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Real-space light-reflection mapping of atomically thin WSe₂ flakes revealing the gradient local strain

Yang Guo^{1,2,4} , Yuan Huang^{1,4} , Shuo Du^{1,2} , Chi Sun^{1,2} , Shibing Tian¹, Hailan Luo¹, Baoli Liu^{1,2,3}, Xingjiang Zhou¹ , Junjie Li^{1,2,3} and Changzhi Gu^{1,2}

¹ Beijing National Laboratory for Condensed Matter Physics, Institute of Physics, Chinese Academy of Sciences, Beijing 100190, People's Republic of China

² School of Physical Sciences, CAS Key Laboratory of Vacuum Physics, University of Chinese Academy of Sciences, Beijing 100190, People's Republic of China

³ Songshan Lake Materials Laboratory, Dongguan, Guangdong 523808, People's Republic of China

⁴ These authors contributed equally to this work.

E-mail: czgu@iphy.ac.cn

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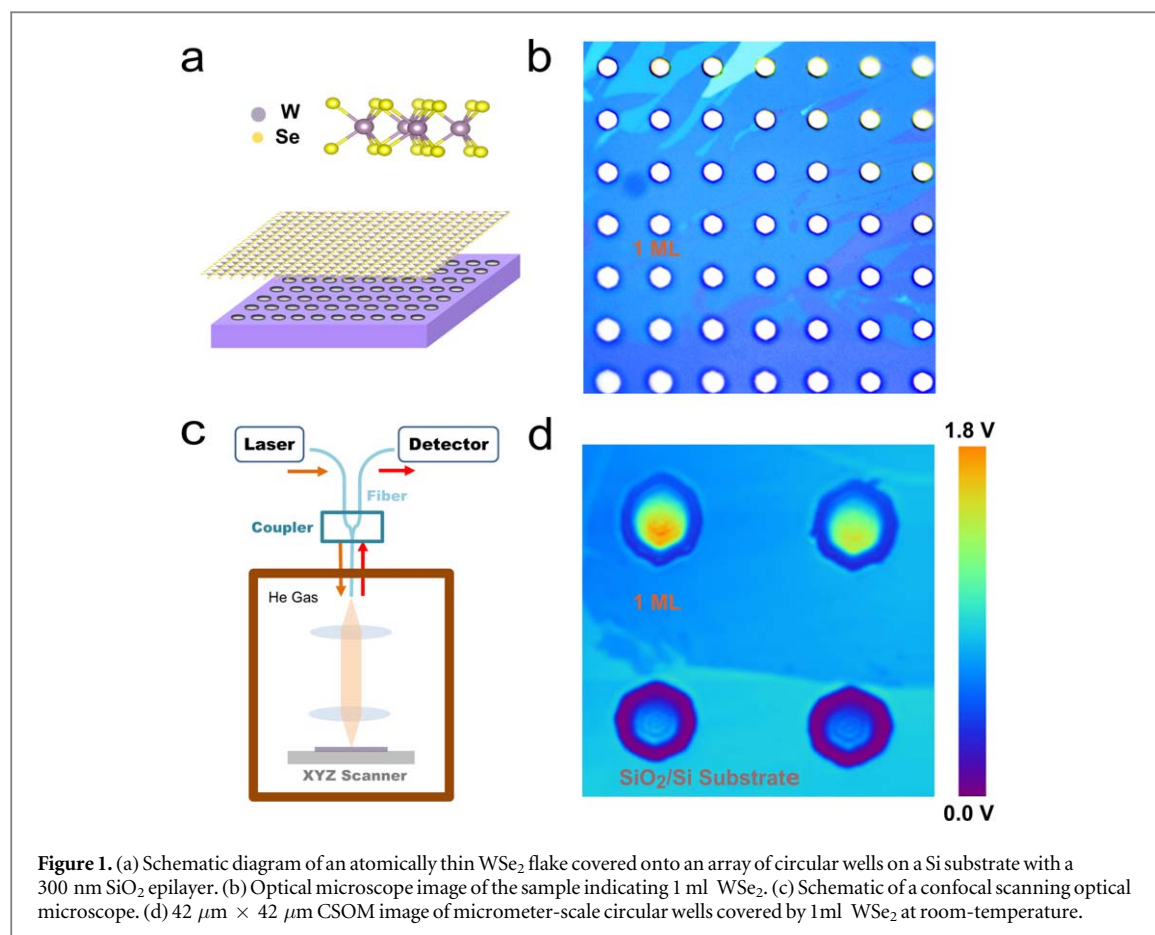
Abstract

