Thickness Scaling Effects on the Complex Optical Conductivity of Few-Layer WSe₂ Investigated by Spectroscopic Ellipsometry

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2D WSe₂ have attracted widespread attentions as an ideal platform for new type optoelectronic and nanoelectronics applications. Understanding the intrinsic thickness scaling effects in complex optical conductivity of 2D, WSe₂ is vital for WSe₂-based photonic and optoelectronic devices. Herein, complex optical conductivities of 1–5 layers in the energy range of 0.73–6.42 eV by spectroscopic ellipsometry with the classical slab model have been determined. Up to eight feature peaks (A–H) are observed in the optical conductivity spectra, and the central energies of these peaks are identified by the differential spectrum analysis method. These central energies exhibit interesting layer dependencies due to the thickness scaling effects. Specifically, for the low-energy feature peaks A–E, their central energies decrease with the thickness increasing. But for the high-energy feature peaks G and H, their central energies first show significant blueshifts and then redshifts as the thickness increases. These novel layer-dependent evolutions are attributed in the optical conductivity of few-layer WSe₂ to the competition mechanism between the decreasing exciton binding energy and the band gap narrowing.

1. Introduction

Up to now, 2D layered materials, such as graphene, transition metal dichalcogenides (TMDCs), black phosphorus, Van der Waals heterostructures, etc., have attracted great interest in diverse electronic and optoelectronic fields. As an important part of 2D materials, 2D TMDCs are widely used in various optoelectronic devices, including the field effect transistors, solar cells, biosensors, absorbers, and photodetectors, which benefit from the flexible tunable bandgap, moderate mobility and on/off ratio, and good thermostability.

Understanding the optical/electrical properties of TMDCs is vital to improve the functionality of TMDCs-based optoelectronic devices. Similar to the bandgap (E_g) of 2D TMDCs varies with thickness, the optical/electrical properties of 2D TMDCs also exhibit striking thickness scaling effects, which poses some challenges to the development of corresponding optoelectronic devices. Therefore, the accurate and quantitative characterization of the thickness scaling effects on the optical/electronic parameters of 2D TMDCs is essential for accurate modeling and design of the TMDCs-containing optoelectronic devices.

In 2005, Novoselov et al., first obtained the monolayer MoS₂ via mechanical exfoliation. In the next ten years, different 2D TMDCs, such as MoSe₂, MoTe₂, WS₂, WSe₂, etc., have been discovered by different preparation methods. Tungsten Selenide (WSe₂), as a representative 2D TMDCs, is attracting growing attentions due to its remarkable properties such as tunable bandgaps, large spin-orbit coupling (≈513 meV), large exciton binding energy, and so on. The optical/electrical properties of 2D WSe₂ have been extensively studied by multiple techniques, such as the microreflectance (absorption) spectrum method, differential reflection spectroscopy method, deterministic method based on reflection coefficients, spectroscopic ellipsometry (SE), and first-principles calculations. Li et al., identified the complex dielectric functions of four kinds of monolayer TMDCs (MoS₂, MoSe₂, WS₂, and WSe₂) from their absolute reflectance spectra over the photon energies of 1.5–3 eV by the Kramers–Kronig constrained variational analysis. Niu et al., determined the complex refractive index spectra of 1–3 layer (L) mechanically exfoliated MoS₂, MoSe₂, WS₂, and WSe₂ samples in the range of 400–850 nm by combining microreflectance spectroscopy with the Fresnel equations. Niu et al., presented a systematic research on the differential reflective spectra of 1–6 L TMDCs (MoS₂, MoSe₂, WS₂, and WSe₂), indicating that the thickness dominates the exciton energies of the prominent features in differential reflective spectra. Jung et al. proposed a deterministic method, which can give a unique and accurate solution to the permittivity of monolayers MoS₂, WS₂, and WSe₂ by using the ratio of reflection coefficients for s and p polarization. With the help of SE, Eichfeld et al. determined the thickness of ultra-thin (<30 nm) WSe₂ by comparing the layer-dependent complex refractive index and found the optical
2. Results and Discussion

