An Inkjet Printing Technique for Scalable Microfabrication of Graphene-Based Sensor Components

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This work was supported by the Novel Bioengineering and Technological Approaches to Solve Two Major Health Problems in Taiwan Sponsored by the Taiwan Ministry of Science and Technology Academic Excellence Program under Grant MOST 108-2633-B-009-001, Grant MOST 109-2639-E-009-001, Grant MOST 108-2321-B-009-007-MY2, and Grant 108-2221-E-009-003-MY2. The work of Ethan B. Secor and Mark C. Hersam was supported by the Air Force Research Laboratory under Agreement FA8650-15-2-5518.

ABSTRACT This paper presents a versatile and precise graphene patterning technique using the combined process of masking and inkjet printing. A graphene-based structure is fabricated by first defining the structural pattern and position using a masking mold, which can be either electroplated copper or deep reactive ion etching (DRIE) silicon shadow mask, followed by inkjet deposition of graphene ink and lift-off. The hybrid technique can realize high-fidelity, high-resolution graphene-based microstructures including free-standing and cantilever beams, four-point resistive measurement structures, and piezoresistive sensing elements with a minimum line width of \( \sim 20 \ \mu \text{m} \). Moreover, this method can facilitate the micropatterning of graphene oxide (GO) and reduced graphene oxide (rGO) on substrates such as polydimethylsiloxane (PDMS) and SiO\(_2\)/Si for selective cell culturing applications. Owing to the characteristics of low chemical usage, low process temperature and complexity, and high flexibility and fault tolerance of inkjet printing, this technique demonstrates compelling potential for a variety of biomedical applications.

INDEX TERMS Microelectromechanical systems, inkjet printing, microstructure, tactile sensors.

I. INTRODUCTION
Graphene, a two-dimensional honeycomb lattice of carbon atoms offering exceptional electrical, thermal, and mechanical properties such as high carrier mobility \([1]\), thermal conductivity \([2]\), and Young’s modulus \([3]\), has sparked considerable research interest in graphene-based microstructure fabrication for various applications. Li et al. \([4]\) applied mechanical exfoliation to form microscale (1\(\sim\)2 \(\mu\)m) free-standing graphene cantilever beams as switches and investigated corresponding dc characteristics and performance potential for nanoelectromechanical systems (NEMS) applications. Lin et al. \([5]\) demonstrated field-effect graphene transistors with a cutoff frequency as high as 100 GHz with potential for mmWave applications using graphene epitaxially formed on a SiC wafer by thermal annealing at 1450 °C. Li et al. \([6]\) employed a chemical vapor deposition (CVD) technique to grow a large-area graphene film, which can be transferred to arbitrary substrates from the initial copper. This process also facilitated the fabrication of dual-gated field-effect transistors with an electron mobility as high as 4050 cm\(^2\) \(\text{V}^{-1}\) \(\text{s}^{-1}\) on a SiO\(_2\)/Si substrates at room temperature. In addition to single-layered graphene devices, Singh et al. \([7]\) demonstrated a multilayered graphene resonator with a quality factor up to 220,000, which is optomechanically coupled with a high-Q superconducting cavity.
for exploring the quantum regime of motion. Ray et al. [8] presented a graphene nanoplatelet embedded polymer microcantilever, which exhibited a high gauge factor for the detection of explosive vapors in a ppb level. Jadhav et al. [9] successfully developed an ultra-low-power electromagnetic microspeaker using a parylene/graphene/parylene composite layer for boosting the speaker’s bass sound performance. Given the broad array of compelling applications for graphene-based devices, scalable and versatile fabrication methods compatible with graphene are highly sought.

