

3.35 Optics with 2D Quantum Materials



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Contents

1 Abstract	2
2 Safety Instructions	2
3 Preparation	3
3.1 Basics of 2D-Transition Metal Dichalcogenides	3
3.1.1 Crystal Structure	4
3.1.2 Electronic Band Structure	4
3.1.3 Photoluminescence of TMDs	5
3.1.4 Fabrication	5
3.2 Hexagonal Boron Nitride	5
4 Tasks	7
5 Measurement Techniques	7
5.1 Start up protocol	7
5.2 Calibration of microscope	7
5.3 Estimation of thickness of Hexagonal Boron Nitride	8
5.4 Identification of Monolayers	9
5.5 Photoluminescence Imaging and Bandgap Estimation	9
6 Protocol	11

1 Abstract

The successful isolation of Graphene, a single atomic layer of carbon, in 2004 started the era of 2D materials. Here the strong bonds between atoms in the same layer are covalent, whereas the layers are held together by weak van der Waals forces to form a layered material. Properties of single or a few layers are governed by quantum mechanics and thus they exhibit distinctly different properties in comparison to large bulk crystals. 2D transition metal dichalcogenides (TMDs) crystals are particularly interesting for optics and electronics. Some examples of TMDs are the semiconductors WS_2 , WSe_2 , $MoSe_2$ and MoS_2 that emit light efficiently when thinned down to one monolayer. Layers of different 2D materials may also be combined to form so called van der Waals (vdW) heterostructures. By choosing which 2 materials we combine in a stacked bilayer and by deciding the stacking the angle between layers we can tune the properties of the new crystal, with properties ranging from superconductivity in multilayer graphene to tunable photoluminescence in TMD heterostructures have been observed.

The goals of your FP experiment are to:

1. Introduce the optical properties of the 2D quantum materials.
2. Develop an understanding of optical microscopy as a powerful tool for the analysis of atomic monolayers and their strong light-matter-interaction.
3. Image, measure and analyse the photoluminescence emission from atomically thin semiconductors and vdW heterostructures to understand the physical nature of different bandgaps and light emission mechanisms in quantum materials.

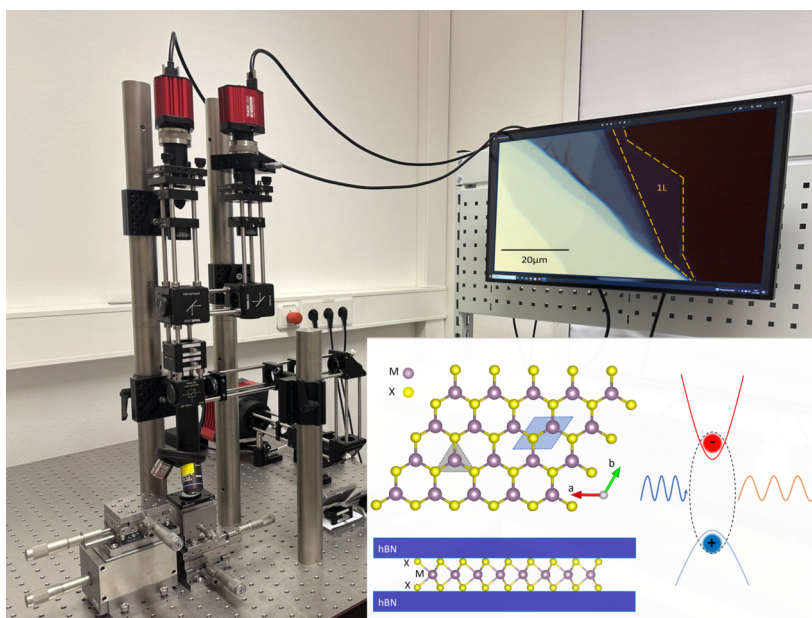


Figure 1: Setup for assembly and photoluminescence imaging of 2D materials.

2 Safety Instructions

- The light source used in the experiment is a high power LED with a typical output power of 8.75 W. Do not look directly into the light source.
- After long periods of usage the housing of the LED gets heated up hence it is recommended to avoid touching the housing directly.
- Always wear the gloves provided while handling optics.
- If there is dust on any optic only remove this by using dedicated optic wipes or the dust blower bulb.
- No drinking or eating is permitted in the lab.

Typical duration of FP – 5hrs (09:00-12:00, 13:00-15:00)

3 Preparation

The following sections summarise essential concepts that the reader must familiarize themselves with in order to perform the experiments successfully. You should be able to answer the following questions:

1. What are 2D materials and why are they an interesting field of study ?
2. What are van der Waals (vdW) crystals?
3. How are 2D materials such as monolayered TMDs different from their bulk form? (Band structure)
4. What is photoluminescence (PL) emission ? Why is measuring PL an essential tool to characterize 2D materials?
5. What are the different experimental techniques that can be used to identify monolayers?
6. What are van der Waals heterostructures (vdWhs) ?
7. Which fabrication techniques are most commonly used for the fabrication of 2D layers and vdWhs?
8. What is hexagonal boron nitride (hBN) and why are 2D materials typically encapsulated in hBN?
9. How does a white light microscope work?
10. Draw and explain the PL microscope setup.
11. What are the samples that will be analyzed in this experiment?
12. How can the microscope magnification be calibrated?

