hz]}$$

Eqn. 8

where C is the capacitance and f is the frequency. The resistance has a stronger dependency of frequency, but with a large DC value, negligible for practical measurement circuits (figure 30). Following the zero-strain characterization, the change in capacitance with strain was measured (figure 32).
Up until strains of 0.03%, the GF was almost constant and equal to -68 and then it rapidly changed to about -375. For capacitors, GF is expressed by:

\[ GF = \frac{\Delta C}{C} \cdot \frac{1}{\varepsilon} \]  

Eqn. 9

where \( \Delta C \) is the change in capacitance, \( C \) is the initial capacitance, and \( \varepsilon \) is the strain.

**Figure 29.** Best-fit line of the capacitor in the RC equivalent circuit vs. frequency.
Figure 30. Resistive component of the RC equivalent circuit vs. frequency.

Figure 31. Capacitive component of the RC equivalent circuit vs. strain.
3.1.4 Discussion

The goal of these experiments was to show the feasibility of using inkjet-printed conductive polymers for sensing the strain in the flexible substrate they are printed on. Large variations in resistance (for piezoresistors) and capacitance (for the interdigitated structures) are induced by small changes in the strain. The PEDOT:PSS has a lower intrinsic conductivity, which deteriorates the quality of the capacitive sensor and increases the nominal (zero-strain) resistance of the strain gauge. Nevertheless, the large gauge factor obtained for the resistive sensing suggests a piezoresistive behavior of the PEDOT:PSS material. The large nominal resistance is an advantage for DC-operated strain gauges, as it significantly decreases the power consumption, in comparison with equivalent metallic strain gauges.
3.2 Piezoresistor Printed on Polyimide

A next phase was to focus on analyzing the piezoresistive behavior of inkjet-printed PEDOT:PSS, using better structures, printed on polymide. Previous experiments have induced stress into the devices through bending; while the procedure is correct, it offers only a limited set of data points (corresponding to the distinct values of the curvature of the cylinders used), and the induced strain increases linearly with the distance from substrate (non-uniform across the thickness of the device). A more accurate investigation into PEDOT:PSS piezoresistivity has used a calibrated stress-strain equipment for this research phase. In this section, except where it is explicitly mentioned otherwise, sample 2 (from section 2.2.3) was used in experiments.

3.2.1 Equipment and Setup

A measurement setup based on ELVIS II board (National Instruments) was used to characterize the electrical response of the sensors; in addition, a mechanical analyzer, ElectroForce 3100 test instrument (Bose, USA), courtesy of Dr. John Madden’s lab, was used to apply a calibrated stress on substrates and to measure the induced strain. Both the ELVIS II board and the mechanical analyzer were used together in a hybrid measurement system setup. An alternative setup, employed in a previous work, has used substrates mounted in mechanical clamps and weights applied at one end in order to stretch it [55].

For the geometric (topographic) characterization of the inkjet printed layers, an optical profiling system, based on white light interferometry, has been used Wyko NT1100 Optical Profiling System (Veeco, Tucson, AZ, USA) provides accurate, non-contact surface metrology at sub-nanometer vertical resolution. This equipment was courtesy of Dr. Boris Stoeber’s lab.
The test-bench simultaneously measures the electrical resistance and the induced displacement by the application of calibrated longitudinal and transversal tensile forces. The Bose mechanical analyzer is used to programmatically apply and record a calibrated stress or strain, while a National Instruments Elvis II board is used to simultaneously measure the resistance of the patterned structure. Figures 33 and 34 illustrate the full experimental setup.

Treating the polyimide substrate with plasma (plasma cleaning) led to a more linear stress-strain behavior and considerably reduces the hysteresis. The main purpose of plasma treatment is to make the substrate more hydrophilic, so that printed PEDOT:PSS (or any other printed ink) will have a stable adhesion onto it. The polyimide substrate was plasma treated by plasma cleaner (Harrick Plasma PDC-001), with RF adjustable power, which generates plasma from compressed air (figure 35). Treating our polyimide substrate at high power for 10 minutes was sufficient to ensure a good hydrophobic substrate.
Figure 33. Experiment setup: a Computer #1 controlling the mechanical analyzer and recording stress and strain; b Mechanical analyzer; c NI Elvis II board; d Computer #2 recording resistance
**Figure 34.** A close-up shot to the mechanical analyzer: a The substrate being tested; b Contacts; c Clamps to hold the substrate

**Figure 35.** Plasma cleaner [48]
3.2.2 Hysteresis and Young’s Modulus of the Whole System

A better mechanical characterization of the inkjet printed PEDOT:PSS was necessary, as resulting material parameters (e.g. Young’s modulus) may be different from their values for bulk samples.

The losses and relaxation of the polyimide substrate, in addition to slippage when the substrate goes to the relaxation mode, lead to an observed hysteresis in the stress-strain behavior (figure 38). Sample waveforms of the applied force and the measured displacement are illustrated in figures 36 and 37, respectively. The strain-stress graph in figure 39, obtained for a plain polyimide substrate, shows a slight nonlinear but reproducible strain-stress dependence.

![Figure 36. Waveform of force applied in experiment](image-url)
The initial Young's modulus of the plain substrate was about 2.02 GPa (as calculated from a linear fit on the loading section presented in figures 39 and 40). Young’s modulus is the slope of the linear stress-strain graph. If uncertainties are to be included, the measurement of the length and the width of the substrate has an uncertainty of 1 mm (the 25μm thickness is controlled by the manufacturer, and has a smaller relative uncertainty). The force and displacement measurements have uncertainty of 1%, due to the measuring equipment used. As a result, and after taking in consideration all uncertainties, $E_{\text{substrate}} = 2.02 \text{ GPa} \pm 20.33\%$ or $2.02 \pm 0.41 \text{ GPa}$. This is based on a sensitivity analysis procedure:

$$E = \frac{\sigma}{\varepsilon} = \frac{F}{W \cdot T} \frac{L}{\Delta L} \Rightarrow \delta E = \delta F - \delta W - \delta T + \delta L - \delta (\Delta L)$$

According to the manufacturer, the Young’s modulus of this thin film ranges between 2.0 GPa (at 200°C) and 2.5 GPa (at 23°C), which validates the outcome of this experiment [46].
Measuring the load – displacement response to longitudinal (force applied vertically) and transversal (force applied horizontally) stress after the PEDOT:PSS has been printed lead to $E_{\text{trans}}=2.678 \pm 0.48$ GPa and $E_{\text{long}}=2.088 \pm 0.33$ GPa respectively (estimated by linear fit from the large strain measurement points in figures 41 and 43). These values for Young’s modulus were extracted from the experimental stress-strain values on the loading curve (for $\varepsilon \geq 0.0012$) and they correspond to the Young’s Modulus, $E$, of the whole system – i.e. for both the printed PEDOT:PSS film and the polyimide substrate. Nevertheless, it is clear that the strain varies over a smaller range in this case, compared to the one used for the elastic characterization of the polyimide substrate alone. This is the undesired effect of being necessary to change the measuring equipment, after the initial Bose tensile characterization machine broke down.