Inkjet printing is a liquid-phase patterning method in which droplets of ink are jetted from a nozzle onto a substrate [10], [11]. It is such a digital non-contact technique that droplets can be printed in an arbitrary pattern on a substrate in a time- and material-efficient format. Better process control capabilities including accurate positioning, good spatial resolution, low processing temperature, and thick film deposition by multiple printing passes have shown the greatest potential for microelectronics integration and microelectromechanical systems (MEMS) fabrication. Given these advantages, a number of studies have explored inkjet printing of graphene-based inks for functional devices. Secor et al. [12] demonstrated the first inkjet-printed graphene electrodes with high electrical conductivity claimed for flexible electronics and sensors. Li et al. [13] demonstrated all-solid-state graphene-based in-plane micro-supercapacitors where inkjet-printed interdigitated graphene structures served as both electrodes and current collectors. The devices exhibited a capacitance of over 0.1 mF/cm² and cycle life of over 1000 times. Santra et al. [14] integrated inkjet-printed graphene on interdigitated gold electrodes with a complementary metal-oxide-semiconductor (CMOS) MEMS micro-hotplate for humidity sensing. The sensors showed a wide detection range of 10-80 % relative humidity change and excellent repeatability. Vuorinen et al. [15] demonstrated temperature sensors by inkjet printing a graphene/poly (3, 4-ethylenedioxythiophene):poly (styrene sulfonate) (PEDOT:PSS) ink on a skin-conformable polyurethane plaster to monitor temperature changes directly on human skin with a temperature coefficient of resistance higher than 0.06 °C/. Nevertheless, due to the size limitation of inkjet printer nozzles, it remains difficult to realize fine line width for better device performance and miniaturization using standard inkjet printing technology alone. In this paper, we demonstrate size-scalable, conductive graphene-based microstructures with a minimum line width of 20 μm via the combined process of masking and inkjet printing for N/MEMS applications. The proposed printing process overcomes limitations in line resolution for direct inkjet printing of graphene and improves process tolerance to mitigate nozzle clogging for fine feature printing [16].

II. EXPERIMENTAL METHODS

The combined process of masking and inkjet printing comprising three major steps is depicted in Fig. 1. The graphene pattern and position are first defined using a masking mold, which can be either electroplated copper or deep reactive ion etching (DRIE) silicon shadow mask on a substrate. This is followed by inkjet printing of graphene or graphene oxide (GO) flakes onto the mold, and then forming the final structure upon mask removal.

A. MASKING PROCESS

Previously, we demonstrated the combined process of lift-off and inkjet printing (CPLoP) to realize size-scalable printed silver microstructures with 10 μm line width for R, L, C passive component applications [17]. The original CPLoP technique utilized photoresist SU-8 (MicroChem Co., Newton, MA, USA) as a masking layer. Because the Remover PG (MicroChem Co., Newton, MA, USA) used for chemical removal of the SU-8 will also result in the detachment of printed graphene from the substrate, this process was not suitable for patterning printed graphene. Rather, the masking layer for graphene microstructures can be made of electroplated Cu. The Cu mask fabrication started with the deposition of a seed layer of Ti (30 nm)/Cu (120 nm) on a silicon substrate with 1 μm thick SiO₂. A 7 μm thick photoresist AZ-4620 (MicroChem Co., Newton, MA, USA) was then coated and photo-patterned for electroplating a 6.5 μm thick Cu mold. After stripping the photoresist and removing the seed layer using Cu etchant and buffered oxide etch (BOE), the remaining Cu mold on the substrate served as a template for inkjet printing of graphene flakes. To form the Cu mold, this process requires an acidic Cu etchant, which might attack other metals or biomaterials deposited on the same substrate. Thus, for resolving possible process compatibility issues, an alternative masking process was developed using silicon shadow masks fabricated by a conventional silicon DRIE process using a mixture of SF₆ and C₄F₈ gases [18]. The shadow masks were then immersed in a solution of 1 % FAS-17 (Triethoxy-1H, 1H, 2H, 2H-perfluorodecylsilane 97 %, Sigma-Aldrich) for 10 hours to form a fluorine-based hydrophobic surface [19]. The mask treated with the FAS-17 can effectively prevent ink accumulation on the mask and ink leakage along the interface between the mask and the target substrate.