Atomic force microscopy (AFM) images illustrated in Figure 1a–e suggest that the thicknesses of 1–5 L WSe₂ films are 0.67, 1.28, 1.86, 2.37, and 2.98 nm, respectively. The surface roughness over 1 × 1 μm² area of the WSe₂ specimens is less than 1 nm, indicating that the sample surfaces are relatively flat. One of the important factors contributing to the surface roughness should be the fine wrinkles shown in Figure 1a–e, which can be interpreted as the different thermal coefficient between WSe₂ and Au. To check the elemental compositions and proportions of the 2D WSe₂ films, the X-ray photoelectron spectra (XPS) of 1–5 L WSe₂ films (Figure 1f–o) were measured. Central energies of the XPS peaks are consistent with the theoretical binding energies of the corresponding orbital electrons of W and Se elements.

Additionally, W 4f½/2, W 4f½/2, W 5p½/2, Se 3d½/2, and Se 3d½/2 XPS peaks (Figure 1f–j) can be deconvoluted with only one Voigt function, which suggests that there is only one tungsten-(selenium-) containing chemical species in the 2D films prepared by us. We evaluate the mole ratio of Se and W atoms by calculating the area ratio of Se 3d peak and W 4f peak, and find that the mole ratio approximately equals to the theoretical value of 2. It means that the films we synthesized are indeed WSe₂. Besides, the Raman spectra of 1–5 L WSe₂ films were measured to further confirm the crystal quality of the 2D WSe₂ films. As shown in Figure 1p–t, three characteristic Raman modes E(K), A₁g + E₂g, and 2LAM(M) were identified in the Raman spectra, and the central energies of these peaks were agreed well with those of the mechanical exfoliated 2D WSe₂. The above characterization results imply that our 2D WSe₂ films are high quality and suitable for the next spectral measurement.

2D WSe₂ films with variable thickness were investigated by a dual rotating-compensator multi-angle spectroscopic ellipsometer (ME-L Muller matrix ellipsrometer; Wuhan Eoptics Technology Co., Wuhan, China). The basic principles of SE measurement are illustrated in Figure 2a.[43,44] The ellipsometer in our experiment consists of a light source, a polarization state generator (PSG) arm, a sample stage, a polarization state analyzer (PSA) arm, and a detector, as shown in Figure 2b. SE is a fast, non-contact and low-cost measurement technique, and it is widely used to study the inherent optical properties of nanomaterials and nanostructures by detecting and analyzing the change in the polarization state of the polarized light after reacting with materials.[45,46] The polarization state change is usually described by a pair of ellipsometric angles (Ψ, Δ) in the SE, which can be defined as:

$$\tan(\Psi) \cdot \exp(i\Delta) = r_p/r_s = (E_{ip}/E_{ip})/(E_{is}/E_{is})$$  \hspace{1cm} (1)

Where $r_p$ and $r_s$ are the reflection coefficients for the $p$ and $s$ components of the polarized light, respectively. $E_{ip}$ ($E_{is}$) and $E_{ip}$ ($E_{is}$) represent the electric field of the incident and reflected $p$-polarized ($s$-polarized) light, respectively. The ellipsometric spectra of 2D WSe₂ films are obtained over 0.73–6.42 eV by multi-incidence measurement mode (the incidence angles are 60°, 65°, and 70°). In ellipsometric measurements, the spatial inhomogeneities of samples can not only generate depolarization and interference effects in the ellipsometric spectra, but also affect the measured refractive index, extinction coefficient, and thickness in the measurements.[47–50] In order to reduce the effects of
spatial inhomogeneities, micro focusing mode is applied to maximally reduce the effects of surface fluctuations by using a pair of focusing probes, and the diameter of the probing spot is as small as 200 μm. Besides, multiple test points are measured for each sample, and the most reliable data with the highest repetition rate among them are selected for the following ellipsometric analysis. In order to determine the optical properties of 2D WSe₂ films, two models should be constructed to perform the ellipsometric analysis process. One is an optical stacking model to describe the sample, and the other is the optical function model to describe the dispersive properties of the material over the concerned energy range. Figure 2c presents the optical model of 2D WSe₂ on the sapphire substrate, which is a vertical stacking multilayer structure, including the ambient air, the 2D WSe₂, the Bruggeman Effective Medium Approximation (EMA) layer, and the sapphire substrate. Here, the Bruggeman EMA layer is a hybrid layer containing 50% sapphire and 50% WSe₂, which can effectively embody the transition interface between two materials. Besides, the optical function of 2D WSe₂ films over the concerned energy range is parameterized by two Cody-Lorentz oscillators and five Lorentz oscillators. With the constructed optical model and the optical function, the theoretical ellipsometric spectra of 2D WSe₂ films can be obtained by the transfer matrix method. Figure 2d,e illustrates the measured (open circles) and best-fitting (solid lines) ellipsometric spectra of the 4 L WSe₂ films, and Table 1 presents the Mean Squared Error (MSE) values and the sample thicknesses acquired in the SE fitting processes. The extremely low MSE values (less than 3) illustrate that the theoretical ellipsometric spectra agree well with the experimental ellipsometric spectra, indicating that the optical model and optical function established by us are reasonable. Besides, Table 1 also shows that the thicknesses acquired in the SE fitting processes are consistent with the AFM results.

