

## Protocol



# Electrochemical molecular intercalation and exfoliation of solution-processable two-dimensional crystals

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## Abstract

Electrochemical molecular intercalation of layered semiconducting crystals with organic cations followed by ultrasonic exfoliation has proven to be an effective approach to producing a rich family of organic/inorganic hybrid superlattices and high-quality, solution-processable 2D semiconductors. A traditional method for exfoliating 2D crystals relies on the intercalation of inorganic alkali metal cations. The organic cations (e.g., alkyl chain-substituted quaternary ammonium cations) are much larger than their inorganic counterparts, and the bulky molecular structure endows distinct intercalation and exfoliation chemistry, as well as molecular tunability. By using this protocol, many layered 2D crystals (including graphene, black phosphorus and versatile metal chalcogenides) can be electrochemically intercalated with organic quaternary alkylammonium cations. Subsequent solution-phase exfoliation of the intercalated compounds is realized by regular bath sonication for a short period (5–30 min) to produce free-standing, thin 2D nanosheets. It is also possible to graft additional ligands on the nanosheet surface. The thickness of the exfoliated nanosheets can be measured by using atomic force microscopy and Raman spectroscopy. Modifying the chemical structure and geometrical configuration of alkylammonium cations results in different exfoliation behavior and a family of versatile organic/inorganic hybrid superlattices with tunable physical/chemical properties. The whole protocol takes ~6 h for the successful production of stable, ultrathin 2D nanosheet dispersion in solution and another 11 h for depositing thin films and transferring them onto an arbitrary surface. This protocol does not require expertise beyond basic electrochemistry knowledge and conventional colloidal nanocrystal synthesis and processing.

## Key points

- A layered semiconductor crystal that undergoes electrochemical intercalation with alkyl chain-substituted quaternary ammonium cations can be exfoliated into free-standing 2D nanosheets by sonication. The resulting 2D nanosheets in solution are spin-coated to form thin films on versatile substrates for electronic applications.
- The main advantage is the preservation of semiconducting characteristics (e.g., MoS<sub>2</sub>) in the exfoliated nanosheets and assembled thin films.

## Key references

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# Two Dimensional Crystals

**Xu Zhang (Ph. D.)**



## **Two Dimensional Crystals:**

**Two-Dimensional Crystals** A. G. Naumovets, 2012-12-02 This is a self contained tutorial introduction to two dimensional crystal science and technology Including concise descriptions of experimental methods and results from fundamental theoretical concepts this book covers a broad range of two dimensional structures from overlayers to freestanding films All those with an active interest in surface science and statistical physics will find this book to be an essential reference work Presents a coherent overview of experimental methods and theoretical background of two dimensional crystal physics Provides a tutorial overview of continuous melting of two dimensional crystals roughening transitions wetting phenomena and commensurate incommensurate transitions

**Two-dimensional Crystals** Xu Zhang (Ph. D.), 2017 The success in creating atomically thin and mechanically robust two dimensional 2D crystals starting with graphene has unveiled new possibilities for next generation of ultrafast and ubiquitous electronics One critical distinction between 2D crystals and 3D crystals is that 2D crystals are all surface materials Therefore it is essential to understand how 2D materials interact with their environments and how this interaction impacts their electronic properties From a practical perspective it also provides us with a unique tool to tailor the electronic properties of 2D materials through surface functionalization In the first half of this thesis a suite of X ray techniques is used to investigate how the surface functionalizing dopants will impact the electronic and chemical states of graphene Based on this study we develop an effective and non invasive doping method for graphene through plasma based chlorination In order to make system level 2D electronics successful a flexible and ubiquitous energy harvesting solution is indispensable Therefore the second part of this thesis is dedicated to the development of a MoS<sub>2</sub> 2H 1T phase heterojunction based GHz flexible rectifier as an enabling component for wireless energy harvester It is the first flexible rectifier operating up to the X band and it covers most of the unlicensed industrial scientific and medical ISM radio band including the Wi Fi channels By integrating this rectifier with an antenna the MoS<sub>2</sub> enabled rectenna successfully demonstrates direct energy harvesting of electromagnetic EM radiation in the Wi Fi band and lights up a commercial light emitting diode LED with zero external bias battery free Moreover our MoS<sub>2</sub> rectifier also realizes successful frequency conversion as a mixer beyond 10 GHz on flexible substrates This work provides a universal energy harvesting building block that can be integrated with various wearable electronic systems and paves the way towards using the existing Wi Fi infrastructure as an energy hotspot for wireless charging

