Optimization of Microstructure Patterning for Flexible Bioelectronics Application

Ishi Gupta[1], Manika Choudhury[2], G. Harish Gnanasambanthan[3] and Debashis Maji[4*]

[1]*Department of Sensor and Biomedical Technology, School of Electronics Science and Engineering (SENSE), Vellore Institute of Technology, Vellore, Tamil Nadu, India – 632014; ishi.gupta2022@vitstudent.ac.in*
[2]*Department of Sensor and Biomedical Technology, School of Electronics Science and Engineering (SENSE), Vellore Institute of Technology, Vellore, Tamil Nadu, India – 632014; manika.choudhury2022@vitstudent.ac.in*
[3]*Department of Sensor and Biomedical Technology, School of Electronics Science and Engineering (SENSE), Vellore Institute of Technology, Vellore, Tamil Nadu, India – 632014; harish.gnanasambanthan2019@vitstudent.ac.in*
[4]*Department of Sensor and Biomedical Technology, School of Electronics Science and Engineering (SENSE), Vellore Institute of Technology, Vellore, Tamil Nadu, India – 632014; debashis.maji@vit.ac.in*

*__Correspondence:__ *debashis.maji@vit.ac.in*

ABSTRACT- Recent advancements in flexible electronics and wearable sensors have given biomedical technology a new edge overcoming the limitations of traditional rigid silicon-based electronics. Furthermore, high flexibility of these wearable sensors enables it to conformally sit over any uneven surface helping in accurate determination of any physical, chemical, or physiological parameter associate with the surface. Conventionally expensive micro/nano photolithography techniques under strict clean room conditions are used for the development of these flexible and wearable biomedical sensors with high degree of accuracy and sensitivity. However, the developed wearable sensors need not only be extremely sensitive, but also cost effective for its successful usage. To address this, the present work discusses the use of a photo-patternable UV sheet for realization of micro patterns over flexible copper cladded surface eliminating the need of costly clean room facilities. It demonstrates the standardization of various design geometries using the photo-patternable UV sheet over the flexible surface similar to photolithography process and involves optimization of the exposure timing of the UV sheets and their development time towards various design patterns over different thick film metal surfaces. Finally, patterned micro devices like micro-electrodes were successfully realized using the above process to ascertain its efficacy.

General Terms: Flexible electronics, Photolithography,

Keywords: Photo-patternable UV sheet; micro-electrodes; photolithography; flexible electronics; wearable sensors.

ARTICLE INFORMATION
Author(s): Ishi Gupta, Manika Choudhury, G. Harish Gnanasambanthan and Debashis Maji;
Received:24/04/2023; **Accepted**: 15/06/2023; **Published**: 20/09/2023;
E- ISSN: 2347-470X
Paper Id: IJEER240412;
Citation: 10.37391/ijeer.110315
Webpage-link:
https://ijeer.forexjournal.co.in/archive/volume-11/ijeer-110315.html

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1. INTRODUCTION

Flexible electronics has captured attention due to their promising implementation in wearable or implantable electronics, rollable display screens [1]–[4], etc. Due to its highly flexible materials, these are easily bendable, and folded, opening research for wearable electronics for healthcare applications, security, etc. These are flexible and lightweight, and they connect sensing elements to create an interactive interface for various electronics, most of which are wearable [2], [5], [6]. It is well evident that flexible sensor is the upcoming area of interest for tracking down the human biological activities, personalized healthcare systems, and food and security purposes [1]. Researchers are focused on improving device materials, force-sensitive interfaces, and device structures in order to improve the sensing performance of flexible sensors. The device configuration gives it an upper edge due to low-cost, low-power consumption characteristics [7]. The growth of flexible sensors and microelectronics for development of low-cost frameworks for rapid and sensitive detection necessitates the use of reusable, economically viable, and easily manufacturable electrodes. Furthermore, cost - efficient, sensitive, specific, user-friendly, and durable electrodes for sensing applications using a simple strategy are highly sorted after [8]. Various techniques make a device cost-effective and reliable [6]. Printing techniques like screen printing, inkjet printing and photolithography are some effective techniques that helps in fabricating the electrodes [8]–[12]. Printing methods, including photolithography, have some advantages over the others, such as simple processing techniques, reduced material waste, and low cost, making printing methods a very appealing way to fabricate cost-effective sensors [13]. Moreover, there is uniformity, and good quality electrode films are obtained [14].

