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Fakulta matematiky, fyziky a informatiky



Mgr. Jakub Hagara

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Templated Growth of small organic molecules

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Predkladateľ: Mgr. Jakub Hagara
Fyzikálny ústav SAV
Dúbravská cesta 9
841 04 Bratislava

Školiteľ: Dr. Rer. Nat. Peter Šiffalovič, DrSc.
Fyzikálny ústav SAV
Dúbravská cesta 9
841 04 Bratislava

študijný odbor: Kvantová elektronika a optika

Predseda odborovej komisie:

Prof. RNDr. Pavel Veis, CSc.
Fakulta matematiky, fyziky a informatiky
Univerzity Komenského
Mlynská dolina F1
842 48 Bratislava

Introduction

Organic electronics have seen a vast improvement in performance over the last few decades, thanks to dedicated and rigorous research.(1–7) Although organic semiconductors cannot rival their inorganic counterparts, they offer an inexpensive and lightweight alternative to already established silicon-based devices. The main factor hindering the performance of organic electronic devices is the weak bonding of organic molecules in a solid state. This weak bonding negatively affects the electron/hole mobility between neighboring molecules inside organic thin films, limiting their use in high-performance electronics.(8,9) However, the ability to tune the electronic and optical properties of organic semiconductors as well as their compatibility with transparent and stretchable substrates enabled these materials to be used in applications that are challenging for standard silicon-based semiconductors.(10–16)

The performance of organic electronic devices depends mainly on the long-range ordering of organic molecules in a thin film and their intermolecular interactions.(9) Full understanding of thin film structure is necessary to utilize the promising semiconducting properties properly.

In this thesis, I will investigate the structure of thin films of organic molecules usable in organic electronics. When deposited by organic molecular beam deposition, organic molecules assemble into well-ordered thin films. Grazing-incidence wide-angle X-ray scattering method is used to study the structure of prepared thin films. Electronic and optical properties of organic semiconductors depend on the orientation of molecules. Organic electronic devices such as organic field-effect transistors and organic solar cells require specific molecular orientation with respect to the substrate in order to work efficiently. I use growth templates to control the orientation of organic molecules. Templates are thin, supporting layers, typically consisting of only a few atomic layers, which are able to influence the orientation of grown organic thin films; an extreme case being a graphene layer. I use mainly the few-layer MoS₂ template for its ability to achieve both desired configurations of an organic thin film. Depending on conditions during the preparation, it is possible to prepare MoS₂ templates with two different alignments. The influence of MoS₂ templates with different alignments on the growth orientation of organic thin films is investigated.

Growth templates

The most common small organic molecules used in organic electronics are molecules such as acenes, phenacenes, and fused-ring oligothiophenes with a conjugated core, extending in one direction. This extended rod-like structure of OSCs then results in an anisotropy of charge carrier transport. In order to successfully use small organic molecules in organic semiconducting devices, the correct orientation of molecules must be considered to achieve the desired performance. For example, hole transport mobility of acenes is dominant in the direction perpendicular to conjugated rings (e.g., in the direction of π - π stacking), where charge carriers can "jump" between conjugated cores of neighboring molecules. To properly utilize acenes in OFETs, molecules must be oriented perpendicular to the direction of charge carrier transport between source and drain. In other words, a standing-up configuration of a thin film is required to achieve the best possible performance of in-plane OFETs.

On the other hand, the optical anisotropy of organic molecules has to be taken into account when used in organic photovoltaics (OPVs). For this purpose, the correct orientation of molecules with respect to the direction of the incoming light is necessary. Most organic molecules suitable for OPVs have their transition dipole moment located along their long molecular axis. To properly implement an organic active layer into OPVs, the lying-down configuration is required for optimal light absorption.⁽¹⁷⁾ The orientation of organic thin film can be controlled by employing growth templates.

