



Fabrication of graphite grids via stencil lithography for highly sensitive motion sensors



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ABSTRACT

A graphite grid was fabricated using reactive ion etching with a stencil mask. The graphite film was synthesized through chemical vapor deposition on commercial Ni foil and was simply etched with a stencil mask using oxygen plasma during reactive ion etching. It had ~85% optical transmittance at a 550-nm wavelength and ~120 Ω/sq sheet resistance. Graphite grids placed on a human hand used to distinguish its motions showed high strain-sensing performance: its gauge factor ranged from 100 to 350, and its response times for strain loading and for release were short (10 and 80 ms, respectively). Because the graphite-based structures could be prepared in large quantities and because etching with a reusable stencil mask is very simple, mass production of high-performance motion sensors using the well-structured graphite grid is commercially feasible.

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1. Introduction

Recent progress in stretchable and flexible materials has realized the fabrication of wearable sensing devices that can be incorporated into clothing or attached directly to the human body for monitoring body motion [1–6], health [7,8], and therapeutics [9]. In order to develop strain and motion sensors with high sensitivity, the materials should have superior ability for bending, stretching, contracting, and twisting, as well as high durability, high conductivity, and high transparency. Various functional materials have been used for strain and motion sensors, including inorganic films [10], organic semiconductors [7,8], graphene [2,5], carbon nanotubes (CNTs) [1,3], piezoelectric composites [4,11,12] and conducting elastomers [6].

There has been considerable interest in the use of the aforementioned materials as strain-sensing materials, in particular, carbon-based materials such as CNTs and graphene, because of their advantageous optical, electrical and chemical properties, as well as their flexibility and stretchability [1–3,5]. CNTs exhibit good strain-gauging performance, conductivity, and transparency [1,3],

and graphene has additional advantages such as uniform structural integrity. Graphene can also be used in the fabrication of scalable devices via top–down approaches that are compatible with current techniques for semiconductor fabrication. Furthermore, a grid structure fabricated from CNTs and graphene has greater optical transparency and strain sensitivity as compared with those of normal uniform films. This enhancement is due to transmission of visible light through empty regions of the grid patterns, as well as significant modulation of the electrical properties of carbon channels under strain by the edges and intersections in the grid lines [2,5,13].

In spite of intensive efforts in this field, carbon materials that are currently studied are critically limited in their application, are expensive, and have relatively poor durability. In addition, their fabrication process is complex and lacks scalability. These attributes of CNTs and graphene grids are the main hurdle to their use in practical applications. For example, expensive metal grid catalysts (Cu or Ni) must be etched away by an etchant to transfer the graphene grid onto target substrates after chemical vapor deposition (CVD) [2,5]. In addition, graphene grids are too delicate to sustain their structures and are easily broken during the transfer process. Therefore, developing a simple approach for large-scale fabrication of high-performance carbon grids with controlled structures for use in strain sensors is vital.

Herein, a graphite grid for high-performance strain and motion

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sensors with high sensitivity is reported for the first time. The graphite film was synthesized through CVD on commercial Ni foil and was simply etched with a stencil mask using oxygen plasma during reactive ion etching (RIE). Exposed regions of the graphite film under the stencil mask were etched away to leave a graphite grid with the shape of the stencil mask. The fabricated grid showed outstanding strain-sensing performance; its gauge factor was ~ 350 at a strain of $\sim 15\%$, and its response times for strain loading and release were short (10 and 80 ms, respectively). Such performance is superior to that of other materials, including metal grids, normal graphite films, commercial metal gauges, conductor/polymer composites, and doped Si strain sensors [14–16]. Furthermore, hand motions could be distinguished through an array of strain sensors using the graphite grids. Because the graphite-based structures could be prepared in large quantities and because etching with a reusable stencil mask is very simple, mass production of high-performance motion sensors using the well-structured graphite grid is commercially feasible.

2. Experimental details

2.1. Synthesis and transfer of graphite film

Nickel foil (25 μm , Goodfellow) was positioned at the center of a thermal furnace. A gas mixture of hydrogen at 50 sccm and argon at 500 sccm was introduced to attain a final pressure of 350 Torr. And then the temperature of the chamber was increased to 1000 $^{\circ}\text{C}$. When the temperature of the furnace reached 1000 $^{\circ}\text{C}$, methane gas was injected at a flux of 50 sccm for 5 min. Finally, the furnace was rapidly cooled to room temperature while maintaining a 50 sccm flux of hydrogen and a 500 sccm flux of argon, resulting in the growth of graphite film with ~ 200 nm thickness (Alpha-Step IQ Surface Profiler). After growth, the nickel foil was removed with a 1 M FeCl_3 aqueous solution, and the graphite film was rinsed to remove residual FeCl_3 solution. The rinsed graphite film was transferred onto various target substrates, including glass, silicon, SiO_2 , and a transparent medical band, and then the graphite/substrate assembly was oven-dried at 60 $^{\circ}\text{C}$.

