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2018/5 123 (MM) (DD)

JSPS Fellow's Signature (Handwritten only) :

Research Report (by Fellow) (Cover Page)

I hereby submit the research report of my fellowship.

1	. Name (Print) :	Junhao Lin
12	2. Nationality :	Chinese
/ 3	3. Host Institution : <u>Na</u>	ational Advanced Institute of Science and Technology (AIST)
7.4	4. Host Researcher :	Dr. Kazutomo Suenaga
/ 5	5. Title of Research in Japan Materials under Excitations	
/ 6	5. Fellowship Tenure : From	m <u>2016 / 09 / 01</u> To <u>2018 / 04 / 30</u> (YYYY) (MM) (DD) (YYYY) (MM) (DD)

*Notes for writing the Research Report

*Type this form except the date and the signature.

Please prepare your Research Report in English or Japanese within three to ten pages including this page. The contents should include:

7. Background of Research

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Motivated by the discovery of graphene, diversified layered materials where layers are bonded by weak van der Waal interactions have been recently revisited profoundly. Similar to graphene that shows superior physical properties, novel physical phenomena also emerge in layered materials when they are peeled down to one molecular layer, the so-call two dimensional (2D) monolayer materials. For instance, monolayer MoS₂ was discovered to have surprisingly high photoluminescence response, which is trivial in the bulk counterpart. This is due to the emerging direct bandgap in the electronic structure benefited from the reduced interlayer coupling. The mechanical exfoliation method was then applied to various layered materials to produce a wide range of 2D materials, including most of the TMDCs and layered oxides. Many novel physical properties have been discovered in these 2D monolayers, such as enhanced charge density wave transition, Ising superconductivity and etc, making 2D materials fascinating to a wide range of applications including nanoelectronic and optoelectronic.

It was recognized from the early studies that the low yield and small flake size are the fatal shortcomings of the mechanical exfoliation method, which limit its use only to demonstration of fundamental physics and prototype devices. Aiming for practical applications of 2D materials, large scale chemical synthesis methods were developed in parallel. Common methods like chemical vapor deposition (CVD) and molecular-beam-epitaxy (MBE) growth have been demonstrated in the growth of MoS₂ atomic layers, which were later also reported for the growth of other 2D materials. Nevertheless, chemically grown 2D materials inevitably incorporate various structural defect, such

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as vacancies, dislocations and grain boundaries. These defects can affect the physical properties of the materials severely. For instance, vacancies are usually considered to be the scattering center for carriers leading to a low mobility in 2D semiconducting monolayer, whereas the types and structure of the vacancies play dramatically different role in affecting the transport properties of the materials. Another example is the grain boundary. Depending on the structure of how the two domains are interconnected, the electronic structure of the system varies substantially. Because the physical properties of 2D materials can be profoundly influenced by the presence of structural defects, understanding the atomic structure of these defects, therefore, is of fundamental importance to the research of 2D materials and the related applications.

Moreover, defects in 2D materials can constantly be excited by other energy source like electron beam. Supplying constant excitations to 2D materials and studying the related dynamical processes in an electron microscope offers unparalleled opportunities to connect atomic structures to properties in a dynamic way. It provides valuable information on the structural dynamics, bonding and chemical changes of 2D materials under excitations. Revealing these dynamical process via insitu investigations helps to understand the performance changes in the materials, e.g., how a material functions and degrades under constant electrical biasing or light illuminations. Understanding the property changes correlated with the defect evolution provides direct feedback for the material design, synthesis and application. Therefore, in situ study of defects in 2D materials is indispensable in exploring the various applications of 2D materials.

8. Research methodology

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We aim to study structure-property correlation of defects in 2D material comprehensively, also for their dynamical change under external excitation. The atomic structure of the defects is the essential topic in this project, most of which are characterized by aberration corrected transmission electron microscopy (TEM) and scanning TEM (STEM). Aberration is caused by the imperfectness of the magnetic lens which spreads the beam at the focal plane forming a blurred spot. Modern aberration corrector now can fix up to the 5th order of the aberration in the microscope, reaching a special resolution of 0.43A at 300kV. Specially, atomic-resolution incoherent STEM annular dark field (ADF) imaging is often applied for the direct identification of the defect structures since the intensity is directly interpretable. The image intensity is proportional to the atomic number of the imaged atom; thus, based on the atomic intensity, the image provides direct information on the local atomic configuration and chemical composition of the material. In this project, the acceleration voltage is much lower than the standard which is lowered to 60kV, reducing the knock-on damage which enables the imaging of monolayer materials. Moreover, exciting the evolution of defects and simultaneously imaging the dynamical process can be also realized. The dynamical process can be captured atom-by-atom, where the dynamical results are explained and compared with the density functional calculations (DFT).