**Superconductivity in Two-Dimensional Crystals** Mohammed El-Bana, 2015-09-07 This book would be exciting to people that their interests broadly encompass condensed matter physics as a whole but lie in particular in the area of graphene like 2D materials and in the area of superconductivity The discovery of graphene opens up the possibility of new and exciting physics in a new class of materials It was exhibited that single layer graphene can carry significant proximity supercurrents Fabrication of graphene Josephson junctions enabled me to study the rare intersection of relativity and superconductivity This initial work was extended to investigate 2D superconducting

crystals of NbSe<sub>2</sub> Our developed new mechanical exfoliation protocol enabled fabrication and measurement of superconductivity in several 4 point NbSe<sub>2</sub> FETs devices Results obtained on both graphene NbSe<sub>2</sub> FETs were encouraging and naturally suggest a number of extensions to this work I would be excited to try and explore the combination of graphene and other 2d crystals by fabricating and characterizing such innovative prototype hybrid structures of these graphene like 2D materials I do believe that this research could play an important role in exploring new physics and device applications

Fabrication and Physical Properties of Novel Two-dimensional Crystal Materials Beyond Graphene: Germanene, Hafnene and PtSe<sub>2</sub> Linfei Li,2020-01-03 This thesis reports on essential experimental work in the field of novel two dimensional 2D atomic crystals beyond graphene It especially describes three new 2D crystal materials namely germanene hafnene and monolayer PtSe<sub>2</sub> fabricated experimentally for the first time using an ultra high vacuum molecular beam epitaxy UHV MBE system Multiple characterization techniques including scanning tunneling microscope STM low energy electron diffraction LEED scanning transmission electron microscope STEM and angle resolved photoemission spectroscopy ARPES combined with theoretical studies reveal the materials atomic and electronic structures which allows the author to further investigate their physical properties and potential applications In addition a new epitaxial growth method for transition metal dichalcogenides involving direct selenization of metal supports is developed These studies represent a significant step forward in expanding the family of 2D crystal materials and exploring their application potentials in future nanotechnology and related areas **Frequency Distribution of Two-dimensional Crystals** Charles Moore Askey,1951

**Two-dimensional Crystallization of Macromolecular Complexes** Hans Oliver Ribi,1987 *Probing the Response of Two-Dimensional Crystals by Optical Spectroscopy* Yilei Li,2015-11-09 This thesis focuses on the study of the optical response of new atomically thin two dimensional crystals principally the family of transition metal dichalcogenides like MoS<sub>2</sub> One central theme of the thesis is the precise treatment of the linear and second order nonlinear optical susceptibilities of atomically thin transition metal dichalcogenides In addition to their significant scientific interest as fundamental material responses these studies provide essential knowledge and convenient characterization tools for the application of these 2D materials in opto electronic devices Another important theme of the thesis is the valley physics of atomically thin transition metal dichalcogenides It is shown that the degeneracy in the valley degree of freedom can be lifted and a valley polarization can be created using a magnetic field which breaks time reversal symmetry in these materials These findings enhance our basic understanding of the valley electronic states and open up new opportunities for valleytronic applications using two dimensional materials **The Structure of Two-dimensional Crystals Residing at the Air-Solution Interface** Sharon Grayer,1991

Electronic Properties of Two-dimensional Crystals Under Strain Fields Elena Blundo,2019 Influence of PH on Two-dimensional Crystals of Streptavidin Michael Todd Yacilla,1998 **Discovery** ,1920 *Growth of Crystals* A. V. Shubnikov,N. N. Sheftal',Aleksē Vasil'evich Shubnikov,1969 **Structure, Symmetry, and Stability of**

**Two-Dimensional Crystals** Katherine Elaine Plass,2006 *Mineralogical Abstracts* ,1925 **Crystallography and Practical Crystal Measurement** Alfred Edwin Howard Tutton,1911 **Soviet Physics, Crystallography** ,1989 **Science Reports** Tōhoku Daigaku,1925 **Proceedings of the Royal Society of London** Royal Society (Great Britain),1929 Publishes research papers in the mathematical and physical sciences Continued by Proceedings Mathematical and physical sciences and Proceedings Mathematical physical and engineering sciences **Government Reports Announcements & Index** ,1988 **Two Dimensional Crystals and Nanostructured Materials** ,1998 New organic amphiphiles were designed with cross sectional shapes designed for the formation of crystalline monolayers that would conform to heretofore undiscovered two dimensional packings The viability of the molecules was tested by atom atom potential calculations Those systems that were deemed promising for formation of new crystalline monolayers were subsequently synthesized thereby creating new types of amphiphiles Further examination of the crystalline monolayers was provided by determination of surface isotherms and atomic force microscopy A racemic mixture of a tetracyclic alcohol was shown to undergo chiral phase separation upon transfer to a mica substrate A trinorborane derivative showed within experimental error the first static hexagonal packing of a monolayer Molecules based on the tetracyclic alcohol exhibited surface pressures approaching that of water These films do not appear to collapse at ambient temperature but demonstrate a hysteretic return to nearly the original low pressure areas after collapse

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