The size of an electrode is an important consideration when designing it in order to avoid skin damage and help to reduce edge effects. Since the electrodes are closely designed with less separation, miniature electrodes provide focused stimulations[15]. Interestingly, much research is currently ongoing in the field of fabrication of the microelectrodes for developing extremely sensitive sensors. In this regard, J Gupta et al. discussed a technique of using gold nanoparticles and gold nanoflowers as effective coatings over copper electrodes to enhance its sensitivity. However, the process may not be cost-effective [16]. The accurate and confined estimation of different analyte concentrations enables the prediction of disease[11]. Currently, a simple and stable method for fabricating such microelectrodes over a flexible substrate is yet to be explored, in the race of development of exceptional biological wearing devices. In the present work a simple and cost-effective method of patterning microelectrodes through non-conventional photolithography process using a commercially available UV photo sheet has been reported. Further, the study also aims to perform optimization of microstructure patterns on flexible substrate for various flexible bioelectronics applications.

In the present investigation, to obtain micro patterns on a bendable substrate, copper and UV sheets have been used. Instead of big bulky electrodes, thin electrode sensor design has been printed onto the copper as electrode, and glass substrate, using photolithography and etching. As mentioned earlier, this technique is comparatively adaptable, plain and feasible. Various parameters like UV exposure time, concentration of developing solution, involvement of heat, have been analyzed to standardize the fabrication steps. Conclusively, a micro device has been realized for bioelectronics purpose.

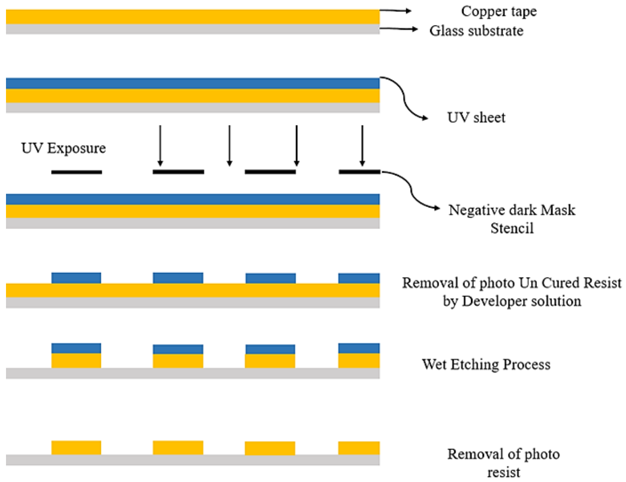
2. MATERIALS AND METHODS

2.1 Chemicals and materials

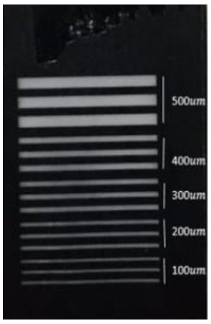
Sodium hydroxide (NaOH) chemical from CHENCHEMs is used as a developing solution. 10 gm NaOH in 100 ml deionized (DI) water. This concentration has been varied to obtain the desired concentration for our purpose. 25 µm thick copper tape was used for fabricating the electrodes. Glass slide from BLUESTAR (Polar industrial corporation, Mumbai, India) was initially used a substrate to affix the copper tape to obtain the pattern. Photo-patternable UV sheets were used to expose the pattern onto the copper tape. Finally, Isopropyl alcohol (IPA) was used to clean the substrate throughout the process.

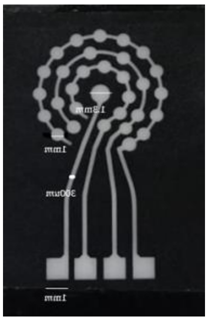
2.2 Experimental procedure

An UV exposure unit from ULTRA VITALUX of wattage 300 W, working in the range 315-400 nm (UVA) wavelength was used to print the patterns on the copper surface. Certain parameters were changed to achieve a standardized fabrication process. To begin, initially, investigation of the effect of simultaneously heating the UV sheet and the copper substrate for proper adhesion of the two layers was explored. Second, the UV exposure time was varied from 5 minutes to 7 minutes. Third, the weight of NaOH used in the development process varied from3 gm in 100ml of DI water to 5 gm, and 10 gm. Finally, to standardize the minimum feature size achievable through the use of UV sheets, the shape and size of the electrodes were also varied to as low as 100 µm width.



