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2D MoS₂ Encapsulated Silicon Nanopillar Array with High-Performance Light Trapping Obtained by Direct CVD Process

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Abstract: Weak absorption remains a vital factor that limits the application of two-dimensional (2D) materials due to the atomic thickness of those materials. In this work, a direct chemical vapor deposition (CVD) process was applied to achieve 2D MoS₂ encapsulation onto the silicon nanopillar array substrate (NPAS). Single-layer 2D MoS₂ monocrystal sheets were obtained, and the percentage of the encapsulated surface of NPAS was up to 80%. The reflection and transmittance of incident light of our 2D MoS₂-encapsulated silicon substrate within visible to shortwave infrared were significantly reduced compared with the counterpart planar silicon substrate, leading to effective light trapping in NPAS. The proposed method provides a method of conformal deposition upon NPAS that combines the advantages of both 2D MoS₂ and its substrate. Furthermore, the method is feasible and low-cost, providing a promising process for high-performance optoelectronic device development.

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Keywords: 2D MoS₂; nano pillar array substrate; CVD; encapsulation; absorption

1. Introduction

Two-dimensional (2D) materials, including transition metal dichalcogenides (TMDs) [1–3], graphene [4,5], black phosphorus [6,7], etc., are attracting ongoing focus due to unique physical properties compared with their block counterparts, owing to the thickness at the atom diameter level and structure [8]. Regarding 2D materials, the optoelectronic property is one of the most attractive topics [9–11]. Two-dimensional materials are applied to develop photodetectors with high quantum efficiency, high responsivity, broadband responses, small dark currents, fast responses and so forth [12,13]. Nevertheless, there is a huge obstacle confusing almost all of those who have tried to develop high-performance detectors based on 2D materials—namely, the extremely weak absorption of incidence [14]. For example, the absorption of graphene is only about 2.3% [15], which means that the overwhelming majority of the incident light is lost, and photoelectronic conversion can only be achieved within the remaining 2.3% of photons absorbed.

Researchers are working hard to bring up various methods to promote the light absorption of 2D material-based optoelectronic devices. Noble metal-based surface plasmon is one of the most utilized approaches to enhancing optical absorption. Jinshui Miao, Weida Hu and Lei Liao et al. studied optical absorption by surface plasmon in few-layer MoS₂ phototransistors with nanostructure arrays, achieving a doubled increase in the photocurrent response from a few-layer phototransistor decorated by 4-nm-thick nanoparticles and a photocurrent of a threefold enhancement by periodic Au nanoarrays [16]. Denis Garoli and Francesco De Angelis et al. studied the optical enhancement for MoS₂ flakes on metallic nanoholes through hybrid plasmonic nanostructures; this strategy can also be applied to 2D materials beyond MoS₂ in sensors based on 2D materials [17]. Andrea Camellini, Giuseppe Della Valle and Margherita Zavelani-Rossi et al. proposed an Au-MoS₂ metasurface structure for a plasmon-enhanced charge transfer, which provides a substantial advancement

toward scalable ultrathin photodetection devices based on hot-electrons technology [18]. These researchers contributed to optical absorption enhancement a great deal, and the noble metal-based surface plasmon was proven to be an effective approach to that issue. Meanwhile, there exist limitations in this method. For example, it is fairly difficult to deposit Au nanoparticles upon 2D materials, especially in few-layer or single-layer, without damaging them, because 2D materials are usually weak in absolute strength [19]. Besides, heavy metals like Au are strictly forbidden in semiconductor fabrication, which means that the fabrication of devices utilizing heavy metals cannot be compatibly integrated with general semiconductor fabrication.

