A perspective on the synthesis and modifications of 2D transition metal dichalcogenides by vacuum methods

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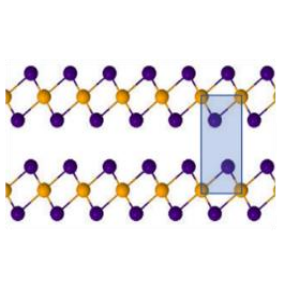
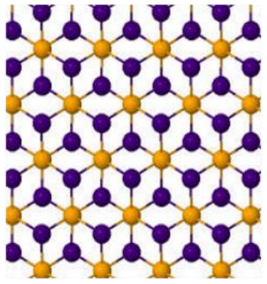
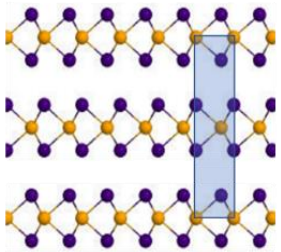
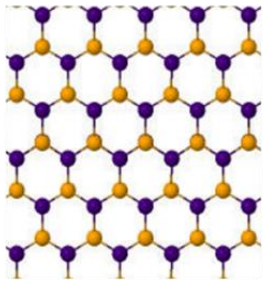
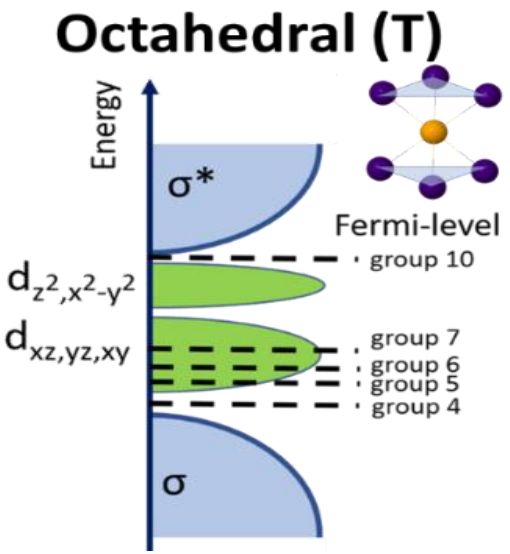
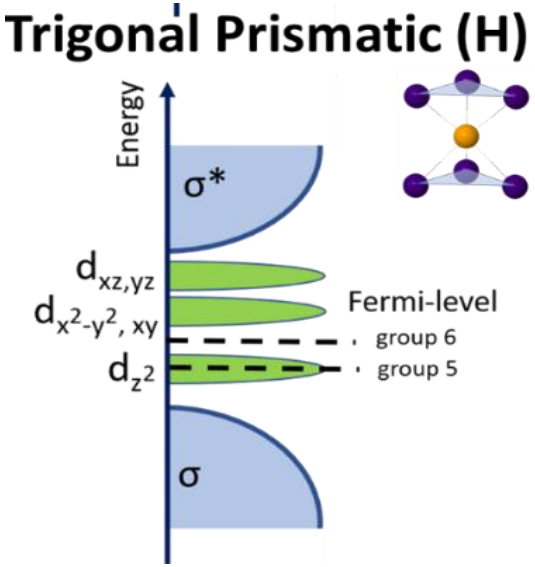
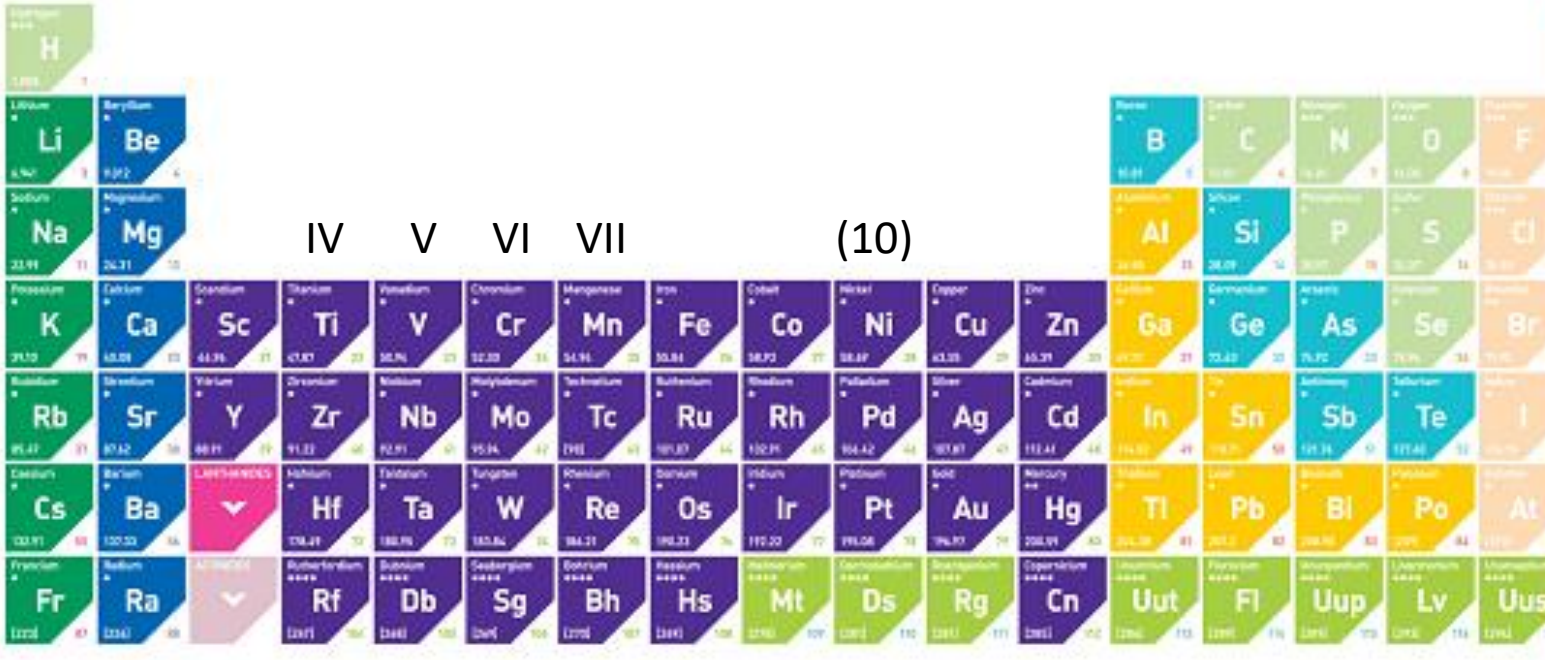
Synchrotron: Maria Asensio, Jose Avila (SOLEIL); Manuel Valvidares; Pierluigi Gargiani (ALBA)
DFT: Ivan Oleynik; Kien-N. Cong (USF); Krzysztof Zberecki (Warsaw University of Technology); Arkady Krasheninnikov (HZ Dresden)

Transition metal dichalcogenides

	Side	Top
1T		
2H		

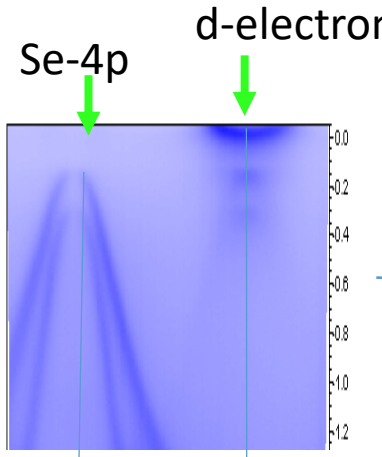


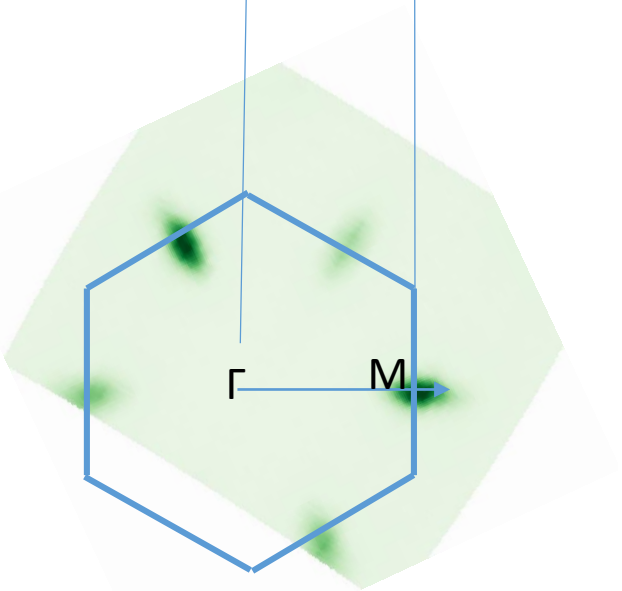


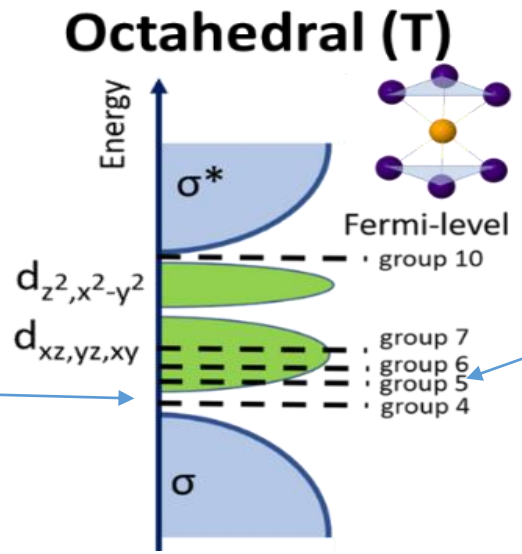


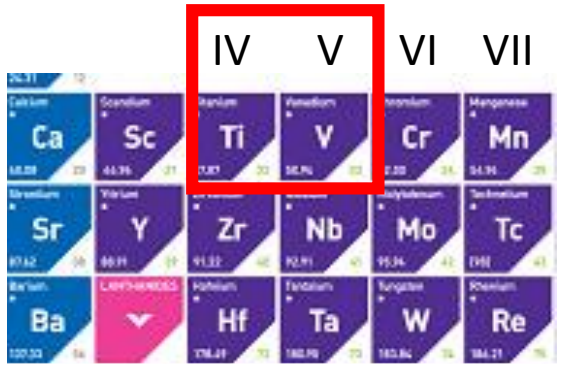
Transition metal dichalcogenides

Group IV: $TiSe_2$

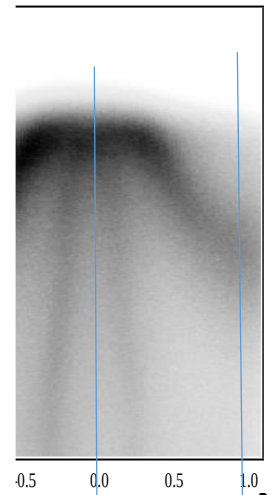


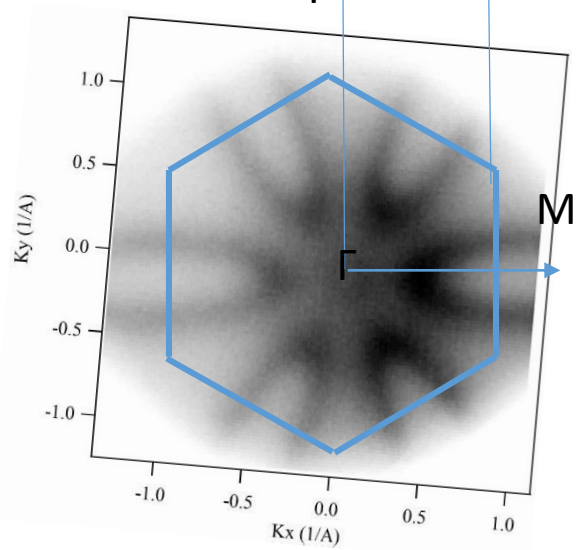






Group V: VSe_2

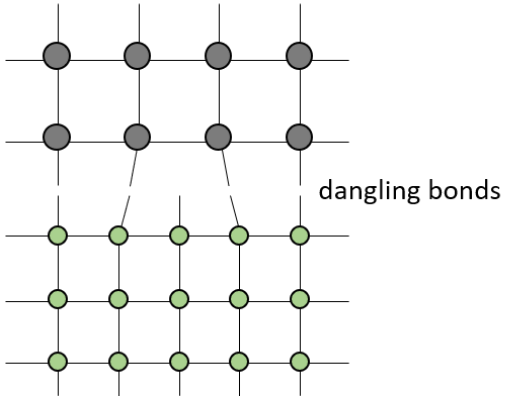




Van der Waals epitaxy

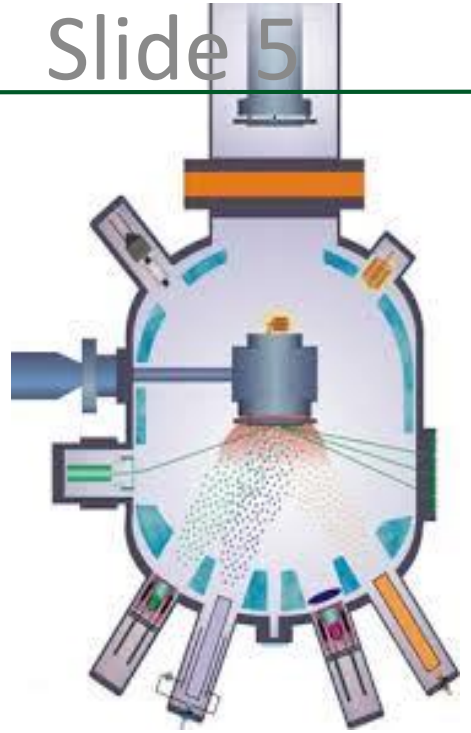
Professor Atsushi Koma, University of Tokyo (from 1984)---- http://van-der-waals-epitaxy.info/

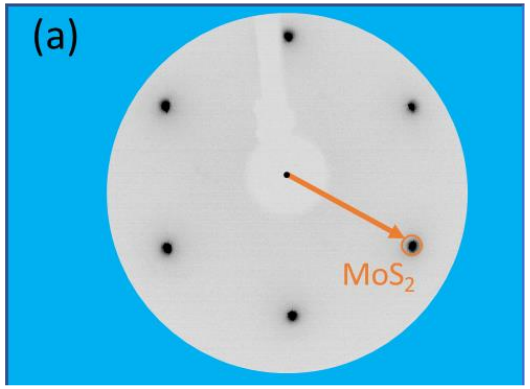
epitaxy

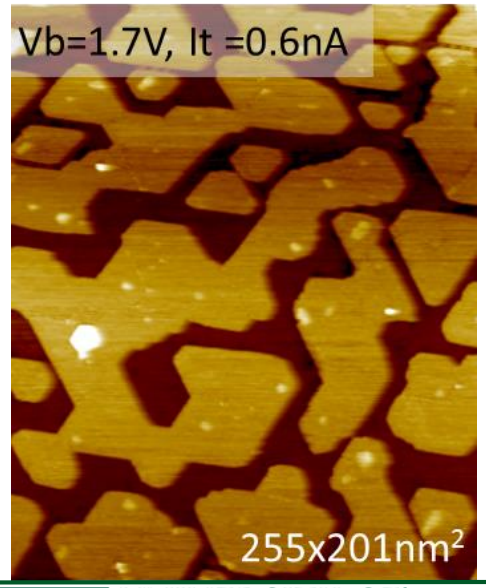


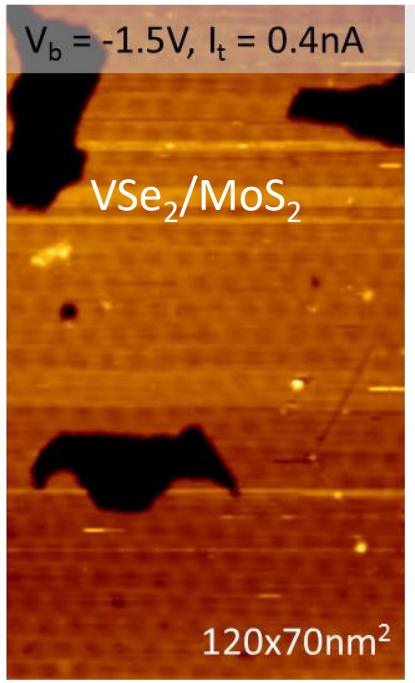
Van der Waals epitaxy

Substrates: MoS_2







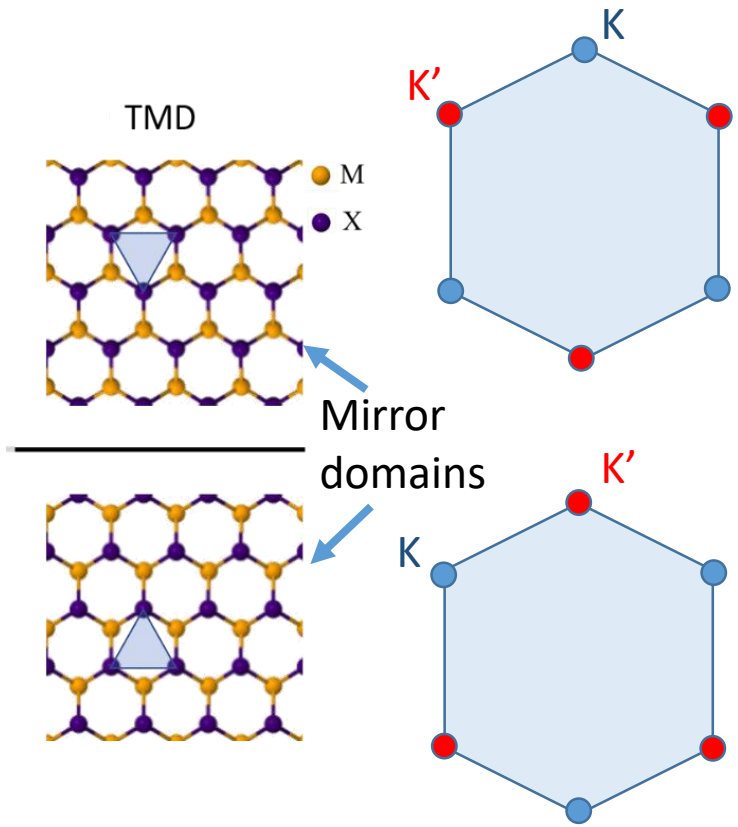


MoS_2-substrate

F. Arnold et al. 2D Mater., 5 (2018) 045009

Van der Waals epitaxy

WS_2 on Au(111)



Outline

1. VSe_2: Competition between ferromagnetic and charge density order?
2. VTe_2: A new monolayer CDW material.
3. $CrTe_2$ monolayer synthesis by van der Waals epitaxy. But is it 1T or 1H?
4. Magnetic defects and dopants: Possible diluted ferromagnetic 2D semiconductors.

Can a transition metal dichalcogenide that is paramagnetic in bulk become ferromagnetic as a monolayer?

Theory:

Evidence of the Existence of Magnetism in Pristine VX_2 Monolayers (X = S, Se) and Their Strain-Induced Tunable Magnetic Properties

Yandong Ma, Ying Dai,* Meng Guo, Chengwang Niu, Yingtao Zhu, and Baibiao Huang

School of Physics, State Key Laboratory of Crystal Materials, Shandong University, Jinan 250100, People's Republic of China.

VOL. 6 ▪ NO. 2 ▪ 1695–1701 ▪ 2012 ACSNANO

Density Functional Theory

		ferromagnetic	H vs. T structure
Y. Ma, … B. Huang	ACS Nano 6, 1695 (2012).	✓ .	T
P. Manchanda, R. Skomski	J. Phys.: Condens. Matter 28, 064002 (2016).	✓ .	H
A.H.M.A. Wasey, .. G.P. Das	J. Appl. Phys. 117, 064313 (2015)	✓ .	H
W.-Y. Tong,…C.-G. Duan	Nature Commun. 7, 13612 (2016)	✓ .	H
J. Du,…. J. Li	Nanoscale 9, 17585 (2017)	✓ .	H
M. Hester, R. Hennig, D.C. Johnson	Phys. Rev. B 96, 235147 (2017)	✓ .	T or H
H.-R. Fuh…H.-T. Jeng	Sci. Rep. 6, 32625 (2016)	✓ .	H
F.Y. Li, …Z.F. Chen	J. Phys. Chem. C *118*, 21264 (2014)	✓ .	T or H
…..		✓ .	

⇒ DFT consistently predicts a <u>ferromagnetic ground state</u> for VSe_2 (also VS_2 or VTe_2).

Magnetism in VSe_2

Exfoliated VSe2

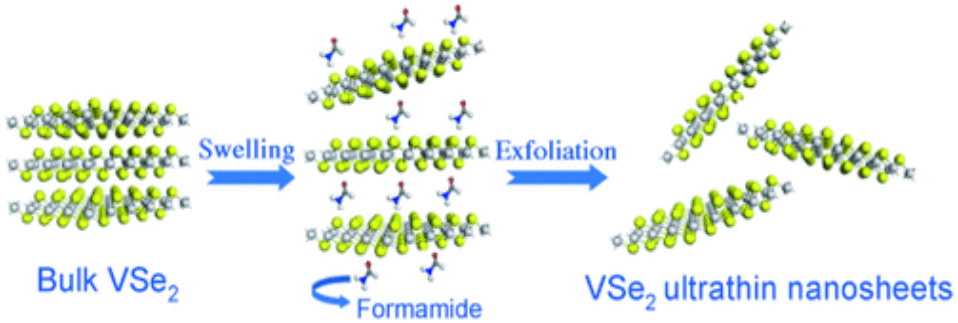
Angewandte International Edition Chemie

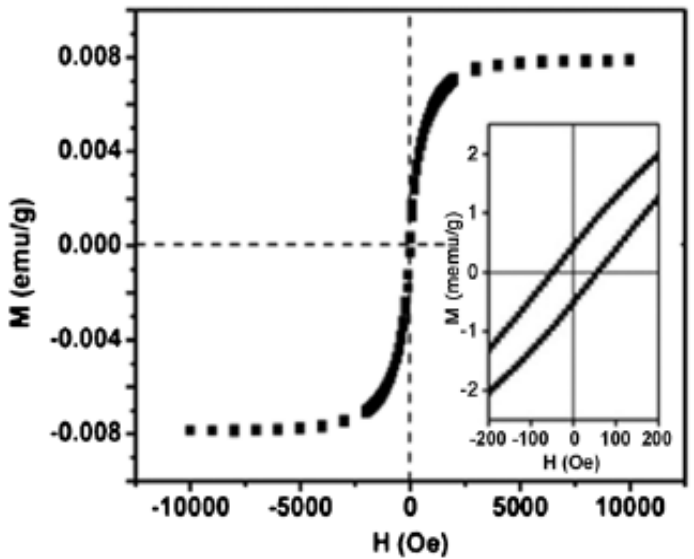
DOI: 10.1002/anie.201304337

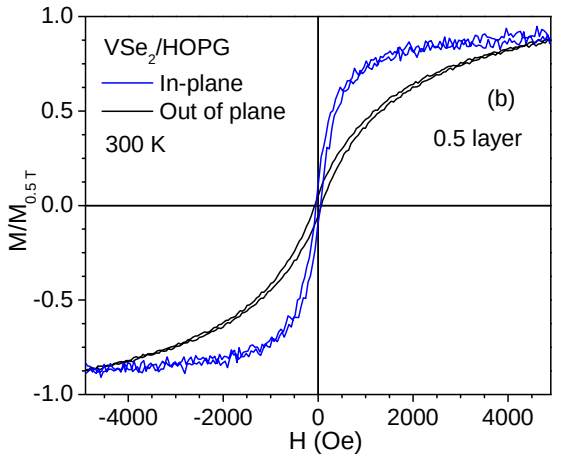
Inorganic Graphene Analogues

Ultrathin Nanosheets of Vanadium Diselenide: A Metallic Two-Dimensional Material with Ferromagnetic Charge-Density-Wave Behavior**

Kun Xu, Pengzuo Chen, Xiuling Li, Changzheng Wu, Yuqiao Guo, Jiyin Zhao, Xiaojun Wu, and Yi Xie**







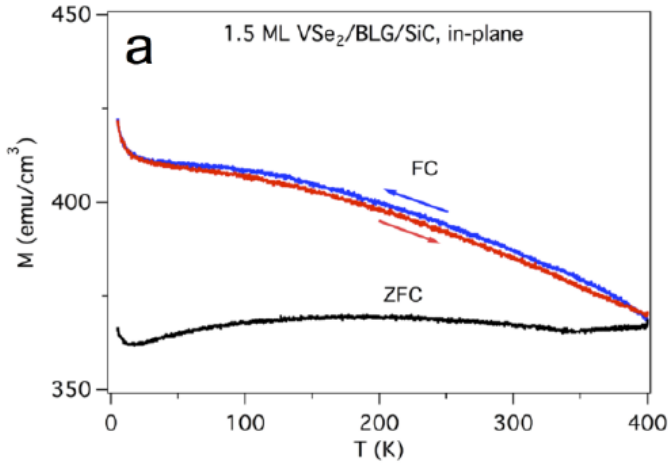
VSe_2/MoS_2 or VSe_2/HOPG

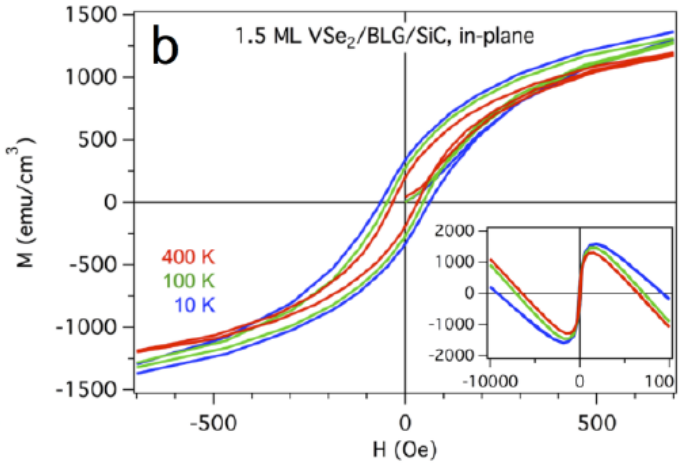
M. Bonilla… M.B. Nat. Nano. 13, 289 (2018)

VSe_2/graphene:

8. Magnetic measurements of 1.5 ML VSe_2

Nano Lett. 18, 5432 (2018)

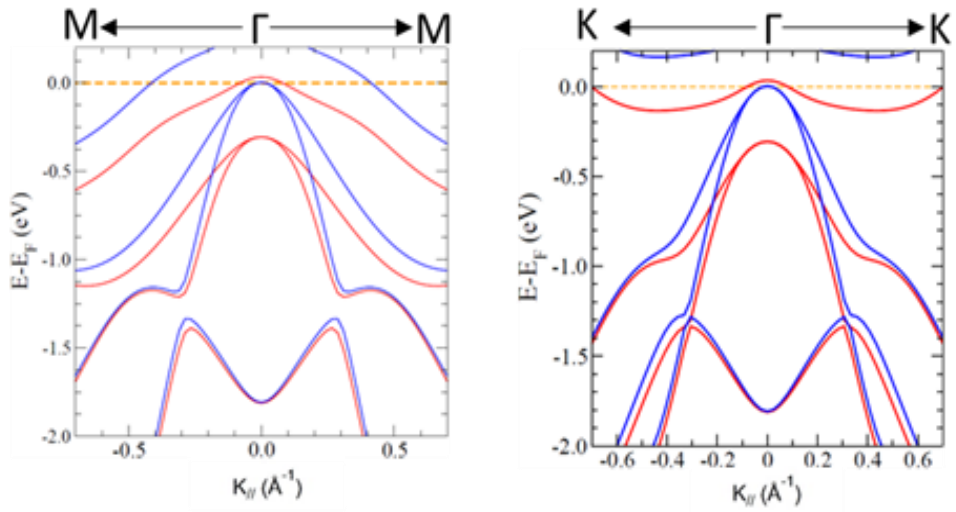




Is monolayer VSe_2 ferromagnetic?