2.2. Etching conditions of graphite and metal film during stencil lithography

For the fabrication of the graphite grid, RF oxygen plasma at a 100-sccm flux with a power density of 80 W was introduced. The base pressure of the RIE chamber was 0.02 mTorr. In the fabrication of the metal grid, a metal layer (gold, silver, and copper) of 100-nm thickness was deposited onto the stretchable medical band by electron-beam evaporation. A nickel grid was positioned on the metal layer, and the metal layer was exposed to the low energy (500 eV) of Ar^+ ion bombardment until unprotected regions were fully removed. It seems that usually 10 min was enough for entire etching of metal film in our experimental condition.

2.3. Characterization

A scanning electron microscope (SEM, Sirion, FEI) was used to observe the surface of the synthesized graphite and the graphite grid on a SiO_2 substrate. To observe the domain structure of the surface of the graphite film, the graphite was coated with 4-pentyl-4-cyanobiphenyl (Sigma Aldrich), and the texture of the liquid-crystal molecules that aligned on the surface of the graphite was observed using polarized optical microscopy (POM; LV100-POL, Nikon). Raman spectroscopy (excitation, 514 nm) (Aramis, Horiba) was performed to monitor the quality of the graphite film and the graphite grid on the SiO_2 substrate. An X-ray diffractometer (XRD,

D/Max-2500, Rigaku) was used to monitor the lattice structure of the graphite film on the glass substrate. XPS (Sigma Probe, Thermo VG Scientific) was performed to analyze the functional groups on the surface of the graphite film and the graphite grid on the silicon substrate. An optical tensiometer (Attention, KSV Instruments) was used to measure the contact angle of a water droplet on the graphite film before and after 3 min of RIE treatment. The optical transmittances of the graphite and metal grid were measured on a spectrophotometer (V-570, Jasco). To measure the electric sheet resistance, 50-nm gold/2-nm chromium electrodes (four lines) were deposited by electron-beam evaporation onto the graphite grid. The electrode width and the gap between electrodes were 100 μm . The electric sheet resistance was measured on a probe station (four-point probe, M150, Cascade Microtech).

2.4. Sensing test on the strain sensor

A commercial stretchable medical band was used as a substrate. After the graphite grid was transferred, dried, and patterned, silver wires were connected by silver paste to form the strain sensor. To observe the response of the strain sensor, the lead wires of the sensor were connected to probes of a probe station (four-point probe, M150, Cascade Microtech), and the electrical resistance was recorded under a voltage bias of 1 V. To measure the sensing speed, the strain sensor was attached to an index finger and then stretched and released, and the delay time for the response was measured. For the sensing of hand motion, a sensor was attached on each of all five fingers, and the real-time responses were collected individually.

3. Results and discussion

The overall scheme for the fabrication of the transparent, flexible graphite grid via RIE with a stencil mask is presented in Fig. 1a–d. First, graphite film (200 nm thickness) was grown on nickel foil through atmospheric CVD, as shown in Fig. 1a. Because carbon easily dissolves in Ni foil at high temperature (~ 1000 $^{\circ}\text{C}$) [17,18], uniform and high-quality polycrystalline graphite films formed on the entire surface of the Ni foil (Fig. S1) [19–21]. Ni foils were etched with an aqueous 1 M FeCl_3 solution, and then the graphite films were transferred to target substrates, such as a transparent, flexible, and stretchable medical band (Fig. 1b) [5,22]. A stencil mask was then placed on the graphite film, as shown in Fig. 1c. In this experiment, a commercial Ni grid film with a 30 μm line width and 300 μm spacing between centers of grid lines was used as a stencil mask. A poly(methyl methacrylate) (PMMA) layer was spin-coated and used as a sacrificial layer to improve contact between the graphite film and Ni grid. The PMMA layer was removed after RIE by simple immersion in acetone solution. After the exposed region of the graphite film was etched during the RIE process, the replica structure of the stencil mask was retained, as shown in Fig. 1d. Scanning electron microscopy (SEM) images of the fabricated graphite grid after 10 min of the RIE process with the Ni grid stencil mask are displayed in Fig. 1e and f. Bright etched graphite regions and dark graphite grid lines are clearly distinguishable from the sharp edges after RIE. These indicate the successful selectivity of the etching process. The SEM results confirm that the feature dimension of the resulting graphite grid is identical to that of the stencil mask (30 μm line widths and 300 μm rectangular voids), thus indicating the successful fabrication of the replica through the stencil-mask approach.

Notably, the Ni grid was stable during the RIE process and was thus reusable. Because stencil lithography does not use an additional resist film, toxic chemical solvents, radian energy, or mechanical pressure, as is the case in conventional lithographic

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