The spatially continuous control of the physical properties in semiconductor materials is an important strategy in increasing electron-capturing or light-harvesting efficiencies, which is highly desirable for the application of optoelectronic devices including photodetectors, solar cells and biosensors. Unlike the multi-layer growth of chemical composition modulation, local strain offers a convenient way to continuously tune the physical properties of a single semiconductor layer, and open up new possibility for band engineering within the 2D plane. Here, we demonstrate that the gradient refractive index and bandgap can be generated in atomically thin transition metal dichalcogenide flakes due to the effect of thermal strain difference. A highly resolved confocal scanning optical microscopy is used to perform a real-space light-reflection mapping of suspended atomically thin WSe₂ flakes at the low temperature of 4.2 K, in which the parabolic light-reflection profiles have been observed on suspended monolayer and bilayer WSe₂ flakes. This finding is corroborated by our theoretical model which includes the effect of strain on both the refractive index and bandgap of nanostructures. The inhomogeneous local strain observed here will allow new device functionalities to be integrated within 2D layered materials, such as in-plane photodetectors and photovoltaic devices.

1. Introduction

In 1957, Kroemer proposed the concept of a compositionally graded semiconductor that became one of the earliest and simplest examples of band-gap engineering [1]. By spatially varying the stoichiometry of a semiconductor, an energy band gap is produced that varies with position. After that, this concept has been widely applied to many optoelectronic semiconductor devices including photovoltaic devices and solar cells by epitaxial growth of specific graded gap structures [2–4]. But, the major drawbacks of this approach are the increased complexity of their fabrication and the high cost of materials. Consequently, there is a need to develop reliable techniques to fine-tune the bandgap within a single semiconducting material. In this context, the strain as a potential candidate for creating a continuously varying bandgap profile within the 2D plane has been theoretically proposed by Feng *et al* [5], since the discovery of 2D layered materials, such as graphene and monolayer transition metal dichalcogenides (TMDs) [6–8]. These materials are able to withstand large strains before rupturing, offering a unique opportunity to introduce large local strains [9] and considered as ultra-strength materials.

Recently, many studies have been reported on the strain effect of 2D layered materials [10–12]. This effect has been proved that the optical band gap can be tuned by up to several hundred meV/% in atomically thin