Density Functional Theory Predictions of magnetism in VSe_2

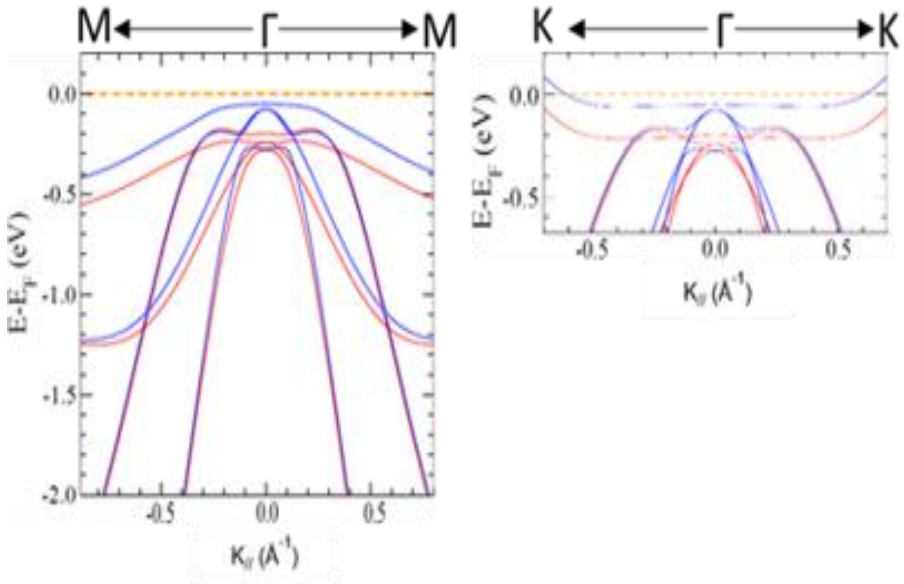
Monolayer VSe_2:



=> DFT consistently predicts an itinerant (band) magnet for monolayer VSe_2.

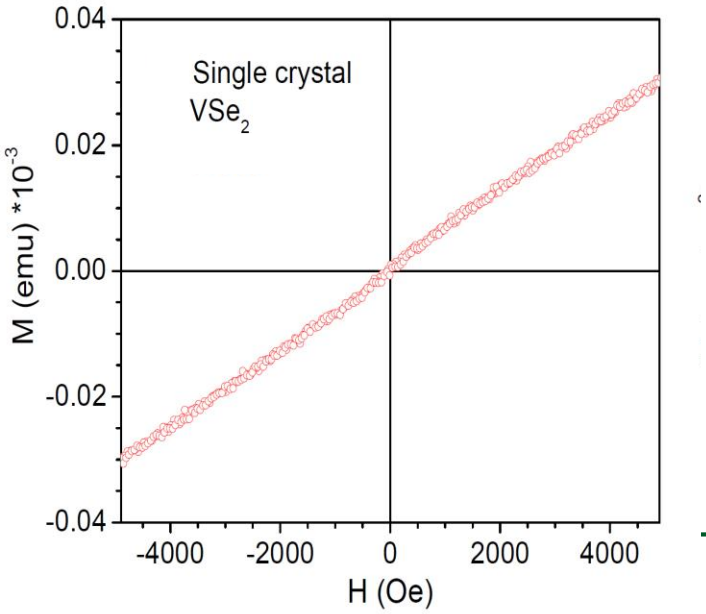
But:

Bulk VSe_2



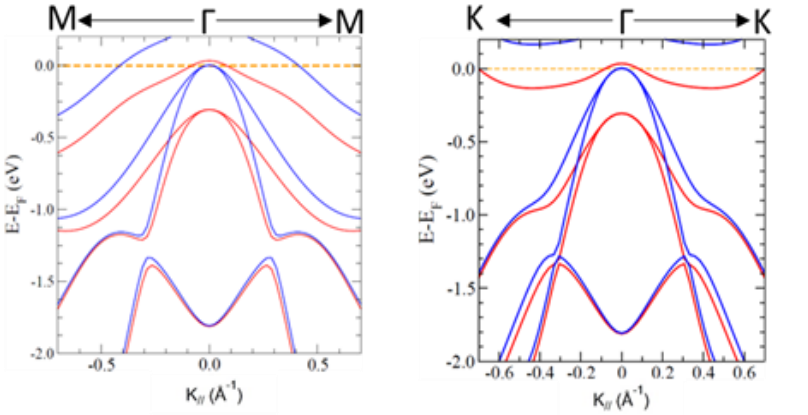
⇒ DFT also predicts an itinerant magnet for bulk VSe_2 (with smaller energy separation between minority/majority bands)

=> Contradiction to experiment? VSe_2 is paramagnetic

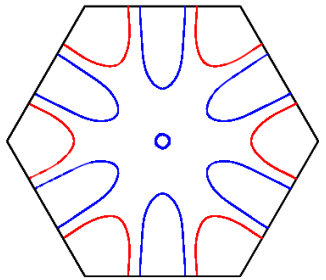


J. Phys. Chem. C 123, 14089-14096 (2019)

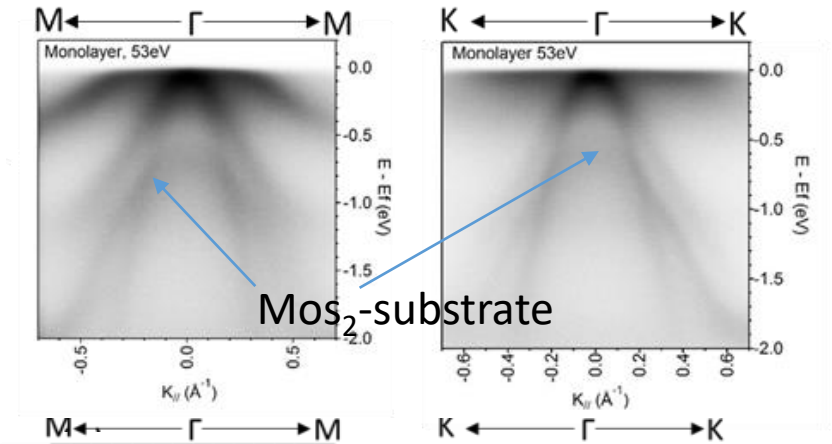
DFT comparison to ARPES

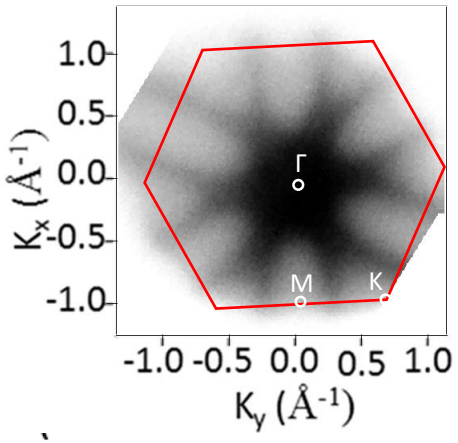


Fermi-surface

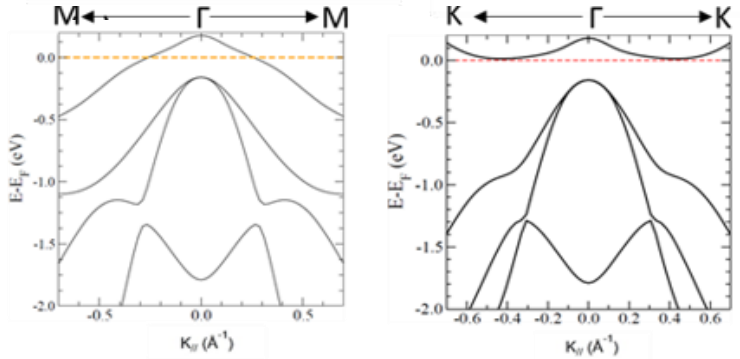


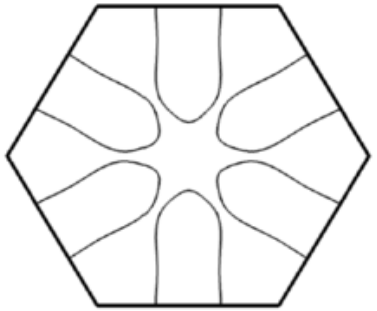
=> Spin polarized DFT predicts spin-split (majority/minority) bands (up to 500 meV).





⇒ No spin split bands are observed in ARPES.
⇒ DFT predictions are wrong, monolayer VSe_2 is not an itinerant magnet.





=> ARPES data, are well reproduced by non-spin polarized DFT. => VSe_2 is not an itinerant magnet.

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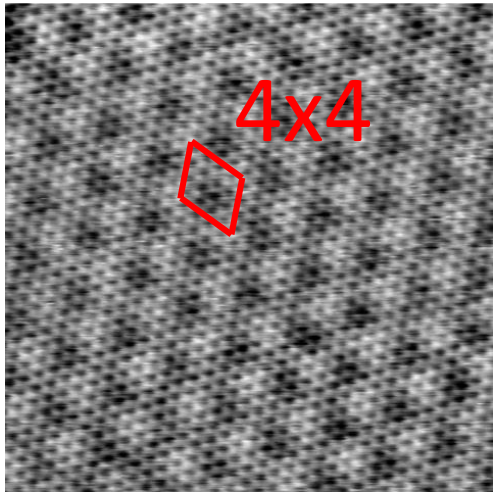
Why does DFT predict a spin-polarized ground state?

Competing ground states?

VSe_2 is known to exhibit a CDW in the bulk.



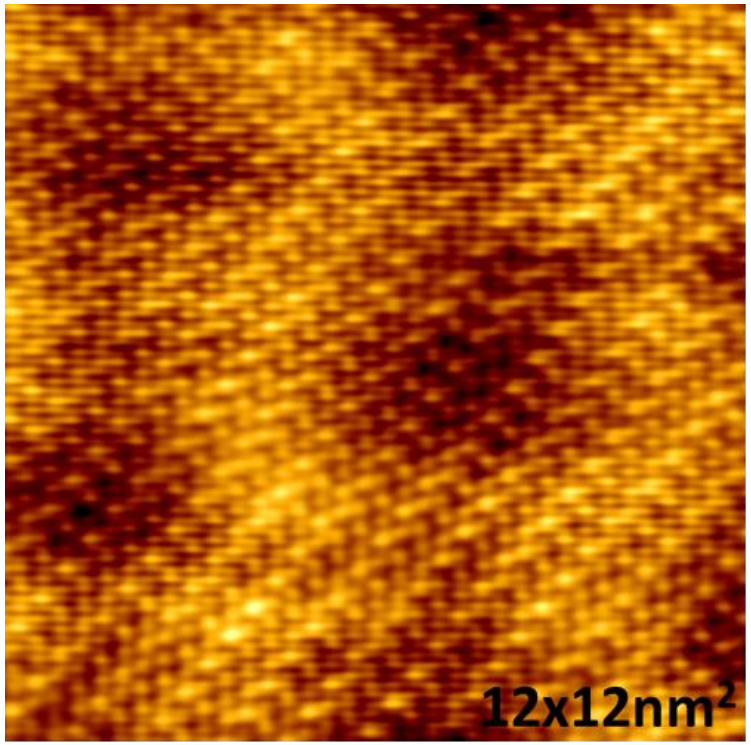
A. Pasztor….C. Renner, 2D Mater. 4 (2017) 041005

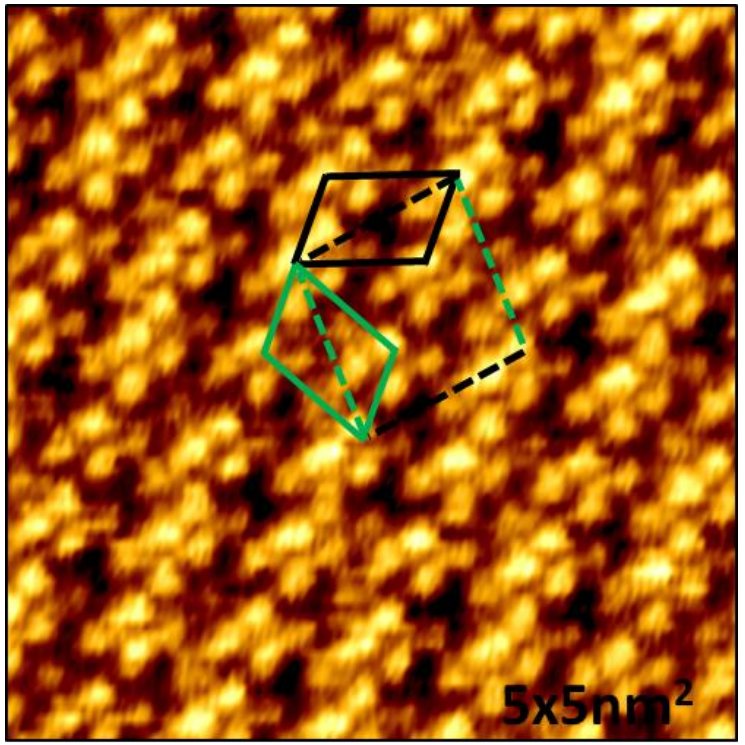
Does the CDW order compete with the ferromagnetic ordering for the ground state?

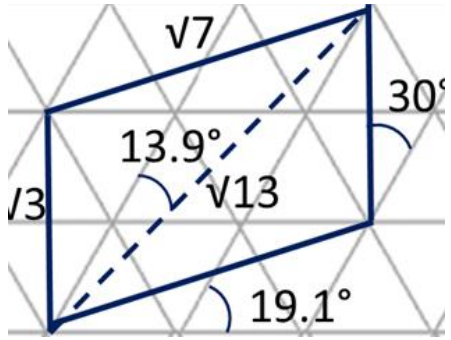
Does the monolayer exhibit a CDW and how does it affect ferromagnetic ordering?

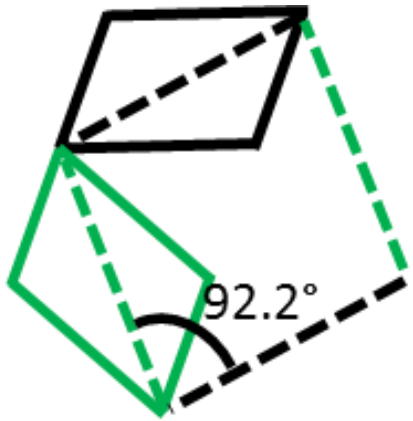
Charge density wave in monolayer VSe_2.

STM at 80 K shows charge density wave superstructure









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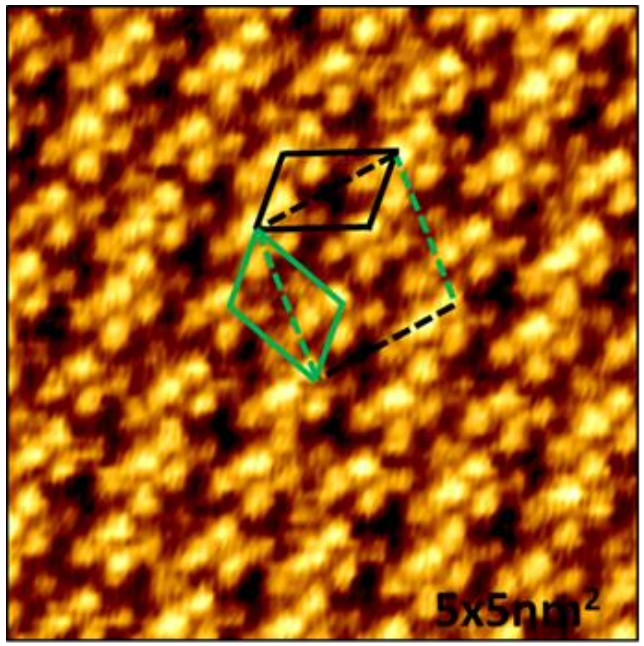
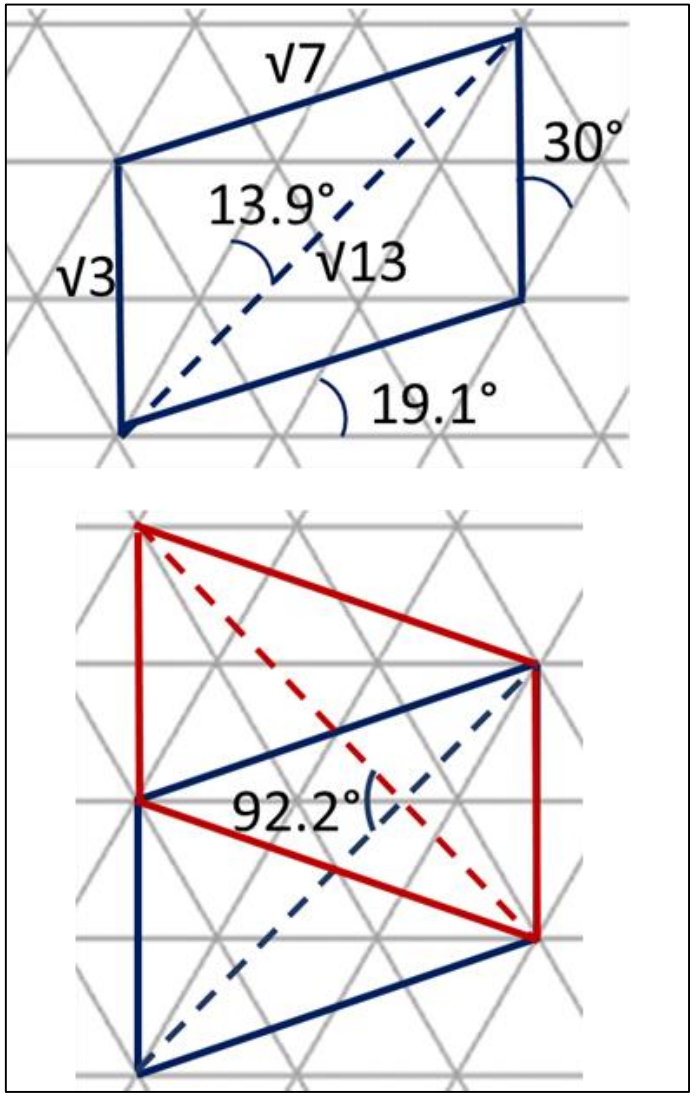
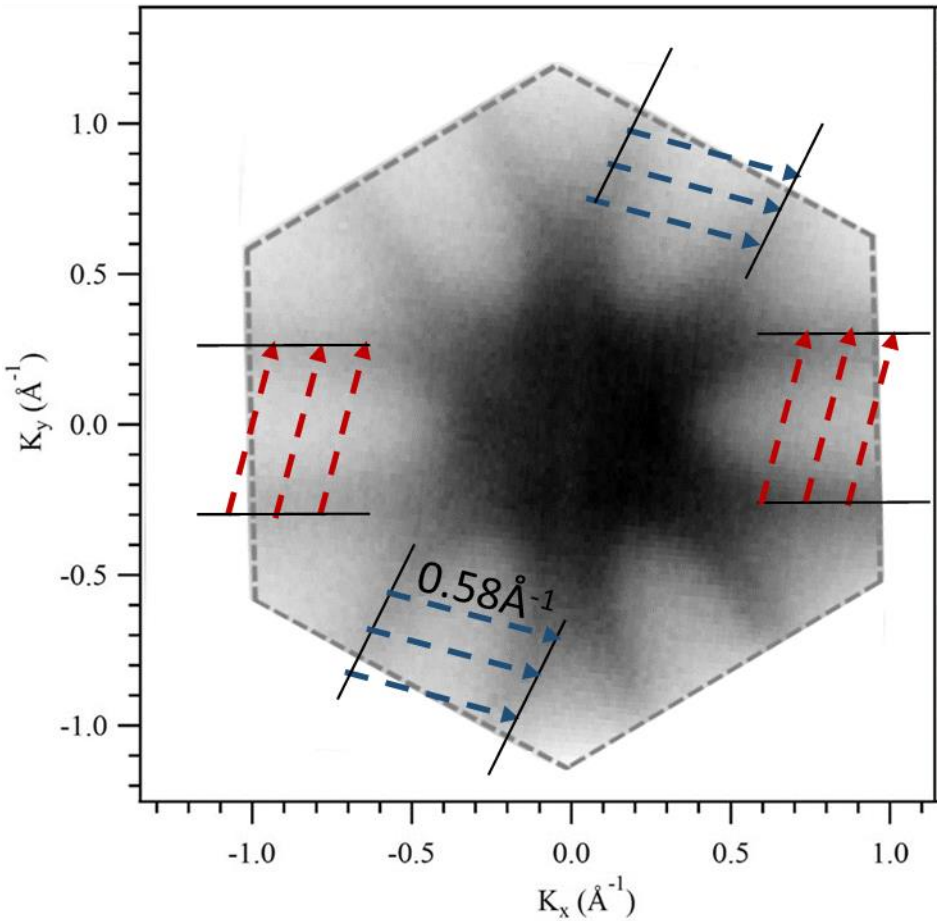
Can CDW be described by Fermi-surface nesting?



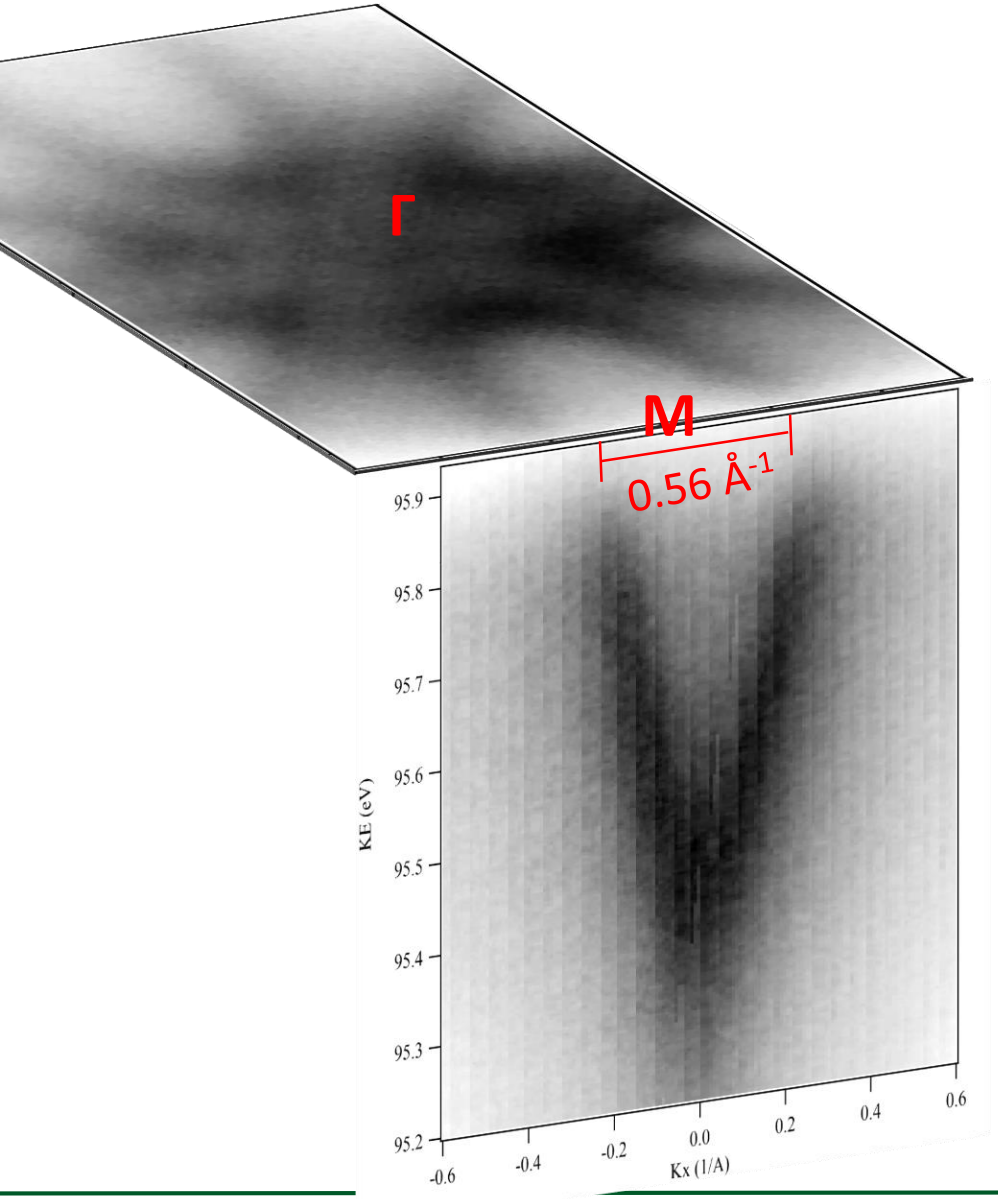
STM at 15 K shows CDW



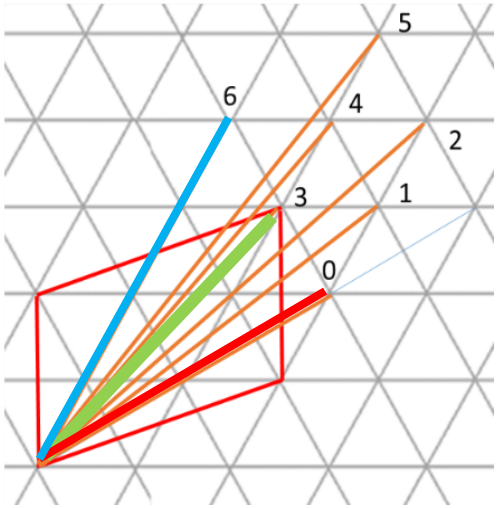


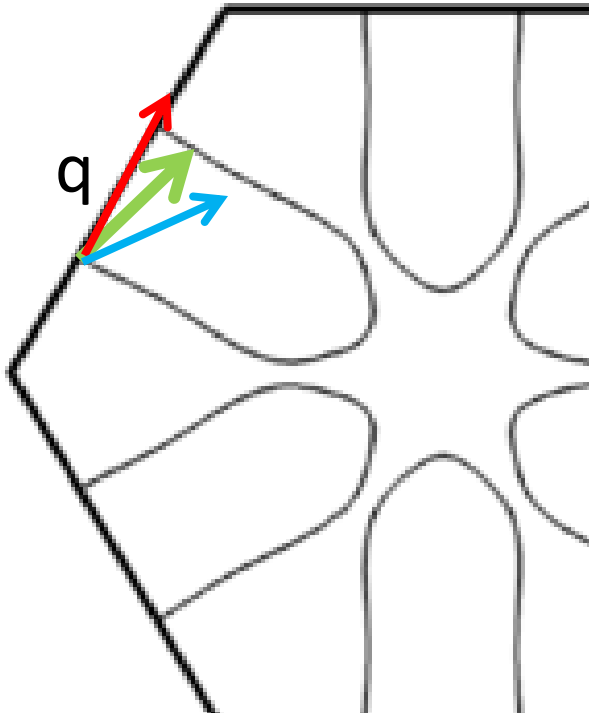
J. Phys. Chem. C 123, 14089-14096 (2019)

Can CDW be described by Fermi-surface nesting?



