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Direct graphene synthesis on LiNbO₃ substrate by C implantation on Cu covering layer

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Abstract

We directly synthesized multi-layer graphene with an area of several hundred square microns on the lithium niobate (LiN, LiNbO₃) substrate by Carbon (C) implantation into the copper (Cu)-covered LiNbO₃. The energy of C ion implantation was optimized per SRIM simulation to ensure that the distribution of C covers the Cu/LiNbO₃ interface. The optimized energy was established at 55 keV, such that the formation of C peaks in the respective materials on each side of the Cu/LiNbO₃ interface. The diffusion of the accumulated C to the Cu/LiNbO₃ interface can form a more uniform C distribution at the interface, which is beneficial to the synthesis of graphene. Following the annealing process and removal of the Cu coating, a multi-layer graphene with an area of several hundred square microns on the surface of LiNbO₃ was identified and characterized using Scanning Electron Microscopy (SEM), Energy-Dispersive x-ray Spectroscopy (EDS), Raman spectroscopy, and Atomic Force Microscopy (AFM). This remarkable advancement encourages the industrialization of direct graphene synthesis on LiNbO₃ substrates via ion implantation.

1. Introduction

The ideal monolayer graphene is a two-dimensional crystal with a honeycomb structure made of carbon atoms [1]. Through sp² hybridization, the three valence electrons of the carbon atom form strong σ–bond with the three carbon atoms that are immediately adjacent, and the unhybridized fourth electrons form large π–bond in an off-domain pattern that span the entire graphene [2]. Graphene’s exceptional performance is a result of its unique structure, which exhibits a very high carrier mobility when it comes to electronics [3, 4]. Optically, graphene has a frequency-independent transmittance of up to 97.7% over a wide spectral range from the visible to the infrared, and its optical absorption is linearly dependent on the number of graphene layers [5, 6]. Graphene also has excellent mechanical properties and an extremely high thermal conductivity [7, 8]. These excellent properties have already led to great advances in the application of graphene in micro-nano optoelectronics, spintronics, photonics and other low-dimensional devices [9].

The application of graphene is based on the graphene/substrate structure, and a large number of related studies have been published in recent years, these studies have primarily focused on the graphene/SiO₂ (Si) structure [10–16]. Currently, a few researchers are turning their attention to optical crystals as substrates, with graphene/LiNbO₃ structures being one of the most promising combinations. LiNbO₃, also known as the optical 'silicon' material [17–19], is an artificial crystal that has a wide range of optical applications and exhibits the characteristics of ferroelectric, piezoelectric, pyroelectric, photovoltaic, photoelastic, and photorefractive [20–22]. Its interaction with graphene has the potential to change the quantum properties of two-dimensional nanomaterials, resulting in new photoelectric properties. The two-dimensional nature of graphene makes it more sensitive to environmental changes, which provides a platform for new functional effects through the influence of substrate materials on graphene. For example, it has been reported that in the graphene/LiNbO₃ combined structure, the ferroelectric polarization characteristics of the LiNbO₃ can change the electrostatic
environment of graphene, adjusting its Fermi level and enabling the electrical 'doping' of graphene [23]. Furthermore, by changing the electrostatic properties of graphene, Surface Plasmon Polariton (SPP) can be excited on graphene under the condition of infrared light irradiation via grating coupling, prism coupling, and near-field coupling [24, 25]. In several reported graphene SPP structures, the Fermi energy level of graphene can be adjusted by the applied voltage or other alternative methods, and the dynamic modulation of graphene SPP is more easily accomplished [23–25]. Our previous research also demonstrated that the graphene-LiNbO3 composite structure had good light-guiding characteristics, the coupling of the LiNbO3 thin-film layer with the graphene SPP can sustained the stable propagation of the guide mode in the waveguide [26]. Up to now, graphene-LiNbO3 structures have exhibit a wide range of potential applications in electronics and photonics devices, such as field effect transistor [27–29] sensors [30, 31], beam splitter [26], optical absorber and modulators [32] etc.

Preparation of graphene-LiNbO3 structures is a prerequisite and basis for exploring device properties and functions. Currently, micro-mechanical peeling, pyrolysis of SiC, reduction of graphene oxide, and chemical vapor deposition (CVD) are the most maturely developed graphene preparation processes. Among them, CVD is the most commonly used method commercially because it enables the preparation of relatively high-quality graphene over a large area and the transfer of graphene to various other substrate materials can also be easily done with the help of conventional methods. Nevertheless, some other methods have been tried to compensate for or improve the CVD approach, including the recently reported ion implantation method. Although the preparation of graphene by ion implantation is far from mature in comparison to CVD, it has several inherent advantages that are particularly beneficial for the preparation of graphene/substrate combination structures [6, 10, 33]:

(a) As a non-equilibrium process, the C capacity introduced by implantation can be independent of the C solubility in material. Therefore, any suitable functional material can theoretically be used as the substrate material.