Because most of the data correspond rather to the initial small strain curve (see Fig 41 versus Fig 40), in order to get a fair comparison, only the measurement points with a strain larger than 0.0012 have been considered in the estimation procedure. This negatively affects the uncertainty of this measurement phase. The Young’s modulus of the assembly polyimide substrate – PEDOT printed structure is necessary for the estimation of the elastic modulus of PEDOT:PSS structure, which will be used in the estimation of the piezoresistive coefficients. The propagation of the uncertainty through the computation chain means that the experimental estimate of the Young’s modulus for the PEDOT:PSS structure has a larger margin of error. Consequently, both the value of this estimate and the values reported in the scientific literature have been used as input data in the procedure yielding the piezoresistive coefficients for PEDOT:PSS and their uncertainty range.

In order to compare the substrate-only elastic characterization (using the initial Bose equipment) with the new measurements, Fig. 42 superimposes a straight line with a slope of $2.02 \cdot 10^9$ GPa
(corresponding to the Young’s modulus of the original substrate) on the measurements obtained using the second experimental setup. This confirms that the points to be used for the estimation are a subset of the measurement points, corresponding to larger strains.

Figure 38. Stress-strain relation of the original substrate
Figure 39. Stress-strain relation of a small linear segment of the original substrate

Figure 40. Stress-strain relation of a small linear segment of the original substrate
**Figure 41.** Stress-strain relation in the longitudinal direction

**Figure 42.** Stress-strain relation in the longitudinal direction
3.2.2.1 Calculating Young’s Modulus of PEDOT:PSS

Piezoresistive properties of PEDOT:PSS have been previously analyzed in the literature [8], [50]-[55], but not for ink-jet printed structures. A justified question is therefore to what extent the reported behavior is maintained when a PEDOT:PSS strain gauge is fabricated through printing. Comparing the thickness of the printed film (<2.4µm) to the thickness of the substrate (25µm) allows us to assume that the printed structure will follow the expansion and contraction of the substrate itself. Thus, for our study, we will assume (like in the other presented studies in the literature) the Poisson’s ratio of PEDOT:PSS is equal to the Poisson’s ratio of polyimide, ν=0.34 [46]. Previous literature reports studied the piezoresistivity of PEDOT:PSS fabricated using conventional techniques such as spin coating and lift-off [8],[50]-[55]. In this paper we
estimated the values of piezoresistive parameters for inkjet printed PEDOT:PSS, to prove that the material does preserve its piezoresistive characteristics after printing. Before this experiment, we were able to show that PEDOT:PSS does preserve its piezoresistivity when printed, in the case of bending-induced strain, but without including a detailed estimate of the piezoresistive coefficients[11].

In Lang et al. [8], Poisson’s ratio was assumed \( \nu = 0.35 \) and GF of 0.48. In Latessa et al. [51], GF=17.8. In Calvert et al., the GF ranged from -5 to -20 [52]. In Lang et al., GF was estimated to be 0.8 [54]. In Schweizer’s thesis, “Electrical characterization and investigation of the piezoresistive effect of PEDOT:PSS thin films,” a comprehensive study was done on conventionally fabricated films and the dependence of piezoresistive coefficients and GF with variation in temperature have been studied [55]. Again, Poisson’s ratio was assumed to be the same as for the substrates. The present approach takes a different path, where the modulus of elasticity is estimated from the experimental data, in addition to inferring the values of the piezoresistive coefficients for inkjet printed PEDOT:PSS. Due to rather large uncertainty associated with this estimation, both this value and the value reported in Lang et al.[9] \((E = 2.8 \pm 0.5 GPa)\) have been used for computing the piezoresistive coefficients from the experimental results.

The test-bench allowed the application of both longitudinal and transversal strains (relative to piezoresistance orientation) and the simultaneous measurement of the resulting stress. In both cases, only positive controlled displacements have been applied (i.e. stretching out the substrate). Negative strains applied to the structure induce a different state of stress (bending the substrate), and therefore a different model is necessary to extract the piezoresistance coefficients from the stress distribution. When the substrate is bent (negative displacements), other factors, such as
non-uniform strain applied to the substrate and the radius of curvature, have to be taken in consideration.

In longitudinal stress case, the electrical resistance increased almost linearly with the positive strain applied; transversally, the electrical resistance decreased. The measured zero-strain resistance value of the printed piezoresistors is $R=2433 \, \Omega$. The gauge factor, $GF$, is identified as the ratio of relative change in electrical resistance to the mechanical strain, $\varepsilon$:

$$GE = \frac{\Delta R}{R} \cdot \frac{1}{\varepsilon}$$  \hspace{1cm} \text{Eqn. 10}

For the stretch-out case, $GF$ was 3.63 and -0.41 longitudinally and transversally respectively. This shows that the printed sensor's resistance increases at a faster rate when strain is applied longitudinally to the substrate, as expected if we would rely on a dominant geometric deformation.

The estimation algorithm has two computation steps. In the first one, a simplified model of the assembly polymide substrate – PEDOT:PSS printed structure is used to estimate the Young’s modulus of the PEDOT:PSS inkjet printed resistor from the experimental measurement points. In the second step, the model of the change in resistance due to strain considers both the geometry and piezoresistive effects. The longitudinal and transversal piezoresistive coefficients are computed from the experimental points after separating the geometric distortion contribution from the overall relative resistance variation. In order to do this, the elastic properties of PEDOT:PSS material must be used. Both the experimentally estimated value (outcome of the first estimation step) and a reference value [9] for $E_{\text{PEDOT:PSS}}$ have been used.

