The Pennsylvania State University

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### A FULLY PRINTED AND PHOTONIC SINTERING DERIVED SILVER-

### NANOPARTICLE-BASED WEARABLE STRAIN SENSORS

A Thesis in

Engineering Science and Mechanics

by

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## ABSTRACT

Traditional strain sensors are widely used in measure systems due to their extraordinary precision and stability; however, their large size and rigidness limit their application in flexible and wearable sensors. New crack-based strain sensors are often fabricated on soft polymer substrates, which enables the easy integration in epidermal electronics for real-time monitoring. In this paper, the low-cost, wearable strain sensors based on silver nanoparticles (AgNPs) inks on Polydimethylsiloxane (PDMS) is designed and manufactured using a commercial Direct Ink Writing (DIW) system in the lab. We expanded the currently narrow measure range of crack-based strain sensors by incorporating serpentine and wavy connections. It could be applied in electronic skins and devices that could be attached to clothing or directly onto the body for motion detection as well as further health monitoring systems.

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### **Chapter 1. Introduction**

### 1.1 Strain Gauge

A SG(SG) is a sensor used to measure strain (relative change in length) on an object. When external forces are applied to a stationary object, stress and strain are the result. Stress is defined as the object's internal resisting forces while strain is defined as the displacement and deformation that occur. Strain is referred as positive if the object is stretched and negative if it is compressed, also defined as tensile and compressive strain. The most common method to measure strain is using a SG, whose electrical resistance varies to the amount of strain in the device. The first SG was developed in 1938 by Edward E. Simmons and Arthur C. Ruge (working independently of each other). The latter invented the device in MIT to help his graduate student complete his investigation of earthquake stress on elevated water tanks. This bonded, metallic wire-type SG had a simple design with a tiny piece of high-resistance filament bent in a zigzag pattern and fixed in a rigid base. The gauge was attached to the object, so any deformation on the surface could be easily detected by measuring the changes in electrical resistance. With supplementary invention of bonded, metallic foil-type SG at 1952, developing distinct advantages such as a reduction in size and production costs, the bonded metallic SG then became the most common type in use today.

## Structure of strain gauge



Figure 1: Schematic diagram of a typical foil strain gauge.

The strain gage is one of the most important sensor of the electrical measurement technique applied to the measurement of mechanical quantities. The strain of a body is caused by an external influence or an internal effect, which might be caused by forces, moments, heat, structural changes of the material and so on. The amount or the value of the influencing quantity can be derived from the measured strain value. Therefore, SG has been widely used in many fields. In experimental stress analysis, the measured strain values state the stress in the material and predict its safety and endurance (e.g., SG bonded on bridge, rail and airplane to monitor their status). In manufacturing of some special transducers, SGs are usually bonded with a pressure sensitive diaphragm to measure forces or other derived quantities like moments, accelerations, vibrations, etc. Besides these traditional applications, SG also plays an important role in some emerging fields, especially in stretchable and wearable sensors in recent years[1].

Wearable technology has been emerging in recent decades, which integrates electronic devices and sensors into clothing and accessories, stuck on human skin like tattoo or even implanted into the body. Physical, chemical and biological status of the human body could be in situ monitored by various flexible sensors, such as body temperature, heart rate, and blood glucose. They are also used for human motion detection and environment monitoring with a high efficiency and low cost. Particularly, wearable strain sensors have been rapidly developed because of their wide applications in continuous heath-monitoring, human motion detection and human-machine interfaces. For example, some wearable strain sensors have been directly mounted on the joints to monitor their state continually and collect the related data[2-4]. When it comes to skin-mountable sensors, several characteristics are vital including high stretchability, flexibility, lightweight, biocompatibility, low energy consumption, etc. Such requirements should be taken seriously in terms of working mechanism, design, material and manufactory methods of the wearable strain sensors.

#### 1.1.1 Type & parameters

There are several types of strain sensors including resistive, capacitive, piezoelectric, optoelectrical, etc. The resistive-type and capacitive-type sensors are mainly chosen for wearable strain sensors due to their simple input/output system, relatedly high resolution and the capability to offer high flexibility and stretchability. Resistive-type strain sensors are typically composed of

electrically conductive sensing components coupled with flexible substrates. When sensing components are stretched, the electrical resistance changes as a function of the applied strains following its sensing mechanism. After release of the strain, restoration of the sensing components to their original statuses recovers the electrical resistance of sensors. Capacitive-type sensors usually contain a highly compliant dielectric layer sandwiched between a pair of stretchable electrodes. Tensile strain brings two electrodes closer and results in an increase of capacitance.

The performance of strain sensors has been characterized by several parameters such as gauge factor (GF), stretchability, linearity, hysteresis, response and recovery time, overshoot behavior, durability, etc. These parameters are crucial for wearable strain sensor considering the high requirements of their possible applications.