(b) Accurate control of the implanted C dose allows for precise control of the quantity of the layers of synthetic graphene.

(c) Promising direct graphene synthesis at the graphene/substrate interface based on tuning the implantation energy to adjust the C distribution.

In the preparation of combined graphene/substrate structures, although subsequent transfer processes can facilitate the transfer of graphene to a variety of different substrates, direct bonding of graphene to the substrate without intermediate processes remains the best solution because it avoids distortion of graphene and the introduction of impurities and defects during the transfer process.

Similar to the idea of growing graphene by CVD, ion implantation method currently used is mainly to implant carbon ions into Ni foil and Cu foil or Ni film and Cu film deposited on SiO2/Si substrate, allowing for the simple production of graphene films under optimized experimental conditions [11, 12, 34, 35]. In contrast to these studies, our group has attempted to obtain composite structures of graphene/LiNbO3 using ion implantation. We reported the direct synthesis of graphene on the surface of LiNbO3 in 2020 by annealing a post-carbon-implantation sample made up of a surface metal Ni coating and a LiNbO3 substrate [36]. This method, which is compatible with conventional integration processes, not only provides a way for transfer-free synthesis of graphene to avoid graphene degradation, but also suggests the possibility of engineering two-dimensional graphene with special confinement by patterned metal film, which is often emphasized in photovoltaic crystals like LiNbO3. However, although graphene structures can be obtained by C ion implantation into Ni/LiNbO3 [36], the resulting structures contain not only multi-layer graphene mixed with turbostratic graphene, but also amorphous C without any lattice structure, making the resulting graphene impractical in term of structural purity.

Analyzing the reasons for this result, we found that the homogeneity of the metal film, annealing conditions and the distribution of C in the two materials are the main reasons affecting the quality of graphene formation. In this paper, we report on the attempts made to improve the structural purity of the formed graphene. By considering the matching of metal films with LiNbO3 substrates, the distribution of implanted C ions and the adjustment of experimental parameters, the growth environment of graphene was improved and optimized, resulting in the direct growth of multi-layer graphene with an area of several hundred square microns on LiNbO3 substrates. This provides an important reference for the preparation of graphene/optical crystal composite structures.
2. Materials and methods

Figure 1 shows a schematic diagram of the experimental steps. The substrate was a Z-cut $10 \times 10 \times 0.5 \text{ mm}^3$ bulk LiNbO$_3$ crystal, and one of the $10 \times 10 \text{ mm}^2$ surfaces was treated by chemical mechanical polishing to make the subsequent coating operation easier. After depositing a Cu thin film as a capping layer on the polished surface, carbon ion implantation was performed at room temperature. The distribution of implanted carbon was designed to cover the bottom of the metal film, the metal/LiNbO$_3$ interface, and the near surface of LiNbO$_3$ crystal. The sample was subsequently annealed, during which the implanted carbon diffused into the interface between the metal film and LiNbO$_3$ crystals. Ideally, the precipitated carbon would form several layers of graphene, and finally the metal film would be removed to obtain the desired graphene/LiNbO$_3$ structure.

Instead of Ni, in the present experiment we used Cu film as the metal cover layer on LiNbO$_3$ substrate. This change came from avoiding a recurring problem in previous experiments where Ni as a cover film for LiNbO$_3$ substrate was easily blistered or peeled after undergoing high temperature annealing. Although both Cu and Ni are face-centered cubic structures, as shown in figure 2, their (111) faces have lattice constants of 5.112 Å and 4.984 Å, respectively. When combined with a Z-cut LiNbO$_3$ substrate with a lattice constant of 5.152 Å [37], the Cu/LiNbO$_3$ structure has a small lattice mismatch and is more likely to form flat and stably bonded metal film, which is essential for the formation of graphene.