Figure 3a–b shows the complex refractive indices $N = n - ik$ of 1–5 L WSe₂ films ($n$ and $k$ are the refractive index and the extinction coefficient). It is obvious that eight absorption peaks (A–H) are existed in the complex refractive index spectra of the 2D WSe₂. Compared with the high-energy absorption peaks E–H,
the low-energy absorption peaks A–D exhibit sharper features and smaller full width at half maximum (FWHM). The underlying physical mechanisms of the phenomenon can be attributed to the different transitions occurring at these absorption peaks. The transitions associated with the formations of the low-energy absorption peaks A–D should be simple and clear, due to that the complex transitions that require high excitation energy have been effectively shielded. On the contrary, the transitions occurring at these high-energy absorption peaks may be multiplicate and intricate, and may include the electron transition, the excitonic transition, and even the defect absorption. Besides, the complex optical conductivities of 1–5 L WSe$_2$ can be calculated with the help of classical slab model\cite{32}:

$$\sigma = \sigma_r + i\sigma_i = id\varepsilon_0 \varepsilon = id\varepsilon_0 N^d$$

(2)

where \(d\) is the thicknesses of the 2D WSe$_2$ determined by SE (Table 1), and \(\varepsilon_0\) refers to the free-space permittivity. Figure 3c–d shows the complex optical conductivity spectra of 1–5 L WSe$_2$. Similar to the above complex refractive indices, eight discernible feature peaks can be also observed in the complex optical conductivity spectra. These feature peaks A–H are relevant to different direct transitions from four highest valance bands to four lowest conduction bands along the \(\Gamma-K-M\) branch in the band structure of 2D WSe$_2$, as detailed discussed in previous works.\cite{23,27,29} It is obvious that real part of optical conductivity (\(\sigma_r\)) increases with the increasing layer number of 2D WSe$_2$, and imaginary part of optical conductivity (\(\sigma_i\)) also exhibits a monotonous increase when the energy is less than 4 eV. The layer-dependent increase of the complex optical conductivity of 2D WSe$_2$ can be explained as the gradually enhanced light absorption and propagation effects, which are expected to be sensitive to the thickness of materials. It is worth noting that \(\sigma_i\) is negative value when the energy is greater than 4 eV (3 eV for the monolayer WSe$_2$). These negative \(\sigma_i\) phenomena are directly relevant to the plasma resonance, and it can be interpreted as the full-filled of the four unoccupied \(d\) bands, which are divided by a tiny gap or a density of states minimum from the \(s\) and \(p\) orbit dominated conduction bands at higher energies.\cite{53} The zero-cross point of \(\sigma_i\) shows a blue shift when the layer number increases from monolayer to bilayer. While the thickness increases from 2 to 5 L, the \(E_{zc}\) shows a slightly red shift. The negative \(\sigma_i\) phenomena are directly relevant to the plasma resonance, and the peaks of energy loss functions show the intrinsic plasmonic excitation. Based on the energy loss function in Ref.\cite{54} we infer that the blue shift of \(E_{zc}\) from monolayer to bilayer may be caused by the obviously enhanced and broaden energy loss peak at 3.5 eV due to the enhanced phonon scattering, and the slightly red shift of \(E_{zc}\) from 2 L to 5 L could be attributed to the gradually red-shift energy loss peak at 3.5 eV.