3.1 Basics of 2D-Transition Metal Dichalcogenides

In the late 1930s scientists argued that in theory two dimensional (2D) materials were thermodynamically unstable and thus could not exist in monolayer form. These arguments were shown to be untrue as at the turn of the millennium a group at the University of Manchester successfully isolated graphene, a single layer of carbon atoms, from a graphite crystal [4]. This was achieved through a simple experimental technique called exfoliation which made use of commonly available adhesive tape. This triggered the discovery and exploration of a vast family of 2D materials and resulted in the physics Nobel prize in 2010.[5]

While all these 2D materials showed unique properties, the class of 2D transition metal dichalcogenides (TMDs) has particular prominence. TMDs have a general formula of MX_2 , where M represents a transition metal such as molybdenum (Mo) or Tungsten (W) and S represents a chalcogen atom such as sulphur (S) or selenium (Se). Of the roughly 60 TMDs that are known to exist, two thirds have a layered structure [10] where each individual X-M-X layer is bound to the next by van der Waals forces, which are considerably weaker than the in-plane covalent bonds. A great deal of research focuses on understanding the properties of monolayers of these materials which have very different optical and spin properties compared to their bulk form. Recently research work has expanded beyond individual monolayers to so called van der Waals heterostructures. These are a combination of two or more individual layers stacked on top of one another, because also different materials can bond between each other due to van der Waals attraction. In the subsequent sections the crystal structure, electronic band structure and the synthesis of 2D-TMDs relevant to your FP experiments are discussed.

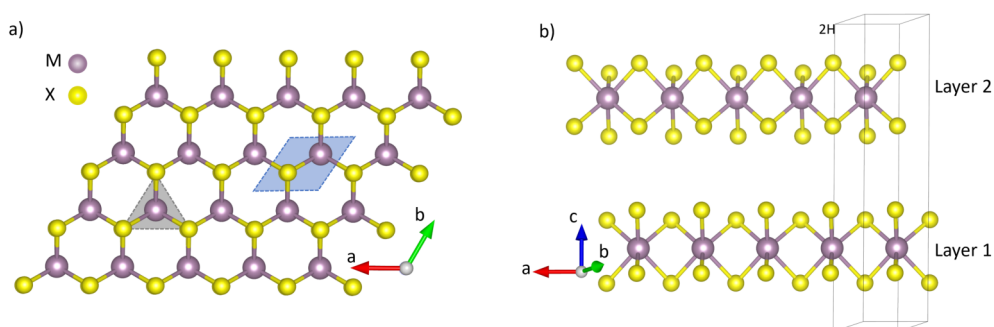


Figure 2: a) Top view of a single layer of TMD with a honeycomb like structure where M represents a transition metal atom and X represents a chalcogen atom. The shaded grey triangular region represents the trigonal prismatic structure and the blue region represents the 2D- unit cell. b) A 2H-TMD bilayer in which one layer is rotated by an angle of 180° with respect to the other and are held together by van der Waals forces.

3.1.1 Crystal Structure

The schematic of a single layer TMD can be seen in Figure 2. The top view of a single layer presented in figure 2a, shows a honeycomb like hexagonal lattice similar to graphene but with alternating M and X atoms at the corners of the hexagon. The grey triangle represents the trigonal prismatic crystal structure of the layer. Each metal atom in a single layer is bound to 6 chalcogen atoms via strong covalent bonds and each individual layer in the bulk is bound to its neighbouring layers via weak van der Waals interactions. Figure 2b shows the side view of a bilayer TMD. The case seen here represents a layer with ABA stacking of atoms and is referred to as the 2H phase. Several properties of 2D-materials, such as their electronic band structure and generation of higher harmonics in non-linear optics are determined by the phase, stacking order and orientation of the layers.

3.1.2 Electronic Band Structure

In term of electronic band structure, the family of TMDs consists of conductors, semiconductors and insulators. The semiconducting TMDs have attracted interest from many researchers owing to their potential in optoelectronic and photonic applications. Their bandgap (energy difference between the top of the valence band and bottom of the conduction band) is in an energy range that results in emission of radiation in the visible part of the optical spectrum (roughly 400 - 750 nm). **In this FP experiment you will explore the optical properties of different semiconducting monolayer TMDs.**

When excited with light of energy above their bandgap energy, an electron in the valance band absorbs a photon and jumps to an excited state in the conduction band. From here it relax to an intermediate state and can then relax back to its ground state in the valance band via the emission of a photon. This process illustrated in Figure 3a is governed by the nature of the bandgap of a material. In general, semiconductors may be distinguished as having either an indirect or a direct band gap. For a direct bandgap the light emission process is efficient, as the high energy electron can emit a photon to reduce its energy and subsequently occupy a state in the valence band. This direct emission process involves one particle : a photon. In contrast a semiconductor with an indirect band gap is not an efficient emitter : in order to conserve momentum, the emission involves a photon and a phonon (a quantum of lattice vibration) at the same time. This 2 particle process is less likely to occur than direct photon emission.

Figure 3b and c show the schematic band structures of a direct gap and an indirect gap semiconductor respectively. In the case of direct band gap semiconductors (as seen in Figure 3b), the highest point of the valence band and the lowest point of the conduction band occur at the same k value and this allows the excitation of an electron into the conduction band directly through the absorption of a photon. In an indirect gap semiconductor (seen in Figure 3c) the highest energy point of the valance band (VB) and the conduction band (CB) do not occur at the same wavevector value in reciprocal space.

For the TMD MoS_2 for example, the thick bulk crystal has an indirect bandgap. However, an MoS_2 monolayer has a direct bandgap. Therefore, a sample with 100 layers of MoS_2 emits less light than a sample which is one monolayer thick - very surprising !

Another distinct property of monolayer TMDs, as depicted in Figure 3d is the splitting of the valence and conduction bands. This allows for there to be two main transitions labelled A and B, across the bandgap. The valence band splitting occurs as a consequence of strong spin orbit coupling in monolayers. One of the ways to experimentally probe the switch to a direct band gap as well as the splitting of the bands is by measuring the photoluminescence from monolayers. In the following section, a brief overview of photoluminescence of TMDs is provided.