Van der Waals (vdW) interactions between the substrate/layer and organic molecules determine the orientation of a grown thin film. If the interactions between conjugated cores of neighboring molecules are much stronger than molecule-substrate interaction, they tend to grow in the standing-up configuration. On the other hand, a strong molecule-substrate interaction can drive a lying-down morphology of a first monolayer. The thin film then continues to grow in this configuration for a few additional layers. However, the lattice mismatch between substrate and thin film causes strain that builds as a thin film thickness increases, until it results in disorder.⁽¹⁸⁾ To preserve the lying-down configuration beyond a few nanometers, the molecule-substrate interaction must be comparable to molecule-molecule interactions of grown molecules. This supports the strain relaxation after the first monolayer, but the interaction is still strong enough to influence the orientation of a thin film.^(17,18)

A growth template can effectively alter the strength of molecules-substrate interaction. A template is a thin interlayer between a substrate and grown organic semiconducting layer. An ideal template only influences the growth orientation of an organic layer but does not change the molecular configuration of a thin film.

Another important role of growth templates is to control the interface between an active organic layer and a substrate. Interface controls the initial nucleation of organic molecules on a substrate during deposition. Growth templates aid the formation of high-quality crystalline thin organic films by limiting the interfacial and structural disorder.⁽¹⁹⁾ Improved crystallinity of thin films leads to a substantial improvement of the charge carrier transport capabilities of OSC active layers.

Finally, a growth template should have an excellent conductivity to provide charge transfer between the active layer and electrodes of an electronic device.

In my work, I used a few-layer Molybdenum disulfide (MoS_2) template to influence the growth of the small organic molecules. It is relatively easy to prepare a large surface area of a few-layer MoS_2 (or even a single monolayer) by the methods such as magnetron sputtering or chemical vapor deposition.

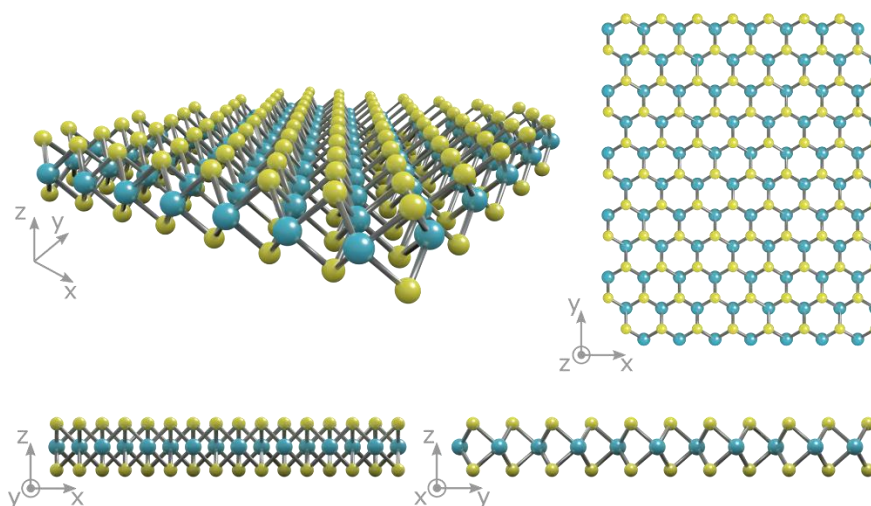


Figure 1: structure of MoS_2 monolayer. (source: www.ossila.com)

Due to its structure, MoS_2 can be used as a growth template for the deposition of OSCs. Strong template-molecule interactions influence the growth orientation of deposited organic molecules without a structural mismatch induced strain. This prevents the disorder of deposited organic films so that organic molecules retain their structure even for a high number

of layers. Additionally, because of its semiconducting nature, the MoS₂ template provides an excellent interface between the organic active layer and electrodes of an electronic device. As shown in the *Results* part of my thesis, it is possible to prepare the MoS₂ growth templates with two distinct crystal orientations. Depending on the alignment of the growth template, organic molecules during the deposition grow in lying-down or standing-up configuration.