In order to simplify calculations for the estimation of the elasticity modulus (first step), we assumed that the whole piezoresistor is just one thin uniform film (i.e. as if there are no gaps between tracks) (figure 44). This model is suitable for a first-order approximation, but it will
need improvements in order to account for elastic behavior differences between longitudinal and transversal axial loading. The printed PEDOT:PSS covers an area of 12 mm by 13.125 mm. The whole polyimide substrate is 2.5 cm by 2.7 cm.

In the first order model, the composite structure has two components: component 1 corresponds to the polyimide substrate, while component 2 corresponds to the PEDOT:PSS printed film. We assume a perfect adherence of the thin film to the substrate, so the displacement compatibility constraint means that the applied strains on both components are the same \( \varepsilon = \varepsilon_1 = \varepsilon_2 \). The resulting axial force, \( F \), is therefore divided between a force component acting upon PEDOT:PSS region, \( F_1 \), and a force component acting upon the substrate, \( F_2 \).

**Figure 44.** The printed piezoresistor is assumed to be one uniform thin film; arrows show the axial force.
As a result, we can estimate the Young's modulus of the printed PEDOT:PSS film using the following relations and using the experimentally measured values for $E_1$ and $E_{\text{whole}}$:

\[ F = A_1 \sigma_1 + A_2 \sigma_2, \]
\[ F = A_{\text{whole}} E_{\text{whole}} \varepsilon_{\text{whole}} = A_1 E_1 \varepsilon_1 + A_2 E_2 \varepsilon_2 \]

with $\varepsilon_{\text{whole}} = \varepsilon_1 = \varepsilon_2$

\[ \therefore \quad E_{\text{whole}} = \frac{A_1 E_1 + A_2 E_2}{A_1 + A_2} = 2.088 \text{ GPa} \]

$A_1 = 2.5 \text{ cm} \cdot 25 \mu\text{m} = 6.25 \cdot 10^{-7} \text{ m}^2$

$E_1 = 2.02 \text{ GPa}$

$A_2 = \frac{2}{3} \cdot 2.47 \mu\text{m} \cdot 1.2 \text{ cm} = 1.976 \cdot 10^{-8} \text{ m}^2$

\[ \therefore \quad E_2 = \frac{E_{\text{whole}} \cdot (A_1 + A_2) - E_1 A_1}{A_2} = 4.24 \text{ GPa} \]

Here $A_1$ is the cross-section area of the polyimide substrate, $E_1$ is the Young's modulus of the original substrate prior to any printing but after treatment, $E_2$ the Young's modulus of PEDOT:PSS film to be calculated, $A_2$ is the cross-section area of the PEDOT:PSS film (parabolic cross-section, obtained from the topographic measurements), and $E_{\text{whole}}$ is the Young's modulus of the whole system (previously measured).

An estimation of the Young’s modulus for PEDOT:PSS based on this model and the experimentally measured load points lead to $E_{\text{trans}} = 2.678 \pm 0.48 \text{ GPa}$ and $E_{\text{long}} = 2.088 \pm 0.33 \text{ GPa}$ for longitudinal and transversal case, respectively. Despite the large uncertainty involved, caused by both model simplification (an uniform PEDOT:PSS area on the top of the kapton polyimide substrate) and a reduced number of valid points (only the larger strains subset was selected from the initial measurement points), the obtained values are comparable with previously reported characterization (in [9], $E_{\text{PEDOT:PSS}} = 2.8 \pm 0.5 \text{ GPa}$, strongly affected by the relative humidity). In consequence, in the estimation of the piezoresistance coefficients we will use the reference values for the mechanical properties of PEDOT:PSS: $E \approx 2.8 \pm 0.8 \text{ GPa}$, $\nu \approx 0.34 \pm 0.03$ [9].
3.2.3 Electrical Conductivity Measurement

Before applying any force to the substrate, the measured resistance was \( R=2433 \, \Omega \) (zero-strain resistance). Knowing that the piezoresistor is formed of 4 equal tracks connected in series, we can assume that the resistance is equally distributed among these tracks (about 608 \( \Omega \) each) as the resistance of the pads is negligible compared to the resistance of the tracks. The resistance, \( R \), is proportional to resistivity, \( \rho \), and length of the track, \( L \), while it is inversely proportional to the cross-sectional area of the track, \( A \).

\[
R = \rho \cdot \frac{L}{A} \quad \text{Eqn. 11}
\]

As observed in the topographical images, the cross section area of printed PEDOT:PSS has a structure close to that of a parabolic shape, with a total value \( A=L_{\text{base}} \times (2/3 \times \text{Height}) \). The average thickness of the printed film has 2.47 microns, and the base has 650 microns. We have, therefore, for one track (i.e. \( L=13.125 \, \text{mm} \) and \( R=608 \, \Omega \)) the following relations (where \( L \) is the length of the track, \( w \) is the width of each track, and \( h \) is its thickness):

\[
\rho = \frac{2}{3} \cdot \frac{Rwh}{L}
\]

\[
\rho = \frac{2}{3} \cdot \frac{608.25 \, \Omega \cdot 650 \, \mu m \cdot 2.47 \, \mu m}{13.125 \, mm}
\]

\[
= 0.00496 \, \Omega \cdot cm
\]

\[
\therefore G = \frac{1}{\rho} = 201.6 \, S/\text{cm}
\]

The manufacturer mentions in the specifications sheet that, for this specific type of PEDOT:PSS, the electrical conductivity is at least 300 S/cm. Most probably the repeated overprinting (3 times) of the PEDOT:PSS layer has increased the concentration of the conductive material and strengthen the bonds. The printed sensor was placed in the oven for about 10 minutes at a
temperature of 200°C only after the last (3\textsuperscript{rd}) print iteration. An alternative procedure would be to thermally cure the structure immediately after each layer is printed. Then the electrical conductivity would have been higher, but it would have required a much better alignment control in order to get a reproducible final structure – few microns misalignment could even result in short circuits between adjacent tracks.