Resonance structures also apply light absorption enhancement. Marco Furchi and Thomas Mueller et al. demonstrated that the optical absorption can be promoted up to 60% by monolithically integrating graphene with a Fabry-Perot microcavity, and they presented a graphene-based microcavity photodetector with responsivity of 21 mA/W [20]. Jianfa Zhang et al. proposed a resonant nanostructure with multilayer configuration based on subwavelength gratings of monolayer MoS₂, realizing perfect ultra-narrowband visible absorption [21]. These efforts provide alternative ways to enhance the absorption in 2D material-based optoelectronic devices, and impressive results were achieved. Regarding the fabrication, these methods still require a relatively complex process and materials—for example, a microcavity-based graphene detector compared with commercial Si-based detectors. Harsh Chaliyawala, Govind Gupta and Indrajit Mukhopadhyay prepared a NIR photodetector of the Schottky junction with graphene deposited on Si-nanowire [22]. The process is feasible, and the device structure is rather simple; thus, quite a few similar devices were developed. The Si nanowire arrays in that type of device were prepared by wet etching—for example, metal-aided chemical etching—resulting in random wire shapes and sizes, making it hard to obtain the controllable fabrication of such devices. Taking the issue of controllable fabrication, Jianting Lu, Zhaoqiang Zheng and Jingbo Li et al. developed an ultrasensitive photodetector with nonlayered 2D In₂S₃ deposited on periodic Si nanopillars that were prepared by classic e-beam lithography and the reactive ion etching (RIE) process. An extremely high responsivity of 4812 A/W was achieved from this proposed device [23].

The above results are encouraging, and the device and process could be further improved. The 2D In₂S₃ in that work was grown on inert mica by the physical vapor epitaxy method and then transferred onto Si nanopillar arrays [23]. Herein, we proposed a strategy of direct conformal growth of 2D MoS₂ upon periodic Si nanopillar arrays. The nanopillar arrays were developed by a commercial semiconductor fabrication process—namely, stepper lithography and RIE. Then, the 2D MoS₂ were directly grown onto the surface of the nanopillar arrays conformally by chemical vapor deposition. The preparation of Si nanopillar array substrate (NPAS) is compatible with the commercial semiconductor fabrication process, the growth of 2D MoS₂ is feasible at a low cost and the device fabrication process is obviously simplified with the 2D MoS₂ transfer omitted. The properties, including the electronic band structure of MoS₂, have been widely studied [24–28], which helps us understand the properties of this material, and here, we mainly focused on the direct deposition of MoS₂ on a nanostructured silicon substrate.

2. Experiments

In our work, the NPAS we used was obtained by commercial available lithography and the RIE process. Figure 1a shows the substrate we used to grow MoS₂ with a boundary above and below, which are the planar surface and the nanopillar array, respectively. Figure 1b is the local detail of the nanopillar arrays. The nanopillars were designed to be cylinders, and the obtained ones show a little inclination leading to slight cones.

Figure 1. Silicon nanopillar array substrate (NPAS) (a) optical image and (b) SEM local detail.

The 2D MoS₂ was directly grown onto the surface of the NPAS by chemical vapor deposition (CVD), as illustrated in Figure 2. A quartz tube was used to provide the space for the deposition, with sulfur powder and MoO₃ powder placed upstream and downstream, respectively, regarding the carrier gas flow. In our experiment, a single heater was utilized to heat the precursors, within the zone of which, the MoO₃ powder was placed. The sulfur powder was placed a few centimeters from the MoO₃ upstream, where the temperature was lower than the direct heating zone, and the temperature there met the requirement of sulfur evaporating into a precursor gas. The experiment was conducted as follows: When both precursors, sulfur and MoO₃, were placed in the quartz tube where designed, the carrier gas Ar was charged into the tube with a rate of 200 sccm and charging time of 15 min to eliminate the air and make sure the precursors were surrounded by Ar. Then, carrier gas Ar was charged at the rate of 20 sccm until the end of the CVD process. The quartz was heated to 775 °C with a temperature rise rate of 30 °C/min and then kept at 775 °C for 15 min. At last, natural cooling was applied with the quartz tube filled with Ar. The growth process of MoS₂ can be divided into five steps [29]: (1) precursors sublime and are transported downstream by a carrier gas, (2) they diffuse from the bulk vapor toward the substrate, (3) they adsorb onto the surface, (4) adatoms of the precursors diffuse along the surface, and (5) they react to form product structures.

Figure 2. The schematic of experiment setup for 2D MoS₂ chemical vapor deposition (CVD) upon NPAS.

In order to characterize the properties of grown 2D MoS₂ and Si NPAS encapsulated by 2D MoS₂, test procedures were conducted. First and foremost, SEM was applied to qualitatively find out how many percentages of the substrate area was covered by 2D MoS₂ and the distribution of the MoS₂ crystal sheets, which gave us an overall estimation of the effect of the MoS₂ conformal deposition. X-ray photoelectron spectroscopy (XPS) measurement was operated to investigate the components of the obtained materials from the CVD process. Raman spectra was utilized to determine whether the deposited crystal sheets are of single-layer or more. As was stated before, the efforts of 2D MoS₂ direct deposition onto nanopillar array substrates were intended to promote optical absorption. When the material was used to develop photodetectors, the optical properties must be performed, including reflection and transmittance of the composite structure.