### **Gauge Factors**

Gauge factor (GF) of a strain gauge is the ratio of relative change in electrical signal to the mechanical strain, which reflect the sensitivity of a strain sensor. GFs are defined by  $GFs = (\Delta R/R_0)/\epsilon$  for resistive-type sensor, and  $GFs = (\Delta C/C_0)/\epsilon$  for capacitive-type sensor. For wearable strain sensors, the value of GFs varies due to the different sensing mechanisms, materials, and structures of the strain sensors. The greater the GF of a sensor, the easier it detects small strain ( $\epsilon$ <1%). General metallic foil-type SGs have GFs in the range of 2–10[5], but some semiconductor or nanomaterial based strain sensors might bear GFs of 500 or larger[6, 7].

### Stretchability

Traditional silicon-based electronical sensors are rigid and brittle. Large deformations would cause structural damage and performance failure. While, the skin coordinates the movements of the body by bending and stretching itself. So wearable strain sensors must be stretchable enough to suit normal body activities and be capable of bending to conform to the topography of the skin. These demands become even more important for epidermal sensors where mechanical compliance like human skin and high stretchability ( $\epsilon > 100\%$ ) are required.[8] Stretchable sensors can be achieved either by using new stretchable materials in conventional structures, or by exploiting new

structural layouts with conventional materials. It is noteworthy that most highly sensitive sensors can only maintain their superiority in a limited strain range, and the sensor with both high stretchability ( $\epsilon \ge 100\%$ ) and high sensitivity (GF  $\ge 50$ ) is still a focus of researches[9].

#### Linearity

Linearity is another important parameter for wearable strain sensors, because it endows the signal detection with accuracy and feasibility. Linearity is the property of a mathematical relationship or function which means that it can be graphically represented as a straight line. For strain sensors, it refers to a good correspondence relationship between the relative change of the output signals and input strains. Most of the resistive-type strain sensors exhibit linearity under low strains but nonlinearity under large strains, which is mainly caused 1by the occurrence of nonlinear heterogeneous deformation upon stretching. Nonlinearity causes a large deviation between the output signal and the input strain, which results in a complex and difficult calibration process. Generally, a trade-off relationship between "high sensitivity" and "high linearity and stretchability" exists in majority of SGs. Since highly sensitive sensors usually require considerable deformations under stretching, even though highly stretchable sensors require homogeneous morphology changes upon stretching. These conflicting requirements enable the development of strain sensors with high linearity, stretchability and sensitivity simultaneously to be a grand challenge.

#### **Hysteresis and Response Time**

Hysteresis and response time are important parameters for dynamical performance of wearable sensors. Hysteresis is a key factor when sensors are under dynamic load, especially for skin-mountable and wearable strain sensors. It is critical to have a consistent sensing performance during loading and unloading in that most body motions are cyclical. Typically, capacitive sensors feature a better hysteresis than resistive strain sensors, since their reliance on the stable overlapped area between opposing electrodes. However, for resistive strain sensors, a larger hysteresis is caused by the viscoelastic nature of polymer substrates as well as the interaction between fillers and polymer substrates. A week interaction would delay the fully recovery of filler, results in a

high hysteresis behavior. While, a weak adhesion is needed to avoid the buckling and facture of fillers at the releasing cycle. Hence, it is still a challenge to optimize the hysteresis performance using novel polymer materials and structural engineering. Response time illustrates how quickly the strain sensors could achieve steady response to applied stimulus. The response delay exists in almost all composite-based sensors due to the viscoelastic nature of polymers. Piezoresistive device usually has a larger response time because they need more time to reconnect percolation network in resistive composites. However, the newly developed crack-based sensors give a good response time (~20 ms) due to the quick connection and disconnection of cracks upon the loading and uploading cycle.[10]

#### **Durability**

Durability determines the endurance of sensors to the long-term stretching/releasing cycles. It is more important for wearable strain sensors since very large, complex, and dynamic strains applied by the human body. The mainly problems affecting durability including fatigue or damaged of substrates under high strains, and fracture, buckling, or even stripping of sensing materials after numerous cycles. Researchers have tried using composite structure and some novel sensing materials to build wearable sensors with very high stability[11, 12]. It is worth mentioning that self-healing may be a potential way to promote durability. For example, embedding external healing agents and catalysts in polymers substrates or using self-healing materials could repair the damaged sensors by releasing healing agents to trigger additional polymerization and link the separated molecules together again[13, 14]. But there's still a long way to go before designing and manufacturing wearable strain sensors with a durability which could meet the requirements of commercialization.

### 1.1.2 Material & fabrication

Choosing the suitable material and fabrication method is the critical consideration of pursuing a wearable strain sensor with good performance. The materials utilized in SGs mainly include sensing and substrate material. Considering the organic materials which have great mechanical flexibility but poor sensing performance, on other hand, inorganic materials which are sensitive but usually rigid and frangible. Therefore, collaboration between different materials can be a solution to achieve high sensing performance, flexibility/stretchability and mechanical robustness in wearable sensors at the same time.