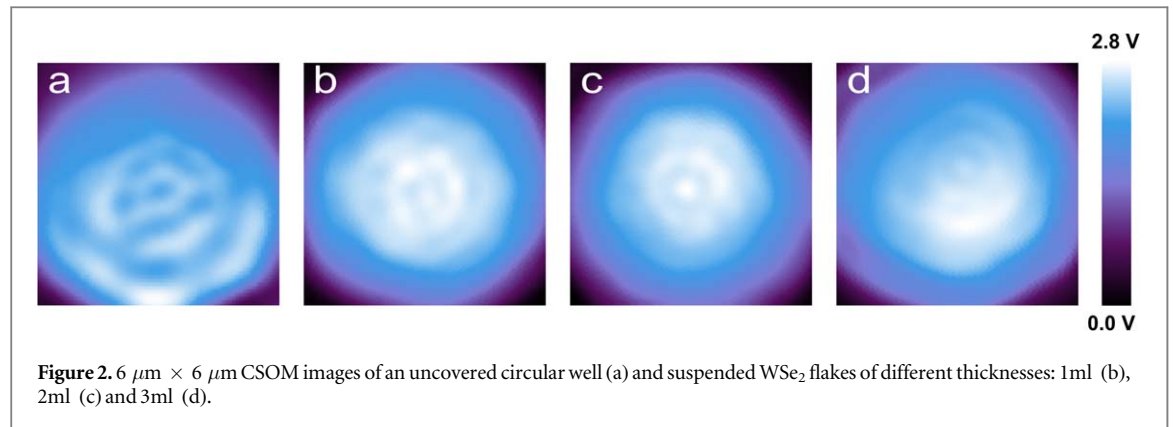


TMDs by local strain [13]. As shown in figure 1(a), monolayer (ML) TMDs have a sandwiched X-M-X structure, with the chalcogen atoms (X = S, Se, Te) in two trigonal planes separated by a plane of metal atoms (M = Mo, W). In contrast to graphene, ML TMDs usually display direct energy gaps in the near-infrared to visible spectral region, which make these materials suitable for optoelectronic device applications. In order to determine the local strain and bandgaps, diverse scanning optical spectroscopy techniques have been applied to investigate the local strain effect in TMD flakes via noncontact and nondestructive measurements, including optical absorption/transmission, photoluminescence (PL) and Raman scattering [14–16]. However, to date, there are few studies of direct observation on continuously varying strain distributions, which give rise to a gradient bandgap profile [17–19]. Here, we utilize confocal scanning optical microscopy (CSOM) to measure local strain profiles induced by the thermal expansion effect on suspended atomically thin WSe₂ flakes of different thickness at the low temperature of 4.2 K. In a CSOM, the use of specific wavelengths of a laser and the use of a pinhole (fiber) to eliminate or reassign out-of-focus light, has significantly increased our ability to resolve and co-localize small structures in high contrast images. With its unique capabilities, we are able to image local thermal strain on a suspended monolayer WSe₂ by detecting the reflected light signals from these flakes.

2. Results and discussion

For this study, an array of circular wells (diameter 10 μm and depth 4 μm) with a spatial period of 30 μm × 30 μm was patterned onto a Si substrate with a 300 nm SiO₂ epilayer by nanoimprint lithography and reactive ion etching, and atomically thin WSe₂ flakes were obtained by mechanical exfoliation from a bulk WSe₂ crystal and translated to the substrate. Optical microscopy was used to examine suspended WSe₂ flakes. Most of the circular wells on substrate were covered by the monolayer WSe₂ flakes to form a series of free-standing membranes as shown in figure 1(b). The thickness of atomically thin WSe₂ flakes can be identified by color or optical contrast [20, 21].

Before cooling down to 4.2 K, we first examine the optical response of suspended WSe₂ flakes in a wide scanning range by using a commercial Attocube confocal microscope as schematically illustrated in figure 1(c). A laser beam with $\lambda = 630$ nm is coupled into one arm of a single mode optical fiber coupler (50:50). The fiber end of the second arm is placed in a ceramic ferrule to guarantee an accurate position with respect to the objective and to minimize optical aberrations. This single mode fiber of 0.13 NA and 3.5 μm core diameter



(Thorlabs 630HP) illuminates the sample and is used as the blocking pinhole aperture when collecting the scattered light from the sample. The sample is mounted on a slip-stick XYZ piezoelectric translation stage with 5 mm range in each direction. The objective lens is a $\times 20$ microscope objective of 0.87 NA and 2.5 mm working distance with good achromaticity throughout the visible and near infrared and is used in infinite conjugate. The best horizontal resolution of this CSOM is about 360 nm with the continuous wave laser of 630 nm.

Figure 1(d) presents a typical $42\ \mu\text{m} \times 42\ \mu\text{m}$ CSOM image of our sample at room-temperature. The reflected light from the sample is measured by using an internal Si-diode detector, and it is transferred to a relative voltage signal. There are two uncovered circular wells (Bottom) and two circular wells covered by 1 ml WSe₂ (Top) in this figure. Each of the covered circular wells shows a closely circular pattern of $10\ \mu\text{m}$ in diameter, in which the center is a bright round region of $\sim 6\ \mu\text{m}$ in diameter with a series of bright and dark rings, and the periphery is a dark ring with a width of $\sim 2\ \mu\text{m}$. The uncovered wells exhibit the similar patterns, which indicates that such type of patterns comes from the topography inside circular wells, and the suspended 1 ml WSe₂ behaves as a uniform dielectric film for optical response at room temperature. As shown in figure S2 available online at stacks.iop.org/MRX/7/035904/mmedia, the reflected CSOM signals from the center of the covered circular wells can be used to identify the number of layers in our study, because they are thickness-dependent and well fitted by the model based on the Fresnel law similar to the one developed by Blake [20, 21].