3. Results and Discussion

The NPASs encapsulated by 2D MoS₂ are shown in Figure 3, in which different coverings can be noted under an increasing carrier gas flow rate from 50 sccm to 120 sccm. It is clear that the area covered by 2D MoS₂ would not increase monotonically with the increase of the carrier gas flow rate; in other words, there existed an optimal gas flow rate leading to the best covering, as is presented in Figure 4. Here, the optimized carrier gas flow rate was 90 sccm, resulting in an encapsulation rate of about 60%. The pictures of the 2D MoS₂-encapsulated nanopillar substrate were used to estimate the encapsulation rate by using optical contrast calculations.

Figure 3. Effect of the carrier gas flow rate on the 2D MoS₂ crystal size and encapsulation rate.

Figure 4. Encapsulation rate variation with the carrier gas flow rate.

In order to confirm that the material deposited on the substrate is monolayer MoS₂ crystal, Raman spectra and X-ray photoelectron spectroscopy were utilized to characterize it, as is shown in Figure 5. Two characteristic peaks related to the Raman vibration modes are presented in Figure 5a, in which the E_{2g} peak is associated with the in-plane vibration mode of molybdenum and sulfur atoms and A_{1g} is associated with the out-of-plane vibration mode of sulfur atoms. The number of layers of 2D MoS₂ determined the frequency variation between the E_{2g} and A_{1g} modes. It can be noted from Figure 5 that the frequency variation between these two modes was around 20.0 cm^{−1}, from 383.2 (E_{2g} location) to 403.2 (A_{1g} location). The results are in accordance with the frequency variation of monolayer MoS₂ demonstrated before, leading to the confirmation of the monolayer of the deposited MoS₂ in our experiment. XPS was applied to examine the components of the deposited sheets. The XPS spectra of Mo 3d and S 2p are presented in Figure 5b,c. The intensity peaks at 229.4 and 232.5 are attributed to the doublet Mo 3d_{5/2} and 3d_{3/2}, while the peaks at 162.3 and 163.3 are attributed to S 2p_{1/2} and 2p_{3/2}, respectively, which was confirmed by previous works [25,30]. The intensity ratio of Mo and S revealed the components of MoS₂. The XPS spectra peaks of Mo 3d at around 229 and 232 eV corresponded to the oxidation state of Mo⁴⁺, which was confirmed by researchers [25]. On the other hand, the peaks at 162.3 and 163.4 eV corresponded to divalent sulfide ions [26]; the ratio of S ions to Mo ions

was calculated as 2.04 according to their peak intensities; thus, the oxidation state of Mo should be quadrivalent.

Figure 5. Characterization of the obtained nanosheets upon the substrate: (a) Raman data and (b,c) X-ray photoelectron spectroscopy (XPS) data.

As was stated, the 2D MoS₂-encapsulated NPAS was intended to promote the optical absorption, so the optical properties were evaluated. When the incident light comes to a substrate, it is clear that the light incidence will either pass through the substrate or be absorbed or else be reflected backward. All the three portions sum up the total incident light. Aiming at the increase of light absorption, the reflected and transmitting light, in other words, should be reduced. Regarding the light transmittance and reflection with incident light of the wavelength from 400 nm to 2000 nm, the results were achieved, as shown in Figure 6. It can be noted in Figure 6a that, overall, the transmittance of incident light was close to zero with visible and near infrared, and it went up obviously when the wavelength was bigger than 1000 nm, which matches the optical properties of monocrystalline silicon. What is important was that the transmittance of NPAS was cut down from 60% to below 40%, compared with a planar one, when the incident wavelength was above 1000 nm. The results were in good accordance with our expectations. What is more, it can be seen that the Si NPAS encapsulated by MoS₂ resulted in lower transmittance (about 30% and below) than that of a bare one, contributing to the essential promotion of MoS₂-based photodetectors. The reflections of three types of substrates were also tested, and the results are presented in Figure 6b. The incident light was reflected at a ratio of 32.4–41.8% within the wavelength from 380 nm to 990 nm, and the reflectivity decreased when the incident wavelength became bigger. However, when the incident wavelength was bigger than 1050 nm, the reflectivity increased again and kept at about 46% within the wavelength from 1200 nm to 2000 nm. The reflectivity of the NPAS was reduced to 20% as high and nearly 2% as low in the visible spectrum. When it went into the near and short-wave infrared, the reflectivity of this kind of substrate was also significantly smaller than the planar one. Depositing 2D MoS₂ onto the NPAS, the reflectivity was further reduced, though the reduction was not that big.