### **Sensing material**

For sensing material, it is important to provide precise detection in a wide range as well as good flexibility and stretchable for wearable strain sensors. Recently, carbon materials, metal nanomaterials and conductive polymers have attracted intensive attention due to their great sensitivity and stretchability. Furthermore, they can be combined with elastomeric polymer to enhance their flexibility. Different allotropes of carbon, such as graphite[15, 16], carbon nanotube(CNT)[17, 18] and graphene[19, 20], have been widely used in wearable sensors for their good performance and various manufacturing methods. However, challenges still exist for carbon materials such as the instability to achieve bulk quantities of CNTs materials, the incorporation of defects in the graphene significantly decreases its conductivity and so on. Conductive polymers, such as PEDOT: PSS, PPy, and PANI/PVDF, are mushrooming due to easy-to-prepare feature and mechanical similarity between them and many insulated substrate polymers. Among them, PEDOT: PSS is one of the promising conductive organics due to its excellent solubility in water, which makes it compatible with many manufacturing techniques, like spinning coating, dippingdrying and inkjet printing. But the dried PEDOT:PSS film cannot sustain continuous bending and stretching cycles so it is often printed and filled into porous substrates to promote the adhesion and durability[21]. Even though the carbon materials or conductive polymers own exciting characteristics, their electrical conductivity is still low. Metal nanomaterial including nano wires (NWs) and nano particles (NPs) are promising candidates for sensing material for wearable strain sensors due to their high conductivity and flexibility. NWs and NPs take advantage of as fillers and conductive inks. For instance, AgNWs can be coated and encapsulated into PDMS to build a crack-induced strain sensors which is highly stretchable and sensitive[22]. The adhesion between AgNWs/AgNPs and polymers is not as strong as carbon materials, hence, they are usually encapsulated between two substrate layers improve its reversibility, reproducibility, and durability. The conductive inks with metal NPs can also be casted and annealed on the substrate surface to form the sensing unit of sensors[6]. Combining with flexible and stretchable structure design such

as serpentine and wavy structures, metal nanomaterials offer an advantageous incorporation of mechanical compliance and compatibility with excellent performance. Cu NWs which have close thermal and electrical properties are also appealing considering its cheaper price, but their poor antioxidation and chemical corrosion should be concerned in the future development.

### Substrate material

The substrate provides the flexible and stretchable support for wearable strain sensors, and directly determines the wearability and durability. Lots of materials have been used including polyethylene terephthalate (PET)[23], polyimide (PI)[24], polyurethane (PU)[25], PDMS[26], Ecoflex[27], tissue paper[28], textiles[21] and cotton[29]. For example, PET is frequently used for its high creep resistance and good transparency (>85%)[23, 30, 31], but it cannot be stretched well for its relatively high modulus (about 2~4 GPa). Among these materials, PDMS is dominated for flexible substrate because of its outstanding comprehensive performance. This commercial silicone elastomer has good mechanical properties (stretchability, bendability and transparency), chemical properties (non-toxic, nonflammability and hydrophobicity) and strong compatibility with other materials[32]. With the development of lithography technology, microstructures can be fabricated onto PDMS films, making them more popular in highly sensitive and microfluid devices. The flexible sensors based on PDMS can be pressed, bended, and twisted with excellent properties, including ultra-low power consumption, fast response time and reliable durability[33]. Owing to its widespread availability and well-studied properties, PDMS could be easily fabricated with a low cost, which also endows it great commercialization potential.

#### Fabrication

With the development of material science and manufacturing process, many approaches have become available for fabricating wearable sensors. The typical fabrication methods include filtration, coating techniques, chemical synthesis and pattern transferring. The manufacture of wearable sensors often contains multiple processes and many different techniques, which combines strategies for substrates and sensing elements. The filtration method is a powerful tool to obtain uniform composite sensing films using filter membranes, which could then be transferred to another substrate. For example, Yan et al. fabricated highly stretchable graphene–nanocellulose composite nanopaper using polycarbonate filter membrane, uniform composite films were obtained after filtration due to the simultaneous extraction of solvent from the evenly distributed track-etched pores in the filter membrane[34]. Coating method use deposition or evaporation methods to make sensing material uniformly grow on the substrate. A textile-based wearable sensor was fabricated by coating PDMS on the surface of the conductive fiber and stacking the PDMS-coated fibers perpendicularly to each other, which obtained a high sensitivity in the low-pressure region and fast relaxation time (<10 ms)[35]. Wang et al. demonstrates a graphene woven fabrics (GWFs) wearable strain sensor by adhering the GWFs on PDMS and medical tape composite[20]. The highly sensitive GWFs were fabricated by the chemical vapor deposition (CVD) method using a crisscross copper mesh as the substrate.

