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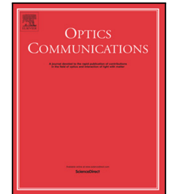

Optical contrast for identifying the thickness of two-dimensional materials

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ABSTRACT

One of the most intriguing properties of two-dimensional (2D) materials is their thickness dependent properties. A quick and precise technique to identify the layer number of 2D materials is therefore highly desirable. In this review, we will introduce the basic principle of using optical contrast to determine the thickness of 2D material and also its advantage as compared to other modern techniques. Different 2D materials, including graphene, graphene oxide, transitional metal dichalcogenides, black phosphorus, boron nitride, have been used as examples to demonstrate the capability of optical contrast methods. A simple and more efficient optical contrast image technique is also emphasized, which is suitable for quick and large-scale thickness identification. We have also discussed the factors that could affect the experimental results of optical contrast, including incident light angle, anisotropic nature of materials, and also the twisted angle between 2D layers. Finally, we give perspectives on future development of optical contrast methods for the study and application of 2D materials.

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1. Introduction

Two dimensional (2D) materials have attracted great attentions for the past decade due to its unique properties and great potential for applications. As an example, graphene, a single layer of carbon atoms arranged with honeycomb lattices, has ultrahigh mobility, superior mechanical and thermal properties, while at the same time is transparent but conductive, which make it promising candidate for electronics and optoelectronics [1,2]. After the discovery of graphene in 2004, different kinds of 2D materials have been explored, including layered transitional metal dichalcogenides (TMDs) [3,4], black phosphorus (BP) [5], boron nitride (BN) [6], antimonene [7], silicene [8], germanene [9]. These materials cover a broad range of electronic bandgap, from 0–~4 eV, which are suitable for different electronic devices. For example, TMDs are great for logic devices with high on/off ratio [3], graphene and BP is ideal for infrared photodetectors [10], BN is suitable as insulator buffer layers or substrates [11,12].

One of the most interesting behaviors of 2D materials is that their properties are strongly dependent on thickness or layer numbers. For example, single layer graphene has linear dispersive electronic band structure and nearly zero electron mass, while bi- and multi-layer graphene possesses massive Dirac fermions and its band structure can be tuned by perpendicular electric field [13]. On the other hand, most of the TMDs materials, including MoS_2 and WS_2, are direct bandgap semiconductors in monolayer, and becoming indirect bandgap materials when their thicknesses are more than two layers [14]. The bandgap of BP can be tuned from 0.3 eV to ~1.8 eV from bulk to single layer [15]. Therefore, it is of great importance to identify the thickness of 2D materials unambiguously and efficiently.

Modern techniques have been adopted to characterize the thickness of 2D materials, including transmission electron microscope (TEM) [16], atomic force microscope (AFM) [17], low energy electron microscopy (LEEM) [18], scanning tunneling microscope (STM) [19] and so on. Although these techniques have very high spatial resolution and can even resolve single atoms, they suffer from low throughput or complicate sample preparation. Optical techniques have the advantage of non-contact, quick and capable for large-scale investigation, which are ideal for the thickness identification of 2D materials. For example, Raman spectroscopy has been widely used to study graphitic materials, and can provide information on layer numbers, strain, doping, defects, and electronic bandstructures of graphene [20]. Photoluminescence (PL) spectroscopy is also frequently used to study the photo emission of 2D semiconductors with different thicknesses [21,22]. For example, the PL intensity of monolayer TMDs is 1–2 orders higher than that of multilayers, due to the difference of direct and indirect bandgap structure [14]. On the other hand, the PL emission can vary from

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~700 nm of single layer phosphorene to ~2 μm for samples of ~5 layers [15]. Second harmonic generation signals of TMDs are distinct for samples with odd and even layers [23]. However, in term of thickness characterization, Raman spectroscopy is normally used for graphene layer number determination of 1–4 layers [24], while PL spectroscopy can be used to identifying some of the direct bandgap semiconductors. On the other hand, optical contrast spectroscopy and images are the most commonly used tools for thickness identification of 2D materials [21,24–31]. They are easily operated, fast, and can be adapted to almost all the 2D materials, including graphene, graphene oxide (GO), TMDs, BP and BN. Different parameters that could affect the optical contrast of 2D materials have been reported and discussed extensively, e.g. refractive index of the multi-layer substrate and top deposition layer, incident angles, twist angles between 2D layers [32–37]. Most importantly, the optical contrast does not require expensive equipment and is ready for large-scale investigation.

