



Fast MoS₂ thickness identification by transmission imaging

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Abstract

Determining the thickness of a few-layer 2D material is a tough task that often involves complex and time consuming measurements. Here we discuss a rapid method for determining the number of layers of molybdenum disulfide, MoS₂, flakes based on microscopic transmission imaging. By analyzing the contrast of the red, blue and green channels of the flake image against the background, we show that it is possible to unequivocally determine the number of layers. The presented method is based on the light absorption properties of MoS₂ and its validity is confirmed by micro-Raman measurements. The main advantage of this method against traditional methods is to quickly determine the thickness of the material in the early stages of the experimental process with low cost apparatus.

Keywords Molybdenum disulphide (MoS₂) · 2D materials · Absorption spectroscopy · Transmittance · Transition metal dichalcogenides

Introduction

Molybdenum disulphide, MoS₂, is a naturally-forming layered transition metal dichalcogenides (TMD) that has a number of unique electronic and mechanical properties useful in many applications: e.g. transistors, flexible displays, optics and wear resistant materials (Rosenkranz et al. 2020; Balabai and Solomenko 2019). Depending on the number of layers, MoS₂ also shows piezoelectric properties, which can be exploited for energy harvesting (Yang and Lee 2019; Wu et al. 2014). Its ability to withstand high stresses also makes it suitable for the manufacture of buckled 2D structures, which have greater efficiency for vibrational energy harvesting (Li et al. 2019; Jiang 2015; Cottone et al. 2015).

The single layer MoS₂ is formed by a plane of molybdenum (Mo) atoms sandwiched and covalently bonded to two planes of sulfur (S) atoms. Few layers and bulk MoS₂, as

other TMDs, are formed by successive stacking of this structure through van der Waals forces forming a weakly coupled sandwich of layers. Due to the weakness of the vdW forces it is possible to mechanically exfoliate MoS₂ crystal down to single layer flakes. This method was successfully used in 2004 to obtain the first graphene flake (Novoselov et al. 2004) and from there on it has been applied to several other crystals to produce atomically thick structures. Although this procedure is fairly simple it presents several limiting problems being the main one the unpredictability of number of layers of the flakes obtained from the original crystal.

The most common and reliable method to identify the number of layers composing the material is based on the use of Raman spectroscopy. The frequency position and the separation between A_{1g} and E_{2g}¹ peaks in the Raman spectrum of MoS₂ are used to determine the number of layers composing the structure (Lee et al. 2010; Tonndorf et al. 2013; Chakraborty et al. 2013; Molina-Sanchez et al. 2015). An alternative to Raman spectroscopy for layer identification are reflectance and absorption spectroscopies. MoS₂ shows characteristic absorption peaks for the different excitons (Shi et al. 2013; Dhakal et al. 2014; Liu and Chi 2015). In this case the position and intensity of the peaks give a clear signature of the number of layers composing the material (Molina-Sanchez et al. 2015). For both techniques, however, as the number of layers increases, the spectral modification decreases, limiting the applicability of these methods to less

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than six layers (Lee et al. 2010; Tonndorf et al. 2013; Chakraborty et al. 2013). Once transferred to a suitable substrate the number of layers can be reliably determined by atomic force microscope measurements since the thickness of the single layer is ~ 0.65 nm (Li and Zhu 2015; Radisavljevic et al. 2011). Imaging methods have also been developed for the thickness identification of MoS₂ and other TMDs flakes transferred over SiO₂/Si surfaces by combining optical imaging with image analysis (Li et al. 2012; Late et al. 2012; Castellanos-Gomez et al. 2013a; Xu et al. 2014). However, the flake transfer increases the constraints in sample preparation and measurement complexity. Moreover, often flakes need to be exfoliated directly on transparent films such as PMMA, PDMS, or PC to make heterostructures (Ali et al. 2020). A method for fast screening based on visible transmission contrast measurements was proposed few years ago (Castellanos-Gomez et al. 2013b) and more recently validated by subsequent studies (Neri and López-Suárez 2018; Zhang et al. 2017; Niu et al. 2018; Taghavi et al. 2019). This method is based on absorption properties of MoS₂. While at macro scales absorption is usually proportional to the thickness, in 2D materials the electronic interactions between layers modify the shape of absorption bands, so that some care is required in applying this procedure. An external calibration is indeed a prerequisite.