In section 2.2.3, we mentioned that two samples have been used in experiments. As mentioned earlier, sample 2 presented more reliable results, and served as base for the extraction of the piezoresistive coefficients. Sample 1 is a 10-layer printed piezoresistor, with a zero-strain resistance of $R=3054\ \Omega$. Knowing that the piezoresistor is formed of 14 equal tracks connected in series, we can assume that the resistance is equally distributed among these tracks (about $218\ \Omega$ each) as the resistance of the pads is negligible compared to the resistance of the tracks.

The average thickness of the printed film has 3.56 microns, and the base has 450 microns (as seen before in graphs 11-13). We have, therefore, for one track (i.e. $L=13.125\ mm$ and $R = 218\ \Omega$) the following relations proving that the electrical conductivity of the printed resistor increases with the number of inkjet printed PEDOT:PSS layers:

\[
\rho = \frac{2 \cdot Rwh}{3 \cdot L} \quad \rho = \frac{2}{3} \cdot \frac{218.143\Omega \cdot 450\mu m \cdot 3.56\mu m}{13.125mm} = \frac{0.001775\Omega \cdot cm}{3} = \rho
\]

\[
\therefore G = \frac{1}{\rho} = 563.361\ S/cm
\]
3.2.4 Piezoresistive Coefficients

The dependence of electrical resistance on mechanical strain is a characteristic of some metals and semiconductors. Nevertheless, when a mechanical force is applied to one of these materials, change in resistance may be due to two types of distinct phenomena: piezoresistive effects (the dependence of the resistivity on the strain) or geometrical effects resulted from physical deformations of the structure (changes in dimensions). According to equation 11, a change in resistance, \( \Delta R \), can be due to a change in resistivity, \( \rho \), and/or a change in geometry (including changes in length, \( L \), width, \( W \), and thickness, \( T \)):

\[
\frac{\Delta R}{R} = \frac{\Delta \rho}{\rho} + \frac{\Delta L}{L} - \frac{\Delta W}{W} - \frac{\Delta T}{T}
\]  

Equation 12 can be divided into two parts: geometric and piezoresistive effects. The geometric effect is dependent on Poisson’s ratio, \( \nu \), and the Young’s modulus of the material. Poisson’s ratio describes the change in dimensions relative to the direction of the applied force (equation 13 and figure 46). On the other hand, the piezoresistive aspect is characterized through a longitudinal piezoresistive coefficient, \( \pi_l \), and a transversal piezoresistive coefficient, \( \pi_t \).

Equation 14 shows the relation between the change in resistivity, the piezoresistive coefficients, and the stress/strain applied. Theoretically, at least for a pure geometric effect, the variation in resistance is much smaller when the axial force is acting transversally on the resistor, compared to the change in resistance when the same force is applied longitudinally.
The change in resistance due to piezoresistivity and geometric effects can be divided into longitudinal and transversal components. With $\varepsilon \ll 1$, introducing $L_{\text{after}}=(1+\varepsilon)L$, $W_{\text{after}}=(1-\nu\varepsilon)W$ and $T_{\text{after}}=(1-\nu\varepsilon)T$ into equation 11 and 12, will lead to the following longitudinal and transversal geometrical effects:

$$\frac{\Delta R}{R} = \varepsilon(1 + 2\nu) \quad \text{Eqn. 15}$$

$$\frac{\Delta R}{R} = -\varepsilon \quad \text{Eqn. 16}$$

In our case, the large variations in electrical resistance with the applied strain led to the conclusion that both geometrical and piezoresistive effects are present. Consequently, we have to take into account as well the longitudinal and transversal changes in resistance due to the piezoresistive effect. The two combined effects result in effective longitudinal and transversal
changes in resistance, which are characterized by equations 17 and 18 that are deduced from equations 12, 14, 15 and 16.

\[
\left( \frac{\Delta R}{R} \right)_{\text{effective}} = (1 + 2\nu + \pi_t E)\epsilon \\
\text{Eqn. 17}
\]

\[
\left( \frac{\Delta R}{R} \right)_{\text{effective}} = (-1 + \pi_t E)\epsilon \\
\text{Eqn. 18}
\]

When force was applied transversally (with an average strain of 0.000868), resistance, \( R_{\text{after}} \), decreased to 2398.8 \( \Omega \) (or 599.7 \( \Omega/\text{track} \)). This shows that PEDOT:PSS does preserve its piezoresistive properties after printing as, according to equation 16, the change in resistance due solely to geometrical effects would be \( -\epsilon = -0.000868 \), whereas the experimentally measured value was \( 0.141 \times 2433 \Omega = -0.0141 \). This suggests that the piezoresistive effect adds its own contribution to the resistance change. As a matter of fact, in this case, the piezoresistive effect contribution (-8.022 \( \Omega/\text{track} \)) is almost double in magnitude compared to the geometric effect (-0.528 \( \Omega/\text{track} \)). Graphs 47 and 48 denote, respectively, resistance vs. strain and change in resistance vs. strain with force applied transversely. It is clear that resistance decreases as transversal force applied increase and the change in resistance is almost consistent after strains of 0.005, and, thus, smaller strains are neglected when \( \pi_t \) is to be estimated. From equation 18, \( \pi_t \) versus strain dependence is illustrated in Fig. 48. As noted previously (Fig. 47), the dependence shows consistency for strains larger than 0.05. The resulting fit for the transversal piezoresistive coefficient gives \( \pi_t \in 3.3 \times 10^{-10}..3.6 \times 10^{-10} \, \text{Pa}^{-1} \) \( (\pi_t \approx 3.45 \times 10^{-10} \, \text{Pa}^{-1} \pm 4.34\%) \).
Figure 46. Resistance vs. strain with transversal force applied

Figure 47. Relative change in resistance vs. strain with transversal force applied
Longitudinal axial loading case. Longitudinal piezoresistive coefficient

We assume again a perfect adhesion to the substrate, so that both the polyimide substrate and the printed film will have identical changes in their displacement. The dimensions of the tracks themselves will suffer changes as the force is applied to the substrate. Length of each track, L=13.125mm, should increase by a factor of (1+ ε); whereas both the width, W=650µm, and the thickness, T=2.47µm, will decrease by a factor of (1- εν). These changes take place because the force is applied longitudinally, along the length of the track and perpendicular to both width and thickness.