Pattern transferring is widely used in producing wearable sensors, mainly including microscale modeling, lithography and printing (e.g., screen printing, inkjet printing, 3D printing) technology. Micro-scale modeling is often employed to fabricate microstructure in flexible substrates and sensing component, especially appropriate for irregular microstructures. In the modeling process, pending materials are poured onto the module and then peeled off after drying with special pretreatment and geometric design. A highly sensitive skin-mountable strain gauges was fabricated by micro-scale modeling processes with two types of conducting elastomers integrated in another insulating elastomer[8]. Lithography is another mature transferring method, which could fabricate high resolution wearable electronics with great performance. This process deposits sensing components onto the substrate and then etches the undesired parts via photolithography. The highly controlled deposition and patterning capability make it suitable for wearable sensors and epidermal electronics, many researches have been done using this method[36-39]. However, the lithographic techniques are limited by their complicated fabrication steps, material availability and labor-intensive processes.

As an alternative approach, printing techniques can simultaneously deposit and pattern materials on various substrates in a simple, rapid and efficient way with desired precision and accuracy. The current printing techniques can be classified into template and non-template based methodologies. Template-based printing processes include screen printing, gravure printing, and many others. Screen printing is a typical templated-based printing process, during which the liquid

ink is forced through a patterned mesh/stencil by a rubber squeegee and transferred onto substrate surface. As the mask moves away, ink will remain on the substrate and form a patterned film.

Non-template printing techniques typically controlled ink dispensing onto the receiving substrate. Direct ink writing (DIW) technologies are being rapidly developed over the recent decades for non-template printing. These technologies basically utilize a computer-controlled motion stage and a pattern generating device (e.g., ink-deposition nozzle) to generate patterns with a specific material composition and structural architecture[40]. Moreover, the CAD-based geometries can be directly printed onto substrate with the help of graphics software and printing equipment. For example, Inkjet printing, depositing small ink droplets (~1-30pL) onto paper, plastic or other substrates by a nozzle, represents a high precision, fast and reproducible nontemplate technique. Bruno et al. has built an all-inkjet printed strain sensors which combined conductive inks with inkjet printing to fabricate both sensing component and substrate, showing a convenient approach for rapid prototyping of wearable sensors[41]. It is worth mentioning that the hybrid fabrication process will be a promising approach for manufacturing high-sensitive and well-patterned devices (e.g., combining printing and lithography). For instance, Wang's group developed a stretchable "island-bridge" sensors, which used screen printing to make the sensitive "island" units and lithography for fabricating the serpentine interconnects[42]. Such new hybrid fabrication strategy enables new way to manufacture wearable sensors that cannot be achieved via individual fabrication routes.

### 1.2 Summary of approach

In this thesis, we design and fabricate wearable resistance-type strain sensors by using a direct writing method to print AgNPs onto PDMS substrate. When tensile strain applied, nanoscale cracks informed in the AgNPs pattern and thus the change of resistance can be sensed to quantify the applied strain. Uniform microscale serpentine AgNPs features are designed for better stretchability, which can be obtained by optimizing the printing parameters including nozzle size, printing ink, surface energy, and printing speed. Furthermore, intense pulsed light (IPL) was employed to sintering AgNPs inks in order to protect the substrate and avoid the formation of thermal stress induced cracks during conventional heat treatments. The AgNPs and PDMS based strain gauge exhibit good stretchability and stable GFs.

### **Chapter 2. Experimental procedures**

### 2.1 Material preparation

As mentioned in the introduction part, metal nanomaterial are promising candidates for sensing material for wearable strain sensors. In this work we choose commercial silver nanoparticles ink (Metalon® JS-A191, NovaCentrix) to print the sensing pattern. The physical property of the ink is listed in the Table 1, it is an electrically conductive ink designed to produce circuits on non-porous, temperature-sensitive substrates including polycarbonate and PET. The ink can be thermally cured or sintering processed and then have a good conductivity according to the datasheet.

|                                  | Value | Units   |
|----------------------------------|-------|---------|
| Ag content                       | 40    | wt%     |
| Viscosity                        | 8-12  | cP      |
| Surface tension                  | 28-32 | dyne/cm |
| z-avg particle size <sup>3</sup> | 30-50 | nm      |
| Specific gravity                 | 1.6   | _       |

General Description ...... Water-based Ag ink Flash Point ......Non-flammable

<sup>3</sup> Malvern dynamic light scattering

Table 1: Physical property of the AgNPs ink.

Here we choose PDMS as the substrate material because of its outstanding comprehensive performance. The first step is mixture of a PDMS pre-polymer and a curing agent (Sylgard 184, Dow corning, USA) with a weight ratio of 10:1. Then the mixture is spin coated on an acrylic board (rpm 600, 30s), and put onto hot plate at 80° for 15 minutes to cure. Here we use acrylic board instead of silicon wafer because the adhesion between PDMS and acrylic board is weaker than silicon wafer, which make it easier to peel off the sensors.