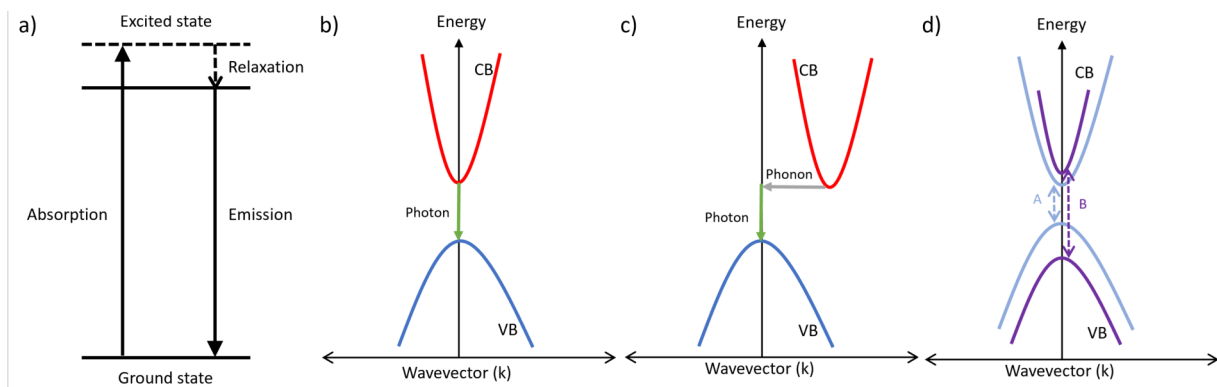


Figure 3: Schematic of a) the process of excitation of an electron and its relaxation back to its ground state through photoluminescence (PL) emission, b) emission from direct bandgap semiconductor (CB - conduction band and VB - valence band) c) emission from an indirect bandgap semiconductor and d) splitting of the valence and conduction bands in 2D-TMDs due to strong spin-orbit coupling.

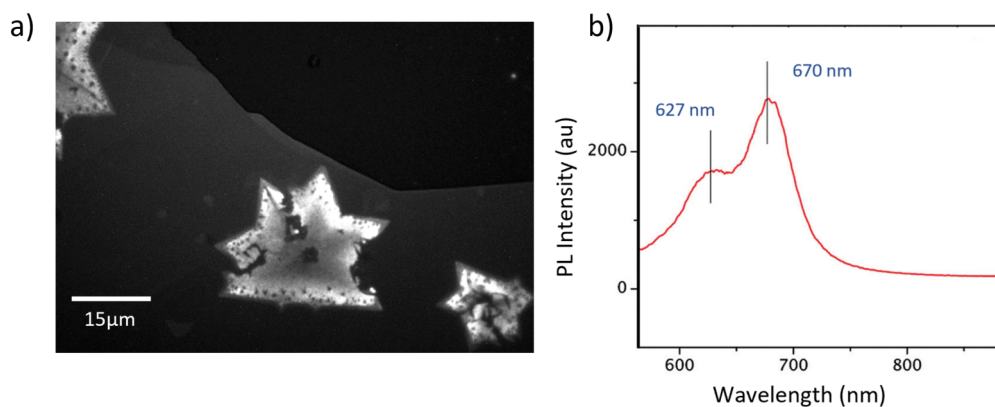


Figure 4: a) PL intensity map of a WS₂ monolayer grown by chemical vapor deposition (CVD). The dark regions within the bright monolayer are regions where the thickness is more than a single layer. b) Room temperature PL spectra from a single layer of MoS₂. The peaks seen at 670 nm (1.85 eV) and 627 nm (1.98 eV) correspond to the A and B transitions due to splitting of the bands. [8]

3.1.3 Photoluminescence of TMDs

Photoluminescence (PL) is the emission of light from a material which occurs after absorption of light by the same material at higher energy. In photoluminescence microscopy (i.e. emission measurements with high spatial resolution), the sample is excited at an energy above its bandgap. This promotes an electron from the valence band into the conduction band. This excited electron relaxes back down to the valence band by emitting a photon. Having a direct bandgap allows the electrons in the conduction band of monolayers to relax directly by the emission of a photon, unlike in the case of an indirect band gap where a phonon is required in addition. Hence a drastic increase in PL intensity is seen for monolayers for instance, **in WS₂ and WSe₂ PL emission from monolayers was reported to be between 100 to 1000 times stronger than in bulk samples with many monolayers [12].** PL intensity plays an important role in identifying monolayers of semiconducting TMDs. It also gives one insight into the electronic band structure of the materials. **In this FP experiment we use PL imaging as a tool to identify monolayers.**

3.1.4 Fabrication

The fabrication of 2D-crystals can be broadly divided into two categories, namely top down (big to small) and bottom up (from small to big) techniques. In top down fabrication, one starts with the bulk form of a material and then removes from the bulk crystal individual or few monolayers, as the van der Waals bond is easy to break. This can be achieved through techniques such as mechanical exfoliation with adhesive tape (similar to commercial tapes from the companies Scotch and Tesa, for example). The technique of mechanical exfoliation was used by the pioneers working on graphene, which resulted in the Noble prize in physics 2010 [5]. In this method, layers of the bulk crystal are mechanically peeled off using adhesive tape. This process is repeated several times until only a single layer remains. Figure 5a) shows an exfoliated monolayer from facilities and TU Darmstadt. Through such mechanical exfoliation monolayers of up to tens of micrometers of lateral size may be obtained. In order to obtain monolayers with a larger surface area in a more scalable manner, researchers have turned to so called bottom up techniques of fabrication. One such bottom up technique is chemical vapor deposition (CVD). During CVD growth, solid and gaseous precursors are made to react with one another resulting in the formation of monolayers on a host substrate, such as silicon with a layer of silicon dioxide (Si/SiO₂). Through such methods, monolayers of sizes greater than 100 μm have been obtained[3]. Figure 5b shows optical images of CVD grown WS₂ monolayer domains on a Si/SiO₂ substrate. These monolayers contain regions with seed crystals. These seeds, that are thicker than a single layer, act as nucleation sites for the growth of monolayers. It can be seen in comparison to Figure 5a, monolayer flakes obtained through CVD are bigger and more uniformly distributed over the substrate as compared to flakes fabricated via mechanical exfoliation. During this FP experiment we shall work with both kinds of monolayers.