As shown in Figure 3c–d, apart from the intensity of the feature peaks in the optical conductivity spectra exhibits obvious layer dependencies, the central energies of these peaks also present attractive layer-dependent shifts. Thus, in the following parts,

![Figure 2. Spectroscopic ellipsometry measurement. a) Basic principles of spectroscopic ellipsometry; b) a photograph of the experimental setup; c) optical model of 2D WSe$_2$ on sapphire substrate; d,e) Ellipsometric analysis results for 4 L WSe$_2$.](image)
we will systematically discuss the shifts and attempt to explore their underlying physical origin. First, the first-order and second-order differential spectra of the complex optical conductivities of 1–5 L WSe2 are calculated and plotted in Figure 4 and 5. The central energies of the feature peaks in \( \sigma \) of 2D WSe2 can be identified by analysing the zero-cross points in the first-order differential spectra and the local minimums in the second-order differential spectra. As shown in Figure 4a–e, the red (blue) solid lines represent the first-order differential spectra of the \( \sigma_r (\sigma_i) \) of the 1–5 L WSe2. The central energies of the extreme peaks A–F in the optical conductivity spectra correspond to the positive-to-negative zero-cross points (Figure 4a–e) with the increasing energy. Further, the zero-cross points marked by grey dots are equal to the central energies of the extreme peaks in the \( \sigma_r (\sigma_i) \) of the 1–5 L WSe2. It is worth noting that the central energies of feature peak B in \( \sigma_r \) of 3–5 L WSe2, peak E in \( \sigma_i \) of 4 L WSe2, and peaks G, H cannot be distinguished in the first-order differential spectra according to the above analysis method. This is because these peaks are shoulder peaks, whose central energies can be determined by calculating the second-order differential spectra of \( \sigma \). As shown in Figure 4f–j and 5, the central energies of these shoulder peaks are equal to the local minimums of the second-order differential spectra. The central energies of the feature peaks A–H in the optical conductivity spectra of 1–5 L WSe2 have been listed in Table 2.

Figure 6a–b show the evolutions of the central energies of the feature peaks A–H in the optical conductivity spectra. For brevity, we use \( E_{0}^{re} \) and \( E_{0}^{im} \) to denote the central energies of the feature peak in the real (imaginary) part of the complex optical conductivity. As demonstrated in Figure 6a–b, with the increase of the layer number of 2D WSe2, the changing patterns of \( E_{0}^{re} \) and \( E_{0}^{im} \) can be roughly divided into two categories. For peaks A–E, \( E_{0}^{re} \) (A–E) and \( E_{0}^{im} \) (A–E) exhibit monotonous redshifts when L increases from 1 to 5. While, for the high-energy feature peaks G and H, \( E_{0}^{re} \) (G–H) and \( E_{0}^{im} \) (G–H) first exhibit a layer-dependent increase and then turn to decrease when \( L > 2 \). For a specific feature peak in optical conductivity spectra, \( E_{0}^{re} \) and \( E_{0}^{im} \) nearly have the same evolution patterns except for peak F. Specifically, the evolvement of \( E_{0}^{re} \) (F) is similar to those of \( E_{0}^{re} \) (G–H), but for \( E_{0}^{im} \) (F), \( E_{0}^{im} \) (F) gradually decreases with the increasing I like the layer-dependent evolutions of \( E_{0}^{im} \) (A–E).

The physical origin of above novel layer-dependent evolutions of \( E_{0}^{re} \) and \( E_{0}^{im} \) can be interpreted as the competition between the decreasing exciton binding energy \( E_{b} \) and the band gap narrowing (namely the decreasing energy gap \( (E_{g}) \), as shown in Figure 6c–e. These two factors can modulate the central energy of feature peaks A–H by changing the exciton transition energy \( E_{excitation} \), which can represent the central energy of peaks in \( \sigma \) largely. In 2D transitional metal dichalcogenides (TMDCs), the increasing layer number will decrease \( E_{b} \) and \( E_{g} \) simultaneously due to the gradually enhanced out-of-plane (direction perpendicular to the surface of 2D TMDCs) dielectric shielding effect.[12] As shown in Figure 6d, the decreasing \( E_{b} \) can enlarge \( E_{excitation} \). On the contrary, the decrease of \( E_{b} \) will reduce \( E_{excitation} \), as presented in Figure 6e. Thus, the competition between \( E_{b} \) and \( E_{g} \) is formed. On the basis of the above analysis, we can conclude that band gap narrowing dominates the evolutions of \( E_{0}^{re} \) (A–E) and \( E_{0}^{im} \) (A–F), which is also consistent with the previous reports that the bandgap of WSe2 also decreases as the WSe2 undergoes a transition from 2D to 3D.[42] For \( E_{0}^{re} \) (F–H) and \( E_{0}^{im} \) (G–H), the decreasing \( E_{b} \) plays an important role in the increase of the