A plasma treatment (45W,1 mins using Tepla M4L<sup>™</sup> Asher) for PDMS substrate is needed to improve the wettability during the printing process. Wettability depends on surface energy measured in mN/m for solid, or surface tension for liquid measured dyne/cm. The surface energy

of the PDMS substrate directly affects how well the AgNPs ink wets the surface. The surface energy of PDMS is ~ 19.8 mM/m while the surface tension of AgNPs is ~30 dyne/cm. For a good wettability between a liquid and a substrate surface, the substrate's surface energy should exceed the liquid's tension by about 2-10 mN/m[43]. Plasma treatment is a useful and environmental method to alter the surface chemistry. Exposure to plasma causes the formation of an oxidized surface layer containing dense SiOx-rich material, which could increase the surface energy of PDMS. However, the oxidized surface layer is brittle and microcrack may happen with increasing exposure time and radio-frequency (RF) power[44]. After several attempts, we finally obtained the proper setting (45W, 1 min) for plasma treatment, which is good enough for AgNPs ink to wet the PDMS surface without the formation of cracking. However, the surfaces would gradually recover their hydrophobicity when it exposed to the air because of hemodynamically favorable orientation of the functional groups[45]. According to the research[46], we storage the treated PDMS substrates in DI water to maintain the hydrophilicity of the surface. We take out the PDMS substrate from DI water before printing each time, the hydrophilicity would remain available for printing around 5-6 hours.

### 2.2 Direct Ink Writing

As we mentioned in last chapter, Direct ink writing (DIW) technologies are being rapidly developed over the recent decades. Here we fabricate our strain gauges by using a commercial DIW machine, SonoPlot® Microplotter II, which is a precision picolitre fluid dispensing system for the microarray and polymer electronics markets. As shown in Fig 2c, the core of the Microplotter is a dispenser that uses controlled ultrasonics to deposit fluid in a noncontact manner. This patented technology can produce picolitre droplets that form features on a surface as small as 5  $\mu$ m wide. When combined with automatic surface height calibration, coefficients of variability for deposited feature diameters as small as 10% can be achieved. The Microplotter's unique ultrasonic liquid dispensing technology lets it print a wide variety of inks, including every ink that a standard inkjet can, plus a wide range of additional ink formulations. There are three requirements for the inks to print: viscosities from 0 centipoise up to 450 centipoises, suspensions with particles ranging from nanometers to up to 15 microns in size and aqueous solutions work

well, as do ones with organic solvents that are less volatile than toluene. The AgNPs we choose meets all three requirements.



Figure 2: DIW system. (a) DIW system; (b) micropipette attached on the dispenser; (c) working principle of printing process; (d) printed dumbbell shape samples.

Microplotter II can draw true continuous features, such as lines, arcs, and bends. These are uniform elements, not made from overlapping droplets like other technologies, and are particularly well-suited to the polymer electronics field. The system is controlled by its own software called SonoGuide through adjusting print parameters and printing process. The printing pattern could be directly designed and drown in its own software called SonoDraw or imported from AutoCAD. The printing process could be automatically executed by SonoGuide or manually controlled by the user. Before printing, surface sensing and calibration function is needed to detect the surface topography of the substrate. The resulting data would be used to adjust the height of the needle during printing process. The attached camera can also monitor and record the printing process as shown in Fig 2a.



Figure 3: Thickness of printed patterns. (a) dumbbell shape; (b) serpentine shape.

Many parameters would affect the printing process, mainly including the tip size of micropipette, dispense voltage, printing speed and the property of the ink. It's important to obtain the proper parameters for printing different inks and substrate, so we would have a detailed discussion about these parameters in next Chapter. In this work, we successfully printed samples of two patterns: dumbbell shape and serpentine shape, the thickness of PDMS and AgNPs pattern are ~0.1 mm and ~0.12 mm, shown in Fig 3 (measure by Nexview<sup>™</sup> 3D Imaging and Surface Metrology System).

### 2.3 Heating and Sintering

After printing, the obtained samples are not conductive because the solvent does not evaporate and the AgNPs are not sintered. Hence, we need to accelerate the solvent evaporation process and sintering the AgNPs to make it conductive. Here we use a drying oven (Jeio Tech ON-02G) to heat the sample and a benchtop Pulsed Light system (X-1100 from XENON Corporation) to sintering the AgNPs. The specific parameters for heating and sintering depend on the condition of printed samples, which would be discussed in next chapter.



Figure 4: Sintering and heating system. (a) Drying oven Jeio Tech ON-02G; (b) benchtop Pulsed Light system X-1100, XENON Corporation; (c) light measurements of optical energy with different voltage and pulse width.

### 2.4 Measurement

After processing the samples, we use a self-built stretching and measurement system to measure the performance of strain gauge samples. The stretching and measurement system includes a home-made programmable stretching platform and a commercial SourceMeter (Keithley 2401). The stretching platform is based on a linear stage driven by a stepper motor, which could be programmed and controlled via Arduino. The program we coded for this work is attached on the Appendix. SourceMeter is used to precisely measure the resistance of strain gauge during the stretching test. Here we use I-V Software to control the SourceMeter and record the measured data.