3.2 Hexagonal Boron Nitride

Hexagonal boron nitride (hBN) is a wide gap material, with layered structure similar to that of graphene. It has an atomically flat surface with root mean square (RMS) roughness of about 0.14 nm and has been synthesised with very low concentration of impurities.[11] These properties have led for its growing use to encapsulate 2D materials and van der Waals heterostructures that are made by combining 2D materials in artificial stacks i.e. without analogy in nature [1]. In the process of encapsulation, the 2D material is sandwiched between hBN stacks. A thick bottom layer of several tens of nm provides a flat substrate surface for

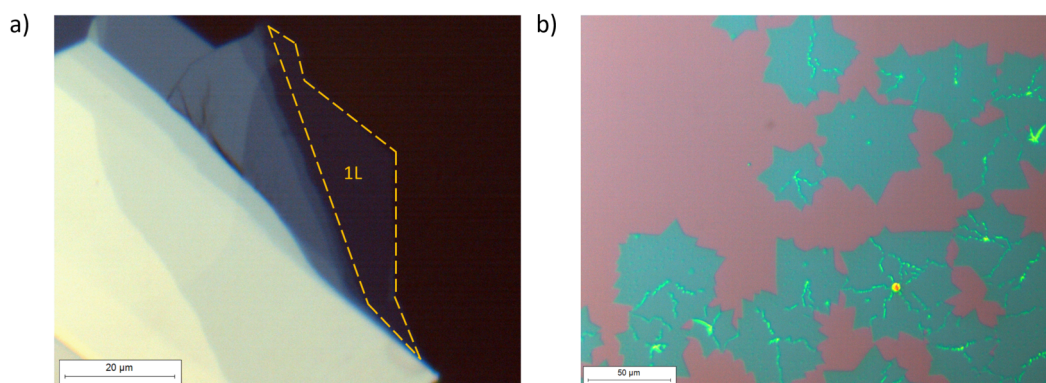


Figure 5: a) White light optical microscopy image of monolayer MoS₂ exfoliated using adhesive tape. Layers of different thicknesses have different color contrasts. The bluish regions are those with fewer layers in comparison to the regions that appear to be white. b) Optical image of WS₂ monolayers grown by the process of chemical vapour deposition (CVD). The greenish blue domains are monolayers and the pink background is the growth substrate which is a silicon (Si) wafer with a layer of silicon dioxide (SiO₂). The brighter regions within the monolayers are due to the seed crystals which act as nucleation sites for the growth of monolayers.

the monolayer and the thinner top hBN layer protects the monolayers from other chemicals landing on the monolayer surface. Encapsulation of a TMD monolayer in hBN allows the monolayer material to be investigated in a clean dielectric environment and free of mechanical strain. Strain may occur when the sample is being investigated directly on its growth substrate such as Si/SiO₂. Mechanical exfoliation is used to isolate hBN layers which are roughly 10s of nanometers thick. A polymer assisted pick up and transfer technique is then used to encapsulate the sample of interest with hBN. Atomic force microscopy (AFM) is one of the most commonly used techniques to determine the thickness of layers. However this process is slow, involves the use of costly setup and may sometimes cause damage to the flakes due to contact with the AFM tip. In order to avoid these, an alternative technique based on color contrast in optical microscopy is often used to estimate the thickness of hBN stack. This technique exploits the concept of thin film interference. When light is incident on a thin film (layer with thickness ranging from nanometers upto several micrometers), light reflected from the air/layer interface and from the layer substrate interface, interfere with each other. When white light is incident on such a layer, based on the thickness of the film only light of a certain wavelength meets the condition for constructive interference. And hence layers of different thicknesses are of different colours. This is the same effect responsible for the colors of a soap bubble and of a film of oil on a surface. In figure 6a, colourful flakes of hBN can be seen. Here every flakes with a uniform color indicates a flake that has the same thickness all over its surface area. This is illustrated more clearly in figure 6b. Here a single hBN flake is seen. Through AFM measurements the thickness of these different regions was found and it can be seen that based on their thickness the different regions appear with different colors. In this FP we make use of a pre-calculated colour scale where the colour of hBN layers of thicknesses upto 750 nm is predicted for different substrates.

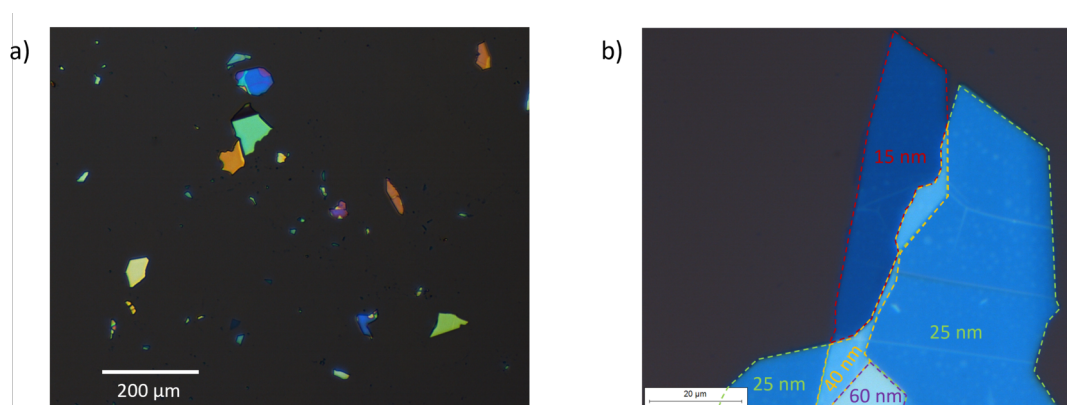


Figure 6: a) hBN flakes of various thicknesses and hence different colours. b) A single hBN flake with the thickness of different regions labelled.

4 Tasks

During the course of this FP experiment you will perform the following tasks:

- Calibration of sizes using different objectives of the microscope
- Identification of monolayers among layers of different thickness
- Estimation of lateral size of monolayers
- Photoluminescence imaging of monolayers
- Identification of material by bandgap estimation
- Colour based estimation of thickness of hBN layers (in protocol)

In the next section, the various measurement techniques used to complete each of these tasks are discussed.