(a)





(b) (c)

Figure 1: (a) Fabrication process flow over copper tape using photo-patternable UV sheet, (b) & (c) shows the negative photo masks used for the present study

2.3 Fabrication optimization

Various parameter optimizations were carried out to successfully achieve desired microstructure patterns over copper tapes. The photomask was developed, and various electrode designs were printed on the mask for the fabrication.

Figure 1(a) illustrates the process flow for fabrication of the desired microstructure which involves placement of the substrate and copper tape adhered on top of it. The glass surface was initially cleaned with IPA, to remove any excess dirt from the surface. UV sheet were thereafter placed over the copper tape and heated adequately. Negative dark mark stencils were preferably used to get the desired patterns, under the UV exposure. *Figure 1(b, c)* depicts the dimensional analysis for the different photomasks used for this work. Furthermore, developer solution was used for the removal of the unexposed region. Later, *via* wet etching technique of uncovered copper, the desired pattern was obtained on the glass slide.

2.3.1 Optimization of heating effect

Proper adhesion of the UV sheet over the copper tape is highly essential for successful realization of the desired patterns. In this context, the UV sheet was placed over the copper tape, and its adhesion with the copper tape was tested by heating the assembly with an iron box for 5 seconds. Adhesion between the two surfaces was also tested without applying any heat and the results were recorded and analyzed.

(a) (b)

Figure 2: (a) and (b) UV patterns obtained without application of any heat over the UV Sheet

2.3.2 Optimization of UV exposure time

A photo mask was centered on the UV sheet, and this setup was exposed under the UV exposure unit for desired period, as shown in the process flow of *figure 1(a)*. The exposure time was varied at 5 minutes and 7 minutes. The negative photomask was employed in this lithography technique, which implies that in the dark region of the pattern, UV light will be blocked, and the underlying sheet will remain un-polymerized, whereas, in the lighted regions, the UV sheet will be exposed, and polymerization of the exposed areas will help in retaining the desired pattern over the substrate.

2.3.3 Optimization of developing solution concentration

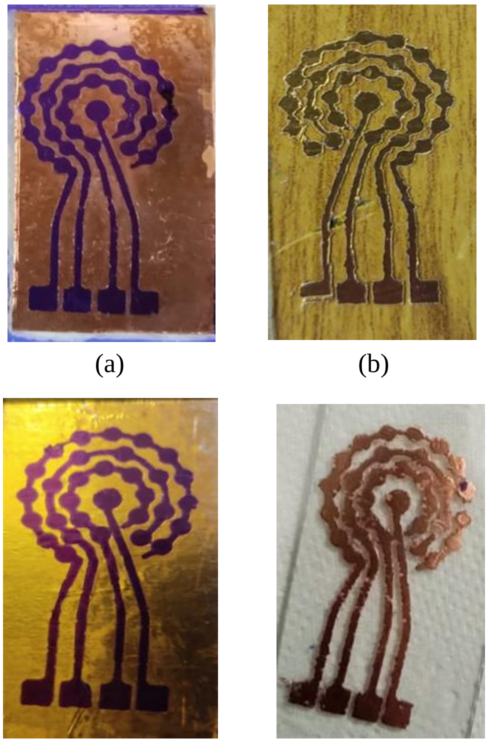
For development of the electrode, 10gm of NaOH by weight in 100ml of DI water was preferred. For experimental analysis, this concentration was varied from 3gm to 5gm, and 10gm respectively. Etching was followed up once the electrode design had been attained post development process of the UV sheet. Ferric chloride as an etchant was prepared, by mixing 15gm $FeCl_3$ in 100 ml of DI water. Etching is the removal of unprotected area of a metal surface with a strong acid to create an incised design in the metal [16]. It will enhance the microstructure pattern on the substrate and wash off all the unwanted residues from the surface, except the desired pattern. Once the etching is done, it was rinsed with acetone to remove the surplus residues of UV sheet from the substrate.