B. INKJET PRINTING GRAPHENE AND GRAPHENE OXIDE FLAKES

The inkjet printing was carried out using a Dimatix DMP-2831 (Dimatix-Fujifilm Inc., Santa Clara, USA) printer with a piezoelectric cartridge (DMCLCP-11610) with 1.5 mL capacity, 21 μm nozzle diameter, and 10 pL nominal droplet volume. During the printing process, the droplet spacing and substrate temperature were kept at 5-20 μm and 60 °C, respectively, to allow rapid evaporation of the
solvent for improved film uniformity. The graphene ink was produced by previously established methods [12] in which a graphene/ethyl cellulose powder was dispersed in a mixture of 85:15 cyclohexanone/terpineol with a concentration of 2.4 wt % solids (~3.4 mg/mL graphene) and viscosity of 10–12 mPa·s at 30 °C. Following inkjet printing, the polymeric binder ethyl cellulose and the solvent were removed through thermal annealing at 260 °C for 30 min to improve structure continuity and electrical conductivity.

In addition to graphene flakes, graphene oxide (GO) can also be patterned using the proposed process. The corresponding ink was prepared by mixing a commercial GO dispersion (Sigma-Aldrich, 4 mg/mL dispersion) with deionized water to form a total of 10 mL GO ink with a concentration of 1 g/L. The GO flakes in the ink were further processed via ultrasonication for 10 min to reduce the size of the GO nanosheets, thereby maintaining GO dispersion and preventing clogging of the printer nozzles.

Following printing of graphene flakes or GO sheets onto the mold and thermal treatment, the Cu mold was chemically etched away or the silicon shadow mask was lifted to yield the graphene-based microstructures on the substrate. For the fabrication of freely suspended microstructures, an additional photolithography step was required using AZ-4620 photore sist to protect the structure pads and open the area for subsequent SiO2 and XeF2 Si etching for structure release. For the process using a silicon shadow mask, the shadow mask and substrate were aligned and pressed to maintain intimate contact using a fixture comprising an upper and lower piece of stainless steel with screws and corresponding threads [20]. An opening was designed in the middle of the upper fixture to provide access for inkjet printing. Following thermal processing, the fixture and shadow mask were removed to obtain the graphene or GO microstructures. Due to the replication characteristic of molding, the masking process can reduce material loss and enable precise microstructures of inkjet-printed graphene and graphene oxide including free-standing graphene beams/sensors and selective cell culturing patterns.

III. PRINTED MATERIAL AND STRUCTURE CHARACTERIZATIONS

A. CANTILEVER GRAPHENE BEAMS

Cantilever beams are an important micromechanical structure of N/MEMS and can be used for a variety of applications due to their linear behavior and high sensitivity [21]. Fig. 2 shows an array of 40 μm wide cantilever beams with the length ranging from 100 to 600 μm fabricated using the proposed combined process of masking and inkjet printing. Fig. 3 shows the measured laser Doppler vibrometer (LDV) spectra of the cantilever graphene beams with 400, 500, and 600 μm length. For a cantilever beam structure, the theoretical resonant frequency can be calculated as follows [22]:

\[
f = 0.162 \times \sqrt{\frac{E}{\rho}} \times \frac{t}{L^2}
\]

where \( \rho \), \( E \), \( t \) and \( L \) are the density and Young’s modulus of the material and the thickness and length of the cantilever graphene beams, respectively. In this case, Eq. (1) can be further simplified as

\[
f = 3.45 \times 10^3 \times \frac{t}{L^2}
\]

in which ideal values for \( \rho \) and \( E \) of 2.2 × 10^3 kg/m^3 and 1 TPa [3], respectively, are used to predict the resonant frequency as a function of beam dimensions. Fig. 4 shows the measured resonant frequency of the printed graphene cantilever beams plotted against the geometric factor \( \frac{t}{L^2} \) in which the slope of the fitted line is 0.45 kHz·m. The measured resonant frequency is thus 7.7 times lower than the theoretical one and, assuming bulk graphite density, the Young’s modulus \( (E) \) is estimated to be 17 GPa, similar to the Young’s modulus of graphite [23], i.e. stacked graphene.