In this paper, we discuss the limits of this method in order to extend its general application and identify a reliable procedure for the calibration. In particular, we assess the contrast analysis of red, blue and green channels of the image of a flake against the background in transmission microscopy to identify the number of layers of MoS₂ flakes. The method is justified on the basis of the absorption properties of MoS₂ in the visible region and the technical specifications of the imaging device. The measured sample thicknesses are compared with those obtained by micro-Raman, which shows the robustness and reliability of the method.

The measurements have been performed on samples ranging from one to four layers. In principle it is possible to determine the number of layers for flakes thicker than four. However, while absorption scales approximately linearly with the number of layers, the transmitted light, which is the actual measured quantity, decreases exponentially with the number of layers. This makes difficult to distinguish between two flakes with high number of layers.

Experiment and results

The MoS₂ samples used in this experiment were obtained by mechanical exfoliation with the Scotch tape method from a single crystal (Graphene Supermarket) and then transferred to a transparent polydimethylsiloxane (PDMS) substrate (Gel Film PF-20-X4 6 mil by Gel-Pak). The prepared

samples were inspected and measured with a transmitted light microscope accordingly modified for transmittance spectroscopy measurements. As in Frisenda et al. (2017) the trinocular port of the microscope has been modified in order to add a 50/50 beam splitter: half of the light goes to the camera, as originally planned, while the other half goes to a spectrometer through a multimode optical fiber. A focusing stage has been used in order to match the focus between fiber and camera sensor.

A schematic of the microscopy setup is presented in Fig. 1 a).

With this setup it is possible to perform micro transmission spectroscopy measurements selecting small circular regions with a diameter ranging from 70 μm down to 3 μm with a 600 μm core fiber changing the objective magnification from 4x to 100x (see Fig. 1 a and b). In Fig. 2 we present the measured absorbance spectrum for MoS₂ ranging from single to four layers. The absorbance is defined as $A = \log_{10} \left(\frac{I_0}{I} \right)$ where I_0 and I are the light intensity on the substrate and on the sample+substrate, respectively. The obtained data are in good agreement with literature and show the three characteristic exciton peaks of MoS₂. The absolute and relative positions of the peaks, as well as their amplitudes, give an effective mark of the number of layers composing the sample.

The obtained numbers of layers have been validated with Raman spectroscopy performed on same samples. Raman characterization was performed using a micro-Raman setup equipped with a solid state laser operating at $\lambda = 532$ nm. The laser light was focused on the sample by a 20X objective with a lateral spatial resolution of about 2 μm . The Stokes region of the back-scattered VV and VH polarized signal was analyzed by a 1800 grooves/mm grating and collected by a Peltier cooled CCD detector. The spectral resolution was about 3 cm^{-1} , however the estimated uncertainty in the determination of the frequency shift between near and well defined peaks is lower (about 1 cm^{-1}). This is relevant because the frequency separation $\Delta\omega$ between A_{1g} and E_{2g}^1 peaks give information on the number of layers of MoS₂ (Lee et al. 2010). As an example Fig. 3 reports the VV and VH spectrum of MoS₂ bi-layer with A_{1g} and E_{2g}^1 peaks, respectively. Also, the PDMS 489 cm^{-1} peak is present in the VV spectrum, and it can be exploited for further spectrum calibration. The inset of Fig. 3 reports $\Delta\omega$ as a function of the number of layers. The results are in good agreement with those obtained in ref (Lee et al. 2010), also reported.