Real vector length (in units of a)	Rotation angle	Reciprocal vector length (in units of a*)	Reciprocal vector length (in Å^{-1})	Experimental separation of the Fermi-sheets (in Å^{-1})	Mismatch
2√3	30°	0.29	0.62	0.56	-10%
√19	23.4°	0.23	0.5	0.56	+11%
√28	19.1°	0.19	0.41	0.57	+28%
√13	13.9°	0.28	0.6	0.58	-3%
√21	10.9	0.22	0.47	0.59	+20%
√31	8.9°	0.18	0.39	0.60	+35%
4	0°	0.25	0.54	0.65	+17%





Commensurate Fermi-surface nesting vectors

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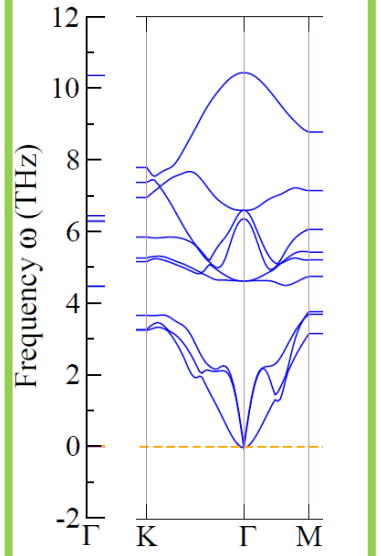
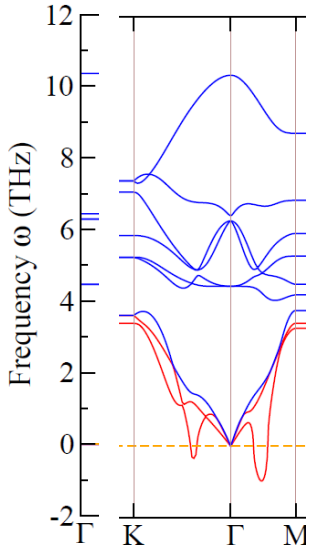
CDW or FM ordering

Phonon modes for spin-polarized and non-spin polarized DFT calculations.

Spin-polarized (magnetic)

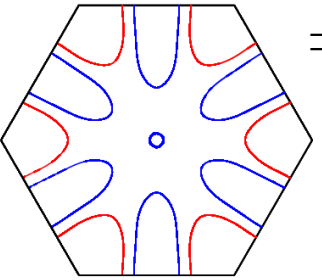


Non Spin-polarized



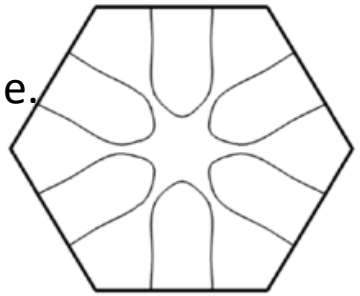
⇒ Only if VSe_2 is non-magnetic a CDW is expected.

⇒ Observation of CDW is consistent with the absence of spin-split bands in ARPES



⇒ No phonon instability, i.e. should not form a CDW

⇒ phonon instability, i.e. CDW is expected=> experiment

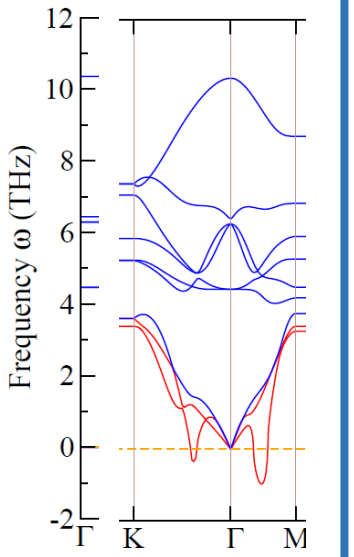


J. Phys. Chem. C 123, 14089-14096 (2019)

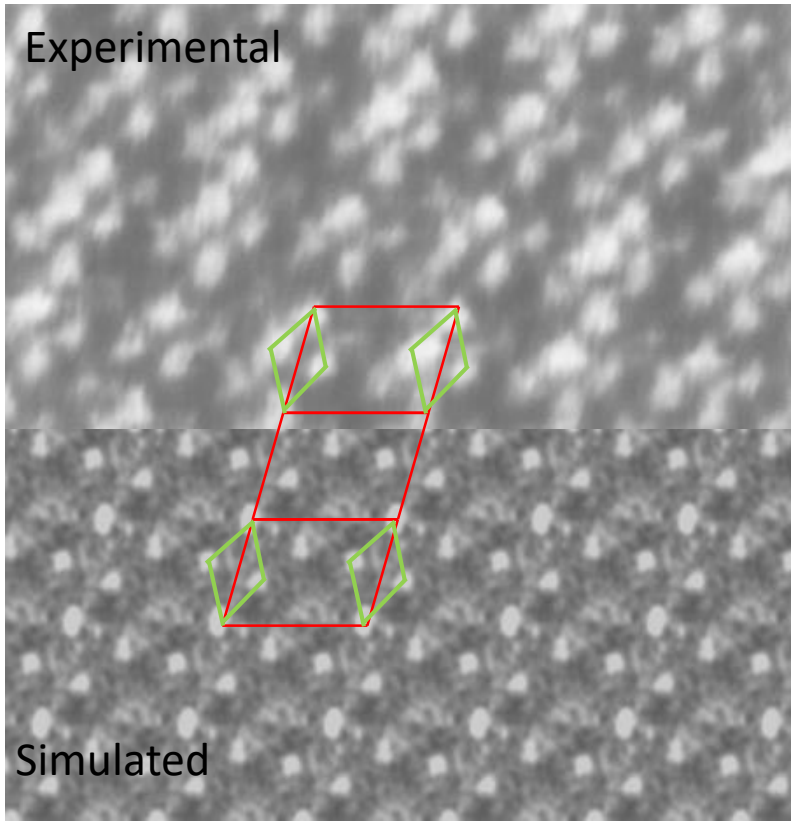
Re-examining DFT predictions

Phonon modes for spin-polarized and non-spin polarized DFT calculations.

Non Spin-polarized



⇒ phonon instability, i.e. forms a CDW=> experiment

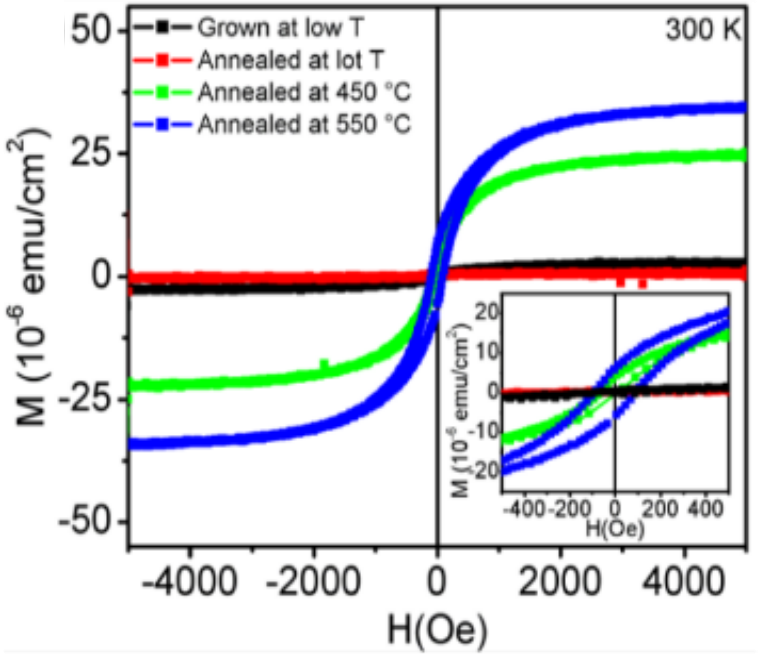


Lowest energy structure search using evolutionary crystal structure prediction method USPEX (with CDW unit cell as only input).

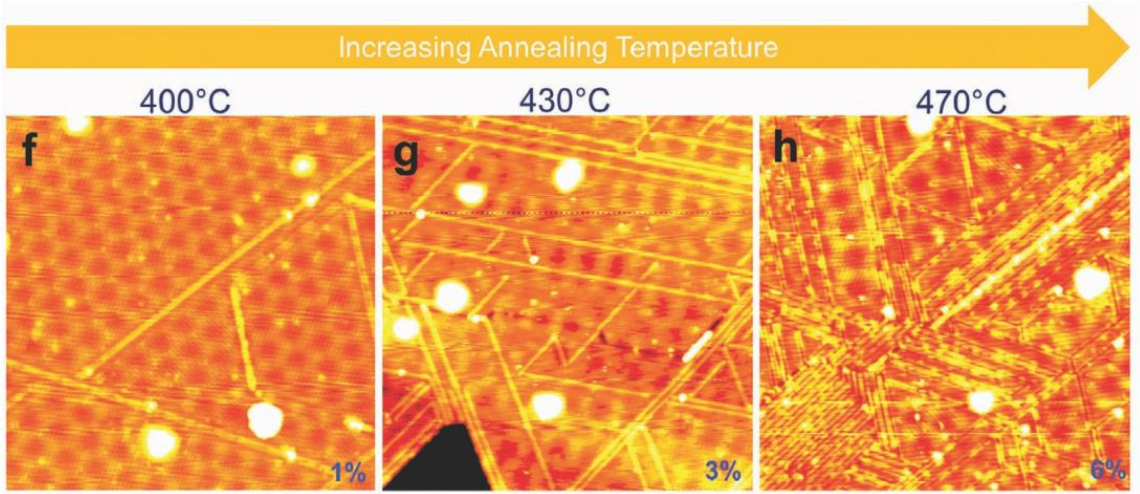
=> CDW order has lower energy than ferromagnetic ordered state.

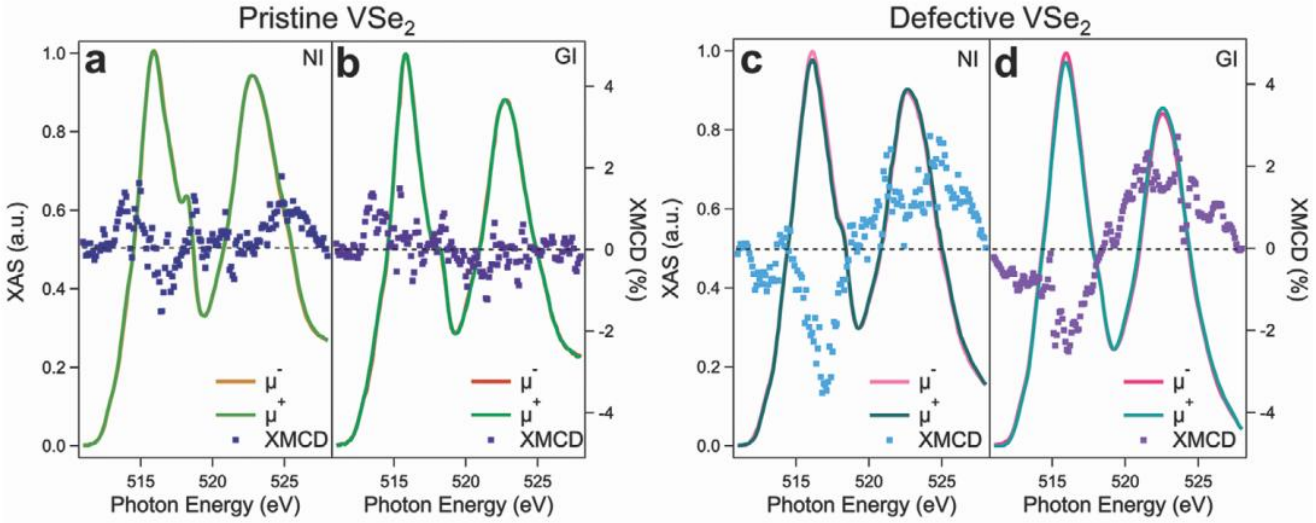
J. Phys. Chem. C 123, 14089-14096 (2019)

Can defects cause magnetism in VSe_2?



R. Chua, … A.T.S. Wee; Adv. Mater. 2000693 (2020)





VSe_2 conclusions

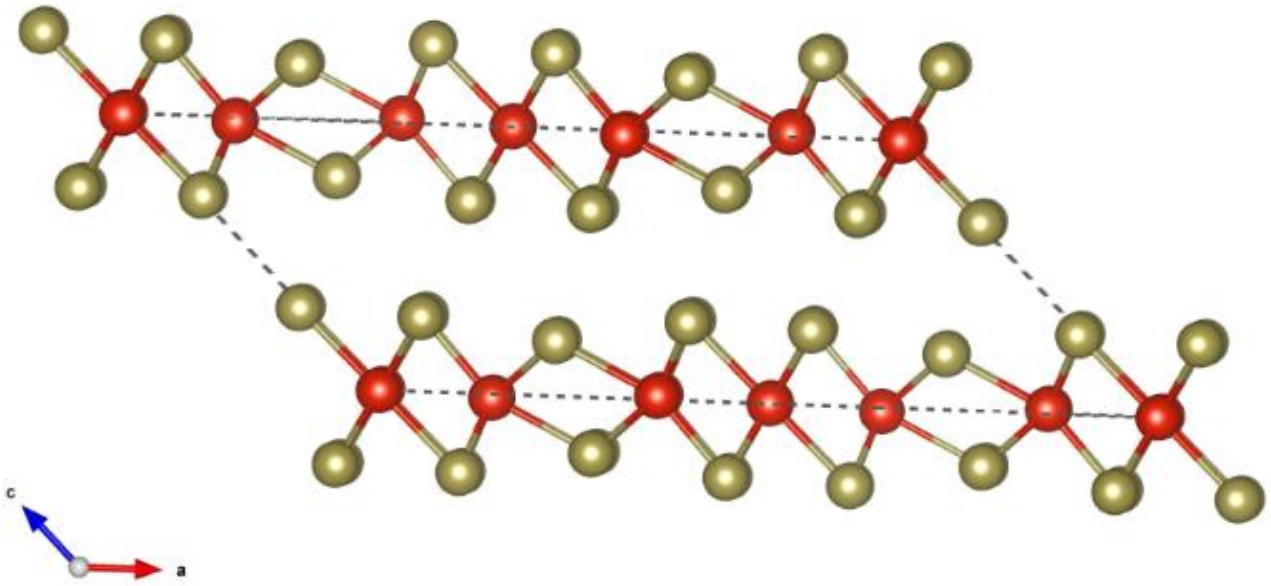
- VSe_2 monolayers on various substrates (after annealing) exhibit M-H hysteresis loops.
- There is mo evidence for itinerant magnetism in ARPES and XMCD shows no magnetic moment.
- Monolayers exhibit a CDW and DFT suggest it is lower in energy than a ferromagnetically ordered state => CDW is ground state.
- Weak magnetization is observed in defective VSe_2.

Outline

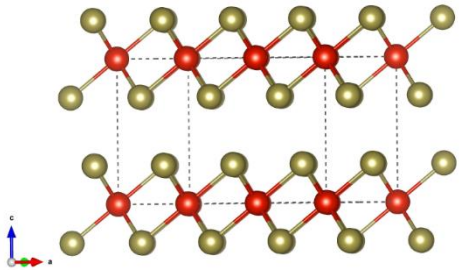
1. VSe_2: Competition between ferromagnetic and charge density order?
2. VTe_2: A new monolayer CDW material.
3. $CrTe_2$ monolayer synthesis by van der Waals epitaxy. But is it 1T or 1H?
4. Magnetic defects and dopants: Possible diluted ferromagnetic 2D semiconductors.

Other vanadium dichalcogenides: VTe_2

Bulk structure of VTe_2 (also $TaTe_2$, $NbTe_2$)

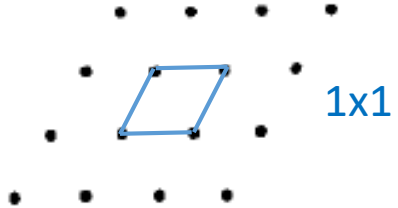


Other vanadium dichalcogenides: VTe_2

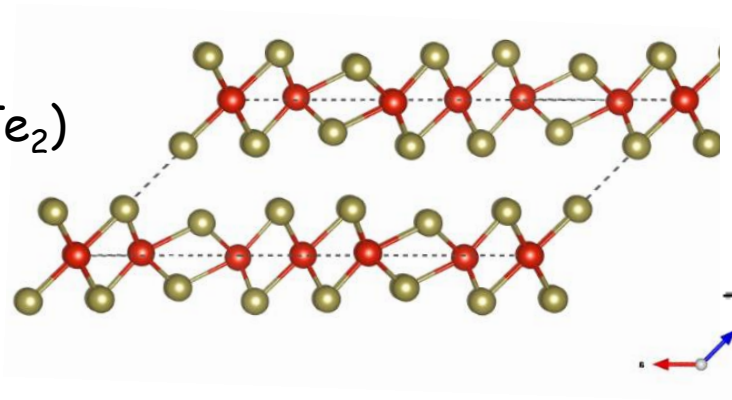


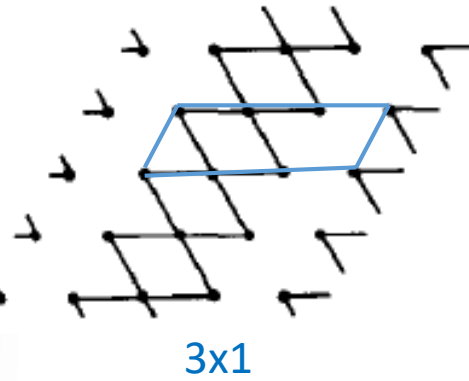
1T

VSe_2- d^1

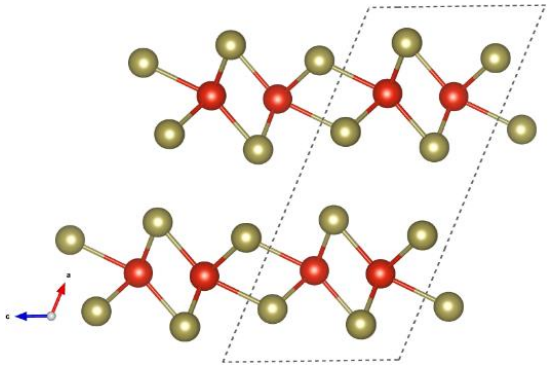


VTe_2
($NbTe_2$, $TaTe_2$)
$d^{1+\varepsilon}$

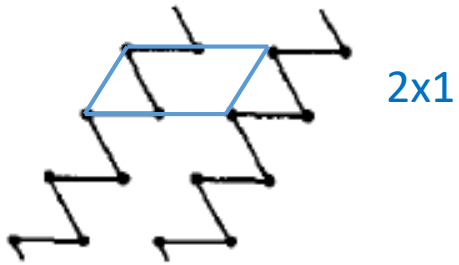




1T'



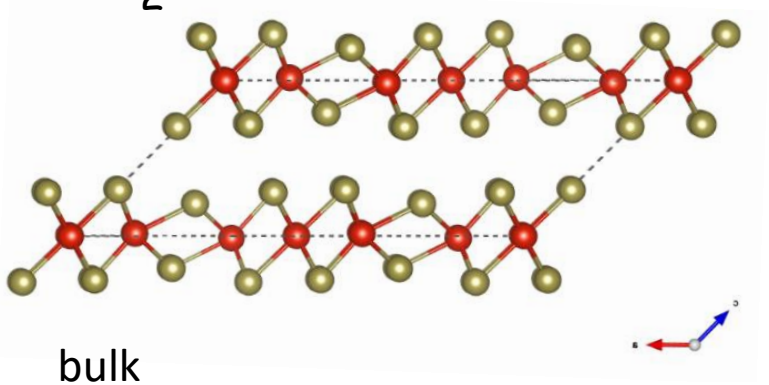
d^2
(e.g.
WTe_2)



Other vanadium dichalcogenides: VTe_2

Are interlayer Te…Te interactions important?