Figure 5: Self-built stretching and measurement system.

As shown in Fig 5, the samples are first loaded on the stretching platform with two ends fixed by double-sided sticky tape rather than clamps to avoid potential damage to the sample. Then the two ends of the samples are connected with SourceMeter through copper foils which attached with silver paste. After the data is stable, the stretching-releasing cycle experiments would begin, and the collected data would be used to evaluate the performance of the samples.

### **Chapter 3. Results and discussion**

### 3.1 Optimization of DIW process

As we mentioned before, many parameters would affect the printing process. In this section, we would explore the key parameters of printing process and summarize an optimized parameter setting for the fabrication of strain gauges.

### 3.1.1 The property of ink

Viscosity, surface tension and the particle size of ink are main factors determine the printing results. Bradley Larson has compared the printing results of two solutions with different viscosity, as show in Fig 6. The red food coloring viscosity is close to that of water, while the blue food coloring is significantly more viscous. To demonstrate the effect of viscosity on deposited spots, the applied frequency and voltage were held constant for the two solutions. Different spot sizes resulted (28 µm diameter for the red spots, 20 µm diameter for the blue spots)[47]. If the viscosity is higher than 450 centipoises, it would be very difficult to load the tip and fluently dispense the ink. For the surface tension, as we discussed in Section 2.1, the substrate's surface energy should exceed the ink's tension by about 2-10 mN/m to obtain a good wettability between ink and substrate during printing. Other than using surface treatment to improve the surface tension. Le et al. have added Triton X-100 in the commercial AgNPs ink and successfully tune the surface tension for a better wettability[48]. In terms of particle size, the ink should be uniform and no particles larger than 10µm is allowed (the inner diameter of tip). Otherwise the tip would be stuck by the big particle. Hence the AgNPs ink needs to be gently shaken before the printing.



Figure 6: Image of printed pattern using ink with different viscosity. (a) A closeup image of the micropipette tip in the process of depositing a pattern of spots. Several spots, spaced ~100  $\mu$ m apart, are shown. (b) A pattern made from red and blue food coloring deposited on an untreated glass slide at the same dispenser voltage and frequency. The red spots are 28  $\mu$ m in diameter, the blue spots are 20  $\mu$ m in diameter, and the center-to-center spacing of the spots is 50  $\mu$ m. The spot size differences are caused by the differing viscosities of the two solutions.

### 3.1.2 Tip size

The tip size of micropipette is another key parameter. The glass micropipette consists of a conical tip and a cylindrical body which was shaped during its manufacturing process. The inner diameter (ID) of conical tip is controllable. Here we tested three different ID (10, 30 and 50  $\mu$ m) micropipettes provided by SonoPlot. Fig 7 shows the image of three different micropipettes printing straight line recorded by the system camera.



Figure 7: Image of three micropipettes with different ID and their printed lines. (a) The ink is loading by the capillary force; ID are (b) 10  $\mu$ m; (c) 30  $\mu$ m; (d) 50  $\mu$ m



Figure 8: Optical microscope imaging of three AgNPs line printed by micropipettes with different IDs. (a) 10 µm; (b) 30 µm; (c) 50 µm.

We measure and compare the width of three AgNPs line printed by micropipettes with different IDs using an optical microscope (ME300, Amscope) as shown in Fig 8. All parameters are consistent for each printing process except the ID. It is obvious that the width of printed line is proportional to the ID. But the width of printed lines is not the same as the ID of microscope because it is determined by several factors such as the wettability, ink property, printing speed, etc.

#### 3.1.3 Voltage control

The Microplotter system could apply dispense and spray voltage to control the printing process. Both types of voltage could control the vibration of the micropipettes, however, they play different roles in the DIW system. The dispense voltage is used to keep the micropipette vibrating continuously during the printing process, ensuring the ink coming out from tip uninterruptedly. This is also an advantage of the DIW compared to the inkjet printing. After the printing, there might be ink residue in micropipette. The spray voltage is used here to spray the residue out from the tips, which could also be used to spray the impurity inside the micropipette before loading the ink. They both have a range of 12V, but the common range of dispense voltage is 0.1-2V while 8V-11V for another one. Because higher voltage would damage the tip during the printing due to the extremely short distance between tip and substrate. On the contrary, the lower voltage would be not enough to spray the residue out.



Figure 9: Optical microscope imaging of four AgNPs line printed with different dispense voltage. (a) 0 V; (b) 1 V; (c) 2 V; (d) 3 V.