B. DOUBLY-CLAMPED GRAPHENE BEAM FOR PIEZORESISTIVE CHARACTERIZATION

Fig. 5 shows the scanning electron microscope (SEM) micrographs of a free-standing graphene-based doubly clamped beam (beam length = 200 μm, width = 30 μm) with the
and thus,
\[ \varepsilon = \frac{\Delta l}{l} = \frac{2h}{l \sin \theta} - 1 = \frac{2h}{l \sin \left[ \tan^{-1} \left( \frac{2h}{l} \right) \right]} - 1 \]  
while \( l > h \),
\[ \sin \left[ \tan^{-1} \left( \frac{2h}{l} \right) \right] \approx \frac{2h}{l} - \frac{4h^3}{l^3} \]  
Thus,
\[ \varepsilon = \frac{2h^2}{l^2 - 2h^2} \]  
where \( h \) and \( l \) are 20 and 100 \( \mu m \), respectively. A B2902A source meter was used to measure the \( I-V \) characteristics of the printed graphene beam at room temperature. The piezoresistivity of the free-standing inkjet-printed graphene beam is derived from the measured resistivity change as a result of the center deformation using a tip manipulator controlled by a stepper motor with a resolution of 0.1 \( \mu m \). As shown in Fig. 7, the measured resistance change of the graphene beam is 44.4 % with a center deflection of 20 \( \mu m \), equivalent to a maximum strain of 8.7% in the beam structure. From the measurement, the average gauge factor \( G = \frac{1}{R} \frac{dR}{d\varepsilon} \), is derived as \( \sim 5.9 \), which is comparable with prior findings [24]–[26] and similar to the gauge factor of graphite [27]. This result can be attributed to the structure of stacked graphene flakes in the resistor, which is patterned and printed via a Cu mold.

### IV. CASE DEMONSTRATION

#### A. GRAPHENE-BASED TACTILE SENSOR SCHEME

Nowadays, minimally invasive surgery (MIS) represents an important trend in medical operations due to its limited body damage to patients and shorter recovery time in comparison with traditional open surgery [28]. After the patient is anesthetized, MIS is performed by directly inserting surgical instruments into the patient’s body via three or four tiny incisions; one is for the insertion of a miniature camera and the other holes are for placing surgical instruments such as...
scissors and pliers in the operating space. In this way, the surgeon works with visual feedback from a screen for clamping, cutting, suturing, and other operations on the diseased tissues. However, valuable tactile feedback could be provided by palpation, a common practice in which a doctor manually feels the texture of a patient’s tissue but that cannot be performed in MIS. Developing endosurgical tools with force feedback sensors to mimic the process of palpation has, therefore, become an important research topic. Surgeons can obtain surgical tool status and organ information via force feedback sensors to identify, for example, lesions or cancerous lumps in healthy tissue, due to their contrasting hardness [29].

Different MEMS-type tactile sensors have been developed and implemented to detect hardness differences based on a tactile feedback stress or strain ratio in MIS [30]–[32]. Nevertheless, none of these sensors can truly reflect the realistic organ sensing experience of actual palpation. The doctor’s hand, in fact, senses two forces while performing palpation, namely the force applied and the force feedback of the tissue. Thus, we propose a back-to-back two-spring linked tactile sensor design to mimic human fingers for MIS applications as shown in Fig. 8 (a) [33]. The two sensing elements linked by a freely standing rod are accommodated on the top and bottom sides of a 3D-printed package. The deformation of the top element will result in a force directly transferred to the bottom element through the rod, causing it to press into the tissue. The bottom element can sense the hardness difference via its deformation resulting from the force feedback from the tissues, allowing measurement of both applied displacement and resulting force with the top and bottom sensors simultaneously.

