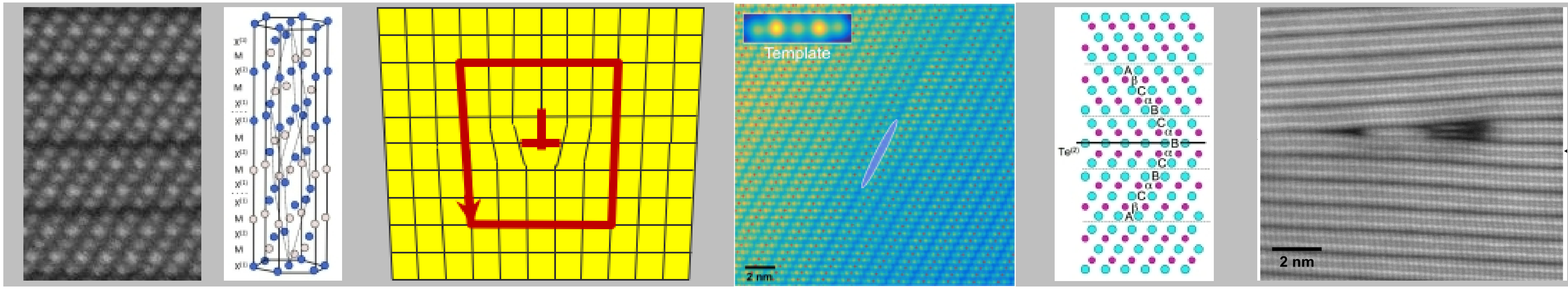
SAND2021-6744PE




# Atomic-Scale Investigations of Interfaces and Line Defects in Chalcogenide Materials


**Douglas L. Medlin**

**Sandia National Laboratories, Livermore, California, USA**

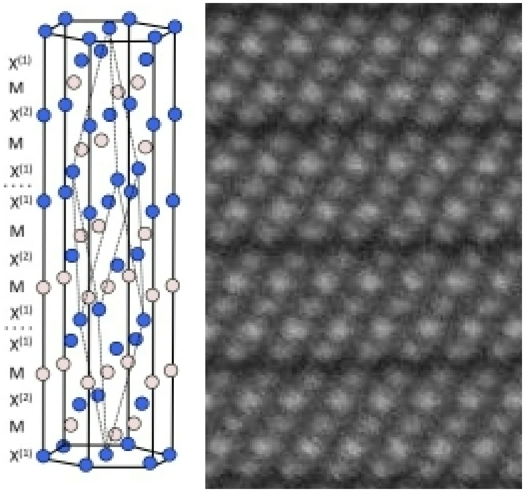
**dlmedli@sandia.gov**