Now we present the observations of CSOM on the suspended WSe₂ flakes at low temperature of 4.2 K and analyze their correlation with local strain induced by thermal expansion effect. Figures 2(a)–(d) presents a set of $6\ \mu\text{m} \times 6\ \mu\text{m}$ CSOM images of an uncovered circular well and suspended WSe₂ flakes of different thicknesses ranging from 1 ml to 3 ml. The image of an uncovered circular well (figure 2(a)) is similar to that at room temperature and shows the topography inside circular wells, but the images of covered circular wells become different and show a continuous change with the number of layers increasing. There appear a few bright spots in a regular arrangement in the central area of the 1 ml suspended WSe₂ (figure 2(b)), and these bright spots become connected (figure 2(c)) and form a circle (figure 2(d)), as the thickness of suspended WSe₂ flakes increases from 1 ml to 3 ml.

In order to clearly show the difference of covered circular wells of different thicknesses, the 3D view of real-space light-reflection mappings of suspended 1 ml (a), 2 ml (b) and 3 ml (c) WSe₂ flakes are displayed in figure 3. These images approximately show a rotary symmetry along the normal direction of surface, but exhibit different reflection profiles. Figure 3(d) compares the CSOM signal profiles along the horizontal centerlines of figures 2(a)–(d). It is obvious that the reflected CSOM signal of the uncovered circular well is almost constant along its cutline (the green curve in figure 3(d)), but ones of suspended 1 ml (the blue curve) and 2 ml (the red curve) WSe₂ flakes strongly depend on the radial coordinate r , while that of suspended 3 ml (the brown curve) WSe₂ flake is weakly dependent on r . Moreover, the CSOM profiles of suspended 1 ml and 2 ml WSe₂ flakes can be approximately fitted by a parabolic relation $R = a - br^2$, where R is the reflected CSOM signal and a and b are constants, as shown figure 3(e). Interestingly, this relation is similar to the previous observation of the deflection shape of a suspended membrane over a circular well, induced by a pressure difference across it, which gives rises to biaxial strain at the center of the suspended membrane [18]. In that case, the mechanism is described by the Hencky's model [22], and the biaxial strain produced at the center of the suspended membrane can be calculated with the formula $\varepsilon = \sigma(\nu)(\delta/a)^2$, where $\sigma(\nu)$ is a numerical constant that depends only on Poisson's ratio ν . δ and a are the deflection height and the radius of a circular well, respectively. However, the parabolic profiles of reflected CSOM signals observed in our measurements originate from the local thermal strain in suspended 1 ml and 2 ml WSe₂ flakes, but not geometric deflection induced by a mechanical load, because there is no external mechanical force applied on the suspended WSe₂ flakes in our experiments (Supporting Information).