Initially (zero-strain case), R is equal to 2433Ω; hence, each track has a resistance of almost 608.25 Ω. After applying the force, the average total resistance became 2467.6 Ω (a resistance of 616.9 Ω for each track) with an average strain of 0.000744, as an example.
The resistance increase of 8.65 Ω for each track cannot be attributed solely to the geometric or piezoresistive effect, but it is rather a combination of both. By referring back to equations 11, 13, and 15, the geometric deformation yields the following (assuming that the cross section maintains a similar parabolic shape, as seen in Figs 11, 13 and 14, after longitudinal loading):

\[
e_{\text{width}} = e_{\text{thickness}} = -\nu e_{\text{length}}
\]

with \( R_0 = \frac{3}{2} \frac{\rho \cdot L_0}{T_0 \cdot W_0} = 608.25 \Omega \)

and \( R_{\text{after}} = \frac{3}{2} \frac{\rho \cdot L_{\text{after}}}{T_{\text{after}} \cdot W_{\text{after}}} \)

with \( L_{\text{after}} = (1 + \varepsilon)L_0 \)

\( W_{\text{after}} = (1 - \nu \varepsilon)W_0 \)

\( T_{\text{after}} = (1 - \nu \varepsilon)T_0 \)

\[
\therefore \frac{R_0}{R_{\text{after}}} = \frac{L_0 W_{\text{after}} T_{\text{after}}}{L_{\text{after}} W_0 T_0} = \frac{L_0 \cdot (1 - \nu \varepsilon)W_0 \cdot (1 - \nu \varepsilon)T_0}{(1 + \varepsilon)L_0 \cdot W_0 \cdot T_0}
\]

\[
= \frac{(1 - \nu \varepsilon)^2}{(1 + \varepsilon)}
\]

\[\Rightarrow R_{\text{after}} = \frac{R_0(1 + \varepsilon)}{(1 - \nu \varepsilon)^2} = 609.0413 \Omega \]

at the same time:

\[
\left( \frac{\Delta R}{R} \right)_{\text{piezoresistive}} = \pi, E, \varepsilon = \left( \frac{\Delta R}{R} \right)_{\text{effective}} - \left( \frac{\Delta R}{R} \right)_{l}
\]

where \( \left( \frac{\Delta R}{R} \right)_{\text{effective}} = \frac{616.9 - 608.25}{608.25} = 0.01422 \)

and \( \left( \frac{\Delta R}{R} \right)_{l} = \frac{609.0413 - 608.25}{608.25} = 0.001301 \)

In this case, the total change in electrical resistance can be separated into a piezoresistive component (7.8587 Ω/track) and a geometrical deformation one (0.7913 Ω/track change).
The experimentally measured dependence illustrated in Fig. 49 shows an increase in resistance with the longitudinal strain. $\pi_l$ is plotted against longitudinal strain (larger than 0.005) in Fig. 51. As previously discussed, smaller strain values were neglected. As a result, the longitudinal piezoresistive coefficient gives $\pi_l \in -5.8 \times 10^{-10} \text{Pa}^{-1} - 4.4 \times 10^{-10} \text{Pa}^{-1}$ ($\pi_l \approx -5.1 \times 10^{-10} \text{Pa}^{-1} \pm 13.7\%$).

---

**Figure 49. Resistance with longitudinal strain**
Figure 50. Relative change in resistance with longitudinal force applied

Figure 51. Longitudinal piezoresistive coefficient [Pa$^{-1}$] vs. strain with longitudinal force applied
3.2.5 Discussion

The goal of these experiments is to show the feasibility of using inkjet-printed conductive polymers for sensing the strain in the flexible substrate they are printed on. Variations in resistance for piezoresistors are induced by small changes in the strain. The PEDOT:PSS has a lower intrinsic conductivity, which increases the nominal (zero-strain) resistance of the strain gauge. Nevertheless, the gauge factor obtained for the resistive sensing suggests a piezoresistive behaviour of the PEDOT:PSS material. Also, the value of the longitudinal and transversal piezoresistive coefficients are at the same order of magnitude of those of silicon (table 5), suggests a noticeable change in resistance with small changes in strain.

Table 3. The 3 independent piezoresistive components for (100) silicon [60]:

<table>
<thead>
<tr>
<th></th>
<th>Longitudinal</th>
<th>Transversal</th>
<th>Shear</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-type</td>
<td>-102.2</td>
<td>+53.4</td>
<td>-13.6</td>
</tr>
<tr>
<td>p-type</td>
<td>+6.6</td>
<td>-1.1</td>
<td>+138.1</td>
</tr>
</tbody>
</table>

The PEDOT:PSS was shown to preserve its piezoresistive properties after inkjet printing as thin films, by showing that the change in resistivity does exist with strain; it is the dominant effect for altering the resistance when the stress is directed either transversally or longitudinally relative to the in-plane orientaton of the printed resistor structure.

In his thesis, Schweizer calculated an average longitudinal piezoresistive coefficient to be $-5.6 \times 10^{-10} \text{ Pa}^{-1}$, and a transversal coefficient of $3.73 \times 10^{-10} \text{ Pa}^{-1}$ (or negative two thirds of the longitudinal one) [55]. In our work, they came out to be $-5.1 \times 10^{-10} \text{ Pa}^{-1} \pm 13.7\%$ and
$3.45 \times 10^{-10} \text{ Pa}^{-1} \pm 4.5\%$ respectively; this shows a good consensus regarding the piezoresistive behavior of PEDOT:PSS, even when the structure is fabricated through inkjet microprinting.
Chapter 4

Printed Movable Structure

2D inkjet printing technology has a steady progress, whether the focus is on analyzing the characteristics of some printed material or on fabricating a specific device. This is noticeable from the wide variation of applications and studies of different printed structures in numerous areas and disciplines already mentioned in the literature review in the introduction. The advances in the field of 2D printing foster a fast evolution of the 3D printing technology as well. By 3D printing, we are referring to printing layers and crossovers of materials on top of each other. The 3D printing of movable microstructures would enable a plethora of microsystem applications that can be ported to this alternative fabrication method, but the topic has not been investigated so far in the scientific literature. Only one relevant publication [36] was found targeting this topic, but the device (cantilever) was not fabricated using exclusively printing technology. As circuits printed on flexible substrates are already a reality, the addition of MEMS devices could enable many promising complex inkjet printed microsystems.

