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Simple and scalable fabrication method of organic electronic devices on textiles

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Abstract:	Today wearable electronics devices combine a large variety of functional stretchable and flexible technologies. However in many cases these devices cannot be worn under extreme daily conditions. Therefore textiles are commonly the best substrates to accommodate electronic devices in wearable use. In this paper we describe how to selectively pattern on textiles organic electroactive materials from solution in an easy and scalable manner. This versatile deposition technique enables the fabrication of wearable organic electronic devices on clothes.
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TITLE:
A simple and scalable fabrication method for organic electronic devices on textiles

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Patterning, textiles, conducting polymers, organic devices, wearable electronics, organic electronics, e-textiles

SHORT ABSTRACT:
In this paper, we present a protocol to selectively deposit organic materials on textiles, which allows for the direct integration of organic electronic devices with wearables. The fabricated devices can be fully integrated in textiles, respecting their mechanical appearance and enabling sensing capabilities.

LONG ABSTRACT:
Today, wearable electronics devices combine a large variety of functional, stretchable, and flexible technologies. However, in many cases, these devices cannot be worn under everyday conditions. Therefore, textiles are commonly considered the best substrate to accommodate electronic devices in wearable use. In this paper, we describe how to selectively pattern organic electroactive materials on textiles from a solution in an easy and scalable manner. This versatile deposition technique enables the fabrication of wearable organic electronic devices on clothes.

INTRODUCTION:

The field of wearable electronics is a fast-growing market expected to be worth 50 billion euros in 2025, over three times the current market. The main challenge facing current wearable devices is that intrusive solid electronic attachments limit the usage of established devices in wearable systems. Using textiles that are already present in everyday life is a very attractive and straightforward approach to avoid this limitation. Due to its elastic capability, some parts of the clothing that we wear are naturally in tight contact with the skin. Many examples of smart clothes available on the market today are based on thin, plastic displays, keyboards, and light source devices embedded in textiles, linking electronics with humans in a fashionable way[1]. In sport practice, health monitoring relies on textile electrodes, which offer comfortable alternatives to commonly used adhesive electrodes and metal wristbands. Here, conductive fibers are directly integrated with stretchy fabrics to prevent skin irritation and other discomforts during extended wear. Additionally, textiles offer a number of opportunities to integrate curvature sensors to capture motion[2], to integrate shear sensors for the development of functional robotic actuators[3], and certainly to integrate biosensors through the detection of an analyte in sweat[4].

Modern wearable technology relies on carbon-based semiconductor materials that deliver electronic devices with unique properties. The “soft” nature of organics offers better mechanical properties for interfacing with the human body compared to traditional solid-state electronics. This mechanical compatibility, paired with mechanically flexible substrates, enables the use of non-planar form factors in devices such as textiles. The use of organics is also relevant in life sciences due to their mixed electronic and ionic conductivity[5]. Besides, organic semiconducting and optoelectronic materials empower a large variety of functional devices with display, transistor, logic, and power capabilities[6,7,8,9]. The main difficulty in the fabrication of such organic devices is the controlled deposition of functional materials on the non-planar surfaces of textiles. Conventional microfabrication techniques are primarily limited by the incompatibility of the deposition process with the structural dimensionality of textile substrates.

Here, we describe a simple and scalable fabrication protocol that allows for the selective deposition of conducting polymers on structured textiles. The presented process enables the fabrication of wearable and conformal electronic devices. The approach is based on the patterning of the commercially available conducting polymer poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS) and an elastomeric stencil material polydimethylsiloxane (PDMS) on textile. This combination allows for the efficient confinement of the aqueous PEDOT:PSS solution, as well as for the retention of the soft and stretchable properties of textiles. This simple and reliable fabrication method paves the way for the fabrication of a variety of electronic devices directly on textiles in a cost-efficient and industrially scalable manner.

PROTOCOL:

1. Patterning conducting polymers on textile

1.1. Fix a 10 cm x 10 cm textile sheet on a planar surface for easy handling during the process. For the textile, use a 100% interlock knit polyester fabric with a thickness of 300 µm and a knit direction stretch capability up to 50%.

1.2. To make a mask containing the patterning design, use a 125 µm-thick polyimide film; an example of the pattern is illustrated in Figure 1.

1.2.1. Use a laser cutter (*e.g.,* Protolaser S, LPKF) to pattern the polyimide mask[10]; the pattern design of an electrode is illustrated in Figure 1.

1.2.2. Coat the PDMS formulation (10:1 base to curing agent ratio) on top of the mask (polyimide film) using an automatic tape casting tool (K control print-coater, doctor blade) with a wet film thickness of 200 µm and at a 6 m/min coating speed. Use about 0.5 mL for a mask of 3 cm x 5 cm. Perform this process under the fume hood.

