Self-assembly of graphene-based planar micro-supercapacitor with selective laser etching-induced superhydrophobic/superhydrophilic pattern

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Abstract

Facile production of graphene-based in-plane interdigital electrode is critical for microscale supercapacitors but remains a great challenge (time consuming, expensive equipment needed, etc). The preparation of fine interdigital electrode planar micro-supercapacitors (MSCs) by selective laser etching of ceramic substrate is proposed, which provides an alternative method for rapid fabrication of planar MSCs. The formation of self-assembled electrode of graphene ink via the laser etching-induced heterogeneous surface wettability was firstly investigated, and then an all-solid-state MSCs microdevice was obtained by adding a polyvinyl alcohol-sulfuric acid gel electrolyte to the interdigital electrode region. The results of cyclic voltammetry test showed that the micro-supercapacitor device could offer an area capacitance of 5.5 mF cm^{-2} at a potential scan rate of 5 mV s^{-1} and the capacitance retention of 88.5% after 4500 cycles.

Keywords Supercapacitors · Interdigital electrode · Self-assembly · Surface wettability

1 Introduction

With the widespread use of portable electronic components, demands for multifunctional, small-scale electrochemical energy storage devices are growing in modern society [1]. In recent years, micro-supercapacitors (MSCs) are gradually becoming one of the emerging and cutting-edge research directions in the study of on-chip energy storage devices [2]. MSCs, as a micropower source, is compatible with various microelectronic components, which has broad application prospects, such as powering for microsensor [3], biomedical implants [4], and micro-robots [5]. The graphene-based two-dimensional materials can be used as electrode materials for MSCs due to

its unique structure and physical behavior, such as excellent surface area and high in-plane conductivity [6]. Liu et al. [7] adopted inkjet printing technology to prepare planar MSCs on different substrates using electrochemically stripped graphene as electrode material. The area capacitance of paper-based MSCs reached 5.4 mF cm^{-2} , which provides a new idea for the development of new flexible MSCs.

Compared with traditional sandwich supercapacitors, graphene-based planar interdigital MSCs have a greater advantage in that they own features of both graphene and planar device geometry. Yoo et al. reported two kinds of graphene-based planar MSCs, i.e., MSCs based either on the single-layer graphene prepared by chemical vapor

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deposition or on the multilayer graphene films prepared by reduced graphene oxide. It was found that the area capacitance of planar MSCs were 80 and 394 $\mu\text{F cm}^{-2}$, respectively, which were much higher than that of traditional sandwich supercapacitors [8]. Combining the photolithography and electrophoretic deposition method, Niu et al. [9] used PVA/ H_3PO_4 gel as electrolyte to produce ultrathin graphene interdigitated patterned electrode on PET substrate, thus flexible and ultrathin solid-state graphene-based MSCs were obtained. Because the electrolyte ions are limited in the narrow gap between the planar interdigital electrodes, the ion diffusion distance is very short and they can be easily transmitted to provide better area capacitance.

However, there are still some problems in the design and production of planar MSCs. For instance, the large distance between the interdigital electrodes will result in poor specific energy and specific power [5, 10]. So, it is still a great challenge to further enhance the electrochemical performance of MSCs by accurately controlling the number and width of interdigital electrodes. Moreover, traditional micro–nanofabrication technologies are usually costly and complex. Wu et al. [11] manufactured a planar interdigitated graphene-based MSCs on silicon wafer by combining methane-plasma-assisted reduction with photolithography microfabrication. These methods require complex equipment and multiple steps, and the treatment process of the generated waste is expensive and time consuming. Therefore, we need to exploit an advanced fine pattern-processing technology during the preparation process of patterned electrode.