We have explored the possibility of fabricating CMUT (Capacitive Micromachined Ultrasonic Transducer) cells through inkjet printing, as a first attempt to make electrostatically actuated movable structures. Fabrication difficulties (detailed in section 2.3) have determined a change in the initial goal (a fully printed movable structure), and a hybrid approach, where a kapton tape has been used as a membrane, was instead adopted. The incipient results are nevertheless promising, as they prove the possibility of obtaining movable microstructures, electrostatically actuated, through alternative low-cost fabrication steps.
4.1 Equipment and Setup

![Figure 52: Complete experimental setup to mechanically characterize a CMUT cell [56]](image)

We have used a laser vibrometer OFV-551 (Polytec GmbH, Germany) which is attached to a micro system analyzer MSA-500 (Polytec GmbH, Germany) and a vibrometer controller OFV-550 (Polytec GmbH, Germany) to determine the resonant frequency of the membrane and whether vibration takes place when an actuation voltage is applied to the device. Figure 52 above shows a typical experimental setup used for characterizing CMUT cells, in order to measure the displacement and resonant frequencies of CMUT cells fabricated in silicon through conventional micromachining techniques (PolyMUMPS fabrication process). The measurement equipment is connected to a PC to analyze and post-process and plot the measurements, using
software provided by the supplier of the equipment, Polytec. A similar setup has been used in the present context, for characterizing CMUT samples fabricated through inkjet printing.

4.2 Theoretical Equations and Simulations

The typical operating range for CMUT arrays used for ultrasound bioimaging is of 1-20MHz. The low stiffness of the kapton membrane used in our case suggests that the resonant frequency of the inkjet printed cells will be much lower. Relevant functional relations for CMUT design and analysis have been derived and presented before in the MASc thesis of Hadi Najar [57]:

\[
\frac{\varepsilon_0 A V_{DC}^2}{2(d_0 - x)^2} = k x
\]

Eqn. 19

\[
k = \frac{16\pi E d_m^3}{3 R^2 (1 - \nu^2)}
\]

Eqn. 20

\[
f_0 = \frac{1}{2\pi} \sqrt{\frac{k}{M_0}}
\]

Eqn. 21

\[
M_0 = \rho_m d_m A (\frac{2\sqrt{2}}{(\lambda_a)_{mn}})
\]

Eqn. 22

\[
f_0 = \frac{1}{R^2} \frac{(\lambda_a)_{00}^2}{2\pi} \sqrt{\frac{E_i d_m^2}{12(1 - \nu^2)\rho_m}}
\]

Eqn. 23

where:

k = the equivalent spring constant of the membrane,

x = membrane maximum displacement,

E = the membrane Young’s modulus,
\(d_m\) = membrane thickness,

\(R\) = radius of the membrane (CMUTs’ membranes are usually circular and not rectangular as in our case),

\(\nu\) = Poisson’s ratio of the membrane,

\(A\) = area of the membrane,

\(V_{DC}\) = the applied DC bias voltage (for static actuation analysis),

\(\varepsilon_0\) = the permeability of free space,

\(d_0\) = the air gap beneath the membrane,

\(f_0\) = resonant frequency of the mode shape considered,

\(M_0\) = effective mass of the clamped membrane,

\(\rho_m\) = the density of the membrane, and

\((\lambda_{a})_{mn}\) = An non-dimensional constant corresponding to the mode shape of the membrane (where \(m\) is the number of concentric circles on the membrane and \(n\) is number of diametric lines).

For the fundamental resonance mode, \(f_0\), both \(m\) and \(n\) are equal to zero yielding a value of 3.196 for \(\lambda\) [56]. Eqn. 23, resulted from combining eqns. 20-22, gives the resonance frequency for the clamped membrane. The displacement shape is more difficult to analyze analytically, so a finite element analysis is indicated for a more accurate analysis of the rectangular CMUT cell. COMSOL Multiphysics® (COMSOL Inc., Burlington, MA, USA) was used for simulating the inkjet printed CMUT.

Table 4 contains a list of estimated parameters of the printed device, while Table 5 contains the parameters of a typical CMUT conventionally fabricated by PolyMUMPS [57]. Clearly, there are major differences in membrane thickness and Young’s modulus; in addition, there is also a large difference in the air gap between membrane and the actuation electrode on the substrate.
The printed device does not need a thin insulation layer to protect against short-circuits when the membrane will suddenly collapse under voltage actuation (pull-in operating mode), as the kapton tape is an insulating material. The differences listed above contributed to the large difference between the fundamental resonant frequencies between the simulated inkjet and Silicon-based CMUT cells.

**Table 4.** List of values of important parameters of the printed CMUT

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Value</th>
<th>Unit</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>$d_m$</td>
<td>63.5</td>
<td>µm</td>
<td>Membrane thickness</td>
</tr>
<tr>
<td>$d_0$</td>
<td>5</td>
<td>µm</td>
<td>Air/Vacuum gap distance</td>
</tr>
<tr>
<td>$f_0$</td>
<td>1.1</td>
<td>kHz</td>
<td>resonant frequency (experimentally)</td>
</tr>
<tr>
<td>$E$</td>
<td>250</td>
<td>kPa</td>
<td>Young’s modulus of the membrane material</td>
</tr>
<tr>
<td>$\nu$</td>
<td>0.34</td>
<td>------</td>
<td>Poisson’s ratio of membrane material</td>
</tr>
<tr>
<td>$\rho_m$</td>
<td>1420</td>
<td>kg/m$^3$</td>
<td>Density of membrane material</td>
</tr>
<tr>
<td>Area</td>
<td>~330*400</td>
<td>µm$^2$</td>
<td>Area of the rectangular membrane</td>
</tr>
</tbody>
</table>
Table 5. List of values if important parameters of a conventionally fabricated CMUT [56]