1.3. Gently transfer the fabric to the PDMS-coated mask. Leave for 10 min, after which the PDMS should be fully absorbed in the textile structure.

1.4. Cure the sample in an air-oven at 100 °C for 10 min.

1.5. Prepare the conducting polymer:PEDOT:PSS dispersion (80 mL), ethylene glycol (20 mL), 4-dodecylbenzenesulfonic acid (40 µL), and 3-methacryloxypropyltrimethoxysilane (1 mL) in the fume hood.

1.6. Brush-coat the PEDOT:PSS solution on the PDMS-free area of the textile until a homogenous penetration of the solution is obtained. Repeat this step to achieve a uniform pattern color. Apply about 1 mL/cm^2.

1.7. Cure the fabric at 110 °C for 1 h to dry the PEDOT:PSS solution. Reduce the temperature to 60 °C for textiles that are sensitive to high-temperature treatment, like nylon.

2. Organic device fabrication

Note: The protocol in Section 1 describes the selective deposition of conducting materials on textiles. The following sections will describe the additional steps needed to fabricate organic devices, like stretch sensors, OECT transistors, cutaneous electrodes, and capacitive sensors.

2.1. To fabricate stretch sensors, shown in Figure 3a, pattern the electrode lines on the textile, as described in Section 1, steps 1.1-1.5.
Note: An example of the pattern design is shown in Figure 3a. The fabrication of such sensors does not require any additional steps.

2.2. To fabricate the transistor design shown in Figure 3b, pattern the transistor arrays on a nylon woven ribbon following the steps described in Section 1. Slightly modify the PDMS annealing and PEDOT:PSS curing steps to avoid the thermal degradation of nylon by curing at 60 °C for a longer time.

2.3. For the fabrication of cutaneous electrodes, shown in Figure 3c, deposit an ionic gel on the patterned PEDOT:PSS textiles.

2.3.1. Prepare an ionic liquid gel mixture containing the ionic liquid, 1-ethyl-3-methylimidazolium-ethyl sulfate; the cross-linking agent, poly(ethylene glycol)diacrylate; and the photoinitiator, 2-hydroxy-2-methylpropiophenone at a (v/v) ratio of 0.6/0.35/0.05, respectively.

2.3.2. Coat the PEDOT:PSS electrode with ionic liquid (20 µL/cm^2) and add the ionic liquid gel mixture from step 2.3.1 (25 µL/cm^2) by drop casting.

2.3.3. Expose to UV light (365 nm) to initiate a crosslinking reaction for 10-15 min, until the gel solidifies. Perform this step in the fume hood. Use a UV-protective cage during UV exposure.

2.4. For capacitive sensor fabrication, use PEDOT:PSS textile electrodes insulated with an insulating material (Figure 3d).

2.4.1. Insulate the keyboard-like PEDOT:PSS electrodes using the PDMS; the keyboard design can be seen in Figure 2b. Dispense the PDMS formulation on top of the fabric and remove the excess with a squeegee.

2.4.2. Place the fabric in an oven at 100 °C for 10 min. Perform this step in the fume hood.

REPRESENTATIVE RESULTS:

Traditional methods for applying colors or patterns to textiles rely on removable masking layers to allow the selective deposition of dyes. In Figure 1, we show the adaptation of such an approach to the patterning of PEDOT:PSS electrodes on textiles. As a masking layer, we used hydrophobic polydimethylsiloxane, which can restrain the non-controllable diffusion of the aqueous PEDOT:PSS solution. Moreover, the softness and stretchability of knitted and woven textiles can be preserved thanks to the elastic and mechanical properties of the PDMS.

In Figure 1, the process starts with the preparation of the patterning master from polyimide film (step 1). The design of the pattern outline is carved onto the film by a laser. Using a tape casting tool, the PDMS is applied on this master (step 2), and the textile is placed on top of it (step 3). The PDMS is then progressively diffused into the textile (step 4). To stop this transfer, a short thermal annealing process is required to cure the PDMS. The viscosity and thickness of the PDMS can be adjusted by using different amounts of the curing agent and coating parameters, respectively, to control the diffusion and to insure the faultless replication of the master design. Finally, the conducting solution is brush-painted on the unprotected textile and baked to dry (step 5). The polyimide master is then delaminated from the textile surface. The results of the fabrication flow are illustrated in Figure 1, on the right. In this case, successful patterning was placed on knitted polyester. The patterning resolution on such a textile is greater than 1 mm. However, lower resolution can also be obtained on tightly knit or woven textiles. Using this deposition technique, the estimated sheet resistance of the conducting textile is close to 230 Ω/sq.