3. RESULTS AND DISCUSSION

The given study accounts to fabricate microelectrodes, using simple photolithography technique, in the presence of UV light using UV sheets.

3.1 Heating effect on the patterns

Figure 2 (a), and (b), shows the images that were obtained when no heating was applied over the copper and UV surface. As a result, it was observed that the UV did not adhere properly on the copper tape, due to which stable pattern was not obtained. The sheet was thereafter placed over the copper tape and heated with an iron box for 5 seconds and the results obtained showed good adhesion and proper replication. The exposure time was kept as 7 minutes, while the developing solution was kept constant at 10gm in 100ml DI water.



(a) (b) (c) (d)

Figure 3: Patterns obtained when the UV exposure time was 5 minutes (a) over UV Sheet and (b) after etching; Patterns obtained when the UV exposure time was 7 minutes(c) over UV Sheet and (d) after etching.

3.2 Exposure time

The extent of exposure on the substrate in the UV exposure unit has a large impact on patterning. It was worth noting that only the exposure time was varied here, with all other parameters remaining constant, i.e., 10 gm NaOH in 100 ml DI water, 15 gm $FeCl_3$ in 100 ml DI water, and 5 seconds of heating to stick the UV sheet over the copper tape. Figure 3(a), and (b) depicts the images when the exposure time was considered as 5 min. The designs were initially found to be proper; however, it was found that after few minutes of development the design peeled off, if not initially cured properly. On the other hand, increasing the exposure time to 7 min, made the developing slightly easy. There was no distortion observed in the developed UV patterns. Fine and thin patterns were notably marked. Figure 3 (c), and (d) shows the results for 7 min exposure time.

3.3 NaOH developer solution optimization

NaOH is an alkali developer solution that removes the excess amount of unexposed UV sheet area i.e. except from the pattern. To obtain fine electrodes without distorting the

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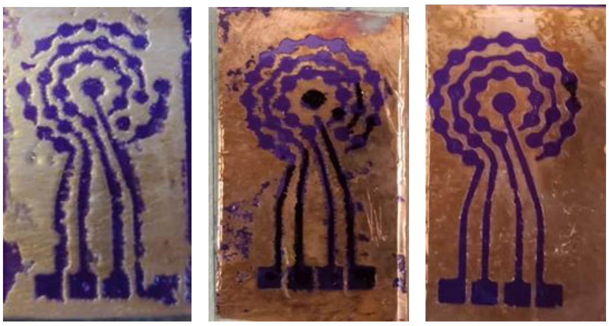
Research Article | Volume 11, Issue 3 | Pages 738-742 | e-ISSN: 2347-470X

pattern, the UV exposed substrate is dipped in the developer solution for a few seconds before rinsing in DI water. After



(a) (b) (c)

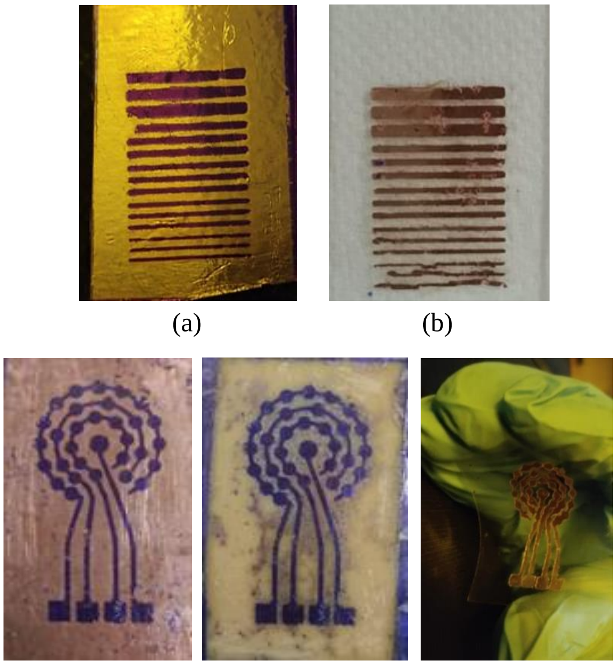
Figure 4: (a) Fabrication with 3% developing solution, (b) Fabrication with 5% developing solution, (c) Fabrication with 10% developing solution