9. Results/impacts

In the 2-year support from JSPS (actually 20 months), my research mainly focuses on the structureproperty correlations in 2D materials *via* the combination of electron microscopy and theory, which consist of several independent projects. I will review the progress and show some representative projects and papers that have high impact in the community.

The most important work starts from a collaboration with the Nanyang Technology University in Singapore. As a starting platform for performing in-situ experiments in various 2D materials, mostly in 2D transition metal dichalcogenides (TMDCs), it is urgent to develop a facile method to prepare various TMDCs 2D samples. However, the current existing methods can only produce a few of them, such as Mo- and W-based TMDCs, which are synthesized via sulfurization, selenization and tellurization of metals and metal compounds. It is difficult to produce many other TMDCs because of the high melting points of their metal and metal oxides precursors. We developed a molten salt-assisted method chemical vapor deposition method for synthesizing a 2D TMDCs library of 47 compounds, including 32 binary (Ti-, Zr-, Hf-, V-, Nb-, Ta-, Mo-, W-, Re-, Pt-, Pd- and Fe-based), 13 alloys (including 11 ternary, 1 quaternary and 1 quinary), and 2 heterostructured compounds. Through the STEM study of various TMDCs samples (Fig. 1), we elaborate the general growing

mechanism of this method and demonstrate that the salt decreases the melting point of reactants and facilitates the formation of intermediate products, guaranteeing the successful reactions. This research paves the way for the multidisciplinary exploration of 2D TMDCs across materials science, physics and various applications, which may result in applications in ultrathin electronic devices, atom-thin superconductor and etc, and more importantly, laid down the foundation for the defect study in various TMDCs through high resolution STEM. This work is published in *Nature* during the supported period (*Nature*, **556**, 355 (2018)).



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Figure 1 | Upper panel: Overview of metals (highlighted in purple) and chalcogens (highlighted in yellow) that can form layered sulfides, selenides and tellurides. Lower panel: Atomic-resolution STEM images of representative monolayer materials in different phases. (a-d), MoS_2 in the 1H phase, $PtSe_2$ in the 1T phase, WTe_2 in the 1T'phase and ReSe₂ in the 1T"phase, with the corresponding FFT patterns and atomic structural models, respectively.

We then studied the defect structures in these TMDCs monolayer through the STEM microscopy. Point defects are the simplest and most abundant types of defects in 2D TMDC materials. 1H phase MoS₂ is a representative monolayer material in the TMD family, and the point defects have been well studied in this material system. Most point defects can be categorized into vacancy and antisite defects. In CVD-grown MoS₂, intrinsic vacancies and anti-site defect presented in either cation and anion site. The formation of these vacancies depends on the growth environment and also the growth method. This is explained by the formation energy of these defects under different chemical potential used in the calculations. For instance, the formation energy of V_S is much lower in a sulphur deficient environment than under sulphur rich conditions, i.e. high concentration V_S is expected if the sample is grown in a Mo-rich condition. The point defect structures found in MoS₂ monolayer (Fig. 1a) could be generalized to other TMDC monolayer materials of 1H phase. In addition, chalcogen vacancy have also been observed in 1T PtSe₂ (Fig. 1b), 1T' phase WTe₂ (Fig. 1c), 1T'' and ReSe₂ (Fig. 1d), while these materials can be easily damaged under electron beam irradiation. To the best of our knowledge, only MoS2, MoSe2, WS2, WSe2 and MoTe2 in 1H phase and ReS2 and ReSe2 in the 1T" phase are stable under ambient conditions, while other TMDC monolayers are prone to oxidation during the sample preparation process before electron microscopy characterization. One of my work during the support of JSPS is that we tried to sandwich the air-sensitive NbSe₂ monolayer, a superconductor material crystalized in the 1H phase, within two layers of graphene to slow down the sample degradation. It could be seen from the lowmagnified STEM image shown in Fig. 2a that, though oxidation still occurs, the pristine triangular morphology preserves and the atomic structure of the defects can be studied. It is notable that the overlaid of graphene does not affect the quality of the imaging due to its light atomic weight. It is confirmed that intrinsic monoselenium vacancy, diselenium vacancy and anti-site defects are also presented in the CVD-grown NbSe₂ monolayer (see Fig. 2b), similar to the ones observed in MoS₂ (Fig. 1a). These point defects, however, do not seem to affect the superconductivity of the CVD-grown NbSe₂ monolayer. This study indicates that graphene protection could serve as a new strategy to study the intrinsic defect structure in air-sensitive TMDC materials. This work is published in *Nature Communications* where I shared the primary contributions with the other collaborators (*Nature Communications*, 8, 394 (2017)).