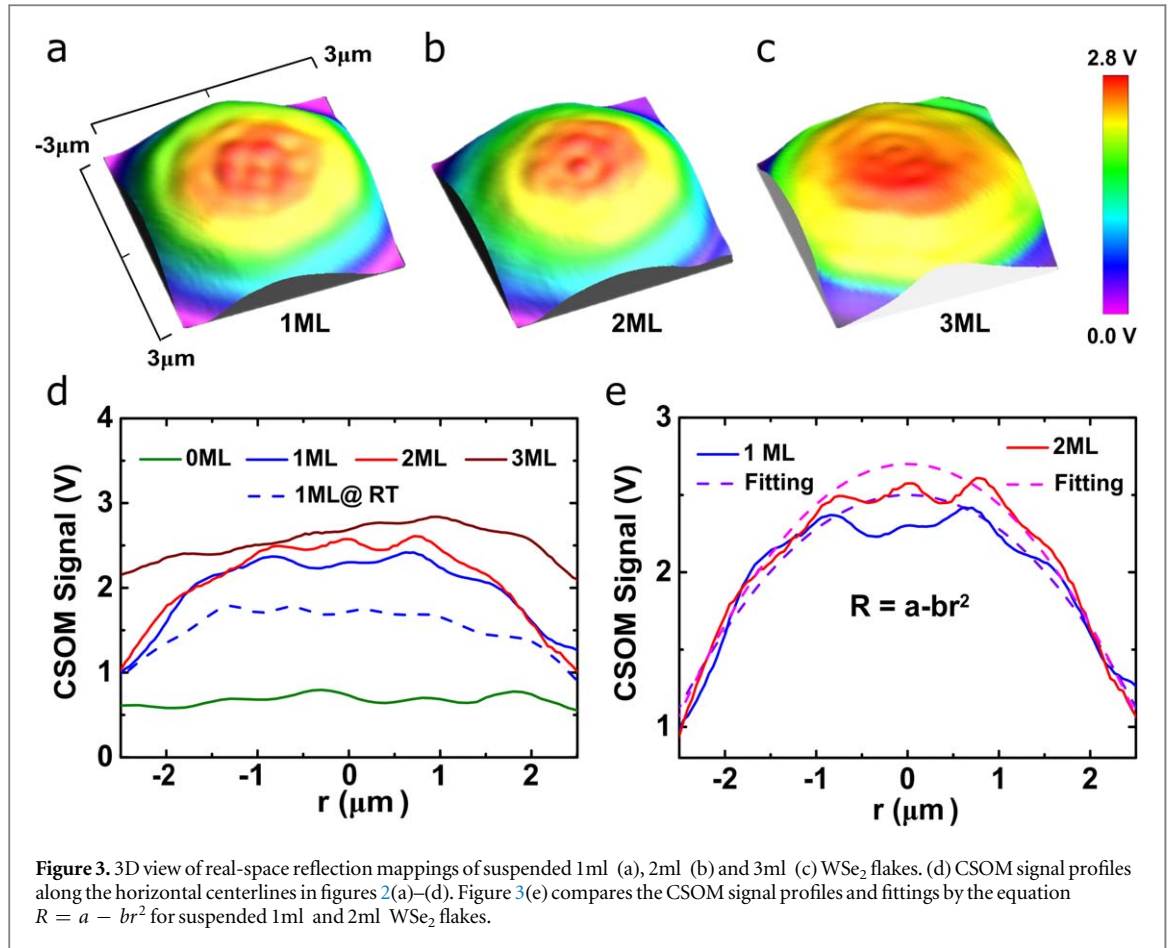


Figure 3. 3D view of real-space reflection mappings of suspended 1ml (a), 2ml (b) and 3ml (c) WSe₂ flakes. (d) CSOM signal profiles along the horizontal centerlines in figures 2(a)–(d). Figure 3(e) compares the CSOM signal profiles and fittings by the equation $R = a - br^2$ for suspended 1ml and 2ml WSe₂ flakes.

In order to verify the thermal strain in our samples, we performed PL measurements at the low temperature of 4.2 K. As shown in figure 4, the direct bandgap of the suspended 1 ml WSe₂ is increased by 45 meV compared to the supported 1 ml WSe₂ (the bandgap 1.65 eV), which proves the existence of compressive strain and the thermal strain difference between suspended and supported 1 ml WSe₂ flakes in our samples. Moreover, in addition to the broad-spectrum emission centered at the photon energy of 1.6 eV of the indirect transition, the second emission centered at the photon energy 1.68 eV corresponding to the direct transition appears in the suspended 2 ml WSe₂ flake, which has been attributed to be strain-induced indirect to direct bandgap transition in multilayer WSe₂ flakes in the previous study [23]. Finally, the PL spectrum of the suspended 3 ml WSe₂ mainly shows a broad-spectrum emission of the indirect transition. This result indicates that the strain effect on suspended WSe₂ flakes becomes weaker as the thickness increases, which is consistent with the observation of the dependence of CSOM signals on the radial coordinate shown in figure 3. Referring to the previous results regarding the strain-induced band gap in atomically thin WSe₂ flakes [23, 24], we have estimated that the average biaxial compressive strains of suspended 1 ml and 2 ml WSe₂ flakes are not less than 1.5% and 1%, respectively.

Next, we discuss how reflected CSOM signals can be used to reveal local strain in atomically thin TMD flakes. The reflected intensity from a multilayer film can be analyzed by the refractive index of each layer, where the relation between two physical properties is accurately described by the approach based on the Fresnel law similar to the one developed by Blake *et al* [20, 21]. By using the modified Moss formula developed by Herve and Vandamme [25], the refractive index of a semiconductor film is represented by its bandgap as

$$n = \sqrt{1 + \left(\frac{E_A}{E_g + E_B} \right)^2}, \quad (1)$$

where E_A is the hydrogen ionization energy ≈ 13.6 eV and E_B is 3.47 eV. This equation applies to the semiconductor films in the optical range of the infrared resonance frequency to the UV resonance frequency. Strain is able to modulate the energy band structures of atomically thin TMD flakes and therefore also influences the light-reflection determined by the refractive index. In order to understand the effect of strain on the bandgap of atomically thin TMD flakes, we introduce a model developed by Ghafouri-Shiraz and Tsuji, which has been successfully applied on In_xGa_(1-x)As laser diodes [26]. In this model, the energy shifts of the valence band E_v , and the conduction band E_c , due to the hydrostatic component of the strain, can be expressed, respectively,