development, the exposed area is insoluble, leaving the desired pattern. 3 gm, 5 gm, and 10 gm of NaOH in 100 ml of DI water was chosen as the variations. In the first case, it was discovered that the development time was ~30 seconds and 3 gm of NaOH caused the pattern to scrape off the substrate surface. Given the considerations, we limited the concentration to 5 gm/100 ml DI water which took ~10 seconds to develop with non-uniform patterns. The results in *figure 4 (a), and (b)* shows that the design was retained but it did not turn out to be appropriate. Finally, at 10 gm of NaOH in 100 ml of DI water led to proper development of UV negative mask and was achieved in ~5 seconds with proper electrode designs over the copper surface, as shown in *figure 4 (c).*

After optimizing the parameters, etching was performed using 15% aqueous solution of $FeCl_3$ (ferric chloride) [17]– [21] for ~25 minutes. The etching was performed using continuous mechanical agitation of etchant at room temperature in a glass container. *Figure 5 (a-e)* shows the final structure on the copper tape, PCB as well as over PET substrate. The excess UV was wiped off with acetone gently, keeping the patterns intact. The structures over PET substrate were further subjected to flexibility test to ensure the structural integrity of the same. The initial resistance of the outer array of electrodes was observed to be 0.141 Ω and the entire electrode array was thereafter subject to periodic bending of over 100 cycles manually. Finally, the resistance of the same outer array was measured after the bending test and was observed to be 0.15 ± 0.003 Ω which was almost like the initial resistance indicating negligible effect on the structural integrity due to the applied bending strain. Microscopic observations also did not show formation of any cracks over the same confirming its mechanical continuity.

3.4 Realization of the micro device

Successfully patterning microelectrodes and straight lines of varying line widths were achieved after various trials and optimization of different parameters as explained earlier. The present study proposes a microelectrode array (MEA) design for use in electrical stimulation for wound healing applications. Besides, these MEAs can also be used in other applications like nerve stimulation, actuation, biosensing and electrochemical applications, and also for various biomedical



(a) (b)

(c) (d) (e)

Figure 5: (a) and (b) line patterns obtained when the UV exposure time increased to 7 minutes, and (c) & (d) electrode patterns obtained on the PCB substrate and (e) over flexible PET substrate using the same optimized conditions.

applications like in bioreactors, 3D printers, microheaters, etc. [22]. These miniaturized devices can open a new research domain for wearable or implantable biosensors, self-powered sensors, and micro device-based system for measuring or analyzing the bio signals or can be used in certain biomedical applications.

4. CONCLUSIONS

The observation in present study is based upon the experimental data performed, by studying the influence of various optimization factors including, UV exposure time, NaOH concentration in developing solution, and heating effect. It was notably found that a standard optimization for the fabrication of micro electrodes has been successfully achieved by incorporating initial heating of the top UV sheet for 5 seconds to promote adhesion with the substrate followed by UV exposure for 7 min for a developer solution of 10 gm of NaOH in 100 ml of DI water which took 10 seconds for proper development. Final patterns were obtained by etching the copper patterns with $FeCl_3$ for 25 min. A 100 µm size was successfully patterned over the glass substrate, which can be performed over polyimide surface, which acts as convenient flexible substrate. Lastly, the obtained standardization technique can be used to fabricate not only microelectrodes, but even thin film patterning based other devices like microheaters, interconnects, physiological sensors, etc. with even other available roll to roll materials like gold, silver, aluminum, etc. for various biomedical applications, where the need of wearable and miniaturization is increasing rapidly. We believe that this experiment will open a new avenue of research in the field of biosensors for the fabrication of micro devices for a variety of applications.

5. ACKNOWLEDGMENTS

The authors are grateful to the Science and Engineering Research Board under the Department of Science and Technology (DST-SERB), India, for sponsoring the funds from the project grant DST SERB: File Number/ECR/2018/001295. The authors would also like to acknowledge the DST-FIST supported MEMS and Chemical sensors lab (TT 717) (SR/FST/ETI-015/2011), VIT Vellore and PCB Lab (TT705), and VIT Vellore for extending its fabrication facilities to aid in the successful completion of this project.

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