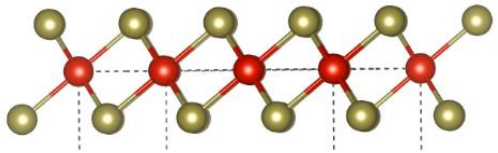
VTe_2 - $d^{1+\varepsilon}$



bulk

Can we get 1T VTe_2 by removing interlayer Te-bonds and avoid Te to V charge transfer?

monolayer



JOURNAL OF SOLID STATE CHEMISTRY 99, 189–199 (1992)

Importance of Short Interlayer Te···Te Contacts for the Structural Distortions and Physical Properties of CdI_2-Type Layered Transition-Metal Ditellurides

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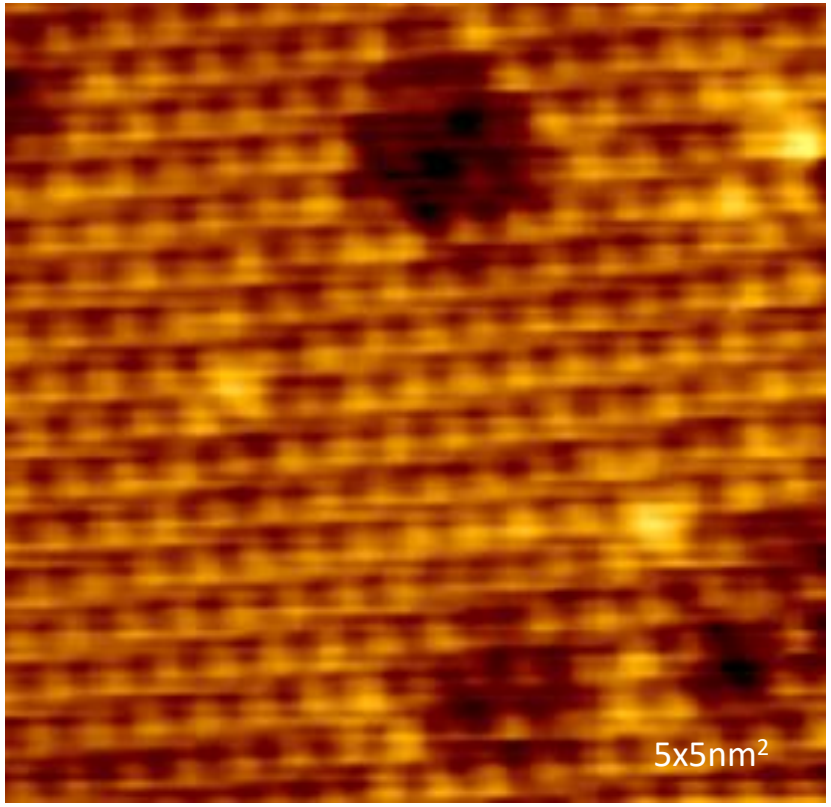
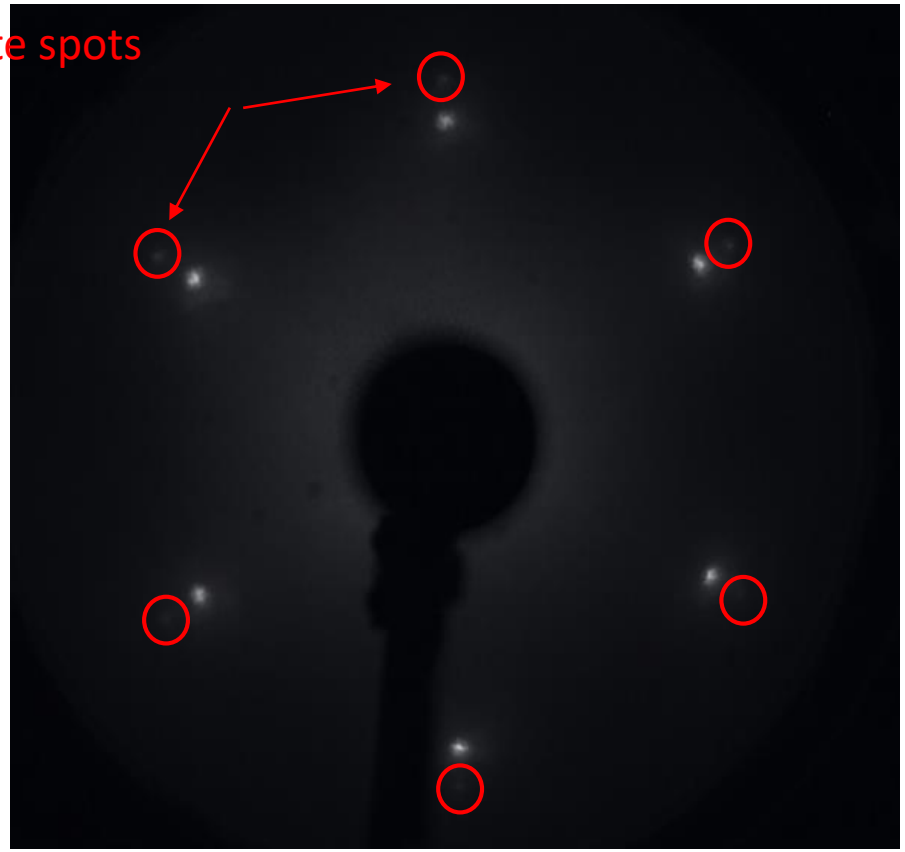
Received October 10, 1991; in revised form January 20, 1992; accepted January 23, 1992

monolayer VTe_2 is 'simple' 1T

STM 300 K



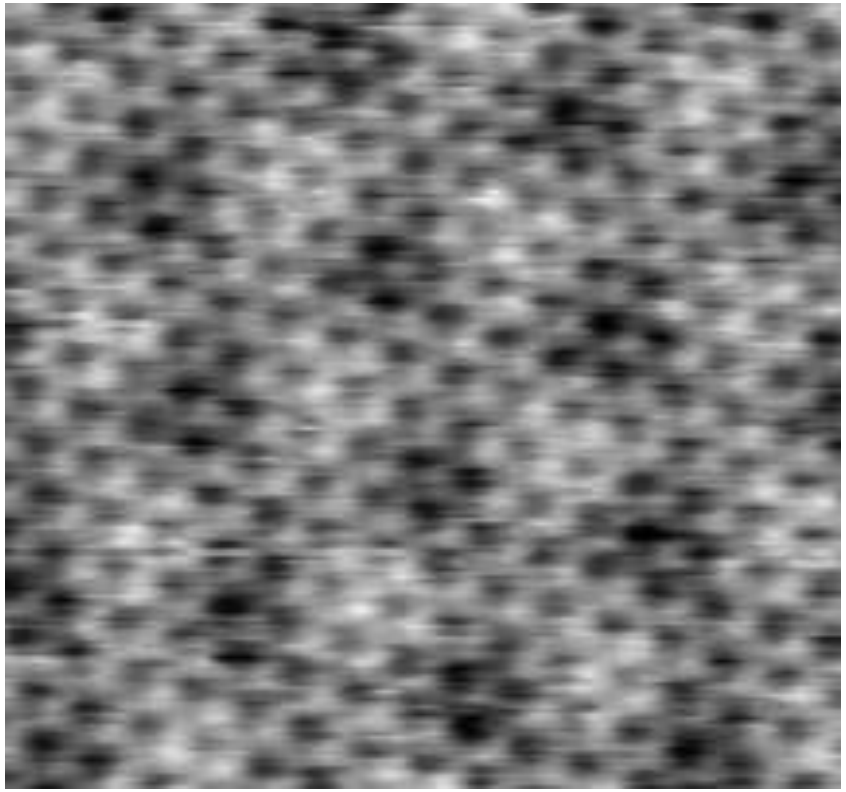


J. Phys. Chem. Lett. 10, 4987-4993 (2019)

CDW in monolayer VTe_2

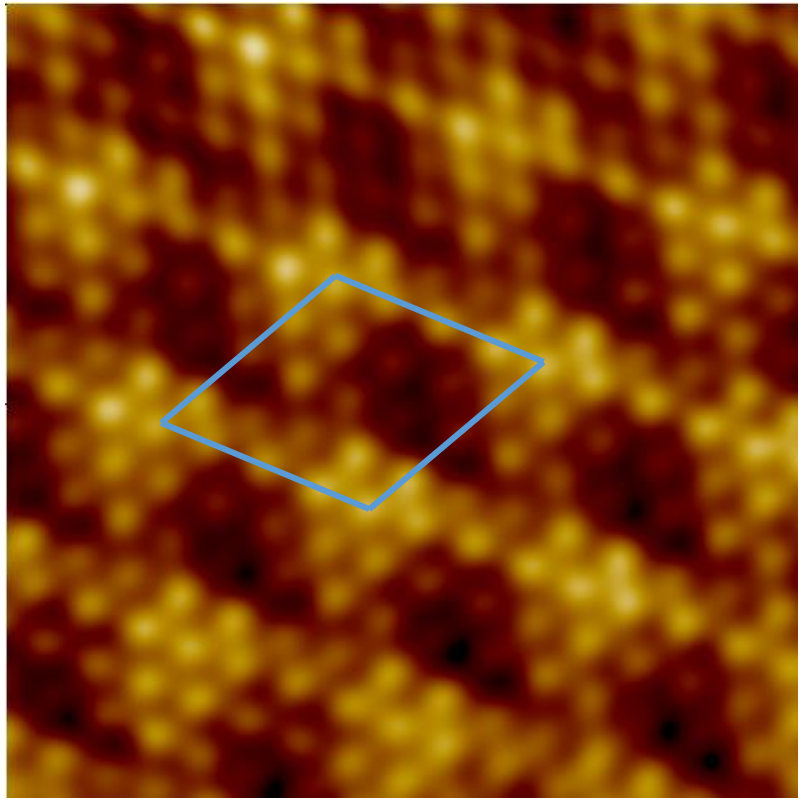
=> Is monolayer VTe_2 a new CDW material?

Same 4x4 CDW as isoelectronic (bulk) VSe_2



A. Pasztor….C. Renner, 2D Mater. 4 (2017) 041005

20 K

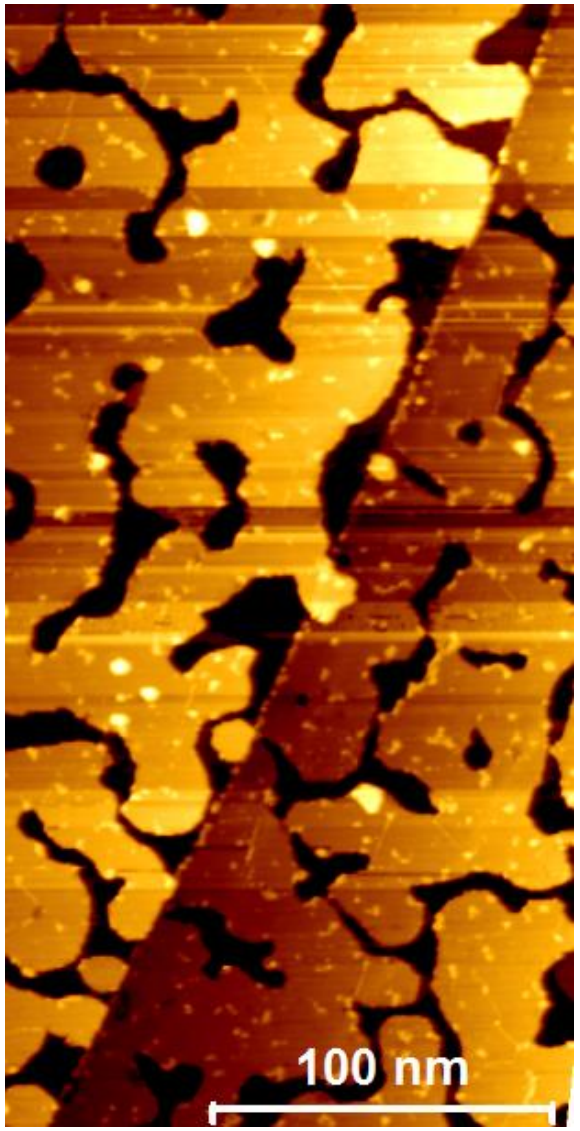


4x4 CDW

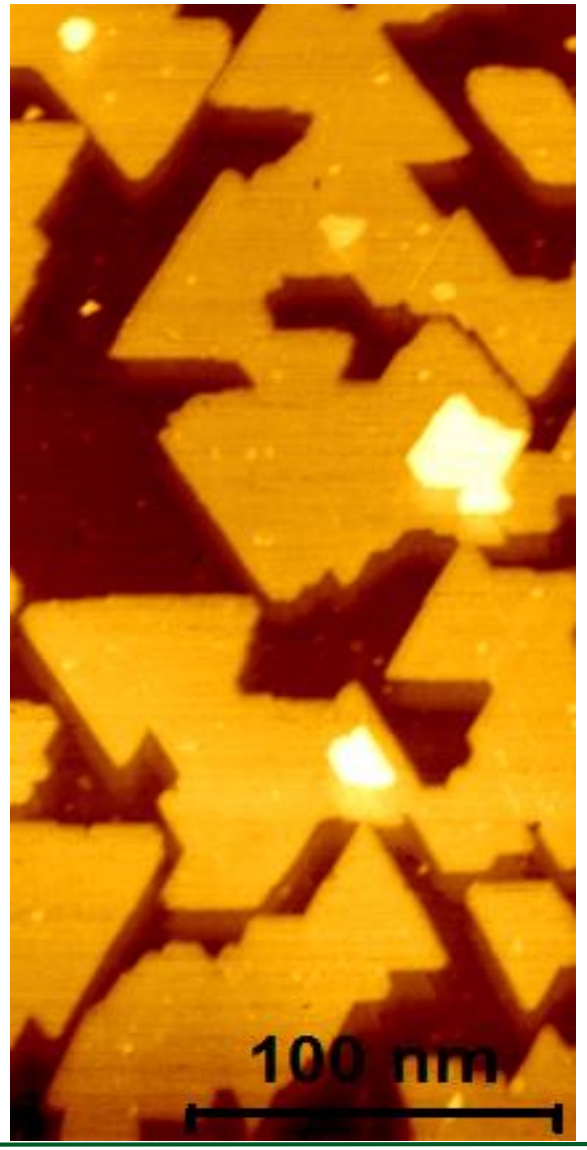
J. Phys. Chem. Lett. 10, 4987-4993 (2019)

Multilayers of V-telluride

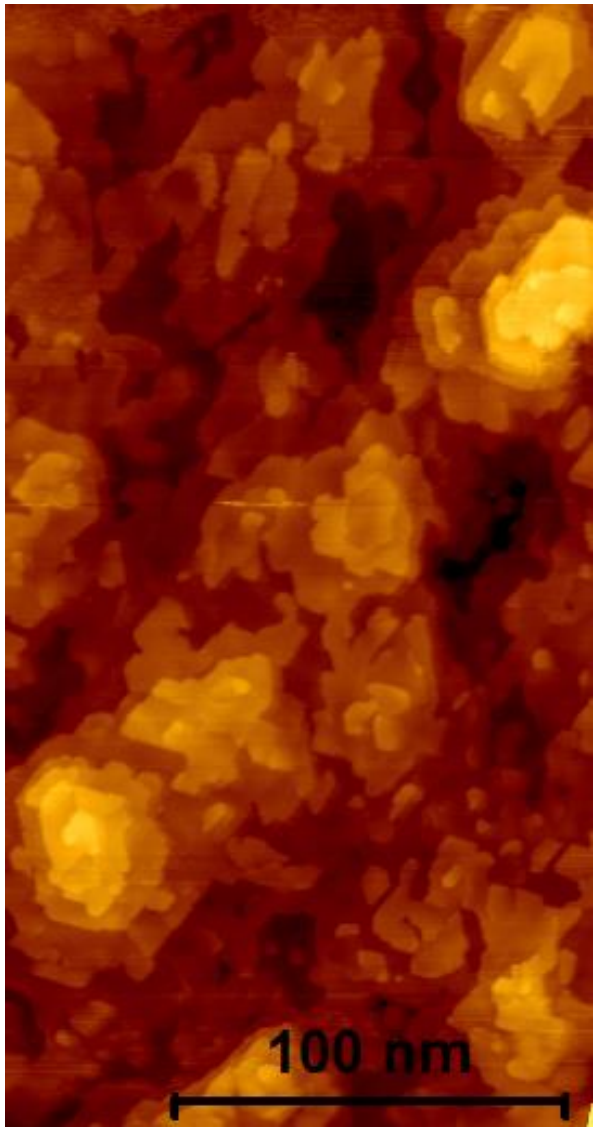
Mono-layer



Mono + Bilayer

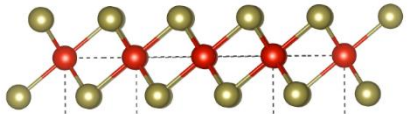


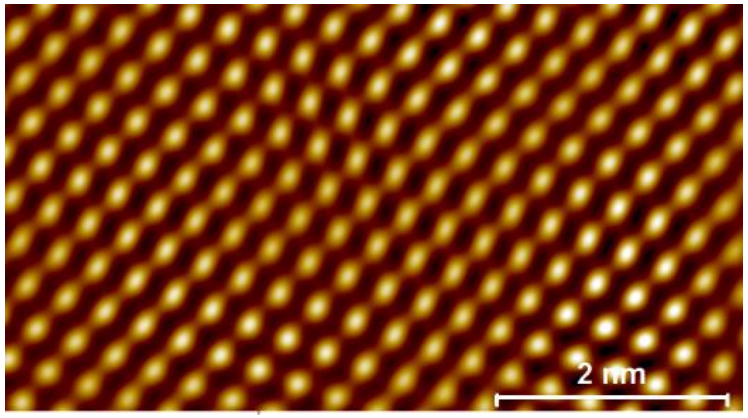
Multilayer

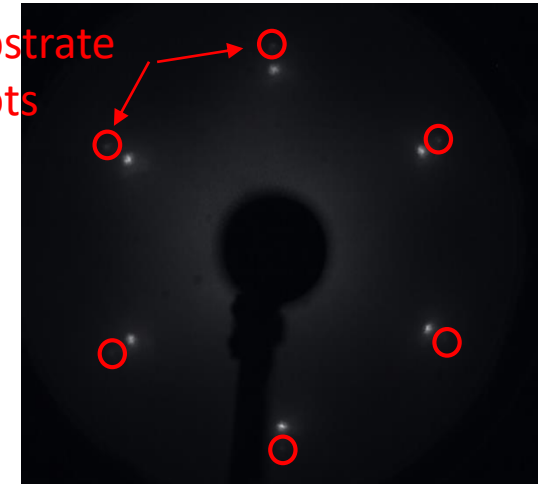


Layer-dependent structure change in VTe_2

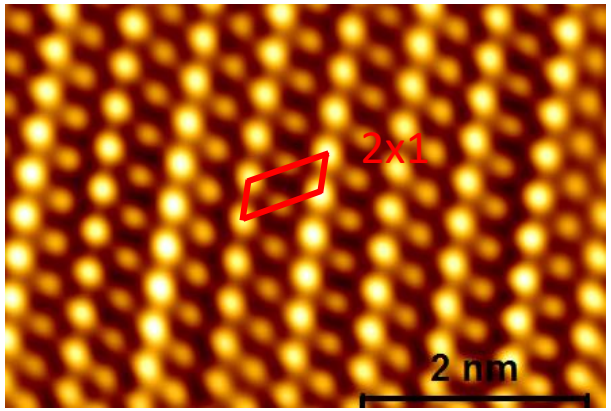
Monolayer VTe_2

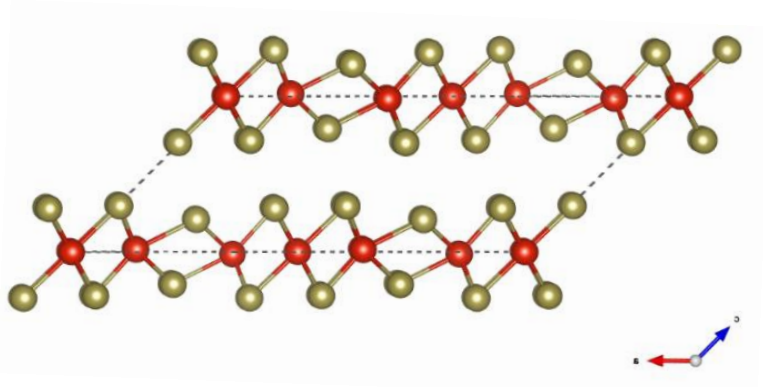


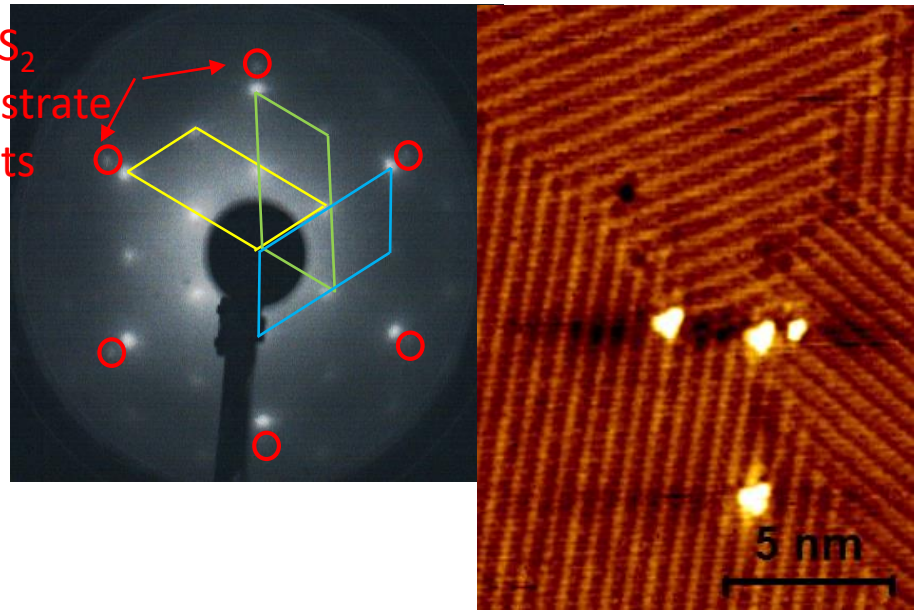


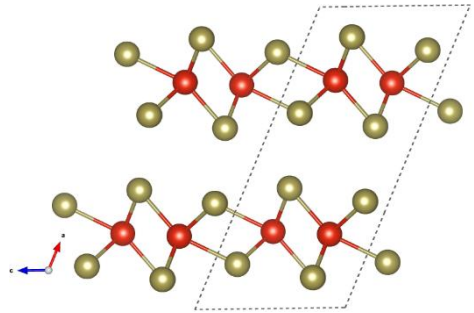


Bi- and Multilayer VTe_2



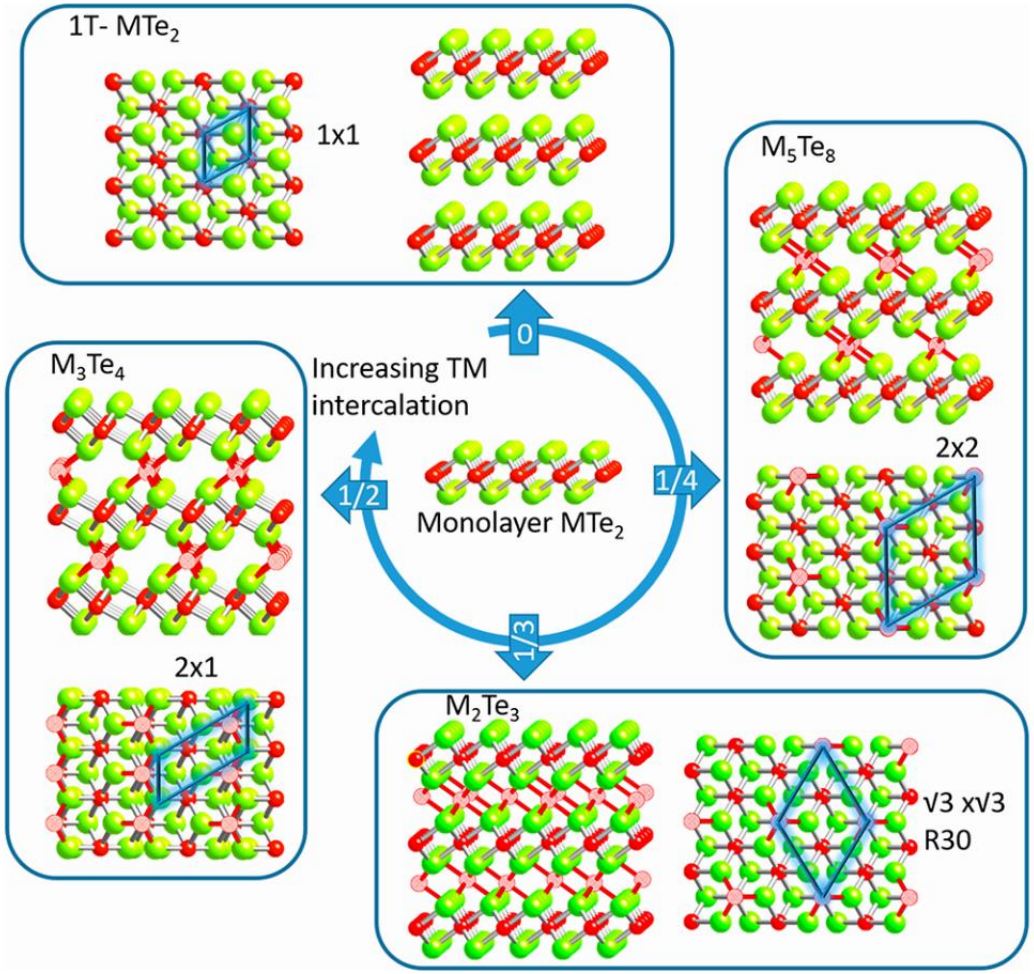


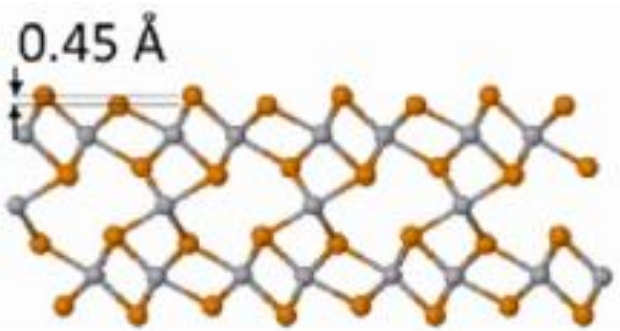


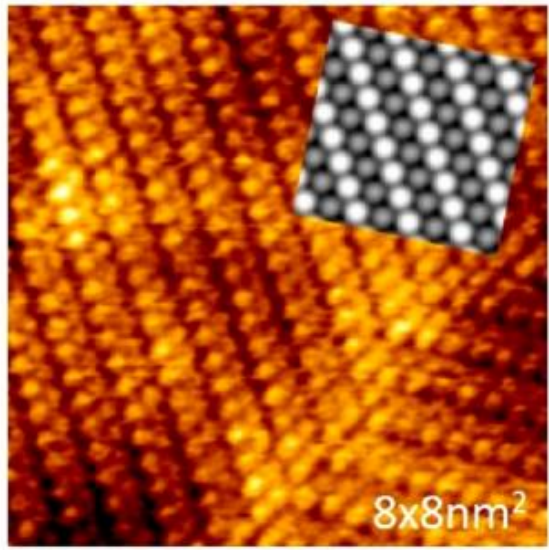


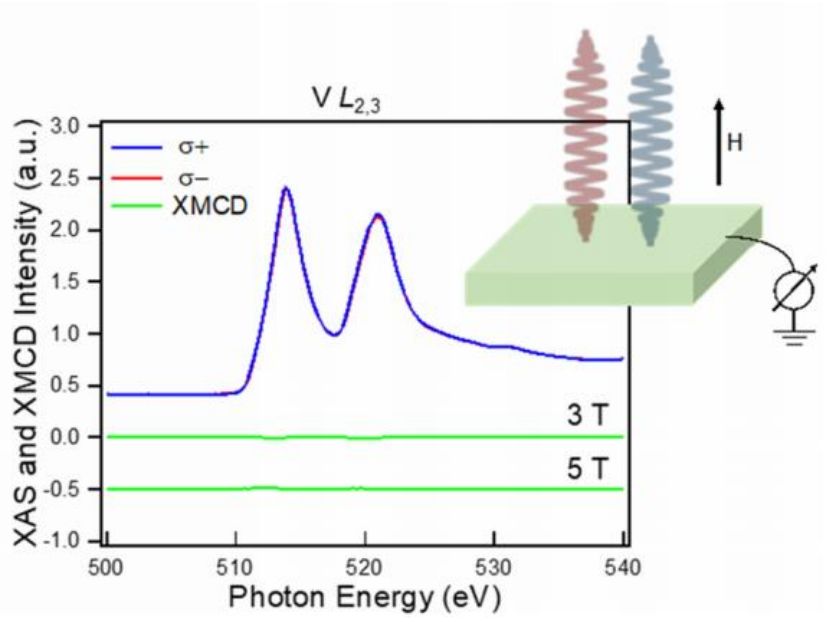
Are multilayers 1T' ?