Objectives of study

The aim of the work will be the preparation of thin organic semiconductor layers suitable for use in organic electronics. The structure of prepared thin films will be investigated by X-ray diffraction methods such as GIWAXS and GISAXS. Furthermore, we will study the structure of 2D materials such as graphene and MoS₂ and their influence on the orientation of deposited thin organic films. Depending on the type of interaction between the 2D layer and the vapor-deposited molecules, it is possible to achieve a different orientation of a thin film. The ability to specifically control the structure of a thin organic film is essential for the use of organic materials in electronics.

Goals:

1. Preparation of thin organic films suitable for use in organic electronics.
2. Study of the structure of thin organic layers employing the GIWAXS / GISAXS method.
3. Study of the possible use of thin films such as graphene and MoS₂ to control the growth orientation of thin organic films.

Methods

MoS₂ growth templates were prepared from the molybdenum precursor by Chemical vapor deposition (CVD). CVD is a process in which a film of desired material is deposited from a gaseous phase by chemical reaction of volatile precursors. In my case, the thin layer of molybdenum on Si/SiO₂ substrate was sulfurized in vapors of sulfur.

Thin organic films were grown employing Organic molecular beam deposition. During the deposition the desired organic material inside of effusion cell is heated up and evaporated. . Due to the high vacuum inside a deposition chamber, evaporated molecules propagate straight to the substrate surface without colliding with the rest gas molecules.(20) On the substrate surface molecules condense to form a homogenous film with a thickness ranging from Ångstroms to micrometers. Thickness of the prepared thin film was measured during the deposition using quartz crystal microbalance (QCM) monitor.

The thin film structure as well as MoS₂ template alignment was characterized by grazing-incidence wide-angle X-ray scattering method. Using laboratory X-ray source, I was able to determine the alignment of growth template and orientation of organic molecules. By employing the synchrotron radiation, it was possible to observe in-situ growth of organic thin film during the deposition, tracking the stress-induced changes of unit cell. Thanks to its high brilliance, even early stages of growth can be observed.

Results

Characterization of growth template

Growth templates are thin layers that can influence the molecular orientation of thin organic films. During the deposition, van der Waals interactions force the growing molecules to adopt one of two commonly observed configurations, either standing-up or lying-down. To achieve the desired configuration, it is usually necessary to select the correct template. In my work, I studied the structures of few-layer MoS₂ films prepared by sulfurization of molybdenum films. I observed that it is possible to prepare few-layer MoS₂ films with two different crystal orientations depending on the preparation conditions. MoS₂ is a semiconducting material with a layered structure, where two atomic layers of sulfur enclose an atomic layer of molybdenum. The alignment of these MoS₂ layers can be determined by GIWAXS measurement. Stacking of individual MoS₂ layers leads to a strong 002 Bragg peak. From the position of this diffraction peak in the GIWAXS pattern, we were able to determine the MoS₂ alignment. I showed that when a 1 nm thick layer of molybdenum film is sulfurized, the resulting few-layer MoS₂ film adopts horizontal alignment (Fig. 2a), where the individual MoS₂ layers are parallel to the substrate surface. On the other hand, after sulfurization of 3 nm thick molybdenum film, we obtained a vertically aligned MoS₂ (Fig. 2b) film with the layers perpendicular to the substrate surface. Few-layer MoS₂ films can be used as growth templates for OSCs. Depending on the alignment, different van der Waals interaction of MoS₂ with deposited molecules is expected.