Examples of functional electronic devices on knit and woven textiles are shown in Figure 3a and b, including successfully fabricated PEDOT:PSS electrodes on knit textiles. The natural horseshoe arrangement of the fibers in knit textiles provides adjustable stretchability to fabrics. This spring-like capability of knit structures can result in highly sensitive strain sensors[11]. A simple deformation in the textile structure is reflected by a change in the electrical resistivity due to the twisting of conductive fibers in the threads. Additionally, by taking advantage of the hygroscopic capacity of the textiles, the array of electrodes in Figure 3b was patterned on textiles to make planar transistors with rectangular channels and different gate widths, which can be used in wearable sweat sensing. Such a geometrical configuration is used in organic electrochemical transistors (OECT) for sensing which channel and gate are linked by a sample of an analyte[12].

The presented patterning technique can be extended to fabricate complex organic electronic devices on textiles. As the PDMS stencil remains in the textile after the patterning process, additional layers can be patterned on PEDOT:PSS-coated conducting textiles. In Figure 2, we present the process in which an ionic liquid gel solution (Figure 2a) and the PDMS formulation (Figure 2b) were applied to functionalize or isolate the surface of a PEDOT:PSS electrode, respectively. Ionic gels are largely used in cutaneous electrodes. The incorporation of an ionic gel in conducting textiles was used to fabricate wearable textile electrodes for electrophysiological monitoring[10] and is illustrated in Figure 3c. Capacitive sensors were made by insulating the textile electrode surface with PDMS. A change in the capacitance was detected when the electrode was touched. Such a touch-sensitive device was used to fabricate an organic electronic textile keyboard[13], as shown in Figure 3d.

Figure 1. Process flow illustrating the patterning of conducting polymers onto textiles.
Process flow illustrating the patterning of conducting polymers onto textiles. Step 1: mask preparation, step 2: PDMS deposition on the polyimide patterning mask defining the outline of the desired design, step 3: transfer of the masking layer by the placement of the textile on the PDMS-coated mask, step 4: transfer of the PDMS into the bulk of the textile, step 5: deposition of conducting polymer solution onto unprotected textile. The pictures on the right show the results of the key steps of the process flow.

Figure 2. Two examples of fabrication organic devices.
Two examples of fabrication organic devices. a) Ionic liquid gel coating on the PEDOT:PSS textile electrode for cutaneous sensing. b) Insulation layer deposition on the PEDOT:PSS textile electrode for touch sensors.

Figure 3. Photographs of organic electronic textile devices.

a) PEDOT:PSS electrodes for stretch sensing b) Array of OECT transistors for wearable biosensing. c) Circular PEDOT:PSS electrode coated with an ionic liquid gel for cutaneous electrophysiology. d) Organic touch sensors for a wearable keyboard.

DISCUSSION:

The patterning of conducting materials is one of the first steps in the fabrication of functional electronic devices. This can become challenging, as the fabrication process needs to take into account the chemical and physical properties of such materials, and the process flow needs to consider the material cross-compatibility between the fabrication steps. In the microfabrication of organic electronic devices, these two aspects are even more significant due to the highly reactive nature of organics. Today, however, organic materials are highly attractive to wearable and flexible electronics for their electro-elastic properties[14,15]. The transfer of such technologies to textiles to obtain fully integrated electronic wearables is limited by their three-dimensional structures. Conventional techniques used in microfabrication are restricted to inkjet or screen printing of conducting inks only on thin substrates and textiles[16,17,18]. Traditional embroidery technique, where a single fiber is sewn into the textile, still lacks industrial production scalability.

The most critical aspect in the patterning of organic materials is deposition without disturbing the electrical properties. The patterning technique described in Figure 1 relies on the direct deposition of organics, with no need to meet specifications of the deposition technique or the tool. The organic materials are formulated to the best of their performances and can then be directly deposited on the chosen fabric structure. The utility of PDMS is key to pattern materials from solution onto textiles. The application of conducting materials from the low-viscosity solution, rather than using paste-like inks, enables a conformal and deep coating in textile structure. However, it limits the selective deposition and leads to the loss of the patterning resolution. We have overcome this limitation by creating a negative pattern from PDMS to restrain the non-controlled conducting solution penetration into the textile. The strategic choice of the PDMS is based on its visco-elastic properties, which maintain the textile stretchability and flexibility. The PDMS is also hydrophobic and allows the control of the spreading of the PEDOT:PSS water-based solution during the patterning. We observed that the conductive patterns fabricated using this protocol demonstrated good electrical conductivity and stability during mechanical deformations. This method allows the future customization of existing garments with smart components that have electronic capabilities. However, one of the critical and, in some cases, limiting points of the proposed approach is still organic material durability in wearable conditions. Some aspects, such as mechanical stress resistance and behavior after the washing and drying of organic conducting textiles, are still unknown.