We measure and compare the width of four AgNPs line printed with different dispense voltage as shown in Fig 9. The printed line in Fig 9a is thin and uneven, even breaks at some points. The reason is that the printing process depends on variation of micropipette and the "moisture bridge" formed between ink and substrate. Condensation from the atmosphere (or from a humidified chamber) causes a moisture bridge to form between the tip and the surface. Solute will then be transferred from the tip, through this moisture bridge, to the surface[47]. If the dispense voltage is zero, the ink cannot be simply printed by the traction of liquid bridge. But if the voltage is too large, the ink will be ejected in a short time, especially on a rough surface as show in Fig 9d. The printing results is better in Fig 9b and Fig 9c, the width of the printed line also increases as the voltage increases. Generally, we would find a proper dispense voltage

between 0.1-2 V based on the property of ink and substrate. Sometimes the dispense voltage could be changed during printing process to obtain components with different size in a single pattern.

### 3.1.4 Printing speed

The printing speed here means the moving speed of micropipette in x and y direction when it is printing. It plays an important role in this DIW system.



Figure 10. Optical microscope imaging of five AgNPs line printed with different printing speed. (a) 500; (b) 750; (c) 1000; (d) 1500; (e) 2000 (Unit: µm/s)

As we just mentioned before, the tip uses the moisture bridge to print instead of directly contacting with the substrate. Therefore, too much speed may cut the bridge and cause the problem during printing as shown in Fig 10 d&e. But the printing speed neither cannot be too slow like Fig 10a, which would print too much ink and effect the geometry of the printing patterns. In our work, we use 700  $\mu$ m/s as printing spend to obtain evenly printed patterns.

In summary, the printing process are mainly affected by the four factors discussed in this section. If the ink and substrate are already decided, we can optimize the printing process by adjusting the tip size, dispense voltage and printing speed.

### 3.2 Post-processing skill

#### 3.2.1 Heating process

As we mentioned in Chapter 2, printed samples must be heated to dry out before sintering. But the heating temperature and time depends on the condition of printed samples, specifically refers to their wetness. The wetness or thickness of printed samples basically depending on parameters discussed in section 3.2, however, it varies for different batches of samples due to some uncontrollable factors in printing process such as changing shape of needle tip, surface roughness and wettability, etc. Therefore, we need setting specific heating parameters for different batches of samples. Generally, the wetter the sample, the longer it takes to heat up. We set the heating temperature as 65°C because it would be difficult for a higher or lower temperature to control the heating process, which would have great risk damaging the simples, or delaying the experiment time. The heating time ranges from 5 mins to 20 mins according to the wetness of samples. If the heating time is not enough, the sample would only be partly dry out. The wet part would be easily blown off the substrate in the following sintering process as shown in Fig 12a. If the heating time is too long, a lot of crosswise cracks would appear on the AgNPs pattern leading to a nonconductive sample after heating as shown in Fig 12b. Besides, these non-conductive samples won't be conductive even after sintering process due to the numerous initial crosswise cracks created during the heating process, which are caused by the significant difference of the thermal expansion coefficient between the AgNPs (bulk silver: 18.9  $\mu$ m  $m^{-1}K^{-1}$ ) and PDMS (Sylgard 184: 310  $\mu$ m  $m^{-1}K^{-1}$ )[5]. This is also the reason why we choose intense pulsed light (IPL) as our sintering method, instead of traditional annealing process whose high temperature would certainly create numerous cracks on our resistance based AgNPs-PDMS strain gauges.

### 3.2.2 Sintering process

The IPL sintering system Xenon X-1110 has the following components: a xenon flash lamp (4.2" spiral lamp), an aluminum reflector and a control system. The intense pulsed light is produced after the gas is ionized by a voltage ranging from 0-3000 V. As shown in Fig 11, the optical spectrum of light from our xenon lamp covers a range of wavelengths from 200-1100 nm. The energy density of sintering could be tuned by changing the voltage and time setting, which was measured and verified by the manufacturer of the xenon lamp when the specimen was 14 mm away from the lamp. The customized recipe of consecutive pulses could also be set from control system. For this experiment, IPL energy was applied at 125 J/cm2, 2500V for a single pulse while the samples were ~30 mm away from the lamp.



(a)



Figure 11: Sintering spectrum and interface. (a) spectrum of a 3600 V - 15 Hz setup 4.2" spiral lamp; (b) operation interface of controlling system.

After heating process, samples are still not conductive since only solvent of AgNPs is evaporated, the AgNPs are not sintered due to the low temperature and short time of heating setting. We use IPL system to sinter the AgNPs and measure the resistance of samples after sintering. It is worth mentioning that we have developed a special sintering method for our samples. We put sample and its attached glass slide upside down for the first-time sintering, and then turn it back for front sintering. If we only sintering the front side of the sample, many cracks would appear on the samples which would lead to an unpredictable resistance, or even non-conductive samples. Our hypothesis for this phenomenon is that attenuation occurs when energy passes from the upper layer of AgNPs to the lower layer (contact with PDMS), so the upper layer is sintered but not the lower layer. However, the energy passed to the lower layer is enough to cause the mismatches between lower layer and PDMS, leading to the cracks on the samples. But if we first sinter the back side of samples, the lower layer would be sintered firstly due to the transparency of glass slide and PDMS. Then the sintering of front side of samples would not cause the cracks on the lower layer. In summary, using this special sintering method, we can obtain sintered samples with few cracks or no cracks at all. After several sintering pulse, the resulting resistance of sintered samples will stabilize between tens to hundred ohms depending on the geometric size of patterns. As far as we know, this is the first time to successfully obtain AgNPs-PDMS strain gauges without initialing cracks during fabricating cracks. Then the fabricated strain sensor was covered by a ~0.1 mm thick PDMS encapsulation layer except the contacting areas to prevent the influence of environment factors.