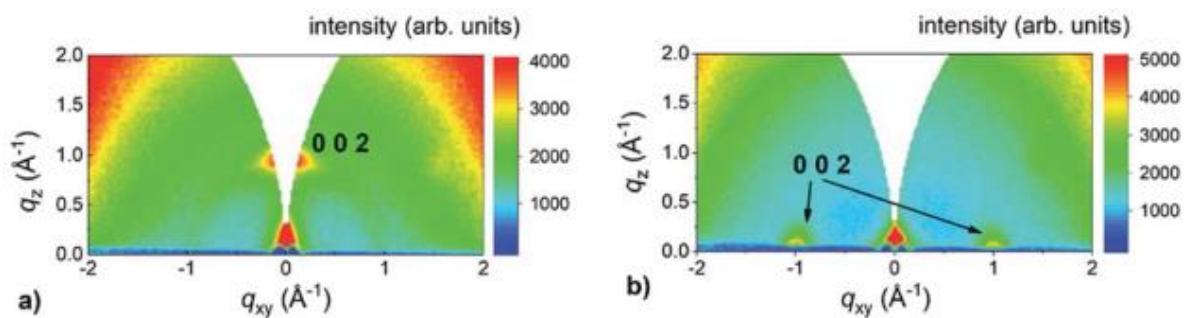


Fig. 2: GIWAXS pattern of MoS₂ growth template with a) horizontal b) vertical alignment.

Growth of small organic molecules on growth templates

As mentioned in the theoretical part of my thesis, graphene is the most frequently used growth template. Since its structure is almost identical to conjugated cores of OSCs, only a small mismatch-induced strain is expected on a thin film. In fact, even a single layer of graphene is able to influence the growth orientation of deposited molecules. Furthermore, it provides an excellent interface to support the nucleation as well as facilitate the charge carrier transport between electrodes and organic thin film. In our work, we studied the growth of 5,5'-bis(naphth-2-yl)-2,2'-bithiophene (NaT2) thin film on the graphene template. NaT2 belongs to the family of oligothiophenes, which are extensively studied for their exceptional charge carrier mobilities. NaT2 consists of an oligothiophene conjugated core that is end-capped by naphthalene groups to enhance the molecular packing in a thin film. We deposited the NaT2 molecules onto a Si/SiO₂ substrate with a graphene growth template on top by OMBD. The structure of prepared thin films was studied by GIWAXS technique. One of the substrates used had unintentional cracks in its graphene template layers, as shown by helium-ion microscopy. In these cracks, the bare Si/SiO₂ substrate was exposed. GIWAXS patterns showed a plethora of pronounced Bragg peaks that result from a well-ordered thin film structure. After the data analysis and unit cell structure calculation, we were able to distinguish between three distinct molecular configurations of NaT2 molecules. Most of the observed Bragg reflections belong to the lying-down configuration of NaT2 molecules. Interestingly, we observed two slightly different orientations of the lying-down molecules. Both of these configurations have an identical unit cell structure. Only a slight difference in orientation with respect to the substrate surface was observed, namely that one structure was rotated by 22° around the *b*-axis when compared to the other observed structure. On the other hand, in cracks with missing growth template to control the orientation, we observed the third distinct orientation. On the exposed Si/SiO₂ substrate, NaT2 molecules grow in the standing-up configuration. This is common for molecules grown on substrates, where substrate-molecule interaction is miniscule. Sharp Bragg spots in GIWAXS pattern suggest that there is no gradual transition between the configurations. After deposition of NaT2 molecules on the second substrate a defect-free graphene template, we still observe both lying-down configurations. The standing-up configuration was absent, which confirms that it was only present on the exposed substrate surface. However, two different lying-down configurations were still present. While their same unit cell parameters suggest the identical molecular packing in a unit cell, the fact that one is slightly rotated might influence the charge carrier transfer at domain boundaries.

We could not determine the reason for the different lying-down configuration. One possible explanation might be the thickness-dependent orientation. Hosokai *at al.*(21) in their work showed the presence of two distinct polymorphs of picene thin films when grown on Si/SiO₂ substrate. Employing different incidence angles during the GIWAXS measurements, they were able to determine the structure at different depths of a thin film. They concluded that depending on the film thickness, the unit cell changes from one polymorph to another. In our case, there is no change of unit cell structure between the two observed lying-down configurations, only a slight orientation shift. This can be explained by template-molecule and molecule-molecule force interactions. At the beginning of the thin film growth, there is a strong vdW force controlling the orientation of the molecules. After the first few layers are fully closed, the molecule-molecule interactions become dominant, slightly changing the orientation of the later NaT2 layers.