The large majority of wearable electronics rely on stretchable devices, where spring-like structures are created to maintain the electrical connection during device deformation. Depending on the textile type, the fibers in knit fabrics are assembled in a horseshoe design, providing mechanical stretchability of the structure. Coating these textiles with conducting materials permits individual fibers to act as strain and motion sensors in smart clothing, as shown in Figure 3a. Moreover, more complex device geometries can easily be patterned, not only on knit, but also on woven fabrics. In Figure 3b, we present an array of OECTs with variable geometries. In conventional photolithography, the simultaneous fabrication of big and small features is almost impossible to achieve without requiring multiple steps. We demonstrate that our patterning technique is able to produce patterns with a resolution that varies from 0.5 mm to about a hundred times greater. Such transistors can be directly used in wearable sweat sensing with an adjustable time response and detection resolution[19].

We have demonstrated that PDMS also enables the consecutive deposition of additional functional layers in a selective manner, as shown in Figure 2. Devices can then be integrated in textiles and become fully integrated on wearable systems. The process in Figure 2a shows the fabrication of a cutaneous textile electrode, where the contact between the electrode and the skin is enhanced with an ionic liquid gel. Wearable electrodes in cutaneous electrophysiology suffer from motion artifacts caused by the electrical contact degradation between the wearer and electrodes during recordings. The possibility to integrate ionic gels on textile electrodes opens an efficient communication channel with the human body, which is desired in wearable healthcare devices. An example of such a device can be seen in Figure 3c.

The consecutive deposition of other active materials can result in devices using a stack geometry, such as organic batteries, capacitors, solar cells, transistors, or sensors. Figure 2b shows the deposition route of insulating or dielectric materials. A wearable organic keyboard (Figure 3d) can be fabricated using this process, where the PDMS is used to create a dielectric layer on top of the electrode. Such a device is capable of capacitive variation sensing between the electrode and a finger, which can have potentially interesting applications in wearable computing and human-machine interfacing.

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DISCLOSURES:
The authors have nothing to disclose.

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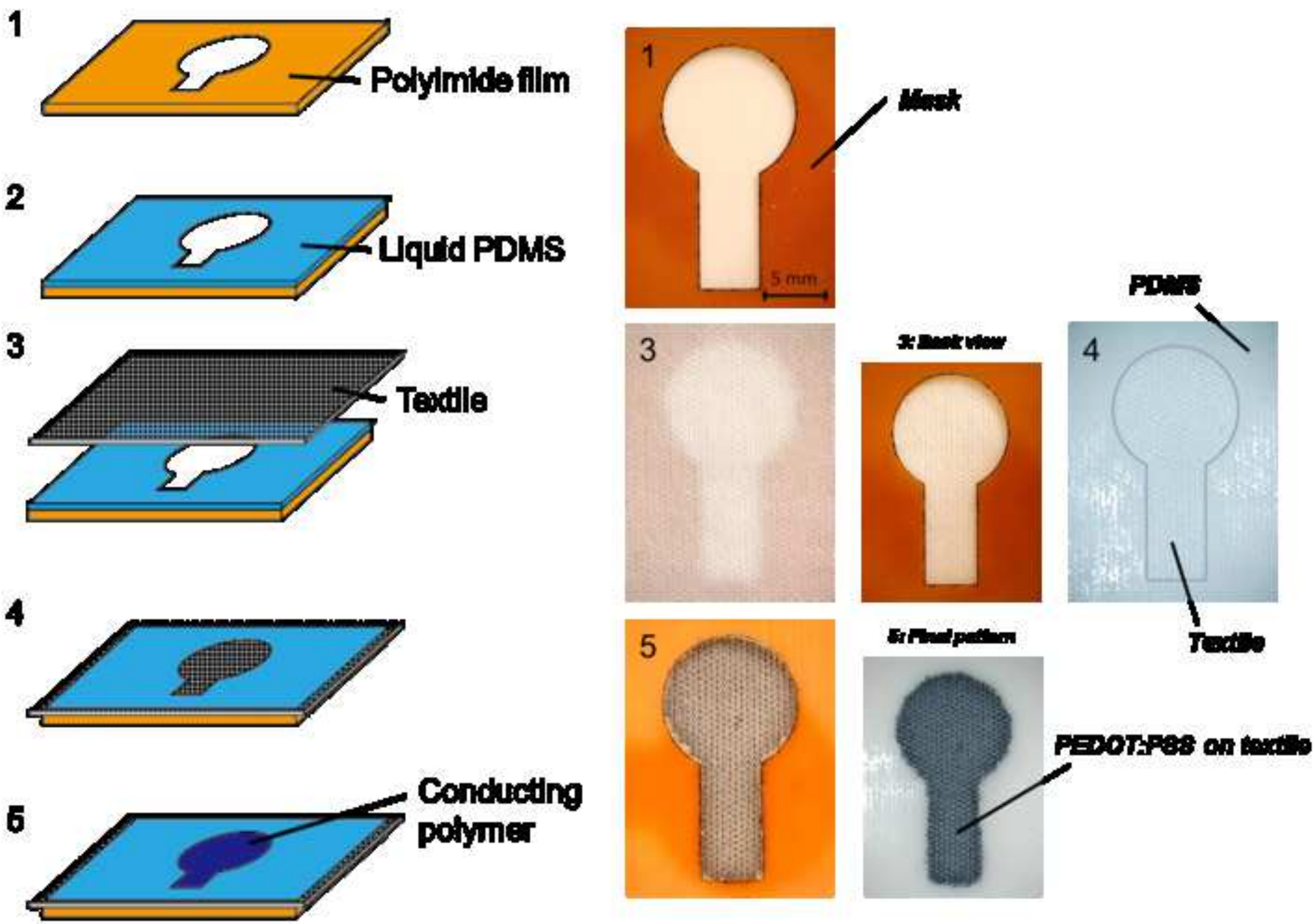
Figure 1