(c)



Figure 12: Samples after post-processing. (a) part of pattern blown off due to high sintering energy and wetness of surface; (b) cracks created because of overheating; (c) sintering using special method.

### 3.3 Strain gauge performance

After 24 hours of PDMS encapsulation layer curing under room temperature, the samples are put onto our self-built stretching and measurement system to measure the performance of strain gauge samples. Firstly, we test the dumbbell shape samples. Strains from 0% with an incremental step of 0.5% (dwelling period 4 s and a strain rate of 0.1% per s for each step) are applied to the samples and then release with same pace. The resistance change would be gigantic (from hundreds  $\Omega$  to G  $\Omega$ ) when  $\varepsilon > 35\%$ , so the stretchability of dumbbell shape strain gauges is ~35%, when is relatively good compared to the previous strain gauges using similar materials[5]. Other parameters are good under 35% strain loading, such as a stable GF (~6), good linearity and quick response time (~ms).



Fig 13: Strain gauge performance. (a)(b) Overall 8% strain load/unload circle; (c) 500 times times cyclic stretching test; (d) part of AgNPs pattern after stretching tested.

Then we test the serpentine shape samples using same settings. The stretchability raise from ~35% to ~45% due to the specific structure design. The overall performance is good, as shown in Fig 13, it exhibits great linearity and almost no hysteresis happen during the totally 8% strain loading/reloading circle. The optical image of serpentine samples after stretching test is also show in the figure, which reveal the nature of our AgNPs-PDMS strain gauges. The cracks would be created and enlarged when the sample is stretched, then they would recovery under the releasing process. The linear change of resistance is caused by this nature. The 500 times cyclic stretching test also shows the good repeatability and stability of our strain gauges, as show in Fig 13.

#### 3.4 Conclusions

In conclusion, we demonstrated a highly stretchable strain sensor based on the AgNPs on the PDMS substrate using the DIW technology and IPL sintering process. This fabrication method is very simple with a fast fabrication speed and a low manufacturing cost. The opening and closure of micro-cracks on the AgNPs by elongation/relaxation cycles enable high stretchability and sensitivity of the sensor. Our strain sensor presented stable sensing performance with fast response/recovery and high sensitivity under both tensile and compressive strains. Additionally, the sensor exhibited good durability against repeated loading cycles. But there are also plenty of work to be done in the future. First, the real mechanism of our hypothesis about special sintering method should be further investigated. Second, the influence of the sample wetness on the formation of cracks also needs to be explored. We observed that cracks rarely appear on the wet part of sample even after stretching test. It may also have the potential to fabricate conductive component if the resistance doesn't change a lot under stretching and compressing. Last of all, our wearable strain sensor is applicable for detecting multiple human motions such as finger and wrist motions. We would try to use our wearable strain sensors to demonstrate a wearable glove system for human finger motion detection in the future work.

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## Appendix

### Arduino code

To control the linear stage in stretching measurement, the motor will step one step at a time, very slowly. You can use this to test that you've got the four wires of your stepper wired to the correct pins. If wired correctly, all steps should be in the same direction.

#include <Stepper.h>

int m=16;

int k=16;

int distance=198.4\*2\*m;//3200 steps=0.5mm

const int steps=400\*2\*4; //stepsPerRevolution =400\*2\*4; change this to fit the number of steps per revolution,2 comes frm 0.9degree, 4comes from 4 steps as a loop

const int speed=1;

String readString;

void setup () {

// initialize the serial port:

```
myStepper.setSpeed(speed);
```

Serial.begin(9600);

Serial.println("Stepper motor control system");

Serial.println("Input cycles number:");

```
}
```

```
void loop()
```

{

```
while (Serial.available()) {
```

```
char c = Serial.read(); //gets one byte from serial buffer
  readString += c; //makes the string readString
  delay(20); //slow looping to allow buffer to fill with next character
 }
if (readString.length() >0)
{
  Serial.println(readString); //so you can see the captured string
  int n = readString.toInt(); //convert readString into a number
  for (n;n>0;n--)
  {
    for (m;m>0;m--)
    {
      myStepper.step(-194.24);
      delay(4000);
    }
    for (m;m<k;m++)
    {
      myStepper.step(194.24);
      delay(4000);
    }
  readString=""; //empty for next input
 }
}
```

```
34
```