Figure 2 | (a) Low-magnified STEM image of graphene sandwiched air-sensitive monolayer NbSe₂. (b) Atomic STEM image of Se vacancy and the anti-site defects.

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Chalcogen vacancy in TMDC is particular important since its aggregation can lead to large structure reconstruction, including interlayer fusion. We studied another new TMDC materials which have a chemical stoichiometry of PdSe₂. It is commonly known that the monolayer form in layered TMDC is typically the same as a single layer of the bulk material. However, the PdSe₂ monolayer has been theoretically shown to be stable, but there have been no reports that monolayer PdSe₂ has been fabricated. We found that the preferred monolayer form of this material amounts to a melding of two bulk monolayers accompanied by the emission of Se atoms so that the resulting stoichiometry is Pd₂Se₃. The Se vacancy play a key role in the two-to-one layer fusion process by pulling the two layers together due to the unsaturated bonding of Pd atoms, which fonds to bond with the dangling Se atom in the other layer. We further show that this new 2D monolayer can be directly fabricated in situ from the PdSe₂ matrix by using the microscope's electron beam. This work provides a first example of an unprecedented way to produce a new monolayer phase by means of controlled modification of stoichiometry (defect engineering) and novel interlayer reconstruction of layered structures. We published such new physical picture of defects induced reconstruction in layered materials in prestigious physical journal of Physical Review Letters (Physical Review Letters, 119, 016101 (2017)).

Another large group of point defect in TMDC materials is substitutional dopant. It is possible to replace either the cation or anion sites in TMDC monolayer with foreign atoms to tune the properties of the materials. Effort has been put in doping monolayer MoS₂ with various elements in achieving enhanced performance towards different applications. Considering the electronic configuration of Mo, transition metals with similar d orbitals are more feasible to be incorporated into the lattice due to similar ionic size, electronegativity and valence. Various transition metal atom has been theoretically considered by DFT calculations to replace the Mo atom in MoS₂. The results show that the binding energy between sulphur columns and transition metal atoms is quite similar to the pristine MoS₂ lattice. We have successfully doped the MoS₂ monolayer and replace Mo atoms with Fe and Co atoms. However, these dopants forms a strange patterns where Fe or Co atoms prefer to form clusters which substitute three Mo atoms in a triangular shape, with a sulphur vacancy in the middle. Such structure maintains the lower formation energy than single substitution. These results are unpublished and still in the process of correlating to the magnetic properties of the monolayer materials.

When doping the MoS₂ lattice with large quantity of foreign atoms, the monolayer material becomes a 2D alloy. 2D Alloy is a promising candidate in electronic application since the electronic structure

can be finely tuned by the alloy concentration. The similarity in the electronic configuration between Mo and W, as well as S and Se, makes it easier to form 2D alloys with combinations of these four elements. The first 2D monolayer alloys experimentally demonstrated are $Mo_xW_{1-x}S_2$ and $MoS_{2x}Se_{2(1-x)}$ in 1H phase. The Mo and W atoms in the 2D $Mo_xW_{1-x}S_2$ alloy was found to distribute randomly in the cation sublattice without any ordering. Similar random distribution was in the $MoS_{2x}Se_{2(1-x)}$ alloy, and quaternary alloy $Mo_xW_{1-x}S_{2y}Se_{2(1-y)}$. 2D alloy with phase transition is also achieved, where the phase is determined by the alloy concentration. Successful demonstrations include anion site alloys such as $WSe_{2x}Te_{2(1-x)}$, with a phase transition from 2H to 1T' at Te concentration around 50%-70%, whereas the sample crystalizes in pure 1H below 50% Te content but in 1T' phase when Te concentration where the phase transition occurs, and indeed confirms there is no phase aggregation happens in a single flake of the sample. This result is first published in *Advanced Materials*, 29, 1603991 (2017)).

A follow-up study of this Te-based alloy 2D materials reveals the profound anisotropic ordering of the anion sites in the 1T' phase. When the alloy is mixing with sulphur or selenium with Te, due to the anisotropic bonding in the two anion sites of the 1T' phase and the ionic sizes of the alloyed atoms, the sulphur or selenium atoms seems not distributed isotopically. It is found in a statistical method that over 95% of the sulphur or selenium atoms occupy the anion sites near the metal atoms than the one away from it. Similar cation site alloy with a 2H-to-1T'' phase transition occurs in the Mo_xRe_{1-x}Se₂ alloy, where the stable phase is determined by the Re concentration, while no obvious ordering of the cation atoms, Mo and Re, is found presumably due to the isotropic alloyed behavior in 1T' phase Te-based layered alloys regardless of their alloy concentration, shining light on fine tuning their physical properties via engineering the alloyed atomic structure. It is published recently in *ACS Nano* (*ACS Nano*, **12**, 894 (2018)).