Laser etching has the advantage of facile fabrication of micropatterned electrodes, which allows the gaps and electrode fingers formed in the micropattern and can be easily adjusted and finely integrated on the same substrate

[12]. Moreover, laser etching can also change the substrate surface property, which will affect the wetting properties. For example, the patterned superhydrophobic (SH) and superhydrophilic (SHL) structure can be prepared on the surface of various materials by laser selective etching. Sun et al. [13] provided a facile method for preparing SH/SHL hybrid patterns on aluminum alloy substrate by using a selective picosecond laser etching. He et al. [14] realized patterned deposition of nanoparticles on SH and SHL composite surfaces fabricated by laser etching on titanium substrates. According to the surface wettability, it is feasible to let the graphene-based aqueous self-assemble into the micro-SHL channel form the hybrid SH/SHL pattern.

In this paper, we employed laser micro–nanomanufacturing technology to fabricate an all-solid graphene-based planar interdigital MSCs. During the fabrication, the graphene-based aqueous can self-assemble along the patterned channels to form ultra-fine conductive interdigital electrodes. The electrochemical behavior of MSCs demonstrates the attractive application prospects of these self-assembled MSCs.

2 Materials and methods

Alumina ceramic plates (96 wt% of Al_2O_3 , Sigma-Aldrich) with dimensions of 40 mm $\times$ 40 mm $\times$ 2 mm were used as the rigid substrates. Aqueous graphene ink (8.9 wt% graphene nanopowder, Sixth Element Material Co., Ltd., China) performed as the conductive ink materials for preparing electrodes. Concentrated sulfuric acid (H_2SO_4) and polyvinyl alcohol (PVA) were purchased from Sigma-Aldrich Company to be used as gel electrolyte.

The preparation processes of all-solid-state graphene-based MSCs on ceramic substrates are illustrated in Fig. 1.

Fig. 1 The schematic of preparation process of graphene-based planar MSCs on an alumina ceramic plate by a laser direct-writing technique

First of all, the periodic surface microstructure was fabricated on a ceramic substrate by laser etching and then treated with a modifier (solution of stearic acid) to form SH surface (Steps 1). Secondly, the surface modifier layer was selectively removed by laser etching again to form the SHL channel on the prepared SH surface, and the different SHL channels will form an ultra-fine interdigitated pattern (Steps 2). After that, the aqueous graphene ink was carefully dropped onto the hydrophilic interdigital pattern area which serves as the electrodes (Steps 3). Finally, 5 μL polymer gel electrolyte of sulfuric acid–polyvinyl (H_2SO_4 –PVA) was carefully dripped into the interdigital electrode gaps and then solidified for 12 h (Step 4). Thereby, the graphene-based MSCs with planar interdigital configuration is completed.

Figure 2 shows the detailed fabrication process of SH and SHL surface by infrared nanosecond fiber laser (YLP, JTL-YLP20W). The substrate was ultrasonically cleaned in ethanol for 10 min before processing. The wavelength, pulse duration, repeat frequency, and focal spot diameter of the used laser were 1064 nm, 25 ns, 20–80 kHz, and 50 μm , respectively. The repetition frequency, scan interval, scan speed, and scan numbers used in the experiments were 40 kHz, 0.1 mm, 200 mm/s, and 10 times, respectively. Then the laser-induced periodic surface structures will be formed on the substrate. To achieve the SH surface, substrate with microstructure was immersed in a 0.02 mol/L stearic acid solution for 1 hour at room temperature and then dried under atmospheric conditions. Stearic acid solution was used to achieve low-surface-energy state for

the laser-ablated area on substrate surface. The contact angle (CA) of the surface was measured with a contact angle meter to evaluate the obtained SH surface. The inset in Fig. 2 presented the microstructure of the SH surface.

The laser scanning process was carried out once again on the fabricated SH surface to fabricate the SHL interdigital pattern. In the laser scanning process, the grid interval of laser scanning was 20 μm , and the scanning speed, repeat frequency, and scanning numbers were 1000 mm/s, 20 kHz and 10 times, respectively. Then, a micropipette was used to drop the graphene ink material into the interdigital pattern to form the electrodes, and the self-assembly behavior according to the different surface wettability is shown in Fig. 8.

The surface morphologies of the SH and SHL were observed by a 3D measuring laser microscope (Olympus, OSL4100) and a scanning electron microscope (SEM, Zeiss, SUPRA55) before and after the laser treatment. Electrochemical characterization of the prepared MSCs was done by means of cyclic voltammetry (CV) test and galvanostatic charge and discharge (GCD) test with an electrochemical workstation.