Magnetron sputtering was used to deposit the layer of Cu thin film on the surface of LiNbO$_3$. The thickness of the Cu film was measured by AFM to be approximately 67 nm, as shown in figure 3. To match the carbon content of the tri-layer graphene, we employed a carbon ion implantation dose of $1.14 \times 10^{16}$ ions·cm$^{-2}$. In order to choose the ideal implantation energy to ensure the formation of the graphene/LiNbO$_3$ structure, the distribution of carbon ions must be set to cover the bottom of Cu, the interface, and the top surface of LiNbO$_3$. Unlike our previous experiments, we tried to adjust the implantation energy of C to obtain a bimodal distribution of C in both materials. The distribution of carbon ions along the depth in the carbon-implanted Cu/LiNbO$_3$ samples were simulated using the SRIM software package, with implantation energies ranging from 45 keV to 65 keV, as shown in figure 4. In order to avoid the channeling effect, a $7^\circ$ implantation angle was used in both simulation and experiment. Comparing the carbon ion distribution in figures 4(a)–(e), it can be found that a carbon distribution peak mainly situates in the Cu layer when the implantation energy is less than 50 keV, and the peak of the carbon distribution shifts to the LiNbO$_3$ layer when the implantation energy is larger than 60 keV. This means that when the implant energy is less than 50 keV or more than 60 keV, there is only one peak of the carbon distribution in either the Cu layer or the LiNbO$_3$ layer. And when the implant energy is between 50 keV and 60 keV, the distribution peak of C splits, as shown in figure 4(a), there is one peak of the carbon distribution in each layer. If the implant energy is selected as 55 keV, two peaks being roughly the same size are formed and locate close to the interface of Cu and LiNbO$_3$. Such a carbon distribution allows for bidirectional diffusion of carbon in opposite direction to the interface during subsequent annealing and promotes uniform carbon distribution at the interface. As shown in figure 4(a), with a relatively high concentration of implanted carbon within the range of 40 ~ 120 nm, a significant number of carbon atoms distributed near the interface are available to form graphene.

Following carbon implantation, the sample was annealed in an annealing furnace. The choice of annealing conditions requires consideration of the diffusion nature of C in Cu and the effect of high temperature annealing on LiNbO$_3$. Our previous studies have found that the C in LiNbO$_3$ can effectively diffuse to the interface when the diffusion temperature lies in the temperature window of 500 °C ~ 650 °C [38], while the aggregated C at the Cu/LiNbO$_3$ interface beyond 700 °C is mainly from the C in Cu [12, 39, 40]. Therefore, the annealing process is set to first increase the temperature to 1050 °C at a rate of 10 °C·min$^{-1}$, with a ramp-up time from 500 °C to 650 °C being approximately 15 min, then held at that temperature for 15 min, and finally naturally cooled to room temperature in situ. The entire annealing process was carried out in N$_2$ environment with a gas flow rate of 100 SCCM and a pressure of $6 \times 10^4$ Pa.
3. Results and discussions

An optical microscope was used to examine the surface of the annealed sample, and no local separation of the Cu film from the LiNbO3 substrate was found. The surface of the sample after implantation and high temperature annealing showed good integrity and flatness. The upper surface of the Cu film was characterized by Raman spectroscopy, and no trace of carbon was found, indicating that the carbon did not precipitate from the upper surface. This is because the majority of the implanted carbon was distributed near the interface between the Cu film and the LiNbO3 substrate, and the precipitation of C tends to occur more at the Cu/LiNbO3 interface.

After etching the Cu layer with FeCl3 solution, we used a SEM to observe the exposed LiNbO3 surface, several membrane-like structures can be observed on the LiNbO3 surface. One of them with a surface area of several hundred square microns is characterized by SEM, EDS and Raman spectroscopy. Figure 5(a) depicts the SEM morphology of it with an acceleration voltage of 5 kV; (b)–(d) depict the respective EDS mapping of C, Nb, and O elements corresponding to the material composition in figure (a) with an acceleration voltage of 15 kV. It can be found that the distribution of C elements is in good agreement with the morphology of membrane-like structures, while the distribution of Nb and O from LiNbO3 substrate is relatively uniform, except for some dark

![Figure 2](image-url). The top-view schematic diagram of (a) a LiNbO3 unit cell, (b) a 2 × 2 Cu (111) layer supercell, and (c) a 2 × 2 Ni (111) layer supercell.
areas covered by the C structure. The results of SEM and EDS reveal that these membrane-like materials are composed of C element.