5 Measurement Techniques

As shown in Figure 5, the typical lateral sizes of CVD grown and exfoliated monolayers that will be investigated in the course of this experiment is in the range of 10-50 μm . To obtain images of layers of such size, we use an optical microscope. This allows us to magnify the monolayers and image them using a camera. All the experimental tasks of this FP will be performed using a microscope. Figure 7 shows the schematic of the microscope setup designed and built at TU Darmstadt for the analysis of 2D-materials. The main components are labelled. This setup contains additional components in comparison to a standard microscope. This allows one to use the system for not only white light microscopy but also PL emission microscopy. Through the next subsection the individual measurement techniques used to complete the tasks in the FP, are elaborated upon. The primary components have been labelled. White light imaging is used as the primary tool to estimate the thickness of hBN layers and monolayer regions of TMDs. For direct band gap, monolayered materials such as MoS_2 , WS_2 , MoSe_2 , and WSe_2 PL imaging can be performed to confirm if the sample thickness is truly a monolayer.

5.1 Start up protocol

In order to start the microscope follow the steps mentioned below.

- Turn on the PC
- Connect the colour camera (see Figure 10d) Camera via USB cables to the PC.
- Launch the ThorCam software on the desktop. (Software manual-[ThorCam Software Manual](#))
- Check if all components are assembled to ensure that light is detected by the colour camera (Name-CS126CU).
- Turn the knob on the LED light source driver to a low intensity (see Figure 10a). Note that pressing down on the knob turns the LED on. Take care to not turn it on while turning the knob.
- Ensure no filters are in the path of the light (see Figure 10a and c).
- Turn on the LED by pressing down on the red knob on the driver.

5.2 Calibration of microscope

In a microscope the image of a sample is created by focusing a magnified image onto a camera. Earlier versions of microscopes used objectives which magnified the images and also focused it onto the camera. However objectives used in current microscopes are so called infinity corrected objectives. In such objectives the light comes out as a parallel beam and has to be focused onto the camera using an additional lens known as a tube lens. The size of the final image seen on the camera is hence dependent on the magnification of objective as well as the focal length of the tube lens. In order to measure the lateral size of a samples seen through a microscope, it is necessary to first calibrate the microscope. This is done by using a calibration target where the size of features on the target is already known. Figure 11b shows a test target used in this experiment. The target comprises of various groups with lines with a specified spacing. By imaging these lines, using different objectives, the size of various features in can be determined. During this experiment, calibration will be performed using the colour camera. The following are the steps to calibrate the microscope.

- Place the test target on the sample stage.
- Image a group of lines such that the lines fit well within the field of view of the camera.

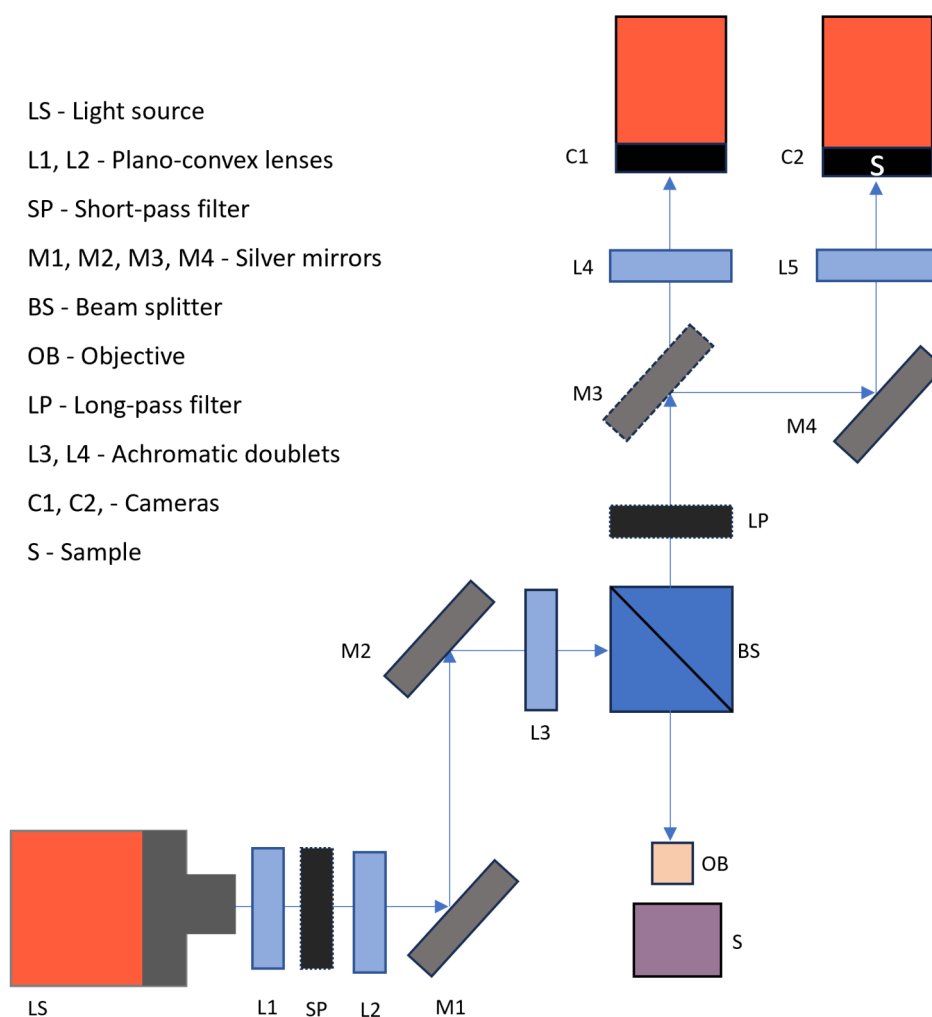


Figure 7: a) Schematic of setup for analysis of 2D-materials.

- Adjust intensity of the LED and exposure time (on the ThorCam software) to obtain a well balanced image i.e. you see contours, no saturation.
- Look up the specifications of the lines chosen on the calibration target on the suppliers website ([Thorlabs Calibration Target](#)).
- Using the "measure" and "annotate" features on the ThorCam software create scale bars in the image.
- Save images in .png format of the lines.
- Repeat the above mentioned steps with every objective. (To avoid crashing into the sample while switching objectives take care to no switch from 5x directly to 50x without first adjusting the stage height!)
- These scale bars can now be used as references in order to determine the lateral size of monolayers. Indicate the sizes for the layers you chose and estimate a realistic error.