3 Results and discussion

Figure 3 presents the characterization results of the surface on ceramic substrates after the former and latter laser treatment. Figure 3a, b shows the 3D profile images of the SH and SHL surfaces. The SH surface which has a

Fig. 2 The schematic of laser direct-writing system and fabrication process of ceramic SH/SHL surface

Fig. 3 Images of laser-ablated ceramic surface. **a, b** The ceramic surface 3D profile. The insets present CA images of SH/SHL surface. **c, d** Cross-sectional profiles of the surface. **e, f** SEM images of the laser etching ceramic surfaces with micro-nanostructures

hierarchical micro-nanostructures is shown in Fig. 3e, and the size of the microscale patterns depends on the laser machining parameters (power density, scanning interval, and scanning speed). The tops of the micropillars are covered with abundant laser-induced nanoparticles as shown in Fig. 3f, which are formed by rapid cooling of the sprayed melt liquid in the nearby melting zone caused by high-temperature laser ablation [15–17]. Generally, the adhesion of SH surfaces mainly depends on two elements: surface of chemical composition and rough structure [18]. The insets in Fig. 3a, b shows the average contact angles of the water droplets measured on the surface of SH and SHL samples, which are 159° and 5°, respectively. The mechanism for the formation of SH surfaces is that laser etching increases surface roughness and stearic acid solution immersion reduces surface energy. After rough

treatment and low surface energy modification, the contact angle of the ceramic substrate surface has changed greatly. The wettability of the ceramic substrate surface could be explained by Cassie–Baxter model [19].

Surface of the prepared ordered micropillars sample exhibited a “petal effect,” and the water droplets presented a mixed wetting state of Wenzel and Cassie–Baxter on this surface [20]. The surface of this wetting state provided a large solid–liquid contact region to get high adhesion, so excess aqueous droplets will adhere to the SH surface and cannot easily roll to SHL area to contaminate the electrodes. As the scanning pitch increased to 100 μm, the surface of the sample showed a protrusion similar to the “Lotus effect,” indicating that the surface was changed to the Cassie–Baxter wetting state. In this wetting state, it is assumed that the rough plate cannot

Fig. 4 Optical images of graphene-based MSCs with an interdigital configuration with **a** 12 (MSCs(12)), and **b** 18 (MSCs(18)) fingers. **c, d** Optical images of the fingers in **a** MSCs(12) and **b** MSCs(18)

Table 1 The specific parameters of the MSCs designed with 12 and 18 interdigital fingers

Number of the fingers in MSCs	12-finger MSCs	18-finger MSCs
Length, l (mm)	5	5
Width, w (μm)	500	250
Interspace, i (μm)	400	400

be completely wetted by the liquid. The average length, height, and width of the microcolumn are 87.94 μm , 63.88 μm , 75.67 μm , respectively, and the average width of the microgroove is 13.75 μm . The microgrooves exhibited the intermittent melting and resolidification effect, which is characterized by a large number of blind holes (Fig. 3e).

In order to study the influence of finger number and width of the MSCs, two kinds of the MSCs devices with different numbers of fingers (12 and 18) were prepared by the identical procedure and the corresponding micro-devices were denoted as MSCs(12) and MSCs(18), as presented in Fig. 4a, b. The finger widths of MSCs(12) and MSCs(18) were 500 μm and 250 μm , respectively, and the finger gap widths and finger length were kept constant, as shown in Table 1. In Fig. 4, it was found that the formed patterned interdigital electrodes seemed relatively regular and uniform.

The cyclic voltammetry (CV) measurements of MSCs(18) were carried out at scan rates ranging from 1 to 200 mV s^{-1} (Fig. 5a–i). Obviously, MSCs(18) demonstrated a classical electrical double-layer capacitance feature with a symmetric quasi-rectangular shape at low scan rates of 1–60 mV s^{-1} , but displayed a nearly symmetric spindle shape at high rates of 80–200 mV s^{-1} . There is no apparent redox peak in the CV curve within the examined potential

window, which indicates that the double-layer capacitance completely provides the capacity of the electrode.