In this review, we will discuss the recent progress on optical contrast of 2D materials. In Section 2, the basic working principle of optical contrast is presented. In Section 3, the use of optical contrast to determine the thickness of different 2D materials will be given, including graphene, GO, TMDs, BP and other 2D materials. In Section 4, an optical contrast image technique which is favored for large-scale thickness identification will be discussed. In Section 5, different factors that would affect the optical contrast of 2D materials will be discussed, including the incident angle of light, anisotropic natures of materials and twist angle between 2D layers. Finally, we discuss the perspective and challenges of optical contrast studies.

2. Working principle of optical contrast spectroscopy

The basic principle of using optical contrast to determine the thickness of 2D materials is shown in Fig. 1. When a 2D material is deposited on a substrate, due to the absorption of the material or change of the length of optical path if there is a multilayer structure, the intensity of reflection light from the sample would be different substrate. As an example, when a single graphene sheet is deposited on 285 nm SiO_2/Si substrate, it looks darker compared to substrate, as shown in Fig. 1(a), which means the light reflected from graphene is weaker compared to SiO_2/Si substrate. This can be easily understood by Fresnel law of multilayer interference as shown in Fig. 1(b). When a beam of polarized light (s-wave or p-wave) is incident at an interface with incident angle θ_1, for example, air/SiO_2 interface, a portion of the beam is reflected from the interface and the rest is transmitted, thus, an infinite number of optical paths are possible as schematically shown in Fig. 1(b). Take air/SiO_2/Si system for example, the total reflected amplitude r_0 is governed by Eq. (1) [24],

$$r_0 = \frac{r_{02} + r_{23} \cdot e^{-2i\phi_2}}{1 + r_{02} \cdot r_{23} \cdot e^{-2i\phi_2}}. \tag{1}$$

Similarly, for air/graphene/SiO_2/Si system, the total reflected amplitude r_1 can be calculated by Eq. (2),

$$r_1 = \frac{r_{01} + r_{01}r_{12}r_{23}e^{-2i\phi_2} + r_{12}e^{-2i\phi_1} + r_{23}e^{-2i(\phi_1+\phi_2)}}{1 + r_{12}r_{23}e^{-2i\phi_2} + r_{01}r_{12}e^{-2i\phi_1} + r_{01}r_{23}e^{-2i(\phi_1+\phi_2)}}. \tag{2}$$

Here, r_{ij} is reflection coefficient at the layer i/layer j interface. At normal incident, r_{ij} can be calculated by $r_{ij} = \frac{\widetilde{n}_i - \widetilde{n}_j}{\widetilde{n}_i + \hat{n}_j}$. $\widetilde{n}_i$ ($\widetilde{n}_j$) is the complex refractive index of layer i (layer j). At oblique incident, in the calculation of reflection coefficient, for s-polarized light, $\widetilde{n}_i$ should be replaced by $\widetilde{n}_i \cos\theta_i$. While for p-polarized light, $\widetilde{n}_i$ should be replaced by $\widetilde{n}_i / \cos\theta_i$. $\phi_2 = \frac{2\pi \cdot n_2 \cdot d_2 \cdot \cos\theta_2'}{\lambda}$ and $\phi_1 = \frac{2\pi \cdot \widetilde{n}_1 \cdot d_1 \cdot \cos\theta_2}{\lambda}$ are individual phase differences when light passes through SiO_2 layer and graphene layer. $\theta_2'(\theta_2)$ is the refraction angle in SiO_2 (graphene) layer. $n_2(\widetilde{n}_1)$ is the refractive index of SiO_2 (graphene) layer. d_2 (d_1) is the thickness of the SiO_2 (graphene) layer. λ is wavelength. The reflection spectrum from air/(SiO_2 on Si), $R_0(\lambda)$ can be calculated by