1
Polyimide film
2
Liquid PDMS
3
Textile
4
5
Conducting polymer
Mask
5 mm
PDMS
3: Back view
Textile
5: Final pattern
PEDOT:PSS on textile

Figure 2

Click here to download Figure image 2.png

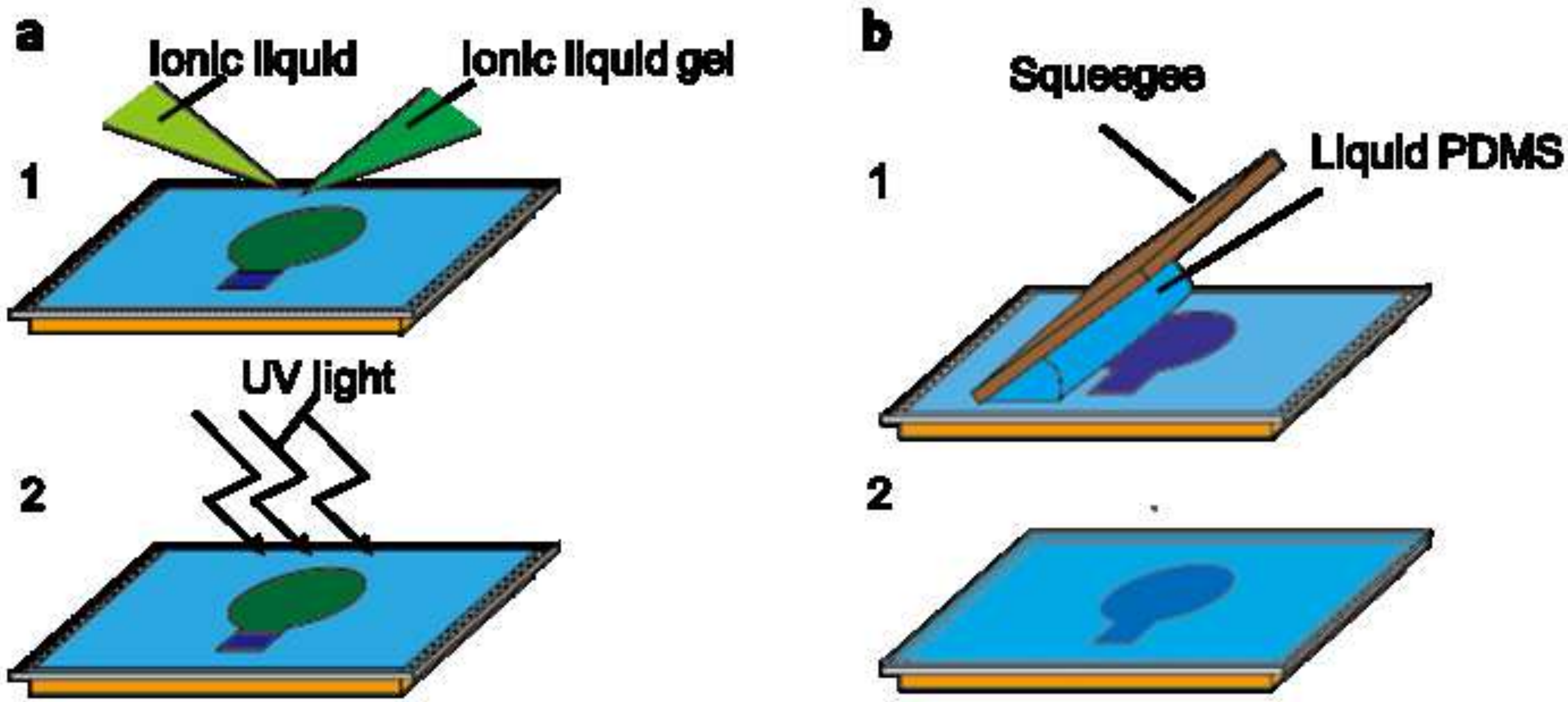
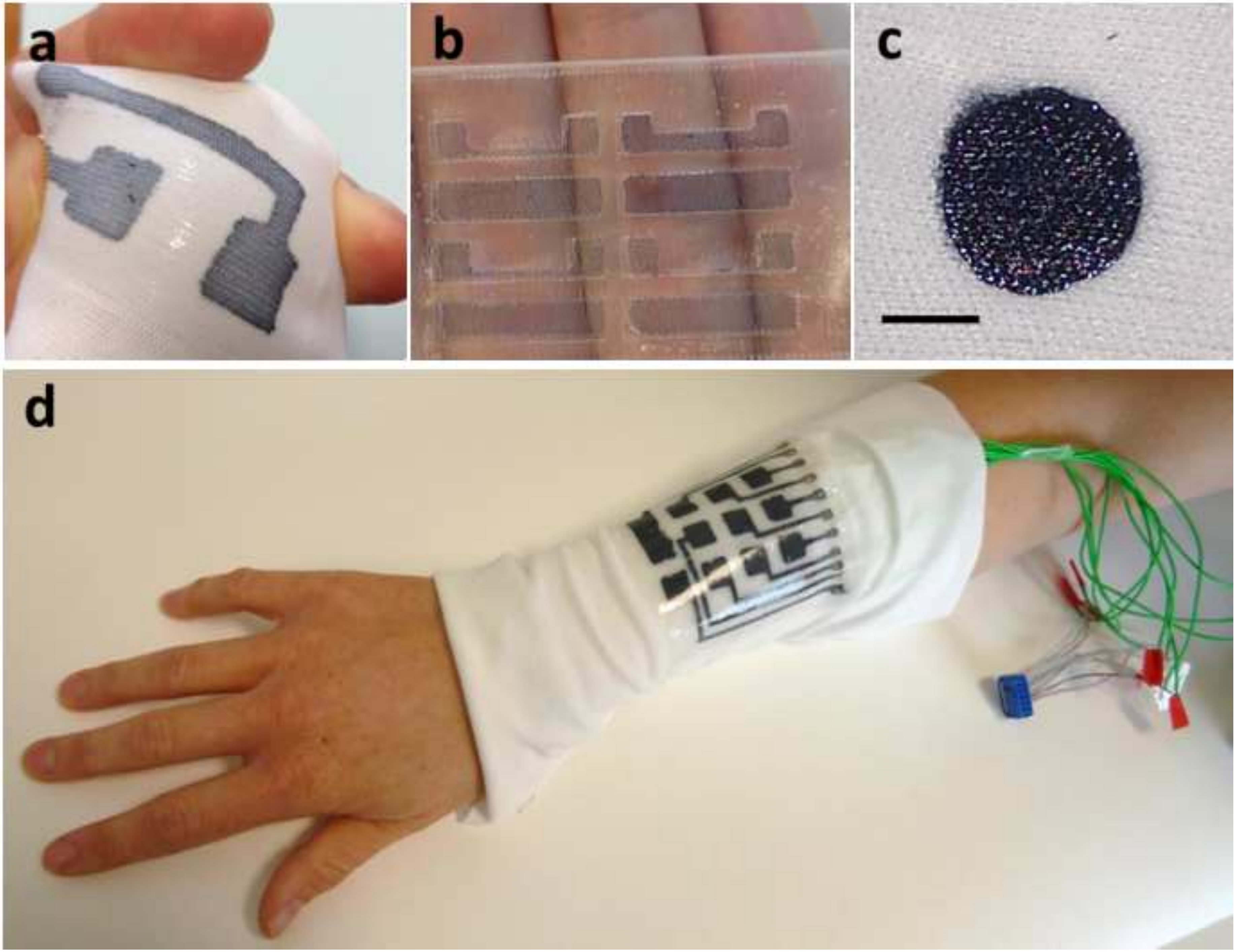


Figure 3 Click here to download Figure Image3.png



Name of Reagent/ Equipment	Company	Catalog Number
SYLGARD 184, Silicone elastomer kit (Base and Curing agent)	Dow Corning	
The conducting polymer formulation		
CleviosTM PH 1000 PEDOT:PSS	Heraeus	
Ethylene glycol	Sigma-Aldrich	03750-250ML
3-methacryloxypropyltrimethoxysilane	Sigma-Aldrich	M6514
4-dodecylbenzenesulfonic acid	Sigma-Aldrich	44198
The ionic liquid gel		
UV lamp DFE 2340	C.I.F/ ATHELEC	DP134
1-Ethyl-3-methylimidazolium ethyl sulfate	Sigma-Aldrich	51682-100G-F
Poly(ethylene glycol) diacrylate	Sigma-Aldrich	455008-100ML
2-Hydroxy-2-methylpropiophenon	Sigma-Aldrich	405655-50ML
The textile fabric	VWR	Spec-Wipe 7 Wipers
The polyimide film	DuPont	HN100