While these two methodologies are powerful tools to determine the number of layers of MoS₂ and other 2D materials they are quite time consuming and require expensive and highly specialized instrumentations. Moreover, flakes produced by mechanical exfoliation can undergo deformation (strain) that persist during the transfer into the substrate.

Fig. 1 Microscope transmission spectroscopy. **a** Schematic of the modified microscope showing the attached spectrometer through a multimode optical fiber. **b** Optical image of a few-layer MoS₂ sample. The two circles highlight the selected regions, i.e. transparent substrate (I_0) and sample+substrate (I). **c** Example of spectra acquired with the spectrometer on the transparent substrate (I_0) and on the sample (I)

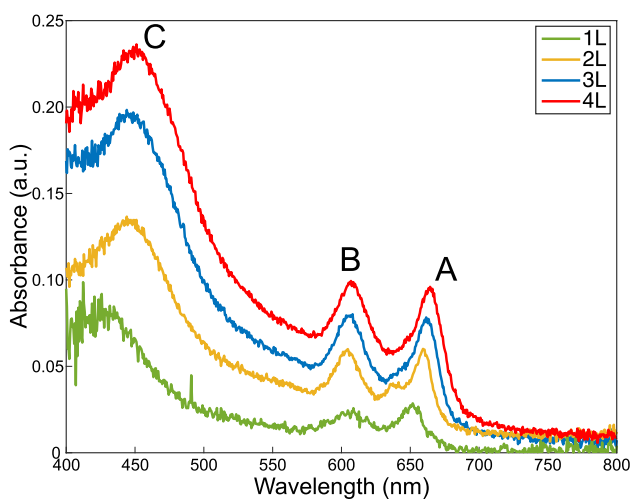
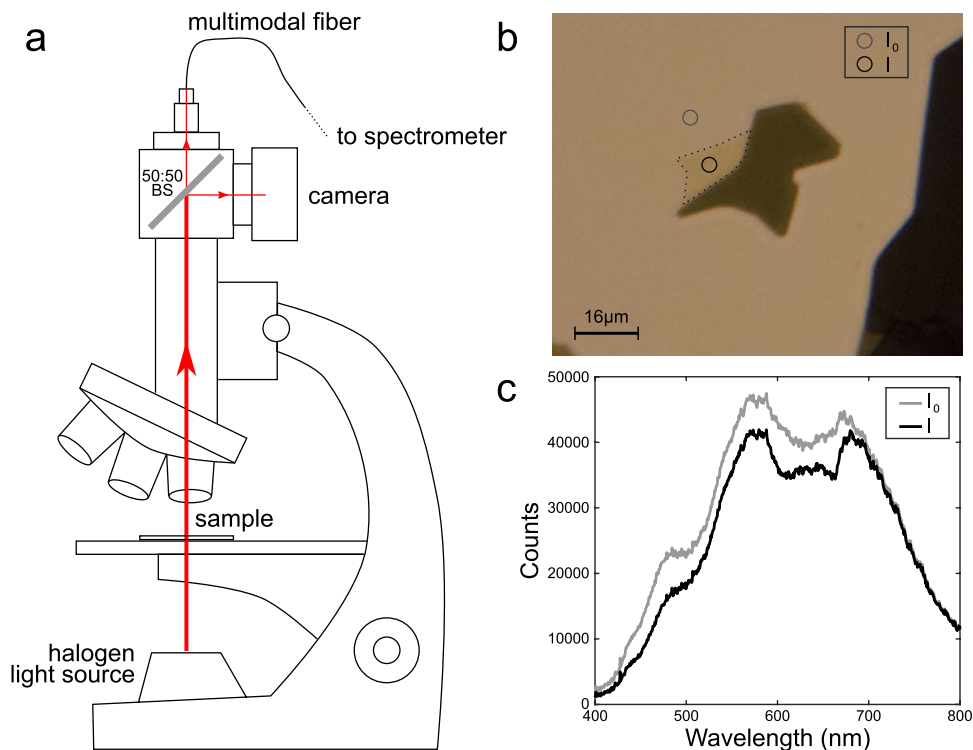