$$R_0(\lambda) = |r_0|^2. \tag{3}$$

The reflection spectrum from air/graphene/SiO_2/Si system, $R_1(\lambda)$ can be calculated by

$$R_1(\lambda) = |r_1|^2. \tag{4}$$

For unpolarized light, one can take an average of the contribution from both s-wave and p-wave. Furthermore, including effect of numerical aperture (NA) of the microscope objective in the calculation of contrast, the reflection spectrum $R_{0,1}(\lambda)$ should be modified by numerical integration of the reflectance values (computed for various angles of incidence) over the solid angle determined by each NA

$$R_{0,1} = 2\pi \int_0^{\theta_m} R_{0,1}(\theta) W(\theta) \sin\theta d\theta \tag{5}$$

where $\theta_m = \arcsin(NA)$ and $W(\theta)$ is a weight function that indicates the angular distribution of the collected light [38,39], e.g., uniform distribution $W(\theta) = \text{const}$, Gaussian distribution $W(\theta) = e^{-\frac{2\sin^2\theta}{\sin^2\theta_m}}$ and so on.

The interference effect is strongly dependent on the wavelength of incident light. Therefore, the intensity of reflection light is also wavelength dependent, as does the optical contrast. This can be clearly shown in the reflection spectra in Fig. 1(c). The reflection spectrum from graphene R_1 is weaker than that from pure SiO_2/Si substrate R_0 in the wavelength range of 500–650 nm, corresponding to the wavelength where interference effect is strongest on 285 nm/Si substrate. The optical contrast spectrum between sample and substrate can be then be derived by following equations: $C(\lambda) = (R_0(\lambda) - R_1(\lambda))/R_0(\lambda)$. Here, R_0–R_1 is used because most of the 2D materials behavior darker as compared to the substrate on multilayer substrate, and this will make the contrast value positive. The contrast spectrum of single layer graphene on 285 nm SiO_2/Si is shown in Fig. 1(d) [24], which has a peak centered at ~560 nm, and has a maximum value of ~0.1 (10%). This contrast peak is the reason why single layer graphene is visible and can be experimental exfoliated and located. There are two origins of such a high contrast of graphene on this specific substrate. Firstly, graphene has a universal optical absorption in visible range, and it can absorb ~2.3% of the incident light even for a monolayer layer. Secondly but importantly, the interference effect from the SiO_2/Si multilayer structure would strongly enhance the absorption of light. On the other hand, the optical contrast of single layer graphene on glass is much weaker (<5%) and can barely be seen, because there are no interference effect and the contrast comes purely from the absorption of graphene [40]. If a single layer graphene is deposited on Si or GaAs substrate [41], the optical contrast is even weaker, and it is not possible to be located by a common microscope or human eye. Methods for improving the optical contrast of graphene on insulating or semiconducting substrates will be discussed in the later section.

3. Optical contrast for identifying the thicknesses of 2D materials

It has been demonstrated above that the optical contrast value of 2D materials is related to substrate, incident wavelength, and most importantly, the thickness of two dimensional materials. Following, we will introduce the identification of thickness of different types of 2D materials by contrast spectroscopy, e.g. graphene, GO, TMDs, BP and BN.

3.1. Graphene

Fig. 2(a) shows the optical images as well as the corresponding contrast spectra of graphene layers with different thicknesses on 285 nm SiO_2/Si substrate [24]. It can be clearly seen that the intensity of contrast peak at ~550 nm increases gradually with the increase of thickness up to 10 layers. The highest contrast values are ~0.09, 0.175, 0.255, and 0.33 for 1–4 layers, respectively. The contrast value decreases for graphene layers >10 and finally negative contrast appears for samples

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