Layer-dependent structure change in VTe_2









K. Lasek....M.B. ACS Nano 2020, 14, 7, 8473–8484

VTe_2 Conclusions

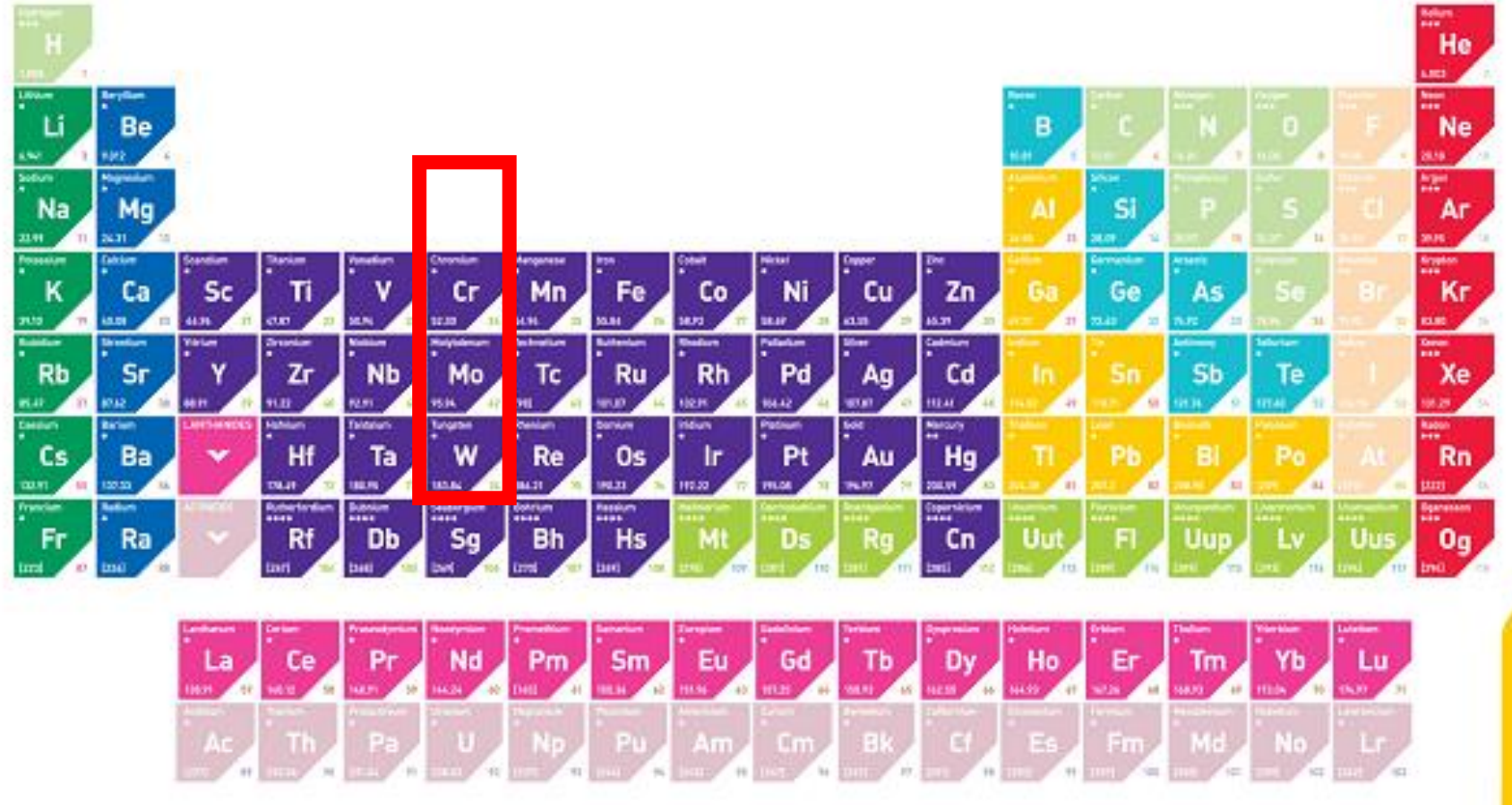
- Monolayer VTe_2 is a simple 1T structure (unlike the bulk that exhibits a distorted 1T" structure).
- The 1T-VTe_2 monolayer undergoes a 4x4 CDW transition- identical to the isoelectric VSe_2 (in the bulk).
- MBE growth of multilayers likely results in the formation of metal intercalated compound with a V_3Te_4 composition. (its not ferromagnetic)

Outline

1. VSe_2: Competition between ferromagnetic and charge density order?
2. VTe_2: A new monolayer CDW material.
3. $CrTe_2$ monolayer synthesis by van der Waals epitaxy. But is it 1T or 1H?
4. Magnetic defects and dopants: Possible diluted ferromagnetic 2D semiconductors.

$CrTe_2$

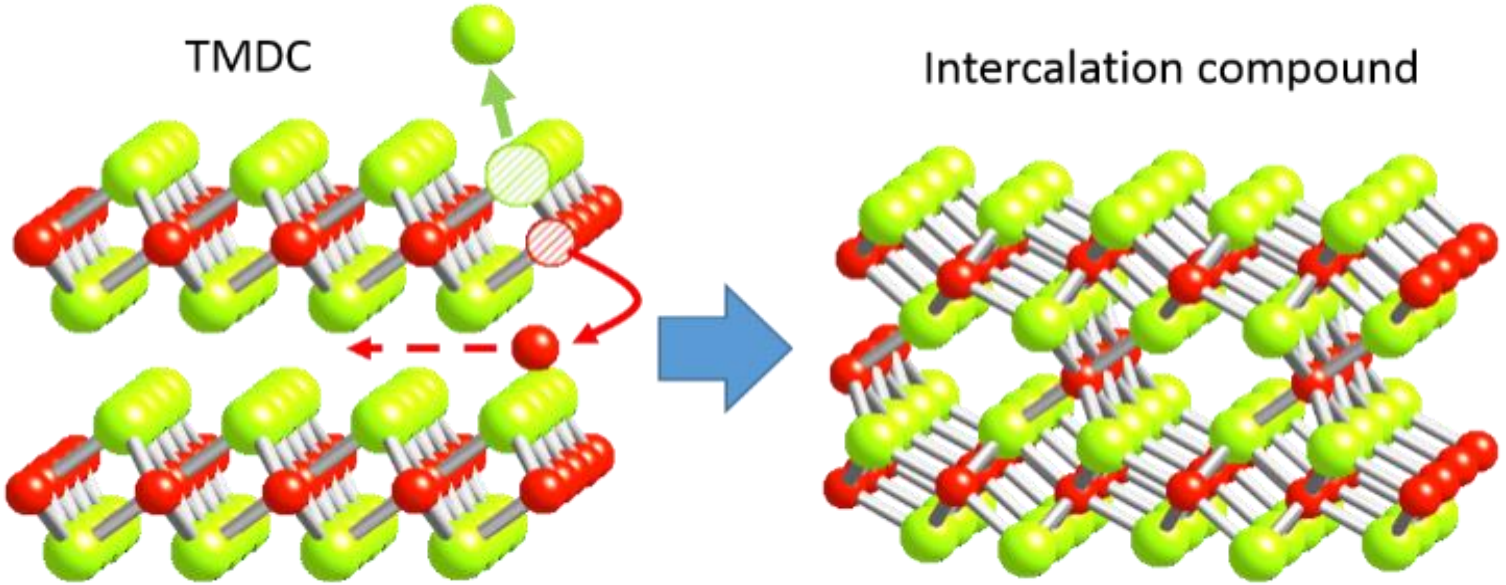
Why are there not more studies on $CrTe_2$?



$CrTe_2$

Why are there not more studies on $CrTe_2$?

Answer: $CrTe_2$ (also $CrSe_2$) is metastable and decomposes at ~330 K (~ 600 K) into an 'intercalation' compound and chalcogens.

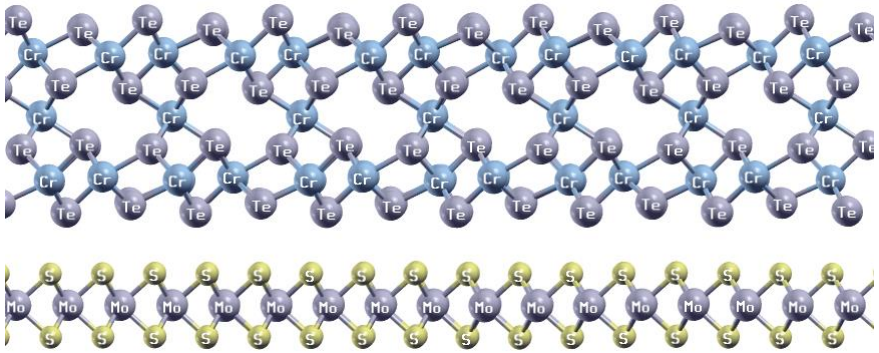


Can we get a monolayer by MBE growth?

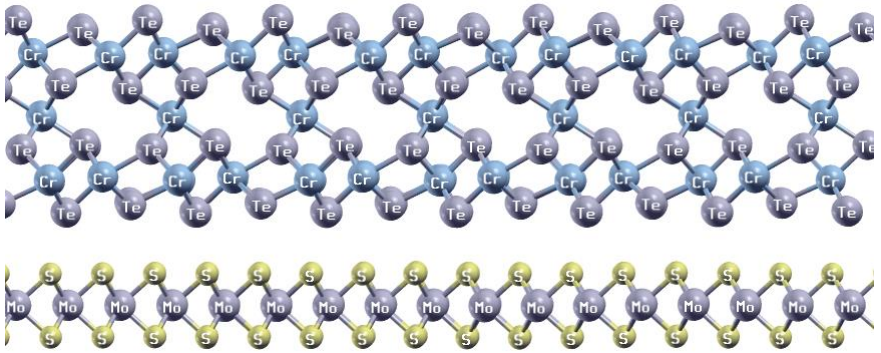
$CrTe_2$

Ab initio MD simulations

Intercalated Cr



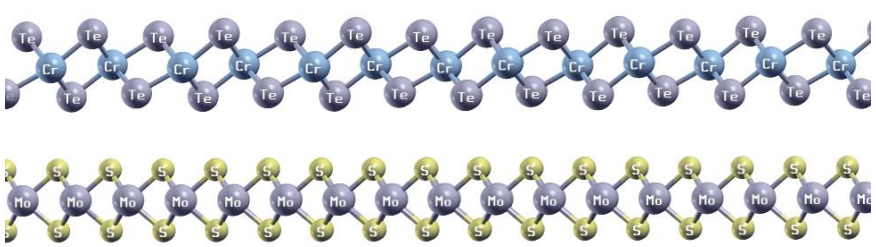
600 K



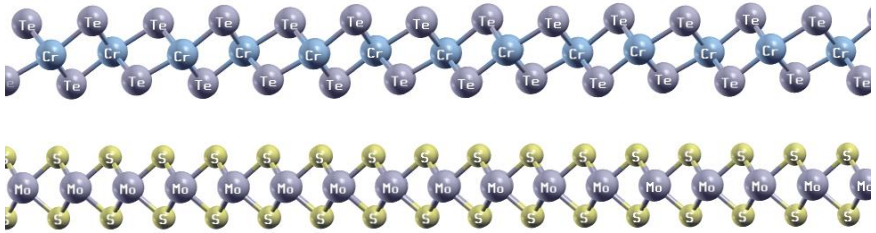
A single layer would only be stable in a $CrTe_2$ composition without excess Cr-atoms (in the vdW gap)

Mono-layer

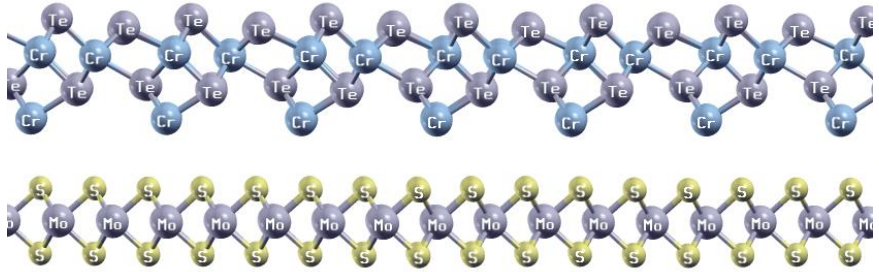
$CrTe_2$



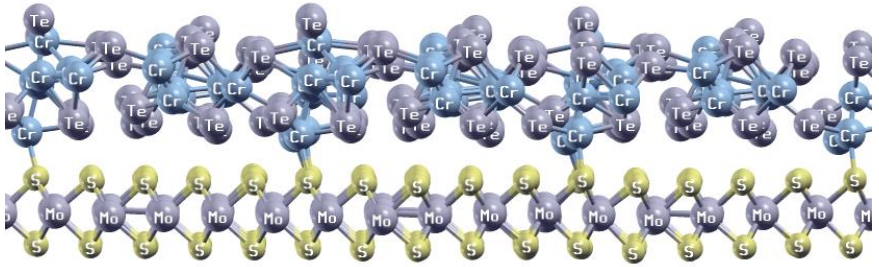
600 K



'Excess' Cr



600 K



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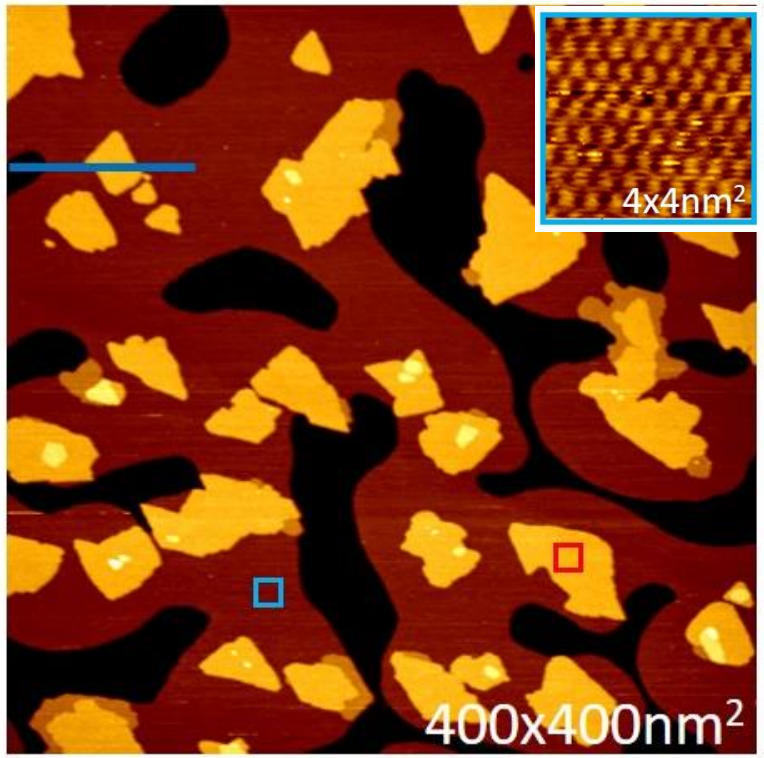
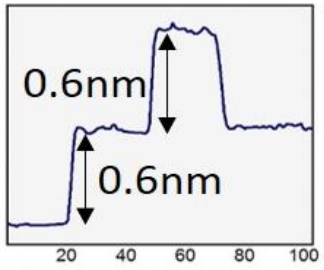
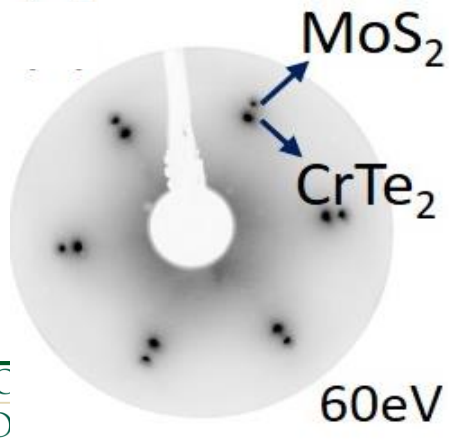
$CrTe_2$

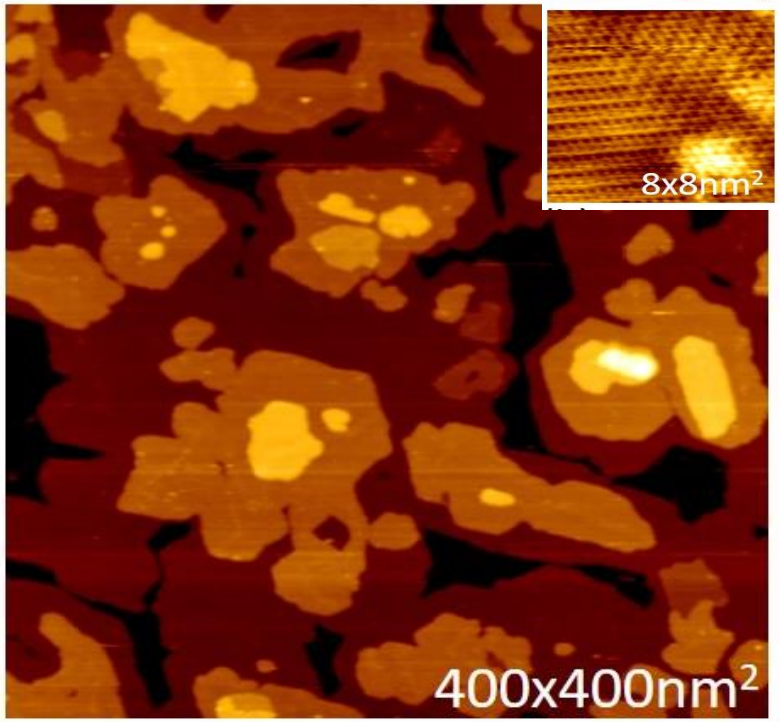
Monolayer (some bilayer)

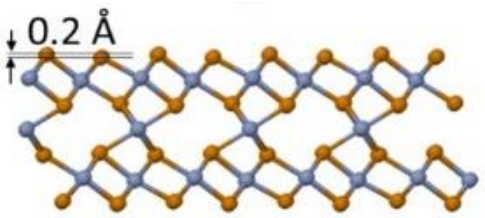


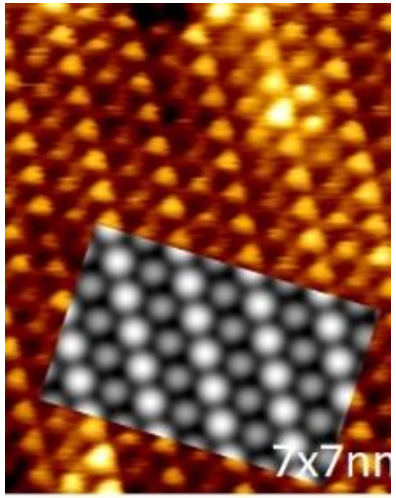


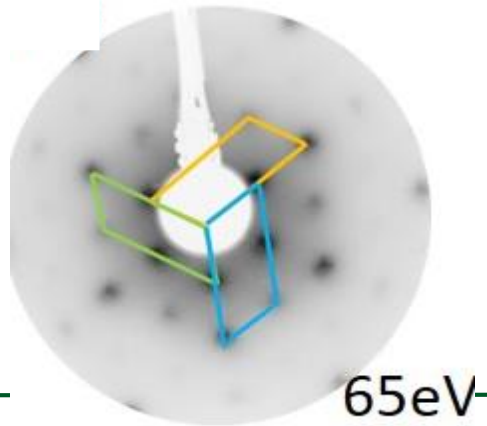


Multilayer films





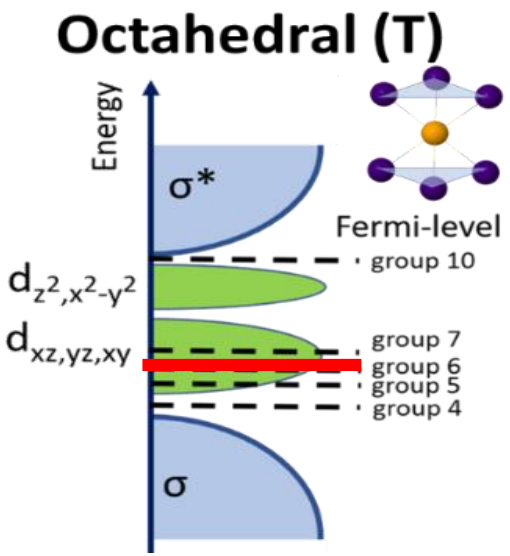


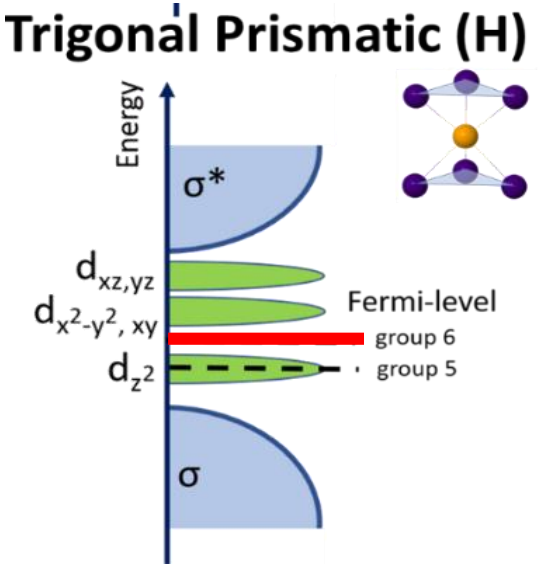


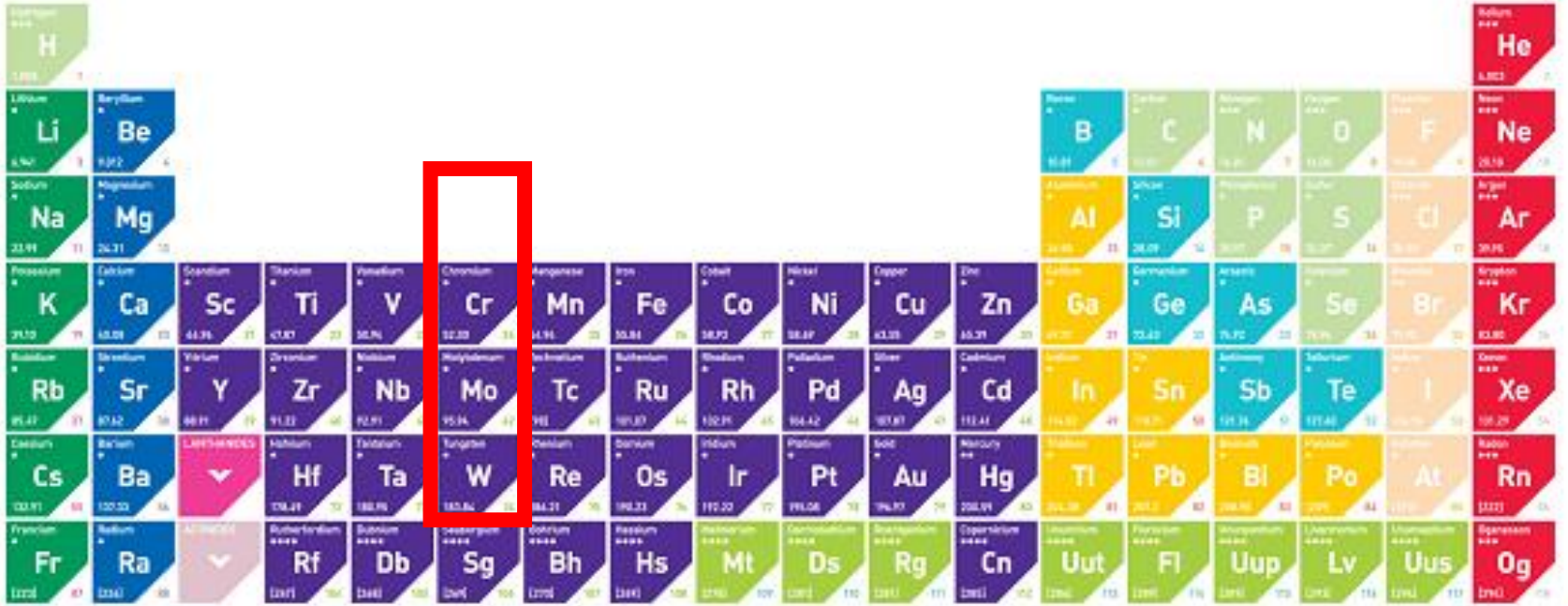
K. Lasek....M.B. ACS Nano 2020, 14, 7, 8473–8484

$CrTe_2$

Is $CrTe_2$ 1T or 2H phase?



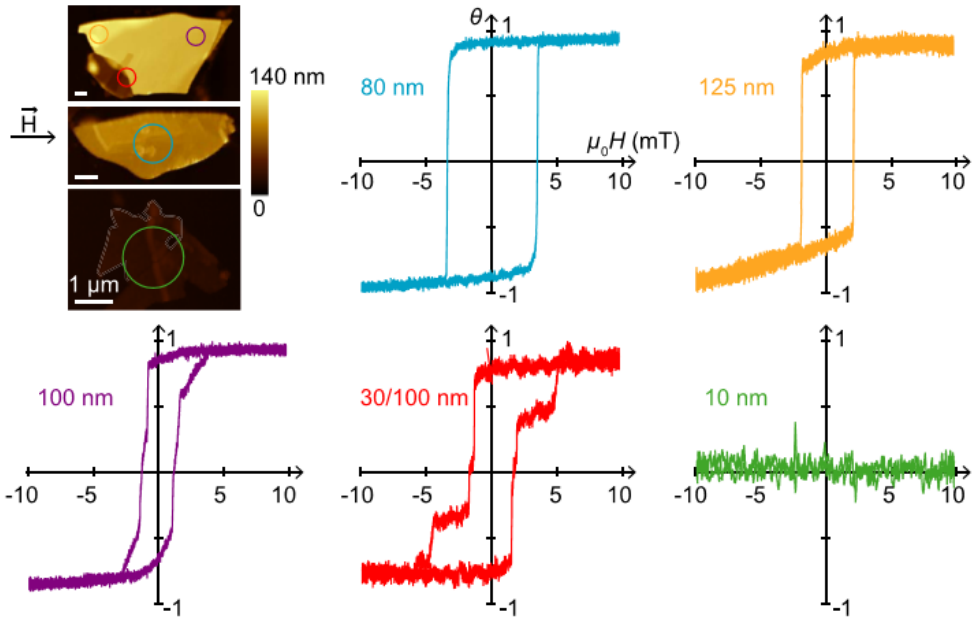




$CrTe_2$

1T or 2H?