5.3 Estimation of thickness of Hexagonal Boron Nitride

The analysis of this task can be done as part of the protocol for this experiment.

- Obtain images of at least three exfoliated hBN flakes.
- Estimate the thickness range ($\pm??$ nm) of the hBN layers by comparing the colour of the flake with the colours seen in the pre-calculated scale shown in figure 9a.

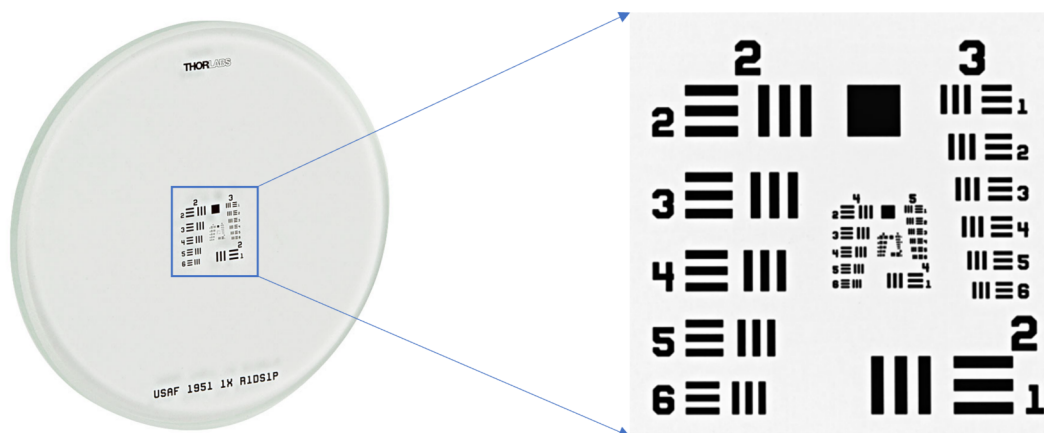


Figure 8: a) Calibration target from Thorlabs GmbH. It consists of various groups lines different spacing. It can be used for calibration of resolution as well as magnification of an imaging system.[9]

- Identify the wavelength of light corresponding to the color of the flake using the visible spectra in Figure 9b.
- Calculate the refractive index of the film using the condition of constructive interference from a thin film given as: $2nd = \lambda/2$ where n is the refractive index of the film, d is its thickness and λ is the wavelength.
- Comment on the order of the interference by comparing the obtained refractive index to literature values([6]).

5.4 Identification of Monolayers

In the next part of the experiment white light microscopy is used to identify monolayers on a substrate with flakes of different thickness. Figure 10 shows the different arms of the setup with every component labelled.

- Place substrate on the sample holder by lifting it carefully with the tweezers provided.
- Bring the sample into focus by adjusting the height of the sample stage with respect to the microscope objective using the micrometer head on the z-stage (see Figure 11).
- To locate the encapsulated monolayers on the Si/SiO₂ substrate, adjust the micrometer heads on the appropriate xy-stage (see Figure 11).
- While using the micrometer heads of the stages take care to not turn it beyond its range. Doing this can either lead to the screw head coming off or could lead to damage to the tip of the screw due to excess pressure on it.
- Once the TMD stack is in the field of view of the camera, re-adjust the intensity of the light source and the exposure time (if the auto-exposure setting is not being used or it doesn't yield a balanced image).
- Save images of regions with mono- or few- layers.
- Follow steps in the next section to obtain a PL image and repeat the above steps for the next sample.

5.5 Photoluminescence Imaging and Bandgap Estimation

Once a region with what is assumed to be a monolayer is found and a white light image is recorded, we proceed with PL measurements, as monolayers are expected to give the stronger emission compared to thicker layers. For the PL measurement the same setup as earlier is used but with filters in the light excitation and light detection path. For the excitation, a 500 nm short pass filter is used. Such a short pass filter only allows light of wavelength less than 500 nm to pass through. Figure 12a shows a combined plot of the emission of the light source (left axis) and the transmission of the excitation filter (right axis). Placing the excitation filter in the path of the LED only allows light in the region in the red shaded region to pass through. Figure 12b shows the combined plots for the PL emission of 4 different TMD monolayers (left axis) and the transmission of the detection filter. The detection filter used here is a 550 nm long pass filter meaning it only allows light with a wavelength longer than 550 nm to pass through. This would hence block out any of the excitation light and allow only PL to pass through and be detected by the camera. The PL imaging is performed using a monochrome camera instead of the colour camera used earlier. This is because the colour camera has three colour (RGB) channels and the quantum efficiency of each channel varies differently with wavelength. In a monochrome camera since there are