Figure 6a, b shows the area capacitance and volumetric capacitance of graphene-based MSCs (18) obtained from the CV measurement. At the scan rate of 1 mV s^{-1} , the area capacitance and volumetric capacitance of MSCs(18) were calculated to be $\sim 8.55 \text{ mF cm}^{-2}$ and $\sim 213 \text{ mF cm}^{-3}$, respectively, both of which were slightly better than some other reports. For example, the planar micro-supercapacitor made by Ding et al. exhibited the area capacitance about 0.114 F m^{-2} at 0.1 A m^{-2} [21]. However, at an higher scan rate of 100 mV s^{-1} , MSCs(18) can only deliver an area capacitance of $\sim 0.34 \text{ mF cm}^{-2}$ and a volumetric capacitance of $\sim 8.5 \text{ mF cm}^{-3}$. As shown in Fig. 6a, b, with the increase in voltage scanning rate, the area capacitance and volume capacitance of MSCs(18) device decrease obviously. We attribute the phenomenon to the larger equivalent series resistance (ESR), which is caused by transport limitations of ions at higher scan rates and the resistance of the graphene fingers [9].

GCD curves were tested at different current densities of 5–15 $\mu\text{A cm}^{-2}$, as shown in Fig. 6c, which reveals the pseudo-linear response feature of a typical MSCs. The presence of a small amount of nonlinear phenomena in the profile means the occurrence of charge transfer, which is due to the surface electrochemically active reaction formed during the laser etching process. At the beginning of each discharge cycle, a slight decrease in IR voltage drop is obvious. We can calculate the ESR of a microdevice by dividing the magnitude of the voltage reduction by twice the specific current [22]. For the decrease in capacitance in CV curve at high discharge rate, we can explicate it with high ESR. As the scanning speed increases, the resistance of the dozens or even hundreds of layers of graphene material restricts the full contact between the electrode and the electrolyte interface, thus restricting the increase in capacitance. In addition, different charging and discharging currents will lead to different charging and discharging time. The larger the current, the shorter the discharging time. Furthermore, the cycling stability of the MSCs(18) was tested up to 4,500 cycles at a scan rate of 80 mV s^{-1} , as shown in Fig. 6d. The CV curves shapes still retained basically unchanged after thousands of cycles and 88.5% capacitance was remained, demonstrating expected stable capacitive behavior.

In order to estimate the impact of the configuration on the performance of the device, we further studied two kinds of graphene-based MSCs(18) and MSCs(12). The area capacitance of MSCs(18) and MSCs(12) were shown in Fig. 7a. At the scan rate of 1 mV s^{-1} , the area capacitance of MSCs(18) was calculated to be $\sim 8.55 \text{ mF cm}^{-2}$, which was higher than that of MSCs(12) ($\sim 4.22 \text{ mF cm}^{-2}$). Moreover, as the scan rate increases, the capacitance of

Fig. 5 CV curves of graphene-based MSCs (18) tested at **a** 1, **b** 5, **c** 10, **d** 20, **e** 40, **f** 60, **g** 80, **h** 100, and **i** 200 mV/s, respectively

MSCs(18) decreases more slowly than MSCs(12). In this case, an area capacitance of $\sim 0.46 \text{ mF cm}^{-2}$ was maintained at 80 mV s^{-1} , while $\sim 0.15 \text{ mF cm}^{-2}$ was achieved for MSCs(12) at 80 mV s^{-1} . Figure 7b shows comparison of the GCD measurement curves of MSCs(18) and MSCs(12). It can be seen that the MSCs(18) has shorter charging and discharging time than MSCs(12), which means the performance improvement of MSCs(18).

In fact, we can effectively reduce the average ion diffusion path between the adjacent fingers by narrowing the finger width and increasing the number of interdigitated fingers of MSCs. Consequently, the electrolyte resistance is reduced under the low ion transport constraints [10]. The results highlight the significant part of the MSCs configuration in determining the electrochemical performance of a device.