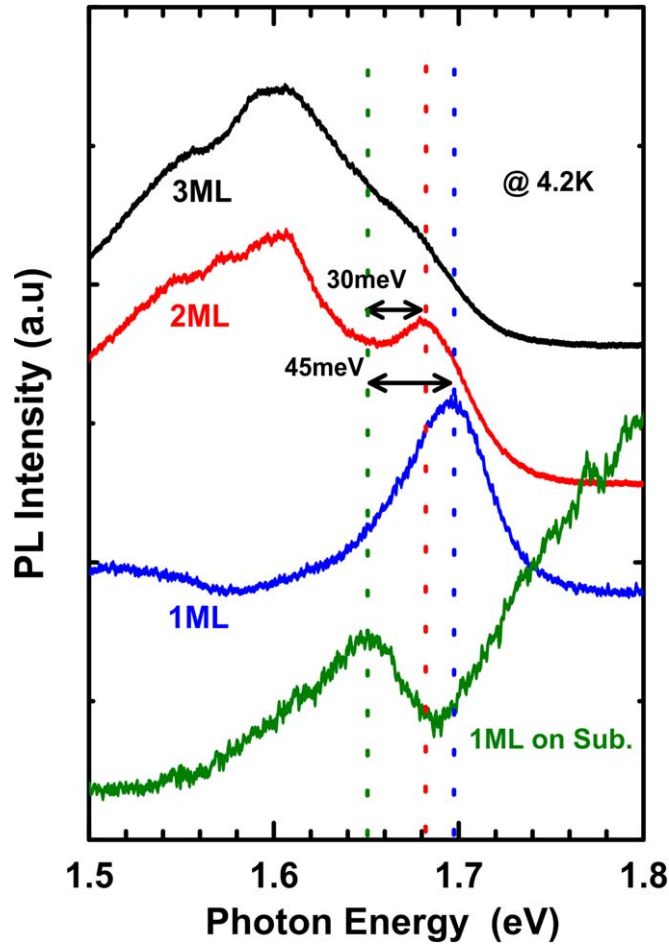
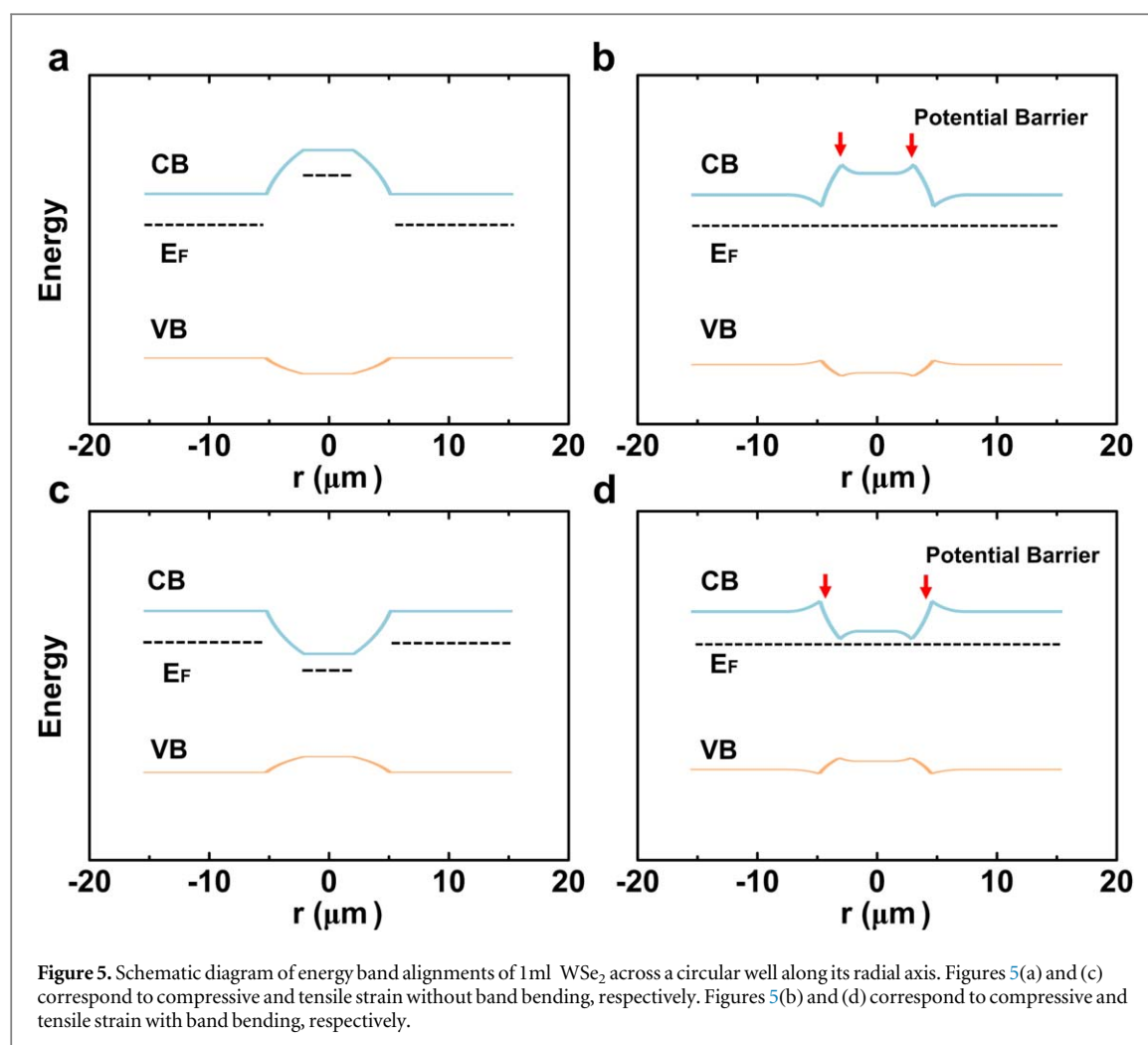