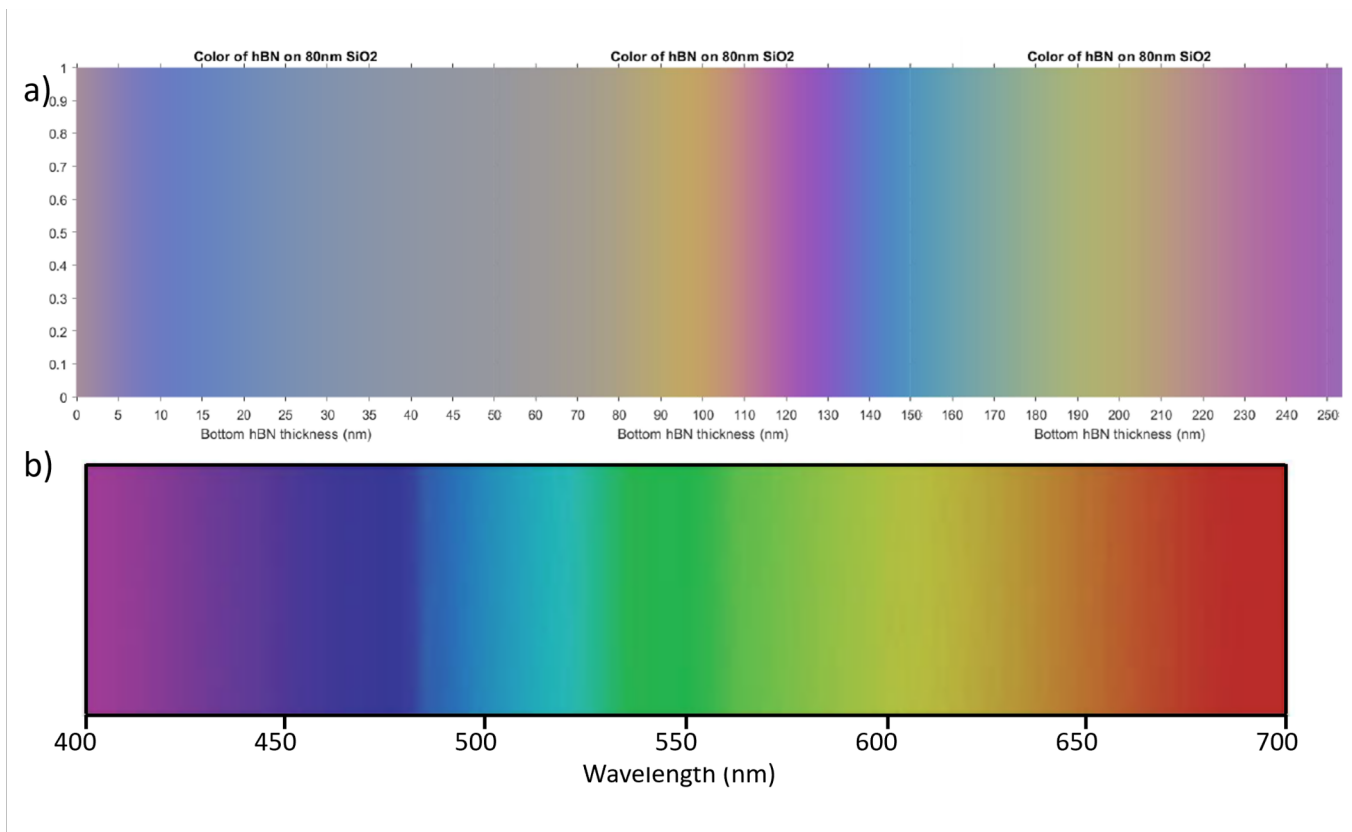


Figure 9: a) Colour of the hBN layers on SiO₂ as a function of thickness. b) Visible spectra colours as a function of wavelength of light, in air.

no colour channels, it is more straightforward to determine the quantum efficiency for a given PL signal. Here are the experimental steps to obtain the PL image of the selected flakes:

- Ensure that the light source is turned off (by pressing down on the knob).
- Remove mirror from the kinematic cage cube in order to ensure detection via the monochrome camera (Name-CS126MU).
- Connect to the monochrome camera via the ThorCam software.
- Turn on the light source with a low intensity.
- At this point a black and white image similar to the earlier observed colour image should be visible.
- Choose and insert excitation and detection filters into the respective filter holders.
- Identify the monolayer region and adjusting the source intensity and the exposure time (on the ThorCam software) until PL emission is visible. Obtain images of these regions. It is suitable to use the 50x objective to obtain PL images. While using high exposure times ensure that the room lights are turned off in order to avoid saturating the camera CCD.
- In order to identify the material, exchange the detection filters and observe when the PL vanishes.
- Once this is observed an estimation (in a 50 nm range) can be made about the wavelength corresponding to the optical gap energy.
- Calculate the estimated bandgap in eV and compare it to literature values (refer to figure 12b).
- Repeat the steps for each sample.

Once the experiment is completed, return all the stage to a neutral position. Unmount the filters from the filter holder and place them in the designated boxes.