Comments/Description

PDMS elastomer

Conductive polymer
Solvent (EG), CAS: 107-21-1

Cros linker (GOPs), CAS: 2530-85-0

(DBSA), CAS: 121-65-3

UV-365nm

Ionic Liquid (IL), CAS: 342573-75-5

Mn 700, CAS: 26570-48-9

Phot Initiator (PI), CAS: 7473-98-5

100% interlock knit polyester fabric

Polyimide film with 125 µm thickness

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Response to Editorial comments

Editorial comments:
The manuscript has been modified by the Science Editor to comply with the JoVE formatting standard. Please maintain the current formatting throughout the manuscript. The updated manuscript (55439_R1_091416.docx) is located in your Editorial Manager account. In the revised PDF submission, there is a hyperlink for downloading the .docx file. Please download the .docx file and use this updated version for any future revisions.

1. Please copyedit the manuscript for grammatical errors and awkward phrasing. This editing should be performed by a native English speaker and is required prior to acceptance.
-Line 38 – "extreme daily conditions" – this is not clear.
-1.1 – "an easy handling"
-1.2 – "the Figure 1" – delete "the"
-1.6 – "Reduce this temperature to 60 °C by using a vacuum oven for other type of textiles," – please clarify. It sounds like a reduction of temperature in the air oven is achieved by a vacuum oven.
-2.1 – Please clarify "consecutively deposit an ionic gel". Only one layer of gel appears to be deposited in the subsequent steps.
-2.2 – "respect the temperature" – awkward phrasing
-Line 163 – "using described method"
-Line 165 – "horseshow arrangement" – do you mean "horseshoe"?
-Line 168 – "percolation" is not the right word here.
-Line 213 – "fiber's three-dimensional arrangement in their structures"
-Line 224 – "processable"
-Line 249 – "in such way"
-Line 253 – "subject for the degradation of the electrical contact during recordings" – please clarify

Response: We have addressed all the coments and introduced corrections in the revised version of the manuscript. The revisions can be seen using the Word tracking tool.

2. Visualization: It is unclear how to visualize 2.2 – 2.4.1. The purpose of these steps is also not clear.

Response: In these steps we describe the fabrication method of the different devices. We have modified the text to underline the purpose of each step.

3. Additional detail is required:
-1.2 – How is the mask made? steps 1.2 and 1.2.1
-1.2.1 – How is laser cutting used to pattern the mask? Please provide a citation.
-1.5 – What is meant by the unprotected area? What part is the protected area?

-1.6 – What is the ink solution? Is this the conducting polymer? Please be consistent with names of materials.
-2.1.2 – How is coating performed? What is “ionic liquid”? Is this the same solution as in 2.1.1? How long is the UV exposure?
-Steps 2.2-2.4.1 are unclear. What is the purpose of these steps? How do they differ from the steps in section 1? It seems like a repeat with no clarity of purpose. Please rewrite this section to clearly state what is being done, as it is not clear what we can film. What actions are the experimenters performing in each step?

Response: We have modified the text adding more details as requested. The comment on steps 2.2-2.4.1 have been addressed in the previous comment (2).

4. Discussion: Please discuss the limitations and future applications of the protocol.

Response: We discuss the limitations and applications now in the manuscript (line 244). This method allows in future customizing existing garments with smart components having electronic capabilities. However one of the critical and in some cases limiting point of the proposed approach is still organic materials durability in wearable conditions. The aspects such as the mechanical stress resistance, behavior after washing and drying steps of organic conducting textiles are still unknown.

Reviewers' comments:
Reviewer #1:
Manuscript Summary:
The authors present a simple and scalable fabrication protocol that allows for the patterning of conducting polymers on textiles. The presented process enables the fabrication of wearable and conformal electronic devices. An elastomeric stencil material polydimethylsiloxane (PDMS) is used to protect textile areas where no impregnation with conductors should occur. This combination allows the precise deposition of conductive structures on rough textile surfaces as well as the retainment of the soft and stretchable properties of textiles.

Major Concerns:
no major concerns.