Figure 4. PL spectra of suspended WSe₂ flakes of different thicknesses compared to the supported 1 ml WSe₂ on the substrate.

$$\Delta E_v = 2A_v \varepsilon_{\parallel} \left(1 - \frac{C_{12}}{C_{11}} \right), \quad (2)$$

$$\Delta E_c = 2A_c \varepsilon_{\parallel} \left(1 - \frac{C_{12}}{C_{11}} \right), \quad (3)$$

where A_v and A_c are the hydrostatic deformation potential for the valence and conduction bands, respectively. C_{11} and C_{12} are the elastic stiffness constants of materials, and $\varepsilon_{\parallel}$ is the in-plane strain, with positive and negative signs indicating tensile and compressive strain, respectively. The bandgap change $\Delta E_g = \Delta E_c - \Delta E_v$ is proportional to the in-plane strain $\varepsilon_{\parallel}$. By using the equations (1)–(3) we can make a conclusion that the parabolic type of light-reflection from atomically thin TMD flakes in our experiments is attributed to continuously varying local strain, which also results in the gradient bandgap and the refractive index (Supporting Information). The inhomogeneous distribution of thermal strain may be caused by the difference in thermal expansion effect between suspended and supported atomically thin WSe₂ flakes, because there exist interactions such as bonding, doping and trapping in supported 1 ml WSe₂ on a SiO₂ layer that has a low thermal expansion coefficient $5.6 \times 10^{-7}/\text{K}$, which is able to change thermal strain in the supported WSe₂ flakes [27–29]. Our previous temperature-dependent Raman investigation on suspended graphene have demonstrated that the thermal expansion coefficient mismatch between graphene and substrate can change the thermal strain in supported graphene to produce a thermal strain difference, which also is suitable for atomically thin WSe₂ flakes [30]. Additionally, van der Waals adhesion at the edge of a circular well should affect the thermal expansion of supported zone in a certain distance, so-called coherent length. Out of this length, the substrate contribution could be neglected. Additional experimental and theoretical works are needed to clarify the coherent length, which is outside the scope of this work. The difference of thermal strain is largest at the edge of circular wells, so the corresponding sharp reflectance change in figure 3, and the effect of the thermal strain difference becomes weaker as r decreases, hence the corresponding slow reflectance change. In particular, it is worth to point out that there are flat CSOM signals in the centers of the suspended 1 ml and 2 ml WSe₂, which correspond to a big discrepancy between the CSOM signal profiles and the parabolic fittings in figure 3(e). This result implies that



the difference in thermal expansion effect between the suspended and supported atomically thin WSe₂ flakes is completely released there.