Available bulk samples are 1T.



Kerr measurements on $CrTe_2$ flakes at RT.

ACS Appl. Mater. Interfaces (2020) 12, 30702–30710

J. Phys.: Condens. Matter 27, 176002 (2015).

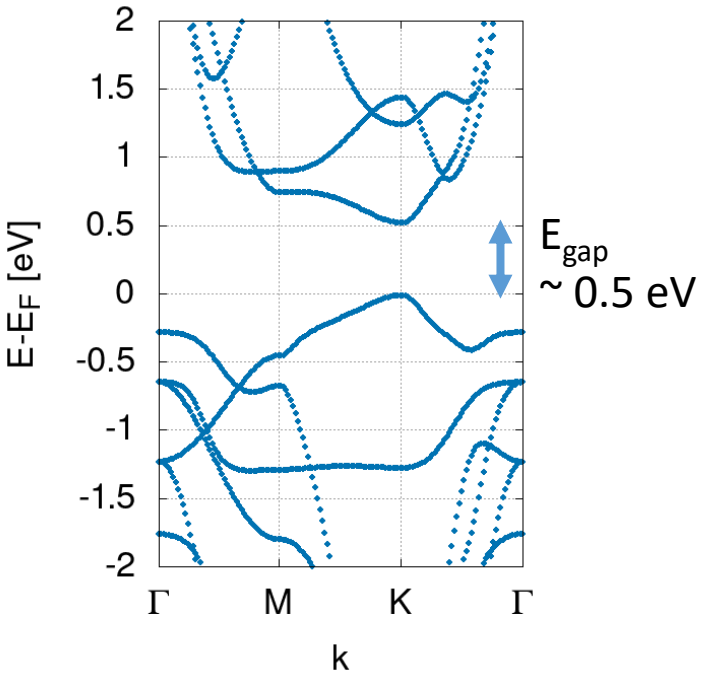
BUT: the synthesis involves formation of a K:$CrTe_2$ intercalation to stabilize the $CrTe_2$-layers and subsequently a low temperature extraction of the potassium atoms. K-doping may stabilize the 1T phase (like for MoS_2).

$CrTe_2$

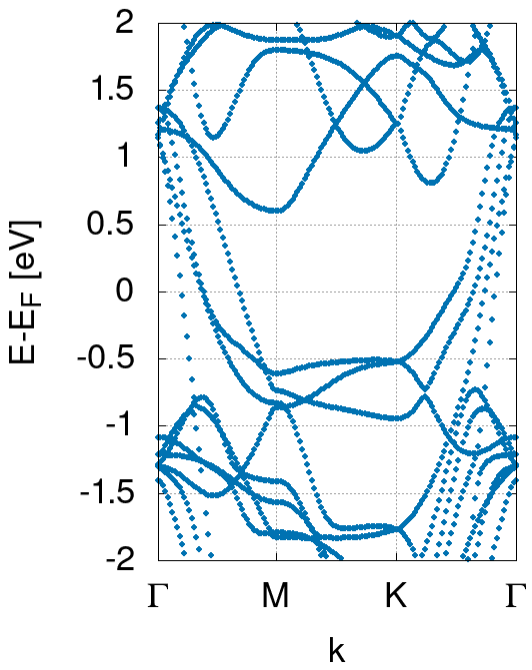
1T or 2H?

DFT-calculations: 1H is favored over 1T (~ 0.5 eV/atom)

1H monolayer

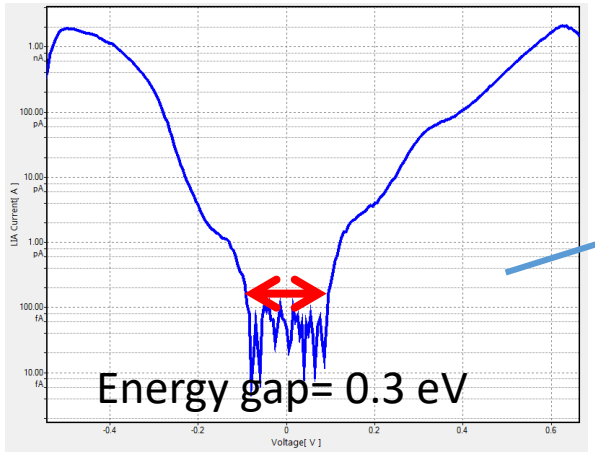


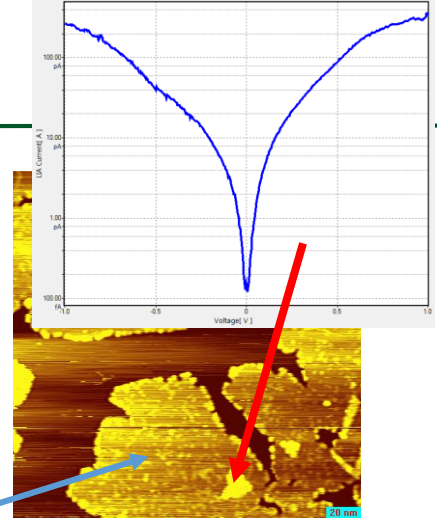
1T monolayer



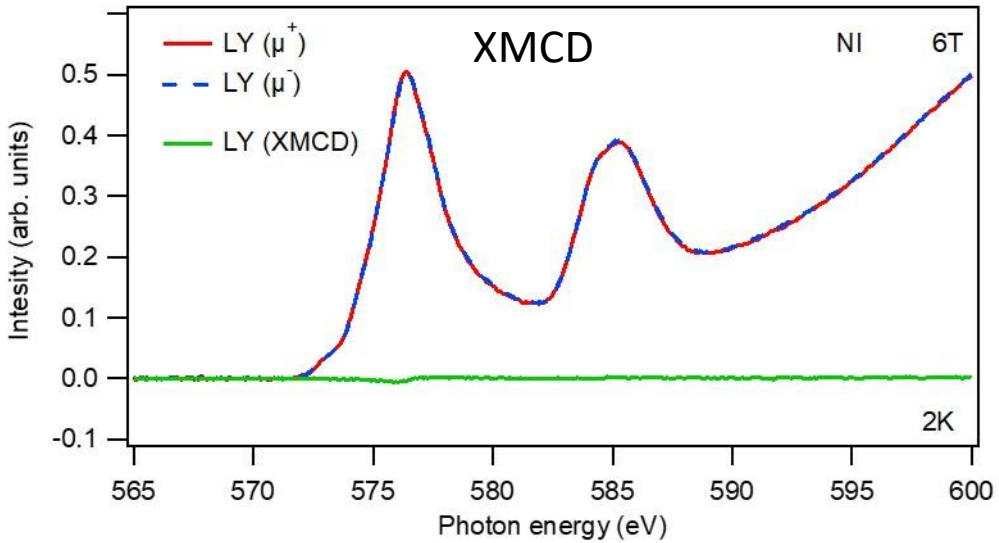
=> MBE grown monolayer (1H) $CrTe_2$ is not ferromagnetic.

Scanning tunneling spectroscopy





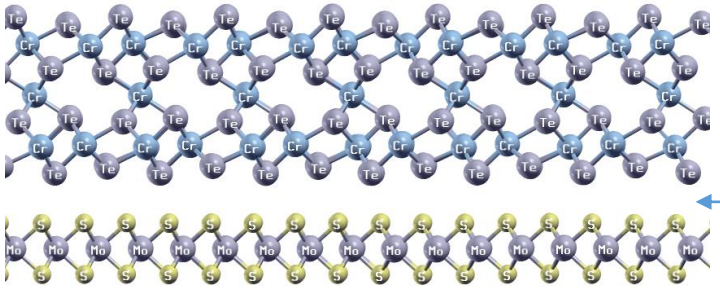
If monolayer $CrTe_2$ is semiconducting 1H, we would not expect any ferromagnetic ordering.



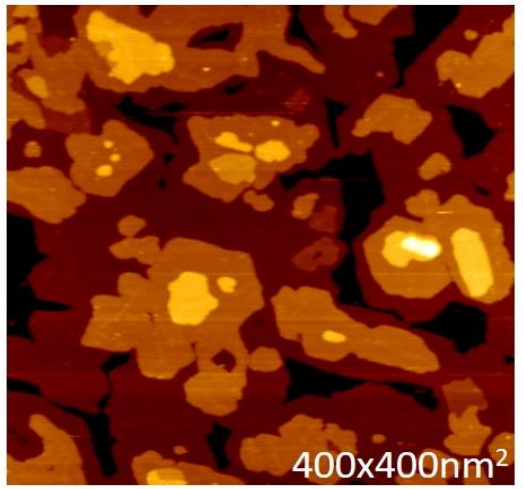
Cr intercalated $CrTe_2$

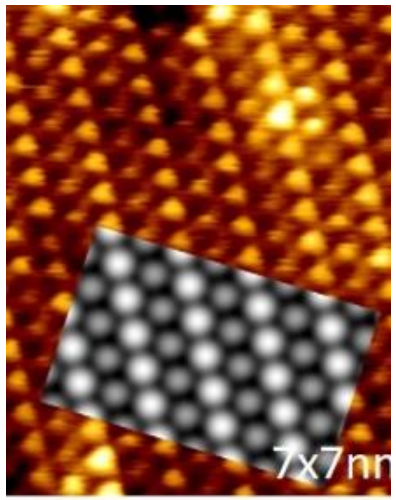
Can Cr_3Te_4 be used as for van der Waals heterostructures?

Ab-intio MD simulations suggest that Cr3Te4 is stable on a vdW substrate with a vdW gap.

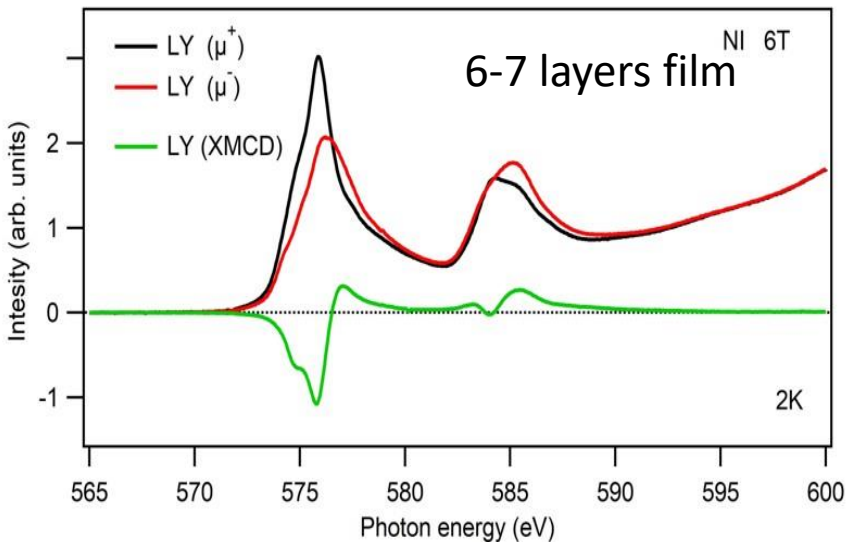


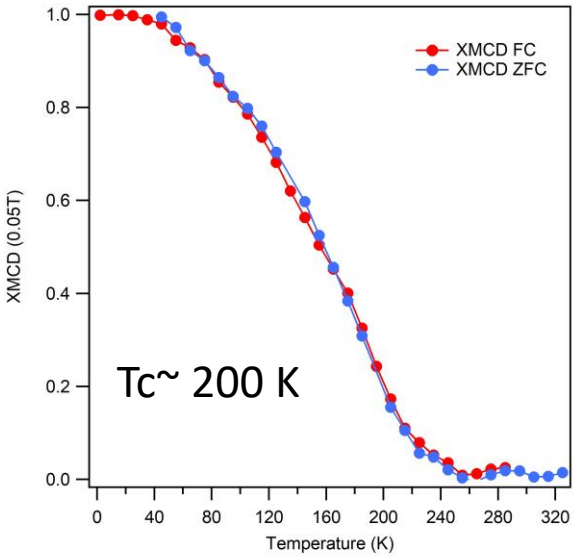
Van der Waals gap

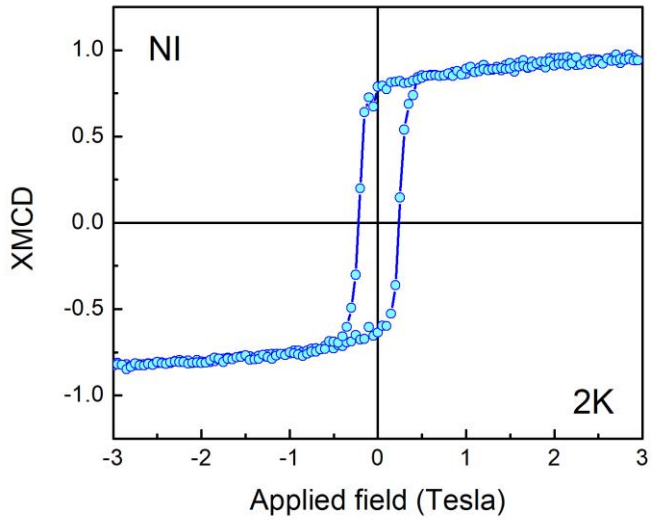




Multilayers of MBE grown Cr_3Te_4 are ferromagnetic with potential for van der Waals epitaxy.







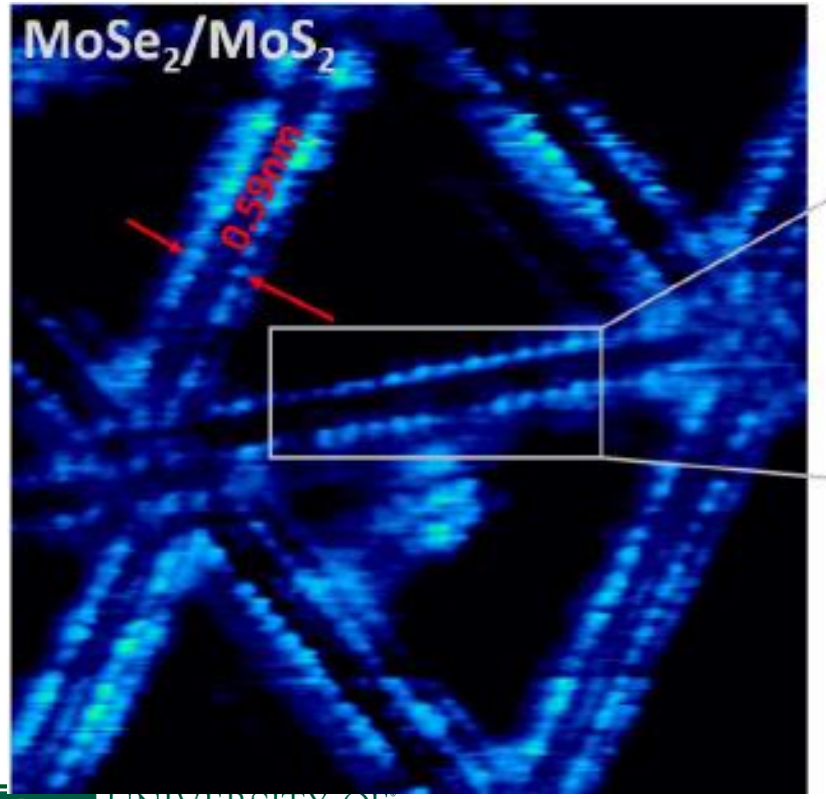
Conclusions for $CrTe_2$

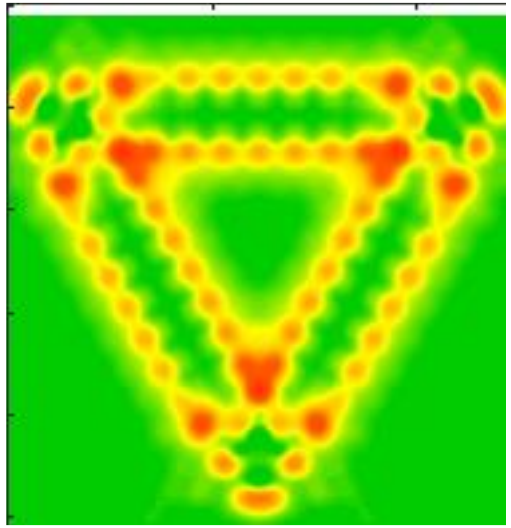
- $CrTe_2$ is metastable but can be stabilized as a monolayer when grown at low growth temperatures (~ 300 °C) by MBE.
- The monolayer is semiconducting 1H-phase with a gap ~ 0.3 eV (smaller than predicted by DFT) and thus is not ferromagnetically ordered.
- Self-intercalated $CrTe_2$ (Cr_3Te_4) may be grown as magnetic van der Waals materials.

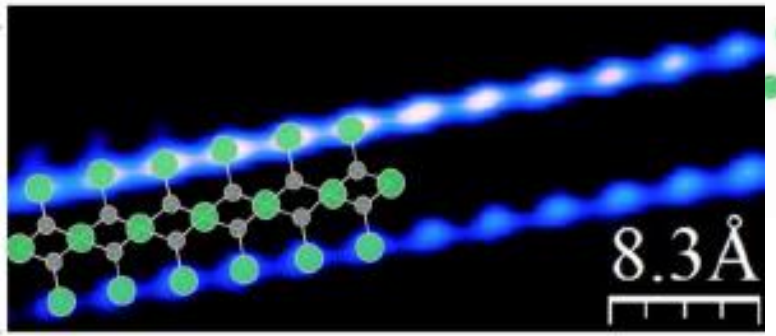
Outline

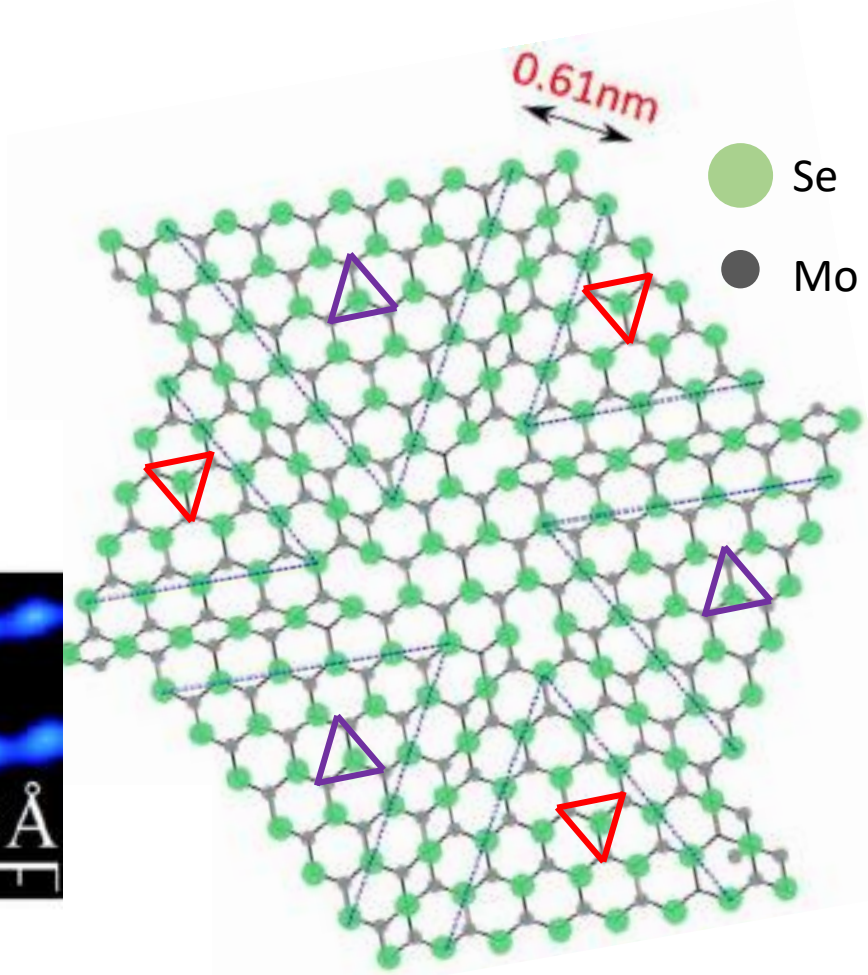
1. VSe_2: Competition between ferromagnetic and charge density order?
2. VTe_2: A new monolayer CDW material.
3. $CrTe_2$ monolayer synthesis by van der Waals epitaxy. But is it 1T or 1H?
4. Magnetic defects and dopants: Possible diluted ferromagnetic 2D semiconductors.

Line –defects in MBE grown $MoSe_2$ and $MoTe_2$



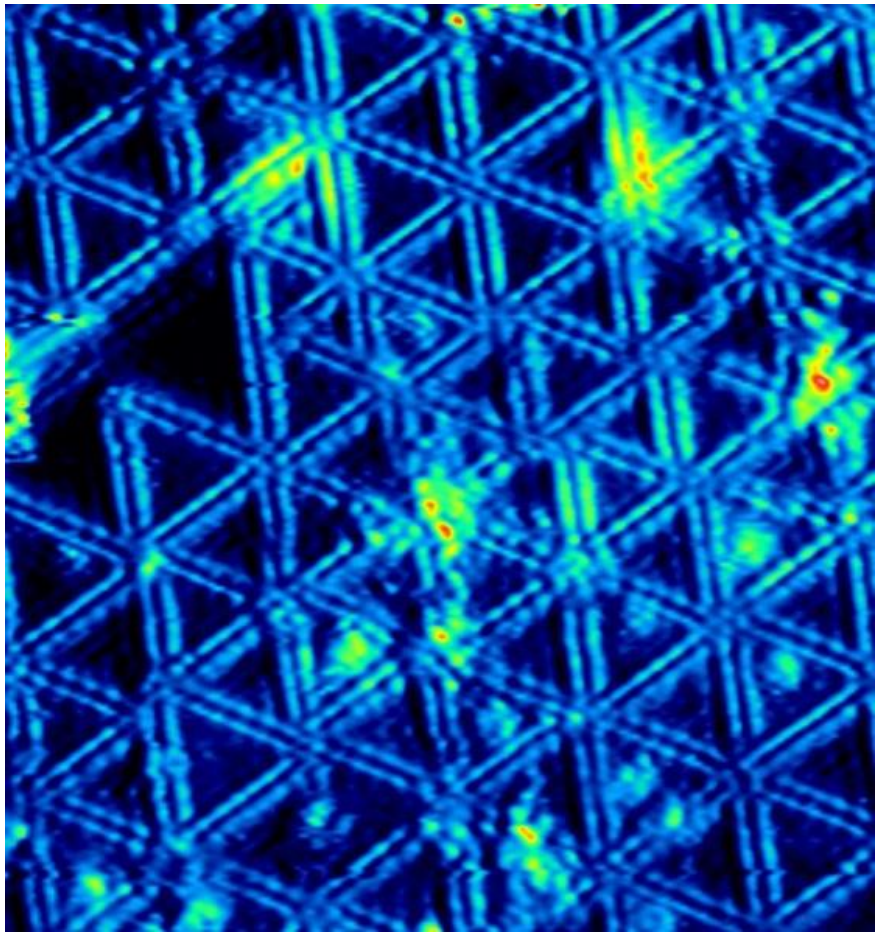


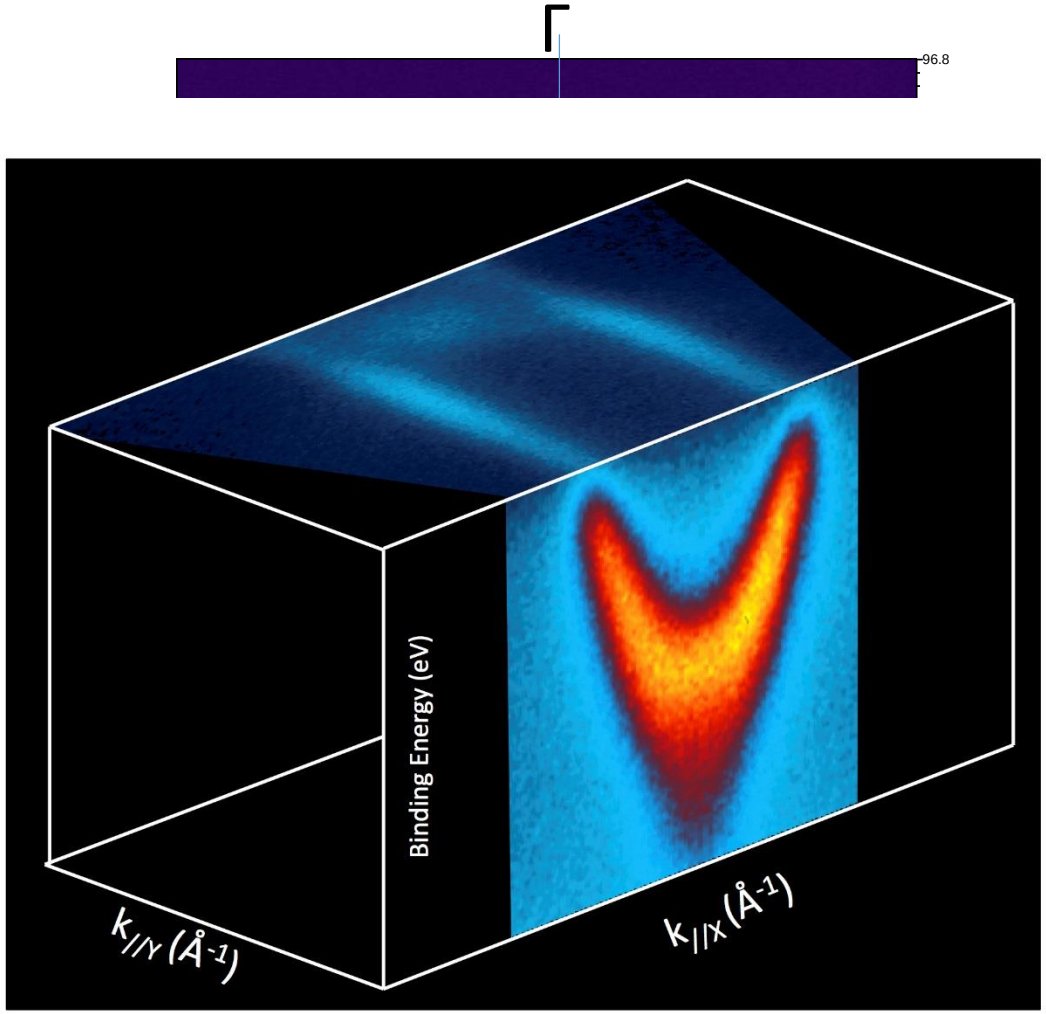




Y. Ma…. M.B. Nature Commun. 8, 14231 (2017)

Are grain boundaries one dimensional electronic systems?

