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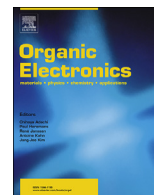

A facile method for fabrication of highly integrated organic field-effect transistors on photoresist-unwettable insulators with remarkable stability

Jincheng Wang, Xiujuan Zhang**, Wei Deng, Qixun Shang, Liang Wang, Liubiao Zhong, Lin Jiang, Jiansheng Jie*

Institute of Functional Nano & Soft Materials (FUNSOM), Jiangsu Key Laboratory for Carbon-Based Functional Materials & Devices, Soochow University, Suzhou, Jiangsu 215123, PR China

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A B S T R A C T

Environmental stability is one of the most important parameters for high-performance organic field-effect transistors (OFETs). Hydrophobic insulators usually possess much better air stability than some conventional inorganic and hydrophilic organic insulators. However, fabrication of devices with high integration by photolithography method cannot be directly performed on hydrophobic insulators due to their photoresist-unwettable properties. In this work, a simple yet efficient metal-assisted photolithography method is developed to achieve large-scale fabrication of highly integrated organic electronic devices on photoresist-unwettable insulators. By using copper (Cu) as sacrificial layer, photolithography can be performed on these insulators with nearly the same resolution, uniformity, and reproducibility as the conventional photolithography. This method shows excellent flexibility and is capable of fabricating high-integrated devices on a variety of hydrophobic insulators including hydrophobic amorphous fluoropolymer (CYTOP), poly(dimethylsiloxane) (PDMS), and octadecyltrichlorosilane (OTS)-modified SiO_2. OFETs based on 6,13-dichloropentacene (DCP) microwires (MWs) with CYTOP as the insulator layer were fabricated, which exhibited excellent device performance. Much improved device stability with very low mobility degradation (less than 9%) was observed after 24 days. While devices fabricated on hydrophilic insulators of poly-(vinyl phenol) (PVP) and bare SiO_2 experienced dramatic performances decay within 24 days. Furthermore, by using metal-assisted photolithography method, flexible OFETs arrays could be further fabricated on polyethylene naphthalate (PEN) substrate, which showed excellent mechanical bending flexibility and stability. This work unveils the great potential of metal-assisted photolithography method for long-term stable high-integration organic electronic devices.

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1. Introduction

To be competitive with their inorganic counterparts, organic field-effect transistors (OFETs) have drawn much attention over the past decades due to their unique advantages of excellent processability, lightweight, low fabrication cost, and mechanical flexibility [1–5]. Highly integrated OFETs are much desirable for practical applications, such as active-matrix organic light emitting display and logic circuits. Particularly, flexible OFETs are of great importance for their wide applications in a variety of fields such as flexible displays, electronic paper, and radio-frequency identification cards [6–9]. One of the most important parameters for high-performance OFETs is their environmental stability. Besides air-stable organic semiconductors [10,11], stability of gate insulators is also vital to achieve environmentally stable OFETs. Thus far, various insulators including inorganic and organic materials are employed in OFETs. Inorganic insulators, such as SiO_2, Al_2O_3, and HfO_2, have been widely utilized owing to their high dielectric constant and compatibility with traditional microfabrication techniques [12–15]. However, a large density of hydroxyl groups exists on the surface of these inorganic insulators, which can absorb water molecules and make semiconductor/insulator interface electrically unstable. Thus OFETs based on inorganic insulators

* Corresponding author.
** Corresponding author.
E-mail addresses: xjzhang@suda.edu.cn (X. Zhang), jsjie@suda.edu.cn (J. Jie).

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usually suffer from performance degradation with time [16–19]. Some polymer insulators such as poly-(vinyl phenol) (PVP) and poly-(vinyl alcohol) (PVA) [20,21], which contain hydroxyl groups, suffer from the same problem and therefore are unsuitable for long-term stable devices. In light of this, hydrophobic insulators, such as hydrophobic self-assembled monolayer (SAM) modified inorganic insulators and hydrophobic polymer insulators, are considered as promising candidates for the fabrication of long-term stable OFETs. Hydrophobic SAMs such as octadecyltrichlorosilane (OTS) can react with hydroxyl groups on the surface of inorganic insulators [22,23], impeding the absorption of water molecules in air. Hydrophobic polymer insulators without hydroxyl groups, such as hydrophobic amorphous fluoropolymer (CYTOP) and poly(-dimethylsiloxane) (PDMS), can also be employed to fabricate stable OFETs [24–26], as they can prevent the devices from invasion of water.

Although use of hydrophobic insulators is favorable for device stability, hydrophobic properties makes it difficult for them to be directly used in photolithography process, since photoresist is nonwettable on the surface of these hydrophobic insulators. Poor film forming ability of photoresist on insulators makes micro-patterning process cannot be proceed. As such, traditional photolithography technique, which is an important microfabrication technology for highly integrated devices [27,28], cannot be applied on hydrophobic insulators. Sakanoue and co-workers recently reported fluorosurfactant-assisted photolithography that enabled photoresist to be spin coated on the surface of CYTOP and Teflon AF [29], but this method was mainly restricted to perfluoropolymers. In our previous work, we developed a two-step transfer printing method to fabricate OFETs on arbitrary substrates with the aid of a sacrificial layer [30,31]. While this method is highly efficient to fabricate the OFETs on flexible substrates, the solution transfer process will influence the device performance and yield by degrading the crystal quality of the organic microcrystals. Metallic shadow mask technology is an alternative micropatterning technique for the fabrication of devices on hydrophobic insulators [32,33]. However, low resolution of shadow mask technology impedes its applications in high-integration devices. Recently, several high-resolution masks, including full-wafer stencil [34–36] and polymer mask [37–39], have been developed to achieve highly integrated OFETs. Nevertheless, the fabrication process is complicated and difficult to control. These masks are also easily damaged and their performance often declines with the use frequency. Considering the overwhelming advantages of photolithography, such as high resolution, wafer-scale capability, and high reproducibility, it is much desired to develop a general technique to enable photolithographic micropatterning process applicable on photoresist-unwettable insulators.