For the palpation application where the Young’s modulus of soft tissues is only tens of kPa [30], the polymer-based tactile sensor with comparable Young’s modulus is preferred for the sake of having detectable deformation. Polymer materials, however, would encounter fabrication process compatibility issues such as low glass transition temperature ($T_g$) and susceptibility to plasma damage, which will increase the process complexity for the utilization of polysilicon piezoresistors in the palpation sensor design. Due to the low temperature compatibility and patterning versatility of inkjet printing, along with the gauge factor of 5.9 for the inkjet-printed graphene comparable with that of polysilicon [34], the process here offers a viable route to fabricate a sensing element based on two cross SU-8 beams incorporated with the printed graphene piezoresistors.

The detailed fabrication process of the proposed sensing elements of the tactile sensor can be referred to [33], which is based on the combined process of Cu masking and graphene printing. Fig. 8(b) and (c) juxtapose SEM micrographs of as-fabricated top and bottom piezoresistive sensing elements, respectively. The SU-8 tip on the bottom sensing element is designed to ensure that the feedback force is directly transferred from the contact object to the sensing beam. While the bottom SU-8 is deformed by feedback force, a micro strain will be induced in the graphene resistor and result in a resistance change. The force load $P$ and deformation displacement $y$ for a doubly clamped beam can be derived from:

$$y = -\frac{PL^3}{48EI} \quad (8)$$

where $I$, $b$, $h$, $E$ and $L$ are the moment of inertia, width, height, Young’s modulus and length of the SU-8 beam, respectively. In this sensor structure, the cross clamped beam is equivalent to two beam springs connected in parallel, so Eq. (8) can be further revised as:

$$y = -\frac{PL^3}{96EI} \quad (9)$$

Fig. 9 shows the characterization results of the top and bottom piezoresistive sensing elements deformed by an in-house
nano-indentation stage, which used a stainless steel tip controlled by a stepper motor with a resolution of 0.1 µm. For the top sensing element, we measured the relation of central displacement vs. the resistance change of the printed graphene resistor. The relative resistance change increases with the induced strain resulting from the tip displacement. Larger deviation between the measurement results and the theoretical calculation derived from the gauge factor and the resistance change at larger displacements can be attributed to the occurrence of non-linear deformation in the SU-8 beams. For characterization of the bottom sensing element, it was placed directly on a load cell and pressed by the tip so that the relation of the resistance change versus the reactive force can be obtained. The measurement results align well with the theoretical calculation up to 4 mN. Fig. 10 shows the piezoresistor responses from the bottom sensing element, illustrating that fat and muscle tissues lead to distinct force feedback characteristics under the same applied translational deformation via the central truss compressed by the top element. The muscle tissue, with a higher hardness than the fat, exhibits a larger force feedback. As a result, the corresponding relative changes of resistance exhibit slopes of $3.4 \times 10^{-4}$ and $1.1 \times 10^{-3}$ mN/µm for the fat and muscle tissues, respectively. The higher slope corresponding to muscle tissue indicates a larger feedback force and thus a harder tissue, validating the proposed palpation sensor scheme for differentiation based on tissue hardness.

**FIGURE 10.** The measurement of actual displacement vs. force feedback from fat and muscle tissues using the back-to-back graphene-based tactile sensor.

### B. SILICON SHADOW MASKING, INKJET PRINTING AND PATTERNING PROCESS (SSMP) OF GRAPHENE OXIDE AND REDUCED GRAPHENE OXIDE FOR SELECTIVE CELL CULTURING APPLICATIONS

Previously, a range of bio-electrode materials has been studied including polypyrrole/collagen, poly(3, 4-ethylenedioxythiophene) (PEDOT), reduced graphene oxide (rGO), and IrO$_x$, among others [35]–[37]. GO micropatterns have even been proposed as an interface between human retinal pigment epithelium (RPE) cells with microelectrodes for facilitating a long-term implantation of artificial subretinal chips [42]. Recent technology development in the field of organ-on-a-chip systems has paved the way for miniaturized microfluidic 3D human organ models including liver, heart, and brain organoids-on-a-chip in which the biological and physiological parameters corresponding to in-vivo counterparts are detected and recapitulated for personalized medicine. This technology can help doctors and scientists in biological and medical fields understand drug dynamics via in-situ observation of the effects of pharmaceutical compounds on organoids [43], [44]. The investigation of multiple biocompatible electrodes for the microfluidic chip are critical in terms of physiological signal detection. Thus, we demonstrate here patterned GO and rGO by employing the size-scalable combined process technology of masking and inkjet printing and show that RPE and PC-12 cells adhere well and grow selectively on the patterned GO films.