Minor Concerns:
Line 95: Deposition of precise volumetric quantities of viscous liquids such as PDMS uncured is difficult. It would be useful if the authors provide more information here.
Response: We have clarified this in the manuscript. 0,5mL was used for a mask of 3x5cm^2

Line 96: The authors should provide details about "an automatic tape casting tool"
Response: This is now clarified this in the manuscript

Line 142: Traditional methods for applying colors or patterns to textiles rely on removable masking layers to allow the selective deposition of dyes.
Is it possible to remove PDMS afterwards?
Response: in our case the PDMS material cannot be removed at the end of the process. While some strong solvents can be used to dissolve PDMS, this can harm the PEDOT:PSS and destroy its conductive properties.

General: How does the process compare to traditional methods such as screen printing? Is there an advantage that pays of the necessity to impregnate with PDMS?

Response: The method described here was developed to pattern low viscosity solutions on thick and knitted textiles, and is able to consistently penetrate the textiles structure and to control their diffusion thanks to the PDMS. Traditional techniques are mostly used for thin and woven textiles. Techniques like ink jet printing or screen printing are limited because of the amount of the ink deposited at the time, or because the paste-like nature of the ink can only be deposited on the top surface of thick textiles.

Additional Comments to Authors:
N/A

Reviewer #2:
Manuscript Summary:
The manuscript deal with the fabrication of conducting polymer devices on textiles. It is well organized and well-written and gives a substantial contribution to the progress of wearable electronics.

Major Concerns:
N/A

Minor Concerns:
What is the role of the silane in the processing of PEDOT:PSS?
Besides the fabrication, why not showing an example of electrical performance?

Response: It is known that a variety of Silane molecules are largely used as surface adhesion promoters. In our case it helps PEDOT/PSS to better adhere to the textile and prevent PEDOT's dissolution in contact with water. This method follows our previous work cited as reference 10. The electrical properties are described in detail in that paper.

Additional Comments to Authors:
Overall, I suggest publication after minor revisions

Response to Editorial comments

The manuscript has been modified by the Science Editor to comply with the JoVE formatting standard. Please maintain the current formatting throughout the manuscript. The updated manuscript (55439_R0_090816.docx) is located in your Editorial Manager account. In the revised PDF submission, there is a hyperlink for downloading the .docx file. Please download the .docx file and use this updated version for any future revisions.

Changes made by the Science Editor:
1. There have been edits made to the manuscript. Please accept (or address) all tracked changes.

Response: We have accepted all changes and addresses the correction concerning the modification of *is* to *are* "We are talking about the field of wearable electronics" so, we changed the phrase in line 46.

Changes to be made by the Author(s):
1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues. The JoVE editor will not copy-edit your manuscript and any errors in the submitted revision may be present in the published version.
Response: We have proofread the manuscript
2. Unfortunately, there are a few sections of the manuscript that show significant overlap with previously published work. Please rewrite the text to avoid plagiarism (including self-plagiarism). Though there may be a limited number of ways to describe a technique, please use original language throughout the manuscript. These sections are highlighted with red text in the revised manuscript.
Response: We have modified the text of these sections. These modifications can be seen in red
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Response: We have removed all commercial symbols from the text and illustrations.
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Response: We have removed them.
5. Please provide an email address for each author.
Response: We now include all emails in the manuscript
6. Please rephrase the Short Abstract to clearly describe the protocol and its applications in complete sentences between 10-50 words: "Here, we present a protocol to …"
Response: We have rephrase the abstract.
7. Please ensure that all text in the protocol section is written in the imperative tense as if telling someone how to do the technique (e.g., "Do this," "Ensure that," etc.). The actions should be described in the imperative tense in complete sentences wherever possible. Avoid usage of phrases such as "could be," "should be," and "would be" throughout the Protocol. Any text that cannot be written in the imperative tense may be added as a "Note." However, notes should be concise and used sparingly. Please include all safety procedures and use of hoods, etc.
Response: We have rephrased the protocol steps that now includ safety specifications when required.
8. The Protocol should contain only action items that direct the reader to do something. Please move the discussion about the protocol to the Discussion.
Response: The protocol is now containing only actions.
9. Please add more details to your protocol steps. Please ensure you answer the "how" question, i.e., how is the step performed? Alternatively, add references to published material specifying how to perform the protocol action.
Response: We now clarify this in the manuscript as much as we could.
10. 1.1: How much is used?
Response: We have clarified this in the manuscript
11. 1.2.1: What pattern is used?
Response: We have clarified this in the manuscript
12. 1.5: How much is brush coated?
Response: We have clarified this in the manuscript
13. Please upload each Figure individually to your Editorial Manager account as a .png or a .tiff file. Please combine

all panels of one figure into a single image file.
Response: The figures are now uploaded separately.
14. Please remove the titles and Figure Legends from the uploaded figures. The information provided in the Figure Legends after the Representative Results is sufficient.
Response: We have uploaded the figures separately. Figure legends are presented after the Representative Results. Additionally, combined figures and legends doc. file is uploaded for demonstration.