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Value</th>
<th>Unit</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R$</td>
<td>32</td>
<td>µm</td>
<td>Radius of the membrane</td>
</tr>
<tr>
<td>$d_m$</td>
<td>1.5</td>
<td>µm</td>
<td>Membrane thickness</td>
</tr>
<tr>
<td>$d_0$</td>
<td>0.75</td>
<td>µm</td>
<td>Air/Vacuum gap distance</td>
</tr>
<tr>
<td>$d_{ins}$</td>
<td>0.60</td>
<td>µm</td>
<td>Insulation thickness</td>
</tr>
<tr>
<td>$f_0$</td>
<td>5.851</td>
<td>MHz</td>
<td>resonant frequency</td>
</tr>
<tr>
<td>$E$</td>
<td>160</td>
<td>GPa</td>
<td>Young’s modulus of the membrane material (poly-silicon)</td>
</tr>
<tr>
<td>$\nu$</td>
<td>0.23</td>
<td>-----</td>
<td>Poisson’s ratio of membrane material</td>
</tr>
<tr>
<td>$(\lambda_{a})_{00}$</td>
<td>3.196</td>
<td>-----</td>
<td>Mode shape constant at fundamental frequency</td>
</tr>
<tr>
<td>$\rho_m$</td>
<td>2332</td>
<td>kg/m$^3$</td>
<td>Density of membrane material</td>
</tr>
</tbody>
</table>

A 3D simplified structure was modeled in Comsol, reproducing the geometry of the fabricated device (figures 53 and 54). The large rectangular block in the middle represents the 63.5µm thick membrane, while the two thinner rectangular boxes model the top electrode and the air gap between the membrane and the substrate. After setting a static deformation with a DC bias voltage of 100V applied, a coupled multiphysics analysis including structural mechanics and electrostatics has been used to determine the primary eigenfrequencies. The simulation results have indicated a fundamental resonance frequency of 9.34 KHz. The first eigenfrequency yielded a maximum displacement (at the centre of the membrane) of 3.723nm (figures 53, 56, 57).
Figure 53. 3D Model of the printed CMUT consisting of top electrode, membrane, and the air gap

Figure 54. Meshing of the model
Figure 55. Side-view of the modeled CMUT actuated at 9.34 KHz.

Figure 56. Top-view of the model showing displacements in the membrane.
Figure 57. Graph of displacement vs. the side of the CMUT zero being the centre of the membrane.

4.3 Mechanical Characterization

The main goal of this experiment was to show that it is possible to get electrostatically actuated movable microstructures fabricated through inkjet printing. A Polytec laser Doppler velocimetry system was used to detect the vibration induced in the membrane through electrical actuation. As it is apparent in figure 58, the membrane vibrates when an actuation voltage (AC and DC bias) was applied between the electrodes. Spectral characterization has revealed a measured resonant frequency larger than 1KHz, with a second eigenfrequency around 2KHz (figure 59).
Figure 58. Vibration of the membrane under electrostatic actuation (experimental characterization)

Figure 59. Frequency analysis with 100V DC bias applied together with the AC excitation voltage
4.4 Discussion

The capacitive actuation generates the displacement of the membrane, confirming the possibility of getting a movable structure (figure 58). The device is nevertheless highly imperfect – exploring the motion of the membrane under electrostatic actuation it became apparent that the kapton (polymide) tape is not properly clamped onto the printed PEDOT:PSS walls. This aspect makes difficult a comparison between finite element analysis and the measured results, but it does confirm the potential of the inkjet printing technology for the fabrication of movable microstructures.
Chapter 5

Conclusion and Future work

Inkjet printing is a versatile microfabrication technique than can be easily adapted into many disciplines to facilitate fabrication of electrical, mechanical, chemical, and biological devices and sensors. This technique can replace conventional deposition techniques as it can be considered a cheaper, less time consuming, more feasible, and environment friendly alternative. Even though this technique is pretty new compared to other techniques like photolithography, for example, numerous reliable devices have been fabricated so far.

We have presented in this work piezoresistors and interdigitated capacitors based strain gauges that were successfully printed on different kinds of flexible and rigid substrates. Even though silver nanoparticles were used in the beginning, we shifted afterwards to conductive polymers, mainly PEDOT:PSS, which proved to be a reliable, and much cheaper alternative. They provide nevertheless lower conductive traces compared with the silver nanoparticle solutions. We have explored in more detail the usability of PEDOT:PSS for fabricating microtransducers through inkjet thin films printing. It was shown that PEDOT:PSS does preserve its piezoresistivity characteristics after inkjet printing. In addition, the estimated piezoresistive coefficients shows that printed PEDOT:PSS is as sensitive as n-doped silicon and spin coated PEDOT:PSS. As we have gained experience in printing different devices, we saw the potential of advancing towards fully printing movable structures, never attempted before, to our knowledge. The trial device was
a rectangular CMUT cell. We have successfully printed non-conductive PEDOT:PSS for the supporting walls, and ZnO as sacrificial layer, but nevertheless the procedure needs significant improvements. The porosity and roughness of the sacrificial layer has forced the use of kapton as membrane; the resulting structures, although imperfect and far from the idealized models used in finite element simulations, have proven a displacement response under electrical actuation, promising future improvements toward a more reliable fabrication flow. The fabricated CMUT cells are not fully clamped to the top of the walls, as was clear in screenshots taken of the vibrating membrane.

5.1 Future Work

The advantages of inkjet printing addressed and discussed in this thesis create tremendous potential for this technique, which attracts researchers to spend more time trying to investigate and study all aspects of exploiting it. Researchers might want to try to integrate inkjet printing and other conventional techniques, to come up with cheaper devices in less time while taking in consideration environment friendly issues, in case printing cannot be the only technique to be used due to some limitations or drawbacks. For the 3D movable structures aspect, a reliable sacrificial layer that preserves a smooth surface after post-processing is needed in place of the rough zinc oxide layers. Potential alternatives can be the photoresist SU-8 (not completely cured) or printable wax. This would enable researchers to try to inkjet print the membrane on the top of the sacrificial layer, and obtain later on movable structures like CMUTs or cantilevers.