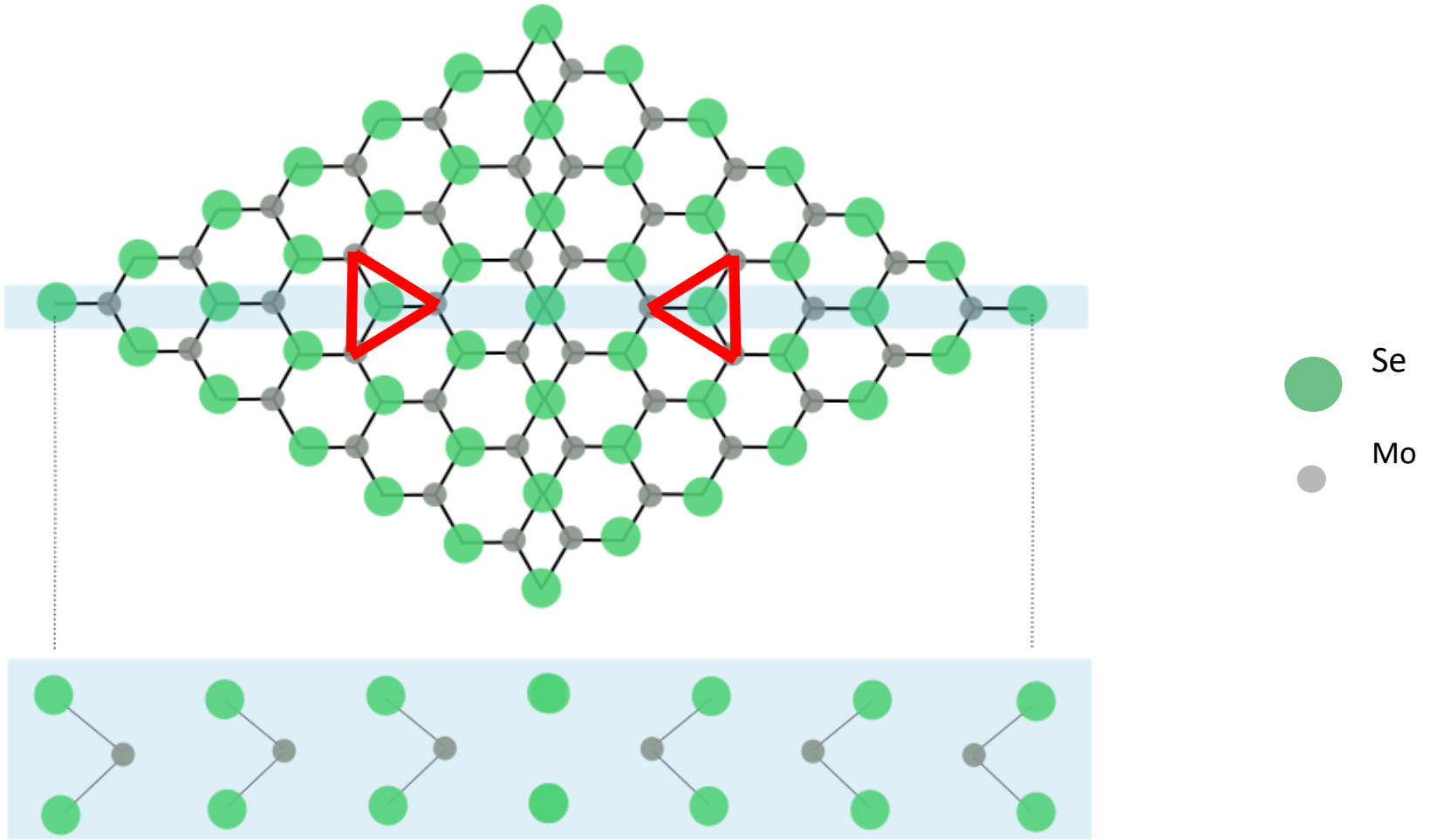




=> these twin grain boundaries are perfect one-dimensional metals?

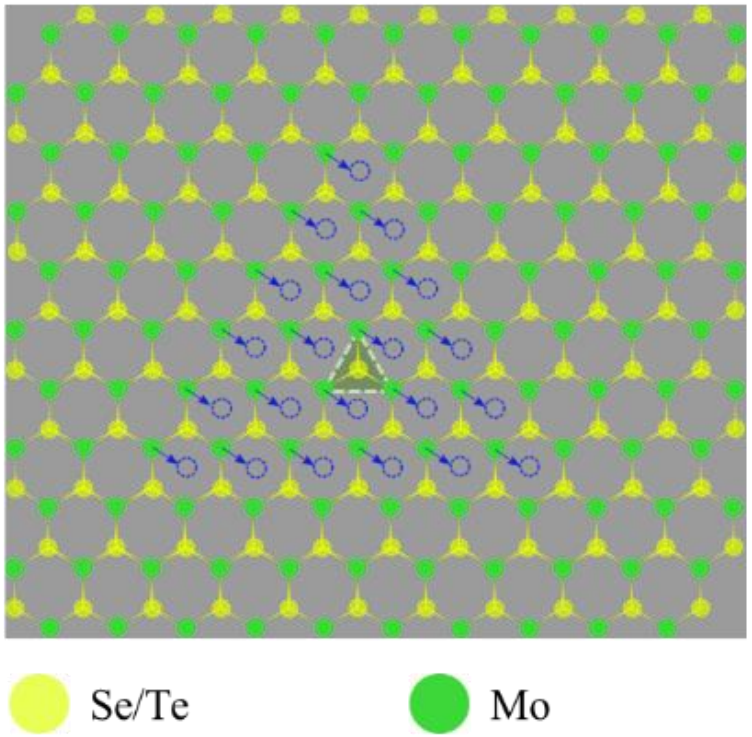
Y. Ma…. M.B. Nature Commun. 8, 14231 (2017)

Twin grain boundaries in TMDs



How do these grain boundaries form?

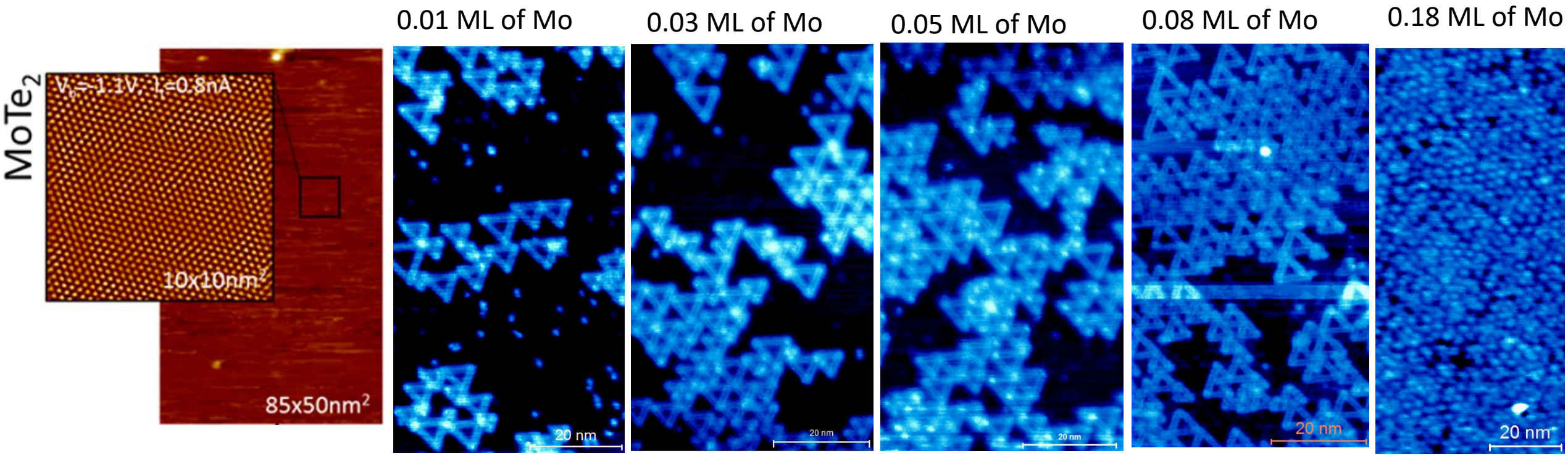
Why and how do these twin grain boundaries form? – Incorporation of excess Mo.



Can we form these grain boundaries by just incorporation of excess Mo into the TMD lattice?

How do these grain boundaries form?

Deposition of Mo on $MoTe_2$ single crystal substrate at 350 °C

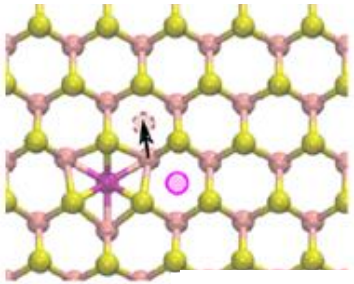


=> Up to ~20% of excess Mo can be incorporated into $MoTe_2$ lattice to form Mo-rich twin grain boundaries

P.M. Coehlo.... M.B.; ACS Nano 12, 4, 3975-3984 (2018)

Proposed formation of mirror twin boundaries by adding Mo

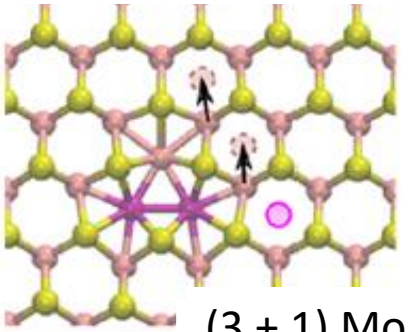
(1 + 1) Mo



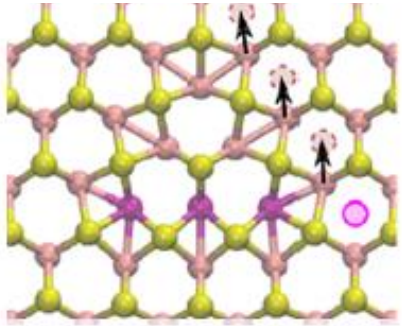
added

shifted

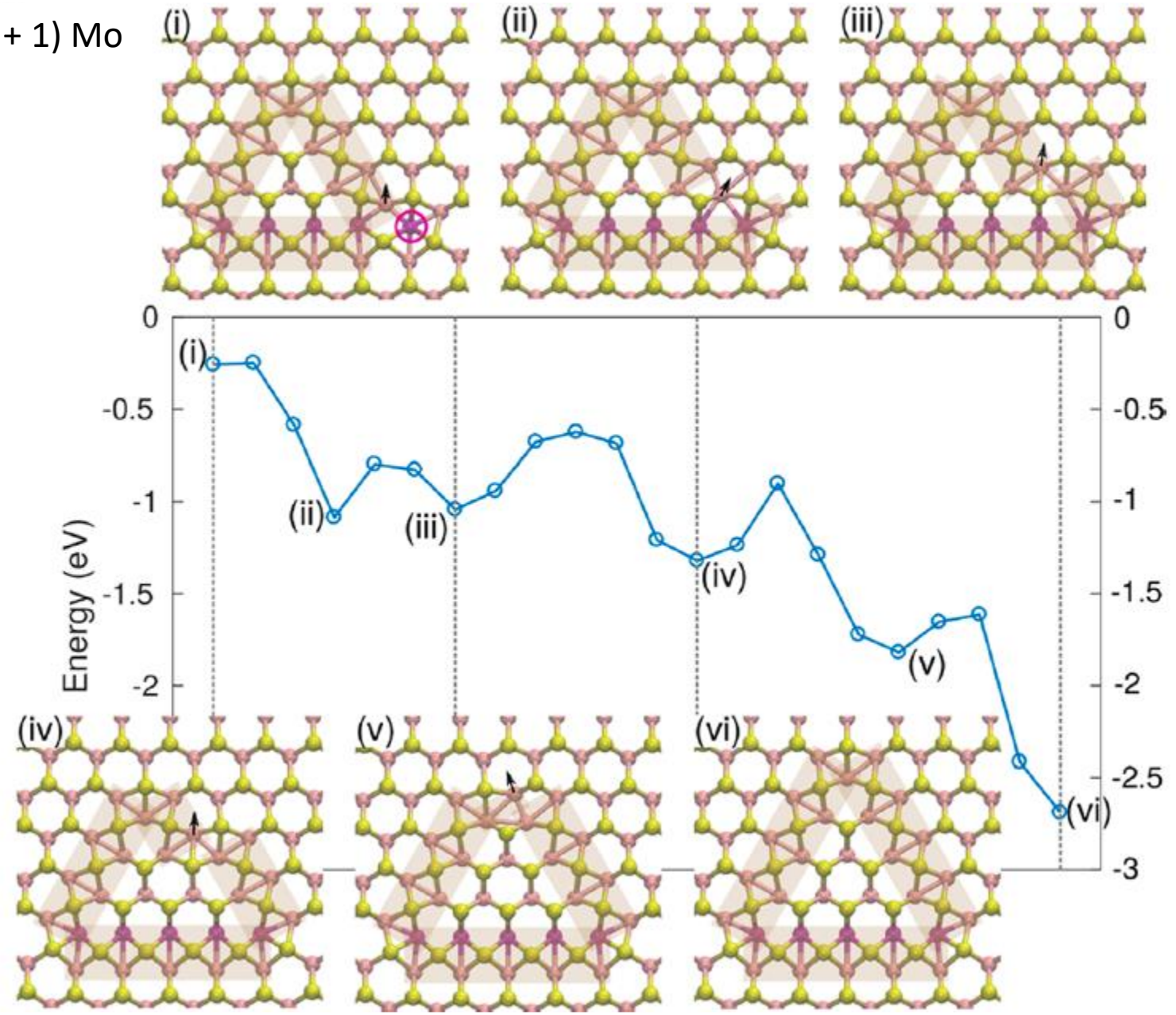
(2 + 1) Mo



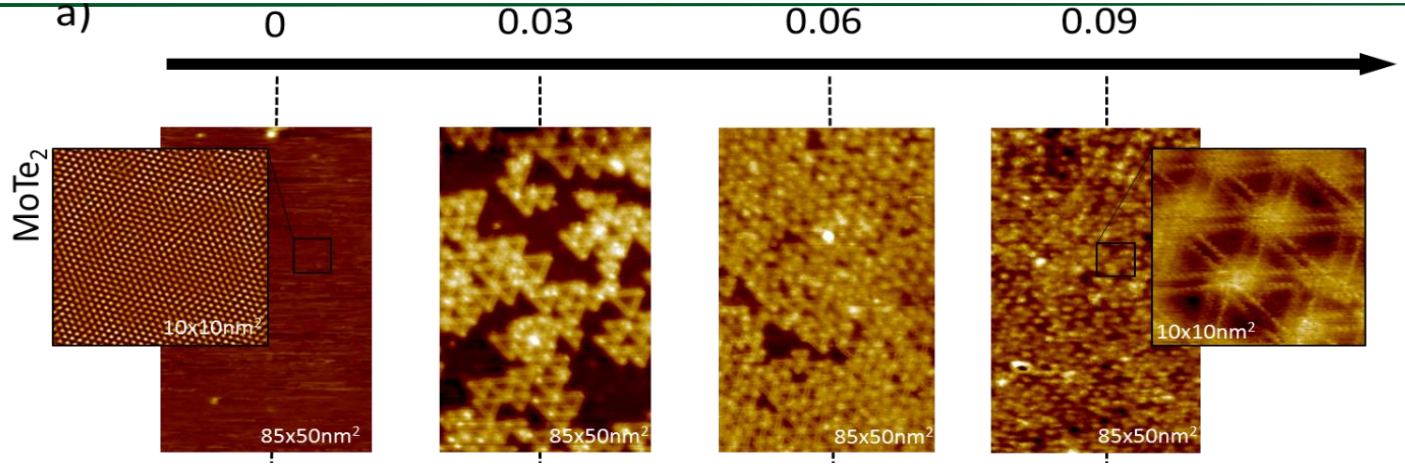
(3 + 1) Mo



(4 + 1) Mo



Comparison between $MoTe_2$, $MoSe_2$ and MoS_2



Twin grain boundary formation by incorporation of excess Mo is only observed for $MoTe_2$ and $MoSe_2$, but not for MoS_2.

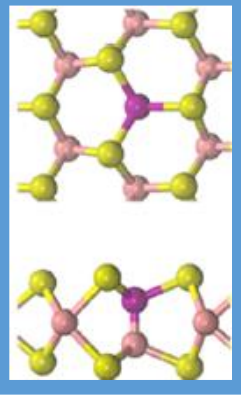
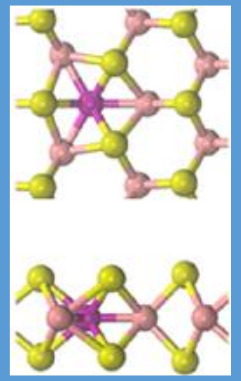
P.M. Coehlo.... M.B.; ACS Nano 12, 4, 3975-3984 (2018)

DFT simulations for Mo-incorporation into Mo-TMDs

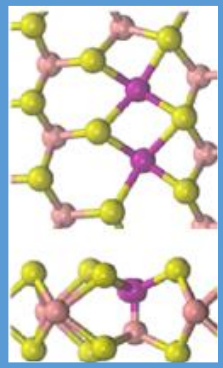
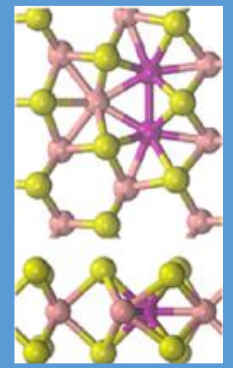
Arkady Krasheninnikov (Helmholtz Zentrum Rossendorf, Germany) & Pekka Hannu-Komsa (Aalto Univ. , Finland)

Two Mo excess atom:

One excess Mo-atom ($\mu_{Mo} = \mu_{Mo}$[atom])

System	Ad-atom	Split-interstitial	Interstitial
			
$MoTe_2$	- 1.08 eV	- 2.15 eV	**- 3.85 eV**
$MoSe_2$	- 1.05 eV	**- 2.26 eV**	- 1.72 eV
MoS_2	- 1.48 eV	**- 2.51 eV**	0.14 eV

Two excess Mo-atoms ($\mu_{Mo} = \mu_{Mo}$[dimer])

System	Ad-dimer	2-Split-interstitial	2-Interstitials
			
$MoTe_2$	- 0.60 eV	0.01 eV	**-4.48 eV**
$MoSe_2$	- 0.46 eV	1.13 eV	**-1.35 eV**
MoS_2	**- 0.74 eV**	0.91 eV	1.45 eV

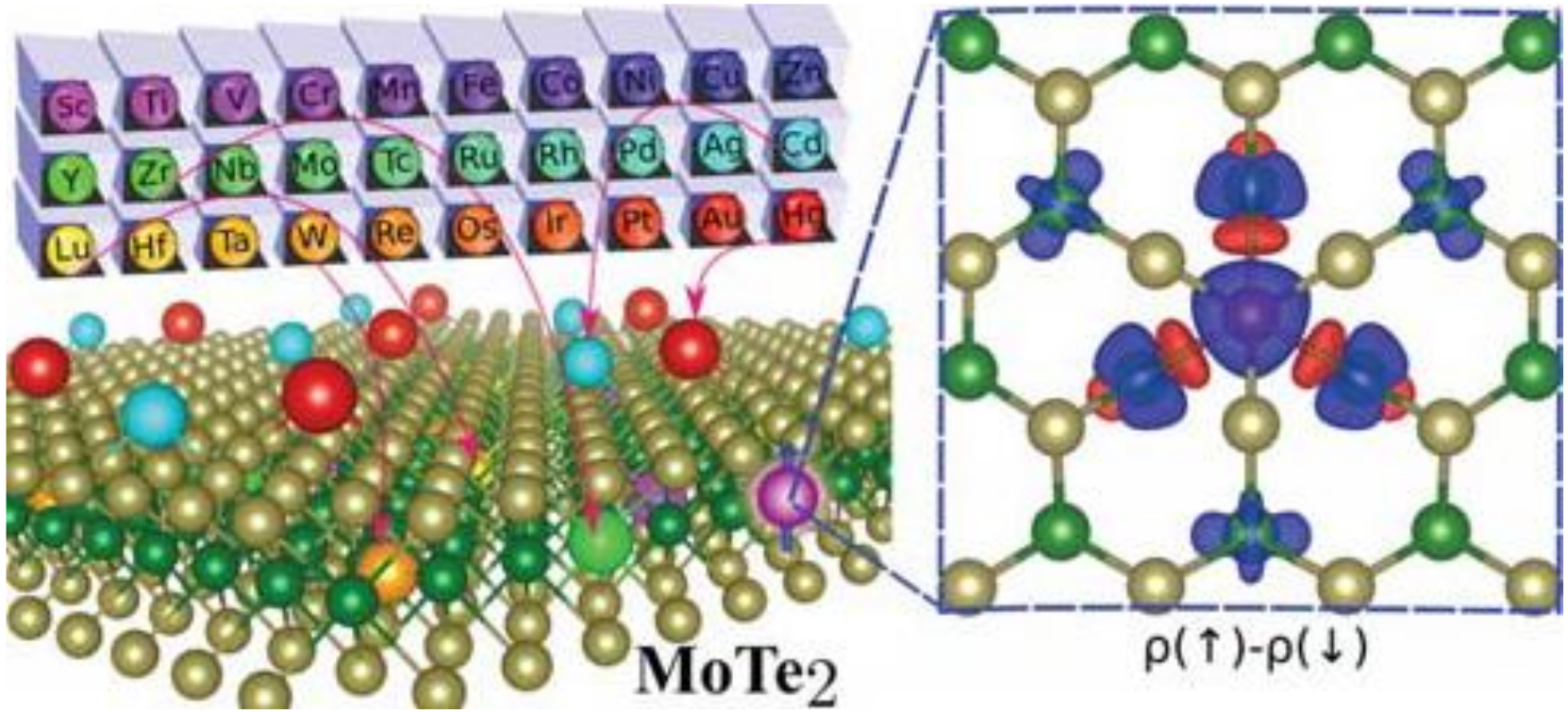
P.M. Coehlo.... M.B.; ACS Nano 12, 4, 3975-3984 (2018)

Impurity doping in Mo-dichalcogenides

NANO LETTERS
Letter
pubs.acs.org/NanoLett

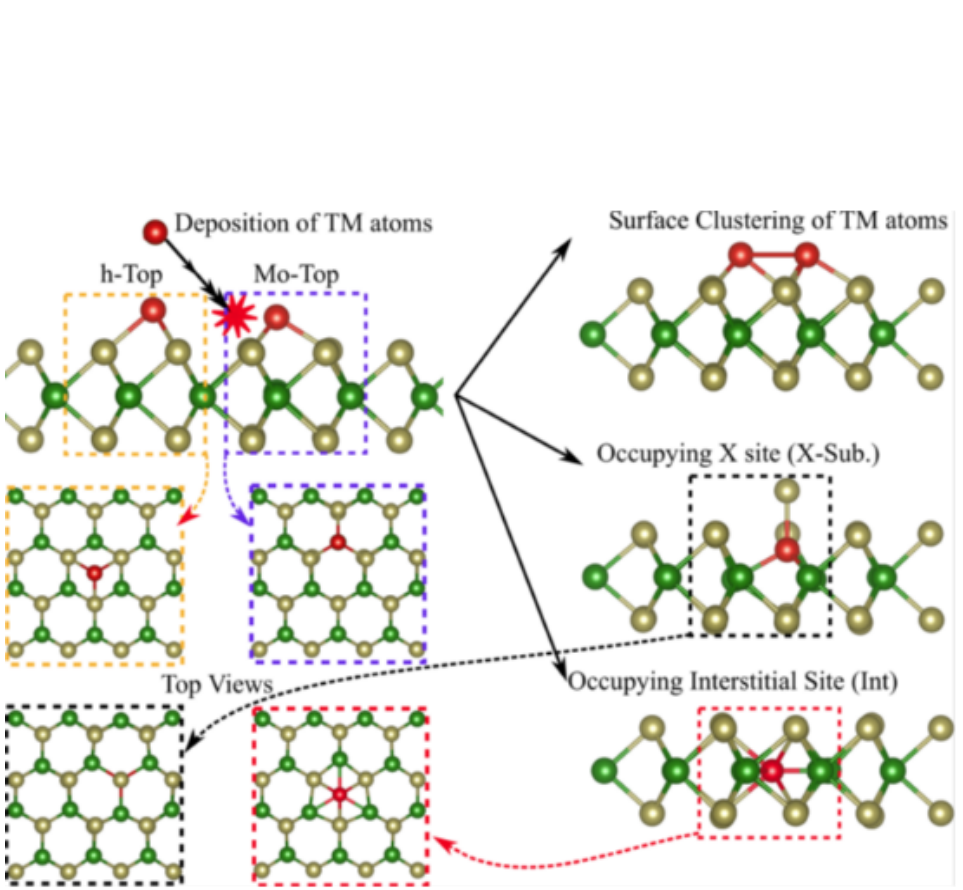
Which Transition Metal Atoms Can Be Embedded into Two-Dimensional Molybdenum Dichalcogenides and Add Magnetism?