Fig. 2 Absorbance spectrum for MoS₂ flakes ranging from single to 4-layers. Labels highlight the positions of the different excitons

In general, the strain induced atomic displacement affects several materials properties from electronic ones (Kansara et al. 2020) to elastic (Caponi et al. 2014) and vibrational ones (Im et al. 2018). In the case of MoS₂, in particular, the induced changes, modifying the frequency of Raman (Wang et al. 2013; Rice et al. 2013) and excitonic (Gant et al. 2019) peaks may lead to a wrong determination of the number of layers.

Unlike micro-spectroscopy, with a high wavelength resolution but poor spatial resolution, a camera sensor has a high

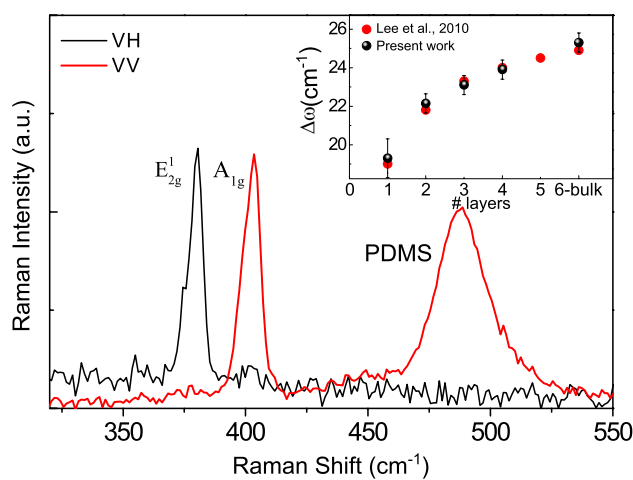


Fig. 3 Raman spectrum of MoS₂ bi-layer showing A_{1g} and E_{2g}^1 peaks. The two curves are relative to VV and VH polarized signal highlighting different features of the measured sample. Inset reports $\Delta\nu$ between A_{1g} and E_{2g}^1 peaks as a function of the number of layers

spatial resolution, determined by the size of the sensor, the number of pixels and the magnification. Light is usually filtered before the camera sensor by three filters (red, green and blue), averaged, and represented as a single pixel. With an image it is therefore not possible to get information on the peaks positions and their intensities, and it can be obtained only an average intensity value that strictly depends on the RGB filter of the camera. Spatially resolved optical absorption can be used to perform transmission spectroscopy

filtering the source light with a monochromatic filter (Castellanos-Gomez et al. 2016).

As a first approximation we can expect the overall intensity of the light captured by the camera to depend monotonically on the number of layers composing the sample: the higher the number of layers, the lower the intensity of the light detected by the device. As a matter of fact, in accordance with the Lambert–Beer law, absorption is proportional to the thickness in all materials. In MoS_2 , and in other 2D materials, however only the average absorption in the visible region is, roughly, proportional to the number of layers, because of the shift and change in oscillation strength of the excitons. This is due to the fact that the electronics properties of 2D materials depend on the number of layers. Note however that any change in the oscillation strength of the excitons is diluted in the average spectrum intensity. In fact, microphotography is integrated absorption in the three RGB bands. As stated before, the absorption coefficient to be used must be empirically determined, and it is strictly dependent on the camera hardware and imaging setting. Furthermore, this presupposes a linear relationship between light intensity and digitized pixel RGB value, which, assuming a linear response of the detector, is true only if the raw image is considered, before any processing (e.g. gamma correction, white balance, and so on)

For sake of clarity, in Fig 4 we have superimposed the RGB filter curve of the camera used in the experiment (Nikon D3200) (Nikon School 2020) and the absorbance spectrum of a candidate sample. But in fact, each camera has its own specific, albeit similar, filters.

We can clearly see that the three filters capture different features of the spectrum. For instance, the light component after the red filter is mostly affected by the A and B excitons.