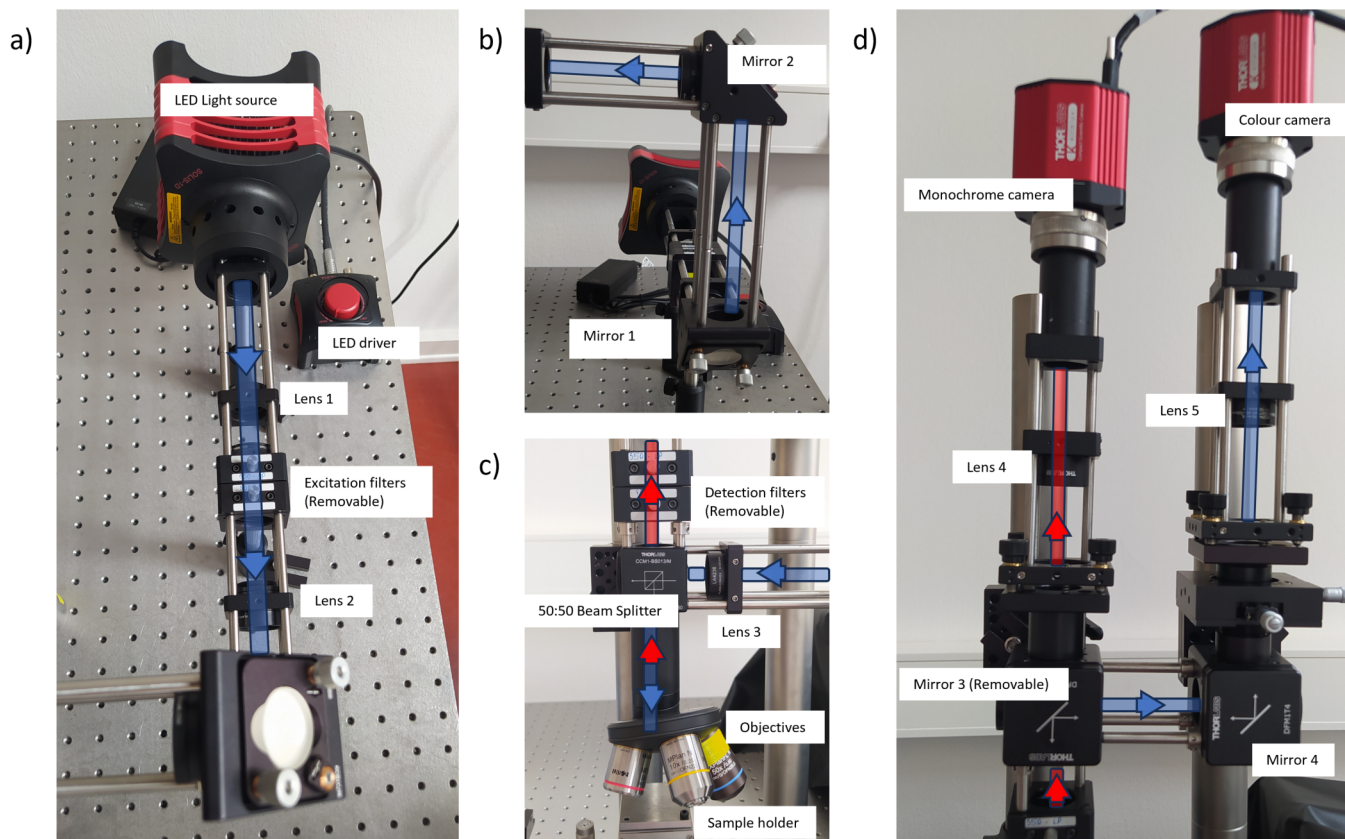


Figure 10: Individual arms of the PL microscope setup. a) Source, excitation filter and lenses for collimation of light. b) Mirrors on kinematic mount to align excitation beam. c) 50:50 beamsplitter, microscope objectives and detection filters. d) Removable mirrors, converging achromatic doublet lenses and cameras.

6 Protocol

- The protocol should begin with a brief introduction explaining the motivation and goals of the experiment performed.
- A background on the properties of 2D-TMDs, with a focus on their PL properties should be included.
- Outline of the experimental setup should be presented.
- Calculate the refractive index of hBN based on the estimated thickness.
- Any external sources from where images or information is used, should be duly cited so we can follow your arguments.

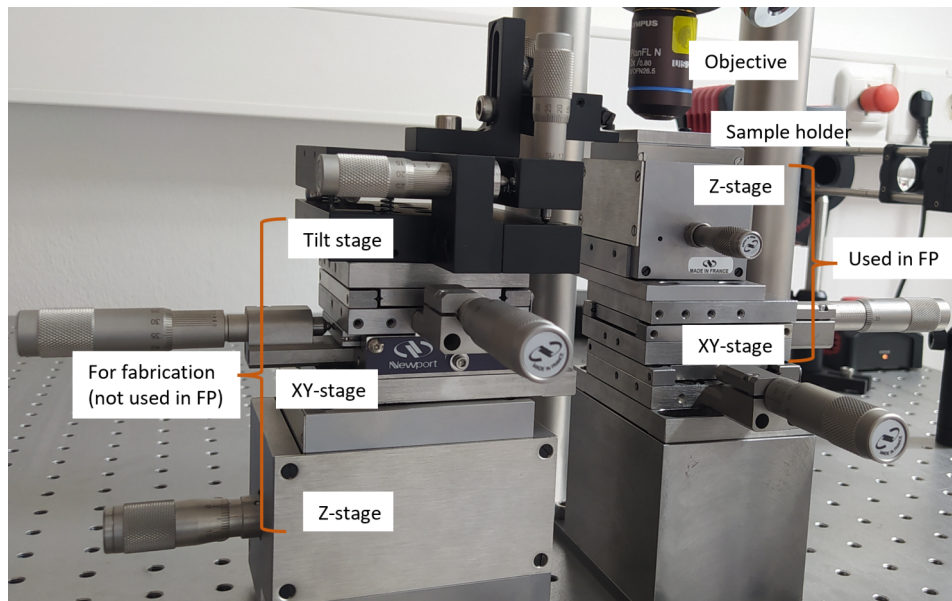


Figure 11: Two sets of manual stages are present in the set up for the placing samples and aligning them. Only the stack on the right side is used during the FP.

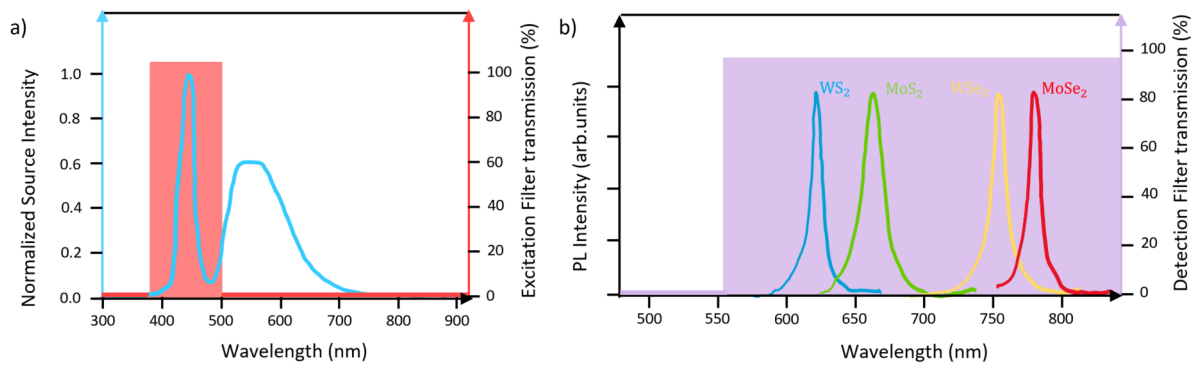


Figure 12: a) Source spectrum (left axis) overlaid with the transmission spectra (right axis) of the shortpass excitation filters. b) PL Emission of the four most commonly studied TMDs (left axis) overlaid with the transmission spectra (right axis) of one of the longpass detection filters.

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