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In addition to STEM imaging of 2D materials, I also focus on the demonstration of the optical properties of single quantum object accessed by valence-loss EELS (VLEELS) in monochromatic microscope. For instance, I discovered that the quantum confinement effect in nanocrystal (NC) ensemble is deviated from isolated individual NCs, which is due to the cooperative effect between these quantum objects. This work provides the microscopic insight in the optical properties of individual NCs, paving the way for nano-optics probed by VLEELS. This work has been published in *Nano Letters* (*Nano Letters*, **16**, 7198 (2016)).

In summary, TMDC layered materials contain a rich inventory of defect structures, and we are just probing the tip of an iceberg. In the family of TMDs, similarity of defect structures can be found in materials of the same phase but consisting of different chemical elements. For example, vacancies and grain boundary structures are quite similar in 2H MoS₂ and NbSe₂, while they are dramatically different in 1T' or 1T' phases such as in WTe₂ or ReSe₂. There remains a large portion of defects in sensitive TMDCs that need sophisticate techniques to tackle with.

Knowing the structure of these defects is an essential step in understanding their effect to the physical properties of the materials on the atomic level. For instance, sulphur vacancies in MoS₂ are revealed to be the major factor that affects the mobility and contact properties. The accurate determination of the atomic structures of vacancies and related vacancy complexes contributes to the quantitative estimation of their effect to the transport properties, while serving as a guidebook for further improvement of the device performance. Combined with theoretical studies based on the defect structures, numerous examples have demonstrated that the understanding of the structure-property correlations is the fundamental way to elaborate emerging physical phenomena that arise from 2D materials. Other atomic characterization technique such as scanning tunneling microscopy (STM) can complement the structural analysis and property measurement on single defect structure. Giving the sulphur vacancy in MoS₂ again as an example, it is possible to probe the electronic states of a single defect should be probed simultaneously for a comprehensive understanding of its correlation to the local properties within the material. Understanding the way that defects interact with the properties can shed light on how to intentionally introduce them to engineer the desired properties.

even at the single atom level.

- Note: As much as possible, describe the contents and results of your research in a manner that is easily understandable to a non-specialist in your field. Provide a concrete description if (1) papers related to your work have been published in major academic journals, (2) particularly outstanding research results were achieved, or (3) patent applications have been made or other tangible outcomes achieved through the research.
- 10. Research Presentations during the period of the fellowship (Name of the conference, title, place, date)
 - (1) Enhanced Data Generated by Electrons (EDGE 2017): 8th International Workshop on Electron Energy Loss Spectroscopy and Related Techniques, Probing the band structure modification in perovskite nanocrystals by low-voltage monochromatic electron energy loss spectroscopy, Okuma, Okinawa, Japan, May 14th - 19th, 2017
 - ② ICDS 2017: 29th International Conference on Defects in Semiconductors, Defect dynamics in monolayer semiconductor, Matsue, Japan, July 31-Aug.4, 2017
 - ③ The 3rd East-Asia Microscopy Conference, Probing the band structure modification in perovskite nanocrystals by low-voltage monochromatic electron energy loss spectroscopy, Exhibition Center 2, BEXCO in Busan, South Korea, Nov. 7-10, 2017
 - ④ 2018 Materials Research Society Spring Meeting, Atomic dynamics and manipulation of defects in 2D materials using scanning transmission electron microscopy, Phoenix, Apr. 2-6, 2018
- 11. A list of paper published during or after the period of the fellowship, and the names of the journals in which they appeared (Please fill in the format below). Attach a copy of each article if available.

Author(s) Title		Name of Journal	Volume	Page	Date	Note

Equal contributors denoted with [†]. Corresponding author denoted with^{*}. High impact journals are highlighted in red.

- Jiadong Zhou[†], Junhao Lin^{†*,} Xiangwei Huang, Yao Zhou, Yu Chen, Juan Xia, Hong Wang, Yu Xie, Huimei Yu, Jincheng Lei, Di Wu, Fucai Liu, Qundong Fu, Qingsheng Zeng, Chuang-Han Hsu, Changli Yang, Li Lu, Ting Yu, Zexiang Shen, Hsin Lin, Boris I. Yakobson, Qian Liu, Kazu Suenaga, Guangtong Liu^{*} and Zheng Liu^{*}, A library of atomcally-thin metal chalcogenides, *Nature*, 556, 355 (2018)
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- 12. Awards during the period of the fellowship (Name of the award, Institution, date etc.) N/A