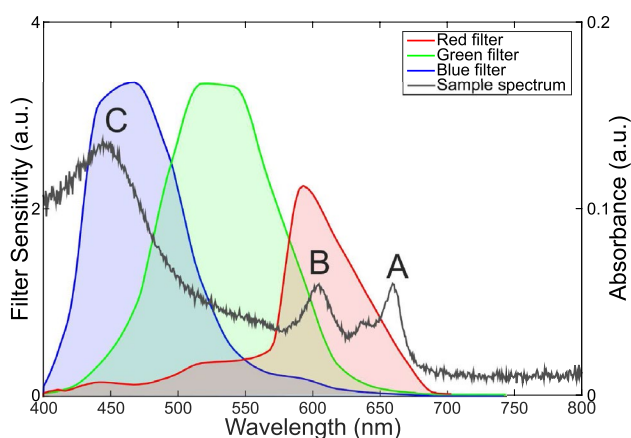


Fig. 4 Filter spectrum of the three R, G and B channels for the Nikon D3200 DSLR camera (Nikon School 2020). The absorbance spectrum of a candidate sample is superimposed indicating the region of interest for each channel. Labels highlight the positions of the different excitons

Similarly, the blue filter well captures all the C exciton. The green filtered signal is only affected by the residuals and slightly by the B exciton. Using an averaged intensity on all bands, therefore requires relying on the equalization procedure of the camera software which is device dependent. On the contrary using a single band intensity as marker requires no exciton moving in or out of the band. In any case the proportionality constant has to be determined for a specific setup

As mentioned above, the increase in the number of layers affects not only the overall absorption, but also changes the position of the peak. This effect, which is weighted by RGB filters, can affect in principle the relationship between light intensity and number of layers for a specific channel. In the Table 1 we report the absorbance value relative to the RGB channel acquired with a DSLR camera (Nikon D3200) for the MoS_2 samples under test.

The presented data follow a linear trend with coefficient (*R*-squared) 0.022 (0.9727), 0.027 (0.9782) and 0.039 (0.9886) for the three R, G and B channels, respectively. In Fig. 5 we present the relation between the number of layers and the absorbance of the three channels (RGB). According to the presented data it is evident that it is possible to accurately determine the thickness of MoS_2 samples based on a one-step transmission imaging method.

Table 1 Absorbance signature of MoS_2 flakes with different number of layers on the R, G and B channels of the DSLR camera

# Layers	R	G	B
1	0.0184	0.0182	0.0318
2	0.0406	0.0475	0.0785
3	0.0566	0.0688	0.1090
4	0.0888	0.1024	0.1571

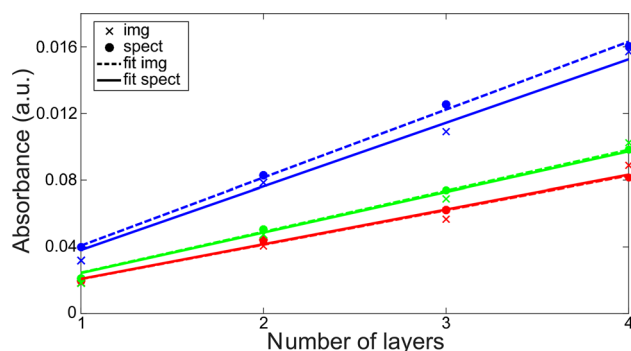


Fig. 5 Absorbance spectra for the MoS_2 flakes under study on the three channels (R, G and B) of the DSLR camera (cross) and computed from the spectrum and filter response (circles). The data are fitted with a linear function showing an excellent fit