According to the above discussions, we schematically draw energy band alignments of 1 ml WSe₂ across a circular well along its radial axis, induced by gradient local strain as shown in figure 5. With it, we try to explain the observation of the central bright spots in figures 2(b)–(c). As reported before [31], compressive strain proportionally widens the band gap of nanostructures, while tensile strain narrows it. For a suspended 1 ml WSe₂ over a circular well, its compressive strain increases as its radial coordinate moves from the edge to the center of the circle well because of the effect of thermal expansion difference as illustrated above, which results in hill-shaped profiles of conduction band (CB) and valence band (VB) as shown in figure 5(a). The central range of CB profile is flat because of the absence of thermal expansion difference. The dash lines in figure 5 indicate the positions of Fermi energy of semiconductors (assuming n-type here). As shown in figure 5(b), the Fermi level of the suspended 1 ml WSe₂ keeps constant over a circular well under thermal equilibrium, which leads to band bending across the slope of the hill-shaped CB profile due to electrons transferring from the center to the edge. Consequently, there is a potential barrier around the flat central range of CB profile (marked in figure 5(b)), which gives rise to a potential well to confine electrons there. Therefore, it is reasonable to suppose that the central bright spots of light reflection in figures 2(b)–(c) could be originated from the surface electrons confined in a potential well and interfered by its edge. The local reflectivity change induced by surface states has been observed and discussed in 2D III-V semiconductor films before [32]. As a comparison, figures 5(c)–(d) shows the energy band alignments of 1 ml WSe₂ across a circular well under local tensile strain, where there is a deeper potential well in the central range of the CB profile as shown in figure 5(d). It is consistent with the concept of funnel-like graded gap structures, which gives rise to lower the threshold of semiconductor lasers by allowing for efficient capture of electrons and holes in the active zone of the quantum well laser [33]. At last, it is worth to note that the effect of the inhomogeneous local strain becomes weaker as the layer number of suspended WSe₂ flakes in our observations increases due to dielectric screening effect for the substrate surface.

3. Conclusion

In summary, we performed a real-space reflection mapping of suspended atomically thin WSe₂ flakes with a high spatial resolution by using a CSOM at the low temperature of 4.2 K. It is observed that the reflected CSOM signals of suspended 1 ml and 2 ml WSe₂ flakes exhibit parabolic profiles over circular wells on the SiO₂/Si substrate and the bright spots at their central areas. We demonstrated that such type of reflection profiles is induced by the continuously varying local strain over a circular well due to the effect of thermal strain difference between suspended and supported WSe₂ flakes, which also atomically to a gradient bandgap profile. Our findings provide fundamental information for future studies in the optical and electrical properties of atomically thin TMDs flakes with gradient gap structures modulated by inhomogeneous local strain.

4. Materials and methods

4.1. Exfoliation and transfer of atomically thin WSe₂ flakes

A WSe₂ crystal (99.999%, HQ Graphene) was used for exfoliation and transfer of atomically thin WSe₂ flakes. Prior to exfoliating WSe₂, the substrate SiO₂/Si with circular well array is ultrasonically cleaned in acetone, isopropanol, and deionized water, and then loaded into oxygen plasma chamber to further remove ambient adsorbates from its surface, such as water and organic molecules. A fresh surface of WSe₂ after cleaving on tape was brought in contact with the substrate, which is similar to the exfoliation process for preparing graphene [34]. The substrate with the attached tape was annealed in air at 373 K for 2 min using a hot plate. After cooling the sample down to room temperature, the adhesive tape was removed. Inspection by optical microscopy shows the successful exfoliation of suspended WSe₂ flakes on the substrate with circular well array.

4.2. PL Measurement

PL measurement of atomically thin TMD flakes were performed at the low temperature of 4.2 K, by using the Horiba HR Evolution. A CW laser of 532 nm was used as excitation source, and the laser power is 0.1 mW, with the light spot size of about 5 μ m.

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ORCID iDs

Yang Guo  <https://orcid.org/0000-0001-8975-9387>

Chi Sun  <https://orcid.org/0000-0003-1475-6442>

Xingjiang Zhou  <https://orcid.org/0000-0002-5261-1386>

Changzhi Gu  <https://orcid.org/0000-0002-2689-2807>

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