Figure 3. Optical spectra of 2D WSe2. a–b) Complex refractive indices spectra of 1–5 L WSe2; c–d) Complex optical conductivity spectra of 1–5 L WSe2.
central energies when \( L \) is less than 3. The excitonic effects at high-energy peaks F–H may be more sensitive to thickness than the low-energy peaks A–E. When the thickness of 2D WSe\(_2\) increases, the dielectric shielding effect gradually increases, the decrease in exciton binding energy \( E_{b} \) of high-energy peaks F–H is strongly greater than the decrease in energy gap (\( E_{g} \)), and causes a blue shift trend in the high-energy peaks F–H. When \( L \) is large than 3, the band gap narrowing dominates the \( E_{g}^{0} \) (F–H) and \( E_{g}^{0} \) (G–H) again. As for the physical origin of different

**Figure 4.** Differential spectra of the complex optical conductivities of 2D WSe\(_2\). a–e) First-order differential spectra over the energy region of 1.5–4.5 eV; f–j) Second-order differential spectra over the energy region of 4.5–6.4 eV.

**Figure 5.** Second-order differential spectra of the complex optical conductivities of 3–5 L WSe\(_2\) over the energy region of 1.8–3.5 eV.

**Table 2.** Center energies of the feature peaks in \( \sigma(E) \) of 1–5 L WSe\(_2\).

| \( L \) | Central Energy | A | B | C | D | E | F | G | H |
|---|---|---|---|---|---|---|---|---|---|
| 1 | \( E_{g}^{0} \) [eV] | 1.67 | 2.12 | 2.46 | 2.89 | 3.38 | 4.09 | 4.71 | 5.77 |
|   | \( E_{g}^{0} \) [eV] | 1.65 | 2.03 | 2.32 | 2.72 | 3.27 | 3.86 | 4.54 | 5.49 |
| 2 | \( E_{g}^{0} \) [eV] | 1.65 | 2.10 | 2.42 | 2.86 | 3.35 | 4.17 | 4.83 | 5.99 |
|   | \( E_{g}^{0} \) [eV] | 1.62 | 2.03 | 2.29 | 2.70 | 3.23 | 3.85 | 4.66 | 5.59 |
| 3 | \( E_{g}^{0} \) [eV] | 1.65 | 2.07 | 2.41 | 2.84 | 3.30 | 4.20 | 4.83 | 5.93 |
|   | \( E_{g}^{0} \) [eV] | 1.60 | 2.03 | 2.27 | 2.67 | 3.16 | 3.83 | 4.69 | 5.49 |
| 4 | \( E_{g}^{0} \) [eV] | 1.64 | 2.06 | 2.40 | 2.82 | 3.21 | 4.16 | 4.76 | 5.91 |
|   | \( E_{g}^{0} \) [eV] | 1.60 | 2.02 | 2.24 | 2.63 | 3.16 | 3.83 | 4.61 | 5.46 |
| 5 | \( E_{g}^{0} \) [eV] | 1.64 | 2.05 | 2.40 | 2.81 | 3.19 | 4.15 | 4.76 | 5.88 |
|   | \( E_{g}^{0} \) [eV] | 1.60 | 2.01 | 2.23 | 2.63 | 3.05 | 3.83 | 4.62 | 5.44 |
layer-dependent evolvements appearing on $E_{Re}^0$ (F) and $E_{Im}^0$ (F), it remains further investigation.