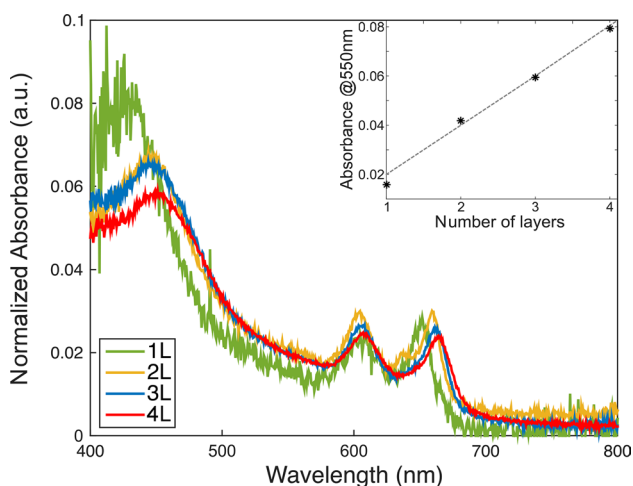


Fig. 6 Absorption spectrum normalized by the number of layers. Inset show the absorption with the application of a band-pass filter centered at 550 nm, width 20 nm

The data obtained from the images are compared with the data acquired by the spectrometer filtered by the camera sensor filter as presented in Fig. 4. For each channel we weighted and integrated the reference and signal counts and then computed the absorbance. Data obtained from image analysis and from spectroscopy are in good agreement. The biggest variation is present in the blue channel, which is the one most subject to noise due to a lower light intensity. This shows that with the sole knowledge of the RGB filter of the imaging camera it is possible to unequivocally determine the number of layers of a MoS₂ flakes by microscope transmission imaging. However, these data are strictly dependent on the RGB filter of the camera and indeed the obtained values vary respect the ones already reported in literature for a similar study (Zhang et al. 2017; Taghavi et al. 2019). To get rid of the dependence on the camera and on the specific RGB filters used, it is possible to filter the light source with a narrow band pass filter acting as a monochromator, making it possible to access a specific feature of the spectrum.

In Fig. 6 we present the absorption spectrum normalized by the number of layers. This is useful to show the region of the spectrum where the absorbance is approximately linearly dependent on the number of layers.

The variation of the excitons positions suggests that the regions around excitons are not good for reference. However, we see that the absorption in the region around 550 nm is approximately linear with the number of layers, with the exception of the mono-layer, which has a slightly lower absorption (see inset of Fig. 6). This suggests that the green channel intensity or the intensity measured using a band pass filter centered in that region could be a reliable proxy for determining the film thickness. Using a filter also allows to design a calibration-free method to determine the number of MoS₂

layers. The fitted absorbance per layers is ~ 0.02 at 550 ± 10 nm, using a narrow band-pass filter around that wavelength it is possible to accurately determine the number of layers $N = \lfloor \frac{\log_{10}(I_0/I)}{0.02} \rfloor$, where $\lfloor \cdot \rfloor$ indicates rounding to the nearest integer, I_0 is the light intensity on the substrate and I is the light intensity on the sample + substrate. Using a narrow band-pass filter permits to get rid of the model by model variations of RGB filters in the cameras, making the method independent of the imaging device used. In general, in different 2D materials, depending on the positions of the excitons, different bands should be exploited for the determination of the thickness.

Conclusions

In this work we have discussed a one-step method based on microscope transmission imaging of MoS₂ flakes to unequivocally determine the thickness of the samples, i.e. the number of layers.

We have shown which features of the absorbance spectrum determine the absorbance of the three RGB channels of the imaging camera. We show that the sole knowledge of the RGB filter response and the absorbance spectrum of MoS₂ are sufficient to determine the number of layers of the flake. MoS₂ also presents a monotonic and almost linear dependence of the absorbance at 550 nm with the number of layers. A narrow band filter around 550 nm could then be used to filter the source light making the imaging technique agnostic on the particular RGB filter used on the camera. We believe that such a technique can be easily extended to other 2D materials and can be exploited to automatically locate flakes with desired characteristics (e.g. mono-layers) from an exfoliated sample.

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Availability of data and materials The data that support the findings of this study are available from the corresponding authors upon request.

Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest or competing interests.

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