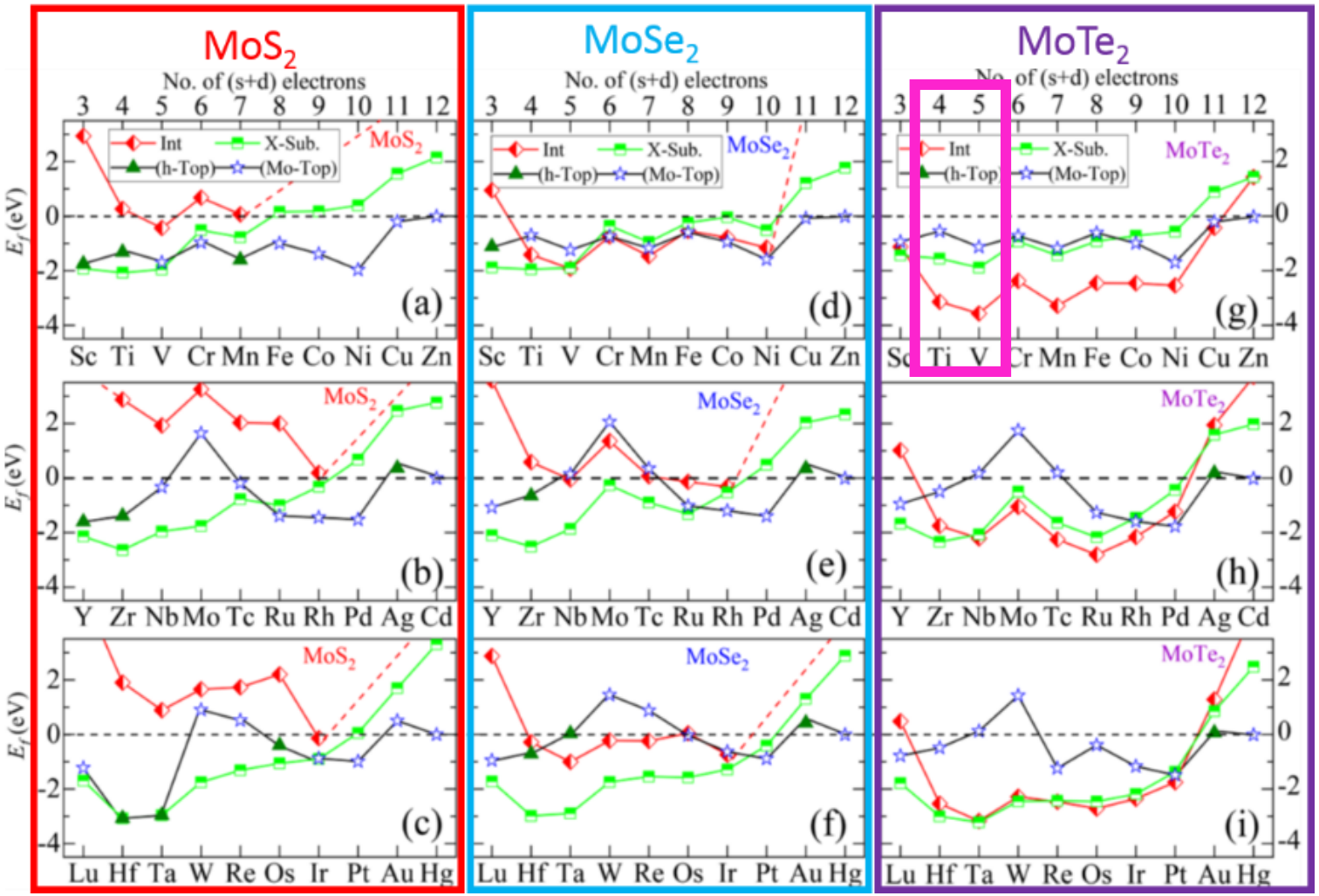
J. Karthikeyan,[†] Hannu-Pekka Komsa,[†] Matthias Batzill,[‡] and Arkady V. Krasheninnikov*[,§,†]



*Nano Lett.*2019 19, 4581-4587

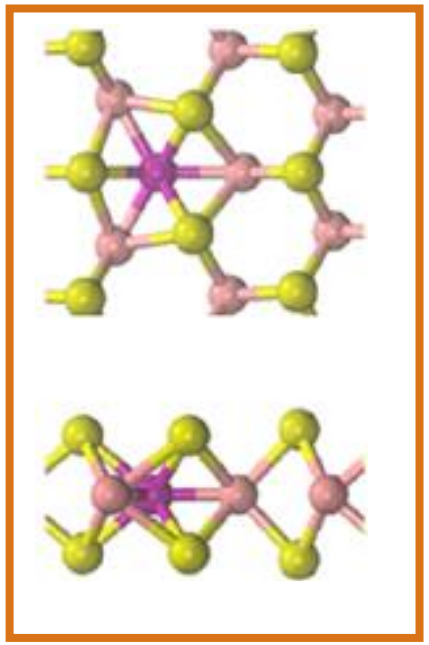
Impurity doping in Mo-dichalcogenides





*Nano Lett.*2019 19, 4581-4587

Impurity doping in Mo-dichalcogenides



V= 1 μ_B
Ti= 0 μ_B

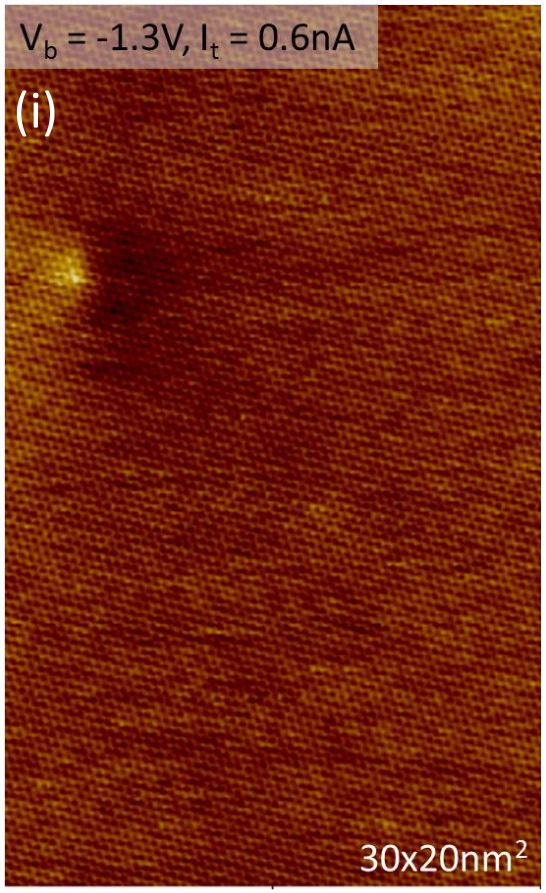
1. Can we dope $MoTe_2$ in interstitial sites with TM by just vapor deposition?

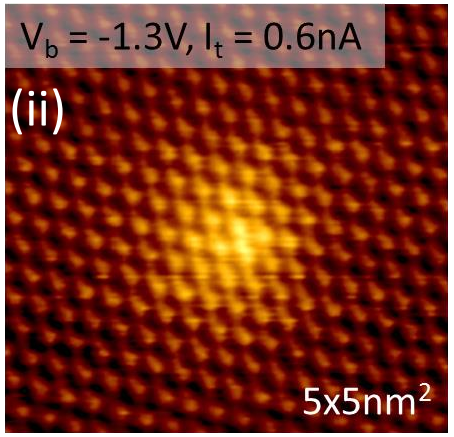
2. Can magnetic dopants introduce long range magnetic ordering in MoTe2, i.e. can we form a 2D dilute ferromagnetic semiconductor?

Adv. Electr. Mater. 1900044 (2019)

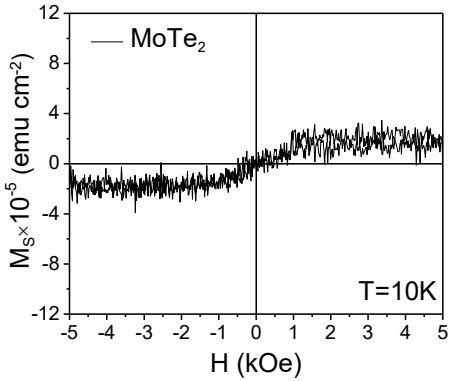
Undoped $MoTe_2$

As received $MoTe_2$

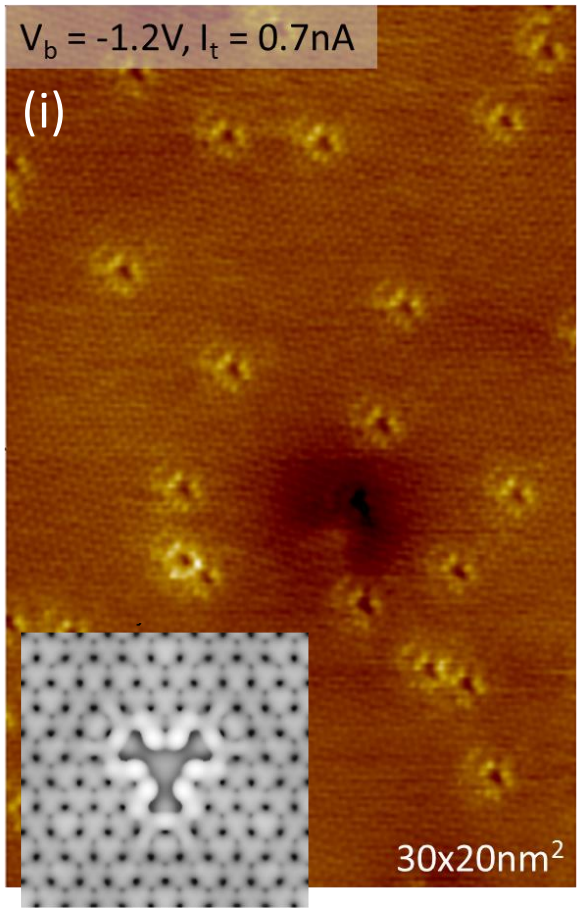


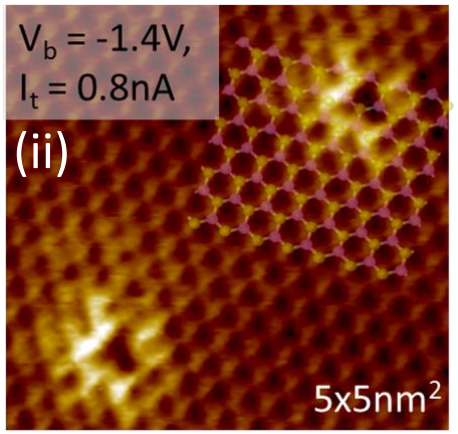


(iii)

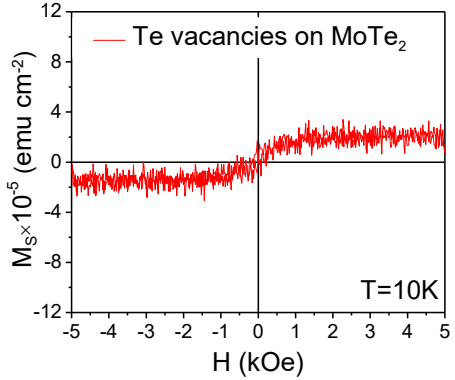


Vacuum annealing to 600 K: Te-vacancies





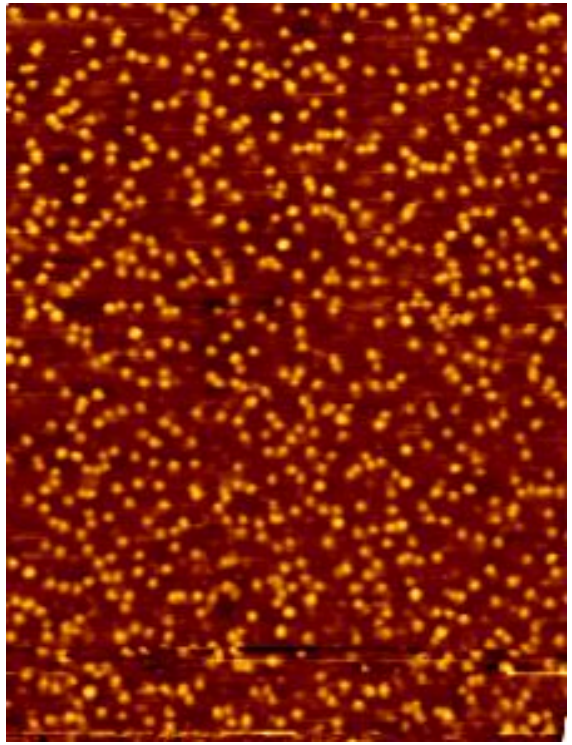
(iii)

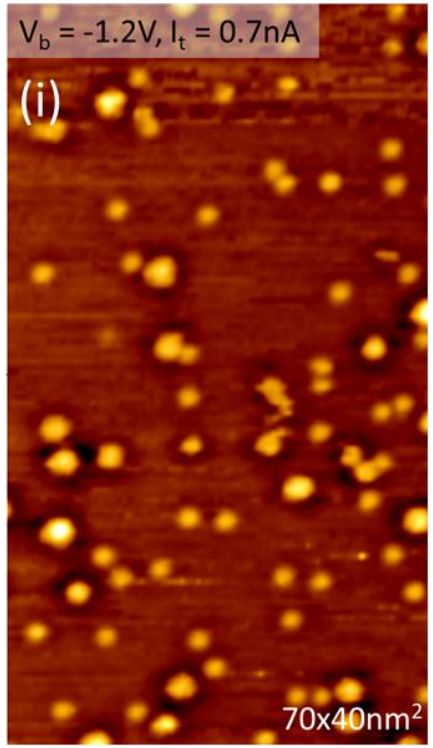


=> Single crystal $MoTe_2$ only shows weak (defect induced) ferromagnetic ordering. Te-vacancies do not change magnetization.

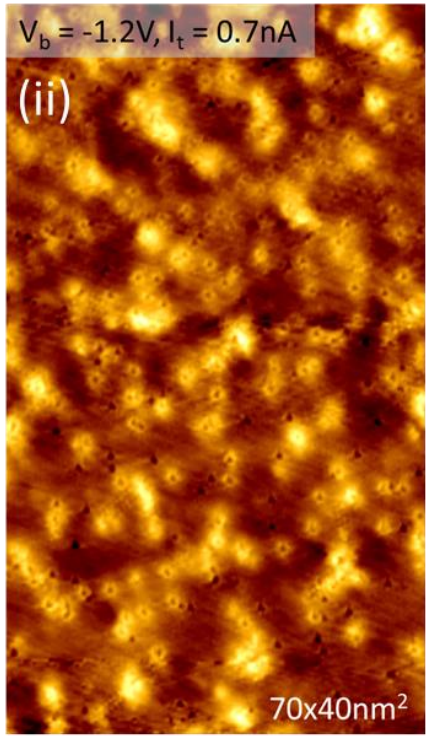
Adv. Electr. Mater. 1900044 (2019)

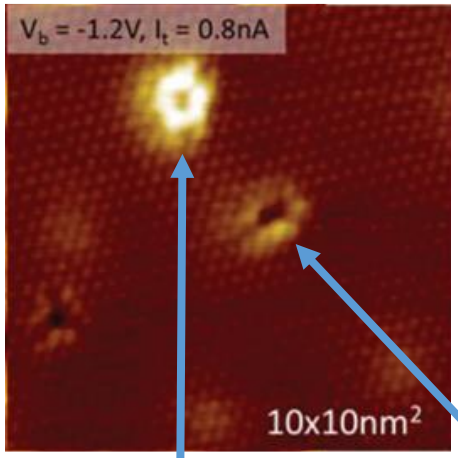
V:$MoTe_2$



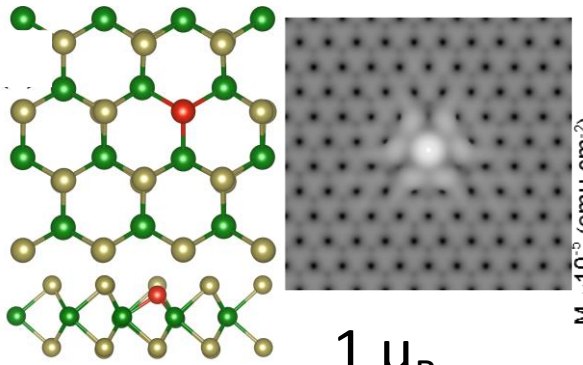


Annealing to 600 K



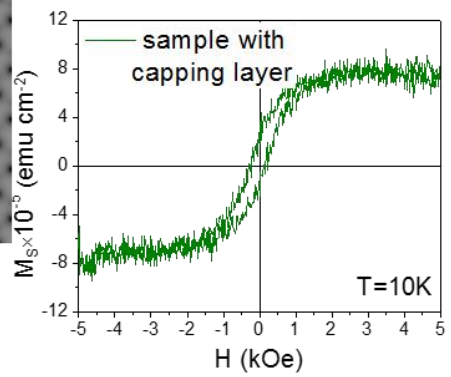


Te-vacancy

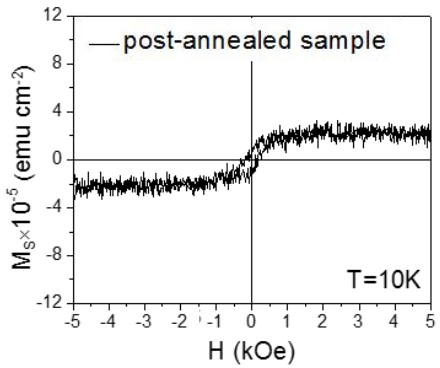


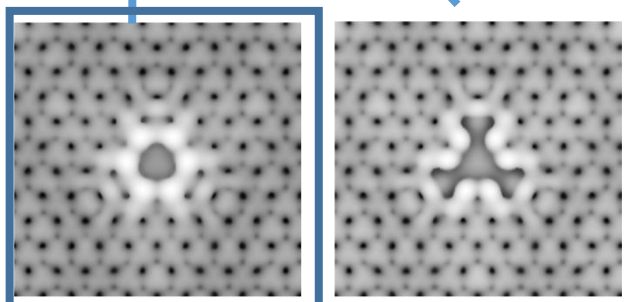
1 μ_B

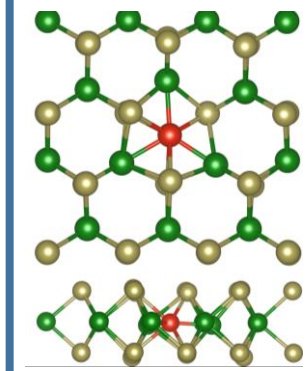
(iii)



(v)







0 μ_B

Te-vacancy with V interstitial

Adv. Electr. Mater. 1900044 (2019)

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Enabling room temperature ferromagnetism in monolayer MoS_2 via in situ iron-doping

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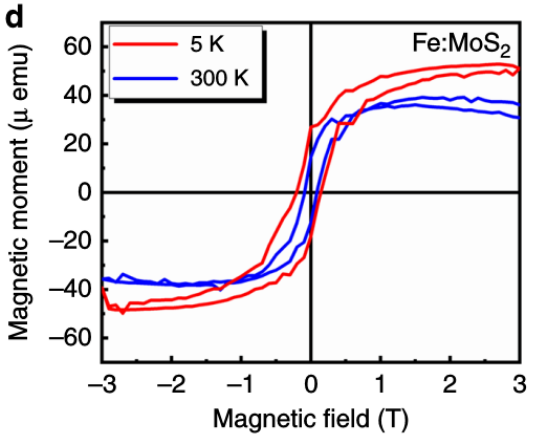

van der Waals epitaxy of Mn-doped $MoSe_2$ on mica

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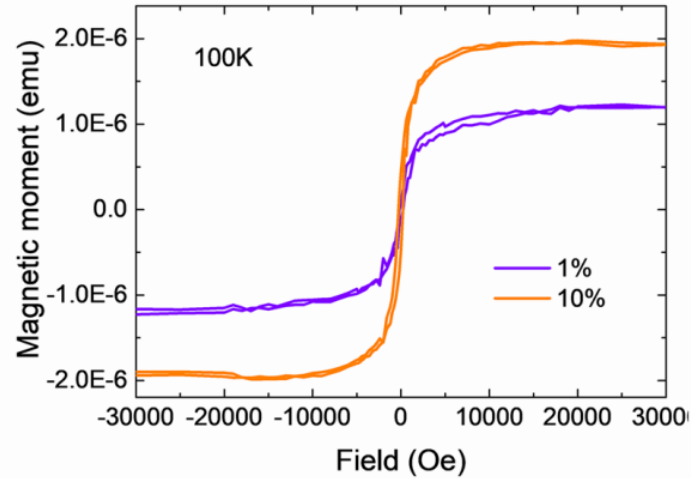

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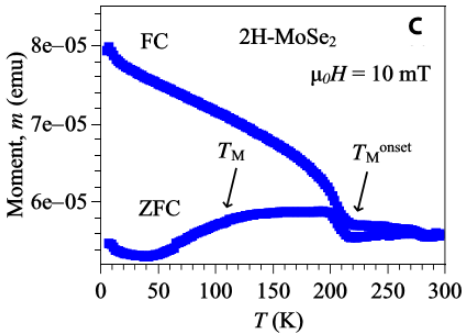

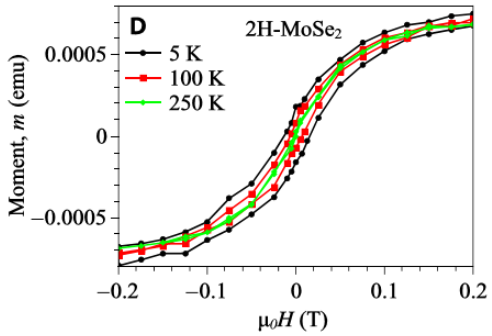

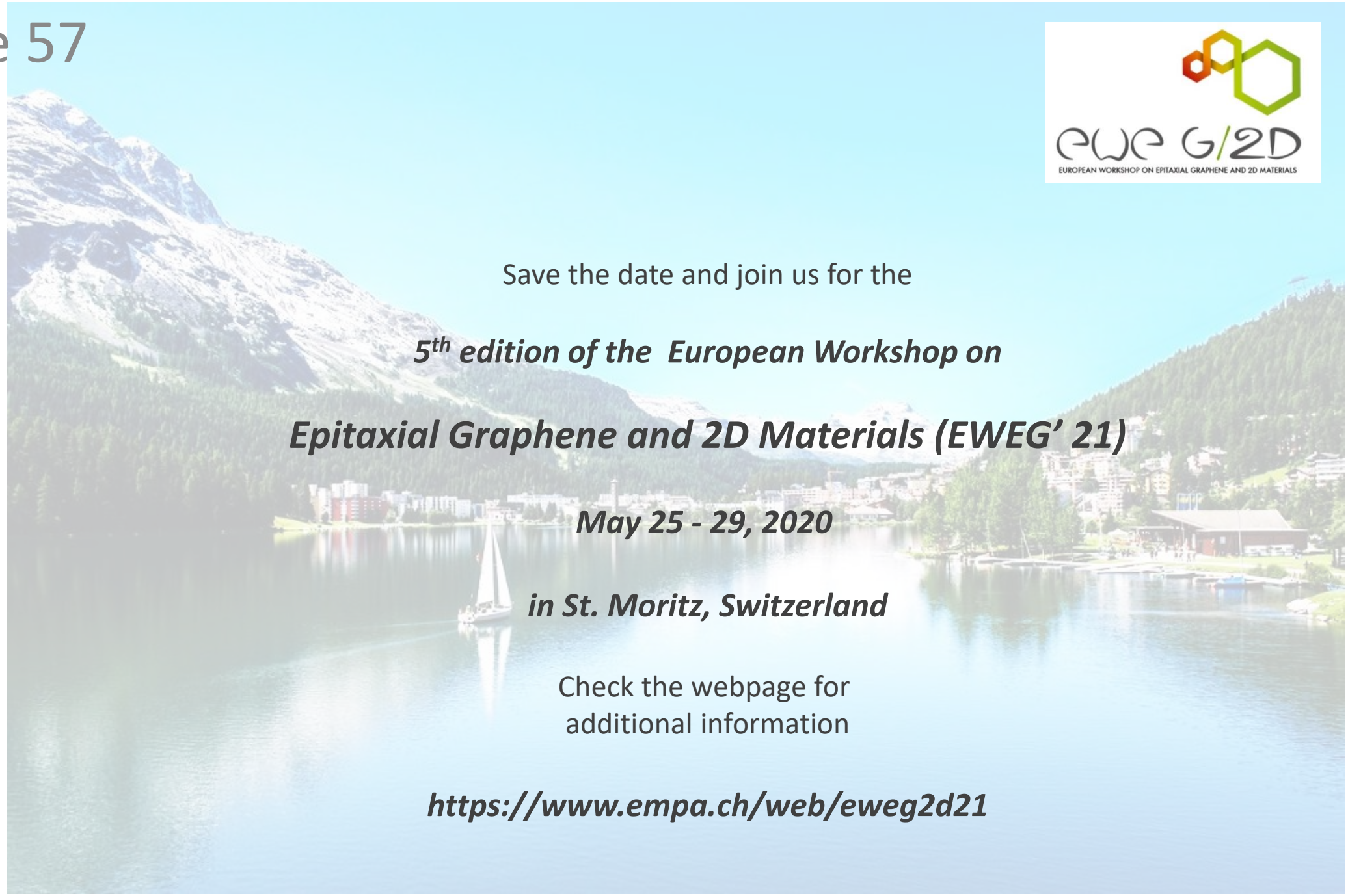

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