3. Conclusions

In summary, we obtained the complex optical conductivities of 1–5 L WSe$_2$ by using the SE and classical slab model over the energy region of 0.73–6.42 eV. The intensity of the complex optical conductivity of 2D WSe$_2$ gradually increased with the increasing L, which was caused by the enhanced absorption and propagation effects. There existed negative $\sigma$, in the complex optical conductivity of 2D WSe$_2$, which was interpreted as the full-filled of the four unoccupied $d$ bands. Up to eight peaks (A–H) were observed in the complex optical conductivity spectra, and the central energies of feature peaks A–H were specifically determined by the differential spectrum analysis method. We plotted the curves of the central energies of peaks in the optical conductivities versus the layer number of 2D WSe$_2$, and found that these feature peaks follow two kinds of layer-dependent evolution patterns. With the increase of L, the central energies of low-energy feature peaks A–E change toward the low energy direction. While their central energies of high-energy peaks G and H first exhibit a layer-dependent increase and then turn to decrease when $L > 2$. We explained these novel thickness scaling effects as the combined influence from the decreasing exciton binding energy and the band gap narrowing. The thickness scaling effects on the layer-dependent complex optical conductivities of 2D WSe$_2$ revealed

![Figure 6. Layer-dependent evolvement of center energies of feature peaks and the physical origin of this evolution. a–b) Layer-dependent center energies of feature peaks (A–H) in the complex optical conductivities of 2D WSe$_2$, where the vertical coordinates $E_{Re}^0$ and $E_{Im}^0$ refer to the center energies of feature peaks in the real (imaginary) part of the optical conductivity; c) Default local electronic band structure of 2D WSe$_2$ with ground state exciton level (blue dash line) between the CB and the VB; d) The decreasing $E_b$ dominates $E_{Exciton}$; e) The band gap narrowing dominates $E_{Exciton}$]
in this work are expected to promote the physically understanding on the fundamental optical/electronic structure of 2D TMDCs, and it is also informative for the optimal design and performance improvement of corresponding devices based on the 2D WSe₂.

4. Experimental Section

Growth of 2D WSe₂ Films: High-quality, large area 2D WSe₂ films with different layers are fabricated by an ultrafast ambient-pressure CVD growth process. First, the large-area monolayer WSe₂ is synthesized onto the polycrystalline Au foil (99.95 wt%, 100 μm thick, 1 × 1 cm² size, Alfa Aesar), which is finely polished and annealed at 1040 °C to reduce its surface roughness. The growth rate of the monolayer WSe₂ on the Au foil is about 26 μm s⁻¹, which is ≈3 orders of magnitude faster than those of most 2D TMDCs grown on non-metal substrates. This efficient preparation process allows us to prepare a large area, high-quality, continuous, monolayer WSe₂ film in just a few minutes. Then, with a non-destructive electrochemical bubbling method, the monolayer WSe₂ is transferred onto a single-side polished sapphire substrate (1 mm thick, 1 × 1 cm² size), whose optical responses over the entire concerned energy region are moderate. Further, the absorption of sapphire is extremely close to zero, and its refractive index is monotonic dispersive and away from that of 2D WSe₂, which facilitate the extraction of the optical parameters of 2D WSe₂. By performing the above transfer operation repeatedly, 2D WSe₂ with different layers (1–5 L) can be fabricated.

Characterization of 2D WSe₂ Films: The X-ray photoelectron spectra (XPS) of our 2D WSe₂ films was obtained by the Escalab 250Xi X-ray photoelectron spectrometer to analyze the elemental composition. Raman spectrometer (LabRAM HR800, Horiba Jobin Yvon) was used to obtain the Raman spectra of our 2D WSe₂ films. The laser wavelength was 532 nm, and the spot size was about 1 μm. AFM (Bruker Dimension ICON) was applied to test the surface roughness and thicknesses of our WSe₂ films in the range of 5 μm × 5 μm and 1 μm × 1 μm. Optical measurements were performed by a dual-compensator compensator multi-angle spectroscopic ellipsometer (ME-L Muller matrix ellipsometer, Wuhan Eoptics Technology Co., Wuhan, China). The measured optical spectra cover the energy range of 0.73–6.43 eV, and the 60°, 65°, 70° incident angles were set for each sample.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

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