The quality of the printed ZnO layer can be also improved through controlled deposition techniques, with the hope of making it suitable for use as sacrificial layer. ZnO may be used, on the other side, for generating piezoelectric devices through these rapid fabrication techniques.
PEDOT:PSS can be used as base material for many other applications, including interdigitated capacitor-based strain gauges. Such capacitors can be printed on flexible substrates, like polyimide, and characterized mechanically and electrically in a similar setup we have used for the test and characterization of the piezoresistive-based strain gauge. The changes in the capacitance induced by the strain field in the substrate will be, unlike piezoresistive sensing, due solely to geometry changes. It is expected that this will generate a lower sensitivity, but more controllable properties when operating in a large range of conditions. Also, for further characterization of the mechanical behaviour of PEDOT:PSS printed films, we suggest modeling the printed piezoresistor presented in chapter 2.2.3 using Comsol or any other finite element analysis software as it will present a more accurate model than the one we presented in chapter 3.2, the uniform thin film. By this, effect of PEDOT:PSS’ stiffness on the polyimide substrate and its effect on the substrate’s characteristics, such as Young’s modulus.
References


18 P. Calvert; G. Jabbour; Y. Yoshioka, “Multilayer Inkjet Printing of Biopolymers, OLED’s and Other Devices.”


43 H.C. Starck, “Chemical Properties of Clevios,”

44 PIRA, “printed electronics,”

45 H.C. Starck, “Product Information,”

46 Dupont, “Technical Datasheet for Dupont Kapton HN,”


Appendices

Appendix A – Scripts Used in Printing Devices

In general, in order to print a line, the following points have to be specified:

SET,DIS,t,x,y,z,xrm,yrm,px,py,rv,rm,fd,hd

Where ‘t’ is dispensing type (1 for pint, 2 for matrix, 4 for microtitre plate), ‘x’ is start position in x-direction (in mm), ‘y’ is start position in y-direction (in mm), ‘z’ is start position in z-direction (in mm), ‘xrm’ is matrix size in x-direction (in mm), ‘yrm’ is matrix size in y-direction (in mm), ‘px’ is number of points in x-direction, ‘py’ is number of points in y-direction, ‘rv’ is matrix offset in mm, ‘rm’ is dispensing pattern, ‘fd’ flow dispensing (No=0 and Yes=1), and ‘hd’ is headnumber for stating position.

One layer of piezoresistor (in our case, this was repeated 10 times):

//pad
set,dis,2,62.5,99,20,0.025,0.025,210,25,0,2,1,1
dis

//line - horizontal
set,dis,2,70,100,20,0.025,0.025,525,12,0,2,1,1
dis

//line | vertical
set,dis,2,70,100.5,20,0.025,0.025,110,12,0,2,1,1
dis
//line-
set,dis,2,70,101,20, 0.025, 0.025,525,12,0,2,1,1
dis
//line|
set,dis,2,60,101.5,20, 0.025, 0.025,110,12,0,2,1,1
dis
//line-
set,dis,2,70,102,20, 0.025, 0.025,525,12,0,2,1,1
dis
//line|
set,dis,2,70,102.5,20, 0.025, 0.025,110,12,0,2,1,1
dis
//line-
set,dis,2,70,103,20, 0.025, 0.025,525,12,0,2,1,1
dis
//line|
set,dis,2,60,103.5,20, 0.025, 0.025,110,12,0,2,1,1
dis
//line-
set,dis,2,70,104,20, 0.025, 0.025,525,12,0,2,1,1
dis
//line|
set,dis,2,70,104.5,20, 0.025, 0.025,110,12,0,2,1,1
dis
//line-
set,dis,2,70,105,20, 0.025, 0.025,525,12,0,2,1,1
dis
set, dis, 2, 60, 105.5, 20, 0.025, 0.025, 110, 12, 0, 2, 1, 1

dis

set, dis, 2, 70, 106, 20, 0.025, 0.025, 525, 12, 0, 2, 1, 1

dis

set, dis, 2, 70, 106.5, 20, 0.025, 0.025, 110, 12, 0, 2, 1, 1

dis

set, dis, 2, 70, 107, 20, 0.025, 0.025, 525, 12, 0, 2, 1, 1

dis

set, dis, 2, 70, 107.5, 20, 0.025, 0.025, 110, 12, 0, 2, 1, 1

dis

set, dis, 2, 70, 108, 20, 0.025, 0.025, 525, 12, 0, 2, 1, 1

dis

set, dis, 2, 70, 108.5, 20, 0.025, 0.025, 110, 12, 0, 2, 1, 1

dis

set, dis, 2, 70, 109, 20, 0.025, 0.025, 525, 12, 0, 2, 1, 1

dis

set, dis, 2, 70, 109.5, 20, 0.025, 0.025, 110, 12, 0, 2, 1, 1
dis

//line-
set,dis,2,70,110,20, 0.025, 0.025,525,12,0,2,1,1
dis
//line|
set,dis,2,70,110.5,20, 0.025, 0.025,110,12,0,2,1,1
dis

//line-
set,dis,2,70,111,20, 0.025, 0.025,525,12,0,2,1,1
dis
///pad
set,dis,2,62.5,111.5,20, 0.025,0.025,210,25,0,2,1,1
dis
//line|
set,dis,2,60,111.5,20, 0.025, 0.025,110,12,0,2,1,1
dis

//line-
set,dis,2,70,112,20, 0.025, 0.025,525,12,0,2,1,1

CMUT: PEDOT:PSS walls around drilled holes (repeated 10 times):

one line: set,dis,2,55,144.60,33,0.1,0.2,50,1,0,2,1,1
dis
other wall: set,dis,2,55,145,33,0.1,0.2,50,1,0,2,1,1
<table>
<thead>
<tr>
<th>dis</th>
<th>set,dis,2,55,144.70,33,0.33,0.33,25,1,0,2,1,1</th>
</tr>
</thead>
<tbody>
<tr>
<td>dis</td>
<td>set,dis,2,55,144.90,33,0.33,0.33,25,1,0,2,1,1</td>
</tr>
<tr>
<td>dis</td>
<td>set,dis,2,55,144.80,33,0.33,0.33,25,1,0,2,1,1</td>
</tr>
</tbody>
</table>
Appendix B – Steps for Printing a Device

1. Specify the ultimate device (design + material)
2. Choose ink(s) to be used accordingly (conductive, insulating...)
3. Dilute ink(s) and add appropriate solvents depending on the final design and nozzle used
4. Write the printing script taking in consideration the droplets’ size and the minimum spacing between them
5. Print and repeat printing procedures depending on the thickness of films anticipated
6. Post processing for printed structures (annealing, drying...); and cleaning nozzles to prevent clogging