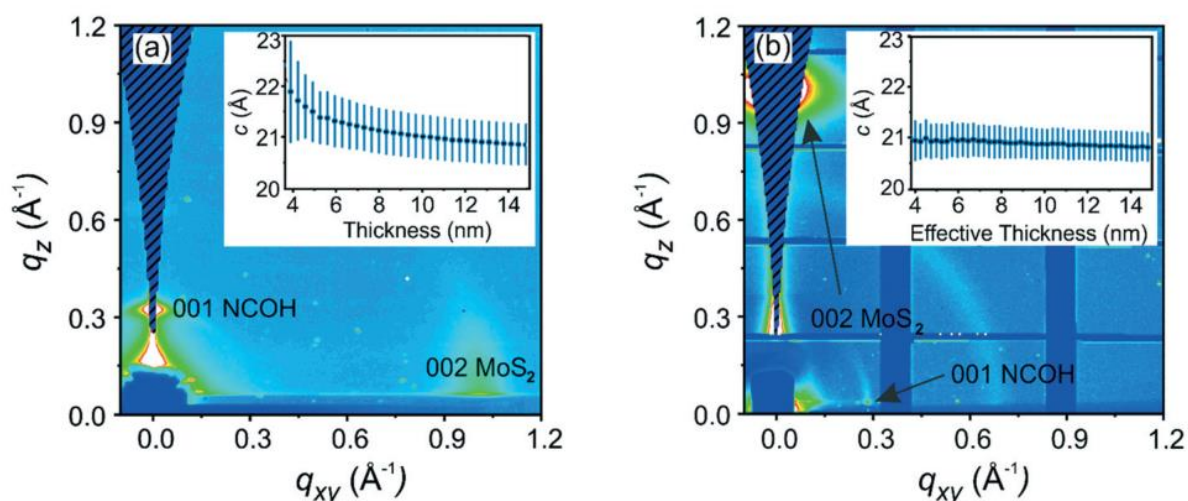


Fig. 3: GIWAXS pattern of NCOH molecules grown on a) vertically b) horizontally aligned MoS₂ growth template.

Summary

The main objective of this work was to prepare and study thin organic films that can be used as an active layer in organic electronic devices. Since the orientation of molecules in a thin film have a significant impact on the performance of electric devices, the possibility of controlling this orientation by using growth templates was investigated.

Thin films were prepared by an organic molecular beam deposition method. OMBD is an ideal method for the preparation of thin, well-ordered organic films with a controllable thickness.

Thin film structure was investigated by GIWAXS measurements. From GIWAXS patterns, I was able to determine the crystallinity of a thin film, its orientation with respect to the substrate and unit cell structure.

Predominately, I focused on the few-layer MoS₂ growth templates to control the orientation of prepared organic thin films. Depending on parameters during the preparation, it is possible to prepare a few-layer MoS₂ template with two distinct crystal orientations. Van der Waals interaction between the template and deposited molecules then controls the orientation of a prepared thin film. When thin films were deposited on the top of a horizontally aligned MoS₂ template, I observed a lying-down configuration of organic molecules. The reason for this is a relatively strong interaction between the template and molecules of the first monolayer. *In situ* GIWAXS measurements during the thin film growth show a progression of slight strain at the early stages of deposition. This is a result of a small mismatch between the template and thin film structure. However, the rapid relaxation of this strain after only a few layers allows the thin film to continue growing with the desired orientation. This would not be the case if the template and thin film structure mismatch was significant, resulting in a disordered thin film. On the other hand, using a vertically aligned MoS₂ template, I observed a standing-up configuration of a thin film. This proves the feasibility of using the MoS₂ layer as a growth template.

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