Herein, in this work, we demonstrated a facile and reliable metal-assisted photolithography method to achieve large-scale fabrication of highly integrated OFETs on photoresist-unwettable insulators. A layer of copper (Cu) film was used as a sacrificial layer on the top of photoresist-unwettable insulator, on which photolithographic micropatterning process could be employed. Patterned Cu layer then served as high-resolution metal mask for the subsequent electrode deposition. This method shows excellent flexibility and is capable of fabricating devices on a variety of hydrophobic insulators, as well as flexible substrates. OFETs based on organic 6,13-dichloropentacene (DCP) microwires (MWs) with CYTOP as the insulator layer exhibited comparable device performance as these fabricated on conventional hydrophilic insulators, but remarkably enhanced device stability was achieved. Our work paves the way toward highly integrated organic electronic devices on photoresist-unwettable insulators.

2. Results and discussion

Fig. 1 schematically illustrates conventional photolithography process and metal-assisted photolithography process on photoresist-unwettable insulator. In conventional photolithography process (Fig. 1(a)), photoresist dropped on the insulator has a high contact angle with the hydrophobic surface of the insulator (Fig. 1(a)-I). During spin-coating process, photoresist will slide down from the substrate (Fig. 1(a)-II), and no film could be formed on the insulator surface (Fig. 1(a)-III). This makes photolithography on the hydrophobic insulator infeasible. In metal-assisted photolithography process, 50 nm Cu sacrificial layer is deposited on the hydrophobic insulator (Fig. 1(b)-I and II). Photoresist windows (electrode patterns) then can be defined on the wettable Cu layer by photolithography process (Fig. 1(b)-III). After that, the substrate is immersed into a saturated ammonium persulfate ($(NH_4)_2S_2O_8$) solution at room temperature for a short time (~10 s) to selectively etch the exposed Cu film away from the photoresist windows, as shown in Fig. 1(b)-IV. Deionized water is used to remove the residual etchant. Deposition of metallic electrodes like gold (Au) by thermal evaporation method is followed (Fig. 1(b)-V). After removing photoresist in acetone, the substrate is immersed into saturated $(NH_4)_2S_2O_8$ solution for additional 10 s to etch residual Cu film. Since Au film is not etched by $(NH_4)_2S_2O_8$ solution (Fig. S1), Au electrode patterns are left behind on the hydrophobic insulators (Fig. 1(b)-IV). It is noteworthy that, by using Cu as a sacrificial layer, direct photolithography on the hydrophobic insulator is avoided. Cu layer with electrode patterns are firstly made, which then serve as a high-resolution metal mask for the subsequent fabrication of metallic electrode arrays on the hydrophobic insulator.

Metal-assisted photolithography method is capable of fabricating device on a variety of photoresist-unwettable insulators. In this study, CYTOP, PDMS, and OTS modified SiO_2 insulators were taken as examples to demonstrate the effectiveness of this technique. Contact angles (θ) of these insulators are measured to be 110.2°, 114.8°, and 106.6° respectively for CYTOP and PDMS coated Si substrate as well as OTS modified SiO_2/Si substrate (Fig. 2(a)-(c)), verifying the hydrophobic nature of the surface. It's noted that hydrophobic properties of the insulators are not affected by the evaporation and etching processes of Cu sacrificial layer; their contact angles are nearly unchanged with the values of 109.8°, 112.6° and 107.4°, respectively, after removing the Cu layer (Fig. S2). Large-scale Au electrode arrays with channel gap of 10 μm are successfully achieved on them by metal-assisted photolithography method, as shown in Fig. 2(d)–(f). The Au electrodes possess smooth and sharp edges, as confirmed by the AFM investigation (Fig. S3). Moreover, channel gaps of the electrode pairs could also be readily controlled. Fig. 2(g)–(i) show electrode arrays with channel gaps of 2, 4, and 10 μm respectively on CYTOP coated Si substrates. This method is capable of fabricating high-resolution patterns. To exploit the potential of this method, electron-beam lithography (EBL) instead of UV photolithography was utilized to fabricate the electrode patterns on CYTOP coated Si substrates. Electrode pairs with channel gaps of 200 and 500 nm are successfully achieved (Fig. S4). Beside Au electrode array, Ag and Al electrode arrays could also be obtained with the same method (Fig. S5). Moreover, the used of nickel (Ni) as sacrificial layer and ferric chloride ($FeCl_3$) as etchant also led to the fabrication of Au electrode array on CYTOP coated Si substrates (Fig. S6). All above results clearly show that metal-assisted photolithography method is robust and has nearly the same accuracy, uniformity, and reproducibility as conventional photolithography.

To assess properties of the insulators after photolithography, leakage current density are measured. Fig. 3(a)–(c) depict typical leakage current density-voltage (J–V) plots of CYTOP, PDMS, and

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