Since the wettability characteristic will disappear after the aqueous graphene ink solidified in the SHL region, we did not cover the graphene interdigital pattern with current collector by SH/SHL property in this experiment,

which prevented our devices from operating at a higher scanning rate.

Usually, conventional photolithographic microfabrication technology is utilized to fabricate graphene-based planar MSCs. Compared with traditional manufacturing methods, the laser direct-writing technology mentioned in Fig. 1 is facile and environmentally friendly since it requires only four processing steps without a metal sputtering process. It is notable that the method we developed here realizes patterned interdigital electrodes automatically through surface wettability differences, which performs a certain degree of self-assembly [23]. As shown in Fig. 8, we only need to drop aqueous graphene ink at any position in the hydrophilic interdigital pattern area, and it will self-assemble along the patterned area. In a short time, aqueous graphene ink can automatically fill the hydrophilic region. What is more, we can increase the thickness of interdigital electrodes by adding more ink again on the same area before it solidifies, which provides a simple method to control the thickness of conductive film.

Fig. 6 Electrochemical characterization of MSCs(18). **a** Area capacitance and **b** volumetric capacitance of MSCs(18) obtained from CV curves versus different scan rate. **c** GCD curves obtained from dif-

ferent current densities of 5–15 $\mu\text{A cm}^{-2}$. **d** Capacitance retention of MSCs(18) measured at a scan rate of 80 mV s^{-1}

Fig. 7 **a** Comparison of area capacitance of graphene-based MSCs(18), and MSCs(12) from different scan rate and **b** Comparison of GCD curves of graphene-based MSCs(18) and MSCs(12) at current densities of 15 $\mu\text{A cm}^{-2}$

In order to explore the relationship between the number of fingers and the self-assembly time of the aqueous graphene solution filling in the hydrophilic interdigitated pattern region, we applied the same process to fabricate

five kinds of interdigitated finger patterns with different number of fingers. As shown in Fig. 9, it can be seen that the self-assembly time is about 12 s when the number of the fingers is 10, and then increases to 10.5 s when the

Fig. 8 Self-assembly photographs of graphene ink in hydrophilic interdigital patterns

Fig. 9 Diagram of the relationship between the self-assembly time of aqueous graphene ink filling the interdigitated pattern and the number of fingers of interdigital pattern

finger number increases to 18. The results show that as the number of fingers increases, the self-assembly time will not increase significantly. In other words, there is no mathematical relationship between the number of fingers and the time of self-assembly. Therefore, we can conclude that the efficiency of self-assembly of the interdigitated electrodes remains constant regardless of the number of electrodes, which is suitable for the preparation of complex devices. This technology provided a new idea for the rapid and convenient fabrication of on-chip energy storage devices and provide potential applications for portable electronic devices such as microsensors.

However, there is a problem to be solved during the procedure. For example, the aqueous graphene ink will diffuse out to the SH area as a result from the wide

interface area between the SH and SHL regions, so that the actual width of the interdigital electrode is large than the patterned width formed by the laser direct writing. This makes it necessary to etch a pattern with a narrower interdigitated width to obtain the electrodes satisfy our desired width. We will study this issue in future experiments, such as using a picosecond or femtosecond laser with nonthermal effect to fabricate patterned interdigital electrodes.

4 Conclusion

A simple and fast method for manufacturing graphene-based planar micro-supercapacitors without mask and photolithographic micropatterning was proposed. The planar MSCs were fabricated via selective laser etching-induced SH and SHL patterns on the alumina ceramic substrate, which allows the aqueous graphene ink rapidly covers the patterned interdigital electrodes area according to the wettability difference. This approach is distinct from the earlier report that the graphene ink can self-assemble on the hydrophilic interdigital pattern area. The resultant microdevices demonstrated relatively good electrochemical characteristics, the area capacitance of MSCs can reach 5.5 mF cm^{-2} and with cycling stability of $\sim 88.5\%$ after 4500 cycles. It is believed that these graphene-based MSCs, as a micropower supply, will have promising applications in miniaturized electronics and other chips.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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