The detailed mask preparation and printing process can be referred to [20]. Although GO exhibits good biocompatibility, its electrical conductivity is too low to be suitable as an electrical sensing material. To improve the conductance of the printed GO, we thermally anneal it at 200 °C in the ambient environment for 10 hrs to form rGO. Fig. 11 shows the $I$-$V$ measurement result of the rGO based on the prior printed four-point electrical resistance measurement pattern indicating a linear ohmic characteristic for a 1000 µm long and 40 µm wide rGO line, exhibiting a sheet resistance of 33.48 kΩ/sq. As compared to the GO with a resistance over 300 MΩ/sq, the 200 °C reduction reaction indeed provides an effective method to enhance conductance. Although the 33.48 kΩ/sq sheet resistance is still too high to be applicable for direct signal detection, it can result in a potential difference within the rGO pattern to assist the neurite outgrowth and orientation control of cultured neuron cells [35].

**FIGURE 11.** The four-point electrical measurement of printed graphene and reduced graphene oxide (rGO) films.
Polydimethylsiloxane (PDMS) is a common structural material in many organ-on-a-chip microfluidic devices owing to the unique characteristics of high permeability for gases and small molecules, optical transparency, and mechanical flexibility for cell culturing. Therefore, inkjet printing of GO on PDMS using a silicon shadow mask with the electrode array pattern in an artificial retinal chip was performed to show its potential for selective cell culturing applications. Here, each substrate printed with the GO pattern was placed in a cell culturing well containing $2 \times 10^5$ RPE cells for 7 hrs, followed by an additional 24 hrs after refreshing the media. Immunofluorescence staining with 4, 6-diamidino-2-phenyl indole (DAPI, blue) was applied to confirm the location of cell nuclei. Fig. 12(a) and (b) shows RPE cells have successfully attached to the GO patterns on both substrates. No cell cytotoxicity has been found in the cell attachment experiment. Meanwhile, by comparing the culturing results in the SiO$_2$ and PDMS substrates, we found that RPE cells adhere well and grow only on the GO and SiO$_2$, which can be attributed to the hydrophilic nature of the surfaces. As a result, the combination of hydrophobic PDMS substrate and the printing technology using the shadow mask can facilitate superior patterning for cell culturing. In addition to GO, Fig. 12(c) shows neurons with axons also well adhered and differentiated on rGO electrodes, which can be utilized for future investigation of cell-to-cell neurotransduction.

**V. CONCLUSION**

In conclusion, we demonstrate a patterning technique to facilitate the fabrication of precise and versatile graphene-based microstructures using the combined process of masking and inkjet printing. Inkjet-printed micromechanical beams have been successfully fabricated and characterized with gauge factor of piezoresistivity and Young’s modulus of 5.9 and 17 GPa, respectively. Leveraging the process versatility, an inkjet-printed back-to-back linked graphene tactile sensor concept has been demonstrated with the capability to differentiate tissue hardness, offering strong potential for practical clinical application in endosurgical palpation. Size-scalable patterned GO and rGO microstructures on substrates for selective cell culturing have also been realized. Neuron cells including RPE and PC-12 cells can well adhere and grow on the GO and rGO patterns to form a cell/electrode interface for implantable retina-stimulated chip packaging and future exploration of cell-to-cell neurotransduction.

**ACKNOWLEDGMENT**

The authors would like to thank the Nano Facility Center (NFC) in NCTU for the support of fabrication facility. The U.S. Government is authorized to reproduce and distribute reprints for Governmental purposes notwithstanding any copyright notation thereon. The views and conclusions contained herein are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, of the sponsors.

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