Figure 6. Transmittance (a) and reflection (b) properties of three kinds of substrates.

Owing to the nanopillar array, light trapping was significantly increased for absorption promotion, which is illustrated in Figure 7 and will be further discussed. Similar results can be found in a previous work [31]. It was clear that, either in visible or infrared, the electric field was greatly enhanced within the Si substrate, especially inside the pillar, reaching up to 1.9 V/m and 1.4 V/m, as was shown in Figure 7a,d, which are the electric field distributions at incident wavelengths of 660 nm and 1350 nm, respectively. Figure 7b,e are the electric field distributions of MoS₂-encapsulated NPAS at incident wavelengths of 660 nm and 1350 nm, respectively, with electric field enhancements up to around 1.9 V/m and 1.3 V/m. Encapsulated by MoS₂, the electric field distribution inside silicon remains concentrated, with little change compared with that of the NPAS not encapsulated by MoS₂. Furthermore, the electric field between pillars was also enhanced in the encapsulated one, especially in infrared. Compared with the planar Si substrate, the electric field enhancement was achieved obviously within NPAS, and the enhancement was not affected too much when MoS₂ was deposited onto the substrate, which could lead to a performance promotion of the optoelectronic device.

Figure 7. Electric field distribution under different conditions. (a) NPAS, (b) NPAS encapsulated by MoS₂ and (c) planar substrate at 660 nm incidence; (d) NPAS, (e) NPAS encapsulated by MoS₂ and (f) planar substrate at 1350 nm incidence.

It can be seen in Figure 7 that the electric fields inside the silicon pillar and in the zone between pillars are enhanced. Scattering cannot be ignored in our work, which contributed to incident light confinement within the surface zone of the nanostructured substrate, leading to electric field enhancements in the space between pillars. Reciprocating reflection and scattering within the space between pillars makes it possible for incident light to travel through MoS₂ many times, leading to an absorption enhancement in MoS₂. Incidence confinement in silicon pillars contributes to photoelectronic conversion promotion, especially near the substrate surface, which can be applied in photoelectronic device designs.

As is mentioned, scattering plays an important role in light trapping [32]. Strong interpillar scattering takes place when the incident light comes to the NPAS. The scattered light will interact with the lateral surface of the pillar and the horizon surface between the pillars repeatedly. Within each interaction process, light absorption occurs. In other words, scattering increases light trapping in the NPAS, contributing to light absorption despite that part of incident light is confined in the space between nanopillars. On the other hand,

each individual pillar acts as a resonator that contributes to light trapping by multiple total internal reflections, leading to electric field enhancement inside the pillar [33]. The two mechanisms of light trapping, which are scattering and total internal reflection, are illustrated in Figure 8.

Figure 8. Schematic diagram of two light trapping mechanisms of scattering and total internal reflection in the light trapping of NPAS (not to scale).

4. Conclusions

Two-dimensional MoS_2 is widely regarded as a promising optoelectronic material due to its excellent physical property; however, 2D MoS_2 -based devices suffer from weak absorption. The nanopillar array is an effective approach to enhance light trapping. In our work, the CVD process was applied to achieve 2D MoS_2 encapsulated onto the surface of Si NPAS conformally in a direct way without a transfer process. Single-layer 2D MoS_2 monocrystal sheets were obtained, and up to about 80% of the area of the Si NPAS was encapsulated with the optimized process parameters. The 2D MoS_2 -encapsulated Si NPAS presented a light transmittance reduction from 60% to around 20% in near infrared and short-wave infrared. The light reflection was cut from around 40% down to 30% and below at the same time using the same substrate, resulting in light trapping, providing an encouraging approach to developing high-performance optoelectronic devices.

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