Raman spectroscopy was performed on the carbon material with an excitation wavelength of 532 nm, and the results are presented in figure 6. The four Raman spectra in figure 6 correspond to the results from four locations indicated in figure 5(a). Each of them reveals the same three spectral features that are unique to graphene structure: a G peak (around 1586 cm\(^{-1}\)) associated with sp\(^2\) carbon bond stretching, a 2D peak (around 2685 cm\(^{-1}\)) sensitive to graphene interlayer interactions, and a D peak (around 1344 cm\(^{-1}\)) arising from symmetry-breaking features such as graphene defects and domain boundaries. For the

![Figure 3. AFM height profiles along the straight line from uncoated notch to the coated area.](image)

![Figure 4. Structural schematic diagram of the coated bulk LiNbO\(_3\) crystal with the distribution of carbon ions along with the depth profiles, simulated by SRIM, when the implantation energy is (a) 55 KeV, (b) 45 KeV, (c) 50 KeV, (d) 60 KeV and (e) 65 KeV.](image)
ideal two-dimensional graphene structure, usually the intensity ratio of the D-peak to the G-peak reflects the degree of disorder, and the intensity ratio of the 2D-peak to the G-peak reflects the number of stacked layers. Comparing the four Raman spectra it can be found that both the D/G and 2D/G ratios from the four different positions of graphene are very close to each other. The relative high intensity of the D-peak suggests the presence of a number of structural defects in the synthesized graphene. The 2D/G ratio in all the spectra is approximately
equal to 0.59, which is consistent with the intensity ratio of multi-layer graphene with 4 ~ 6 layers, demonstrating the relative homogeneity of the formed graphene structure.

The distribution of resulting multi-layer graphene can also be verified by AFM measurement. A relatively distinct graphene boundary can be calibrated by AFM, and the results are shown in figure 7. The tested graphene boundary is located in the red boxed area in figure 5(a). Figure 7(a) depicts a two-dimensional topography of graphene by AFM, and the protrusion in the lower right corner could be impurity particle, residual metals or even folded multi-layer graphene film. The boundary of the layered graphene is also clearly visible in figure 7(a), and the height profiles along the three solid blue lines at different positions spanning the graphene and the LiNbO3 substrate are shown in figure 7(b). The height difference is 31 ~ 37 nm, indicating that the thickness of graphene film is around 34 nm. Although the theoretical thickness of ideal single-layer graphene is 0.35 nm, in practical AFM measurements, the measured thickness of graphene, especially for multi-layer graphene, increases geometrically due to weak interactions between graphene and substrate and the presence of environmental species (nitrogen, oxygen or water) between the AFM probe and graphene. And this effect is magnified for multi-layer graphene due to the greatly increased roughness caused by increased layer number and interlayer spacing [12, 26, 41, 42]. In particular, for image stability in SEM and EDS characterization, we plated a layer of platinum on the sample surface; the thickness of the multi-layer graphene was also influenced by this platinum layer.

We have experimentally demonstrated that multi-layer graphene with an area of several hundred square meters can be formed directly on LiNbO3 substrates, which provides an effective method for the formation of graphene/crystal composite structures. In particular, the method of C-ion implant into metal films allows...
flexible adjustment of the shape of the formed graphene structures, which is particularly important for micro-nano structures using graphene as a functional material. The currently formed graphene still has many defects, by adjusting the implantation and annealing conditions, we hope to improve the quality of the graphene film while reducing the damage to the LiNbO3 lattice caused by the implantation. In addition, the testing and analysis of the optoelectronic properties of this composite structure will be the subject of our future research.

4. Conclusions

Based on a Cu/LiNbO3 combination using metallic Cu as a cover layer for the LiNbO3 substrate, we have synthesised multi-layer graphene structures with an area of several hundred square microns directly on LiNbO3 substrates using the method of implanting C near the Cu/LiNbO3 interface. Before C implantation, a uniform 67 nm thick Cu layer, functioned as a catalyst, was deposited to the polished surface of the LiNbO3 substrate by magnetron sputtering to form Cu/LiNbO3 sample. Based on simulations, the implantation energy of C for the experiment was finally determined to be 55 keV, as the C implanted under this condition can form a similarly C peak distribution in the respective materials on each side of the Cu/LiNbO3 interface, which will facilitate not only the diffusion of C to the interface but also the uniform growth of graphene during the subsequent annealing process. C content of tri-layer graphene was selected as the carbon implantation dose. After implantation, the sample was annealed in an annealing furnace under a N2 environment. Following annealing and removing Cu film, a multi-layer graphene with an area of several hundred square microns on the upper surface of the LiNbO3 substrate was identified and characterized by SEM, EDS, Raman spectroscopy, and AFM. This method of preparing graphene/substrate structures not only provides a more effective way to prepare graphene-based functional devices, but also promotes the industrialization of direct synthesis of graphene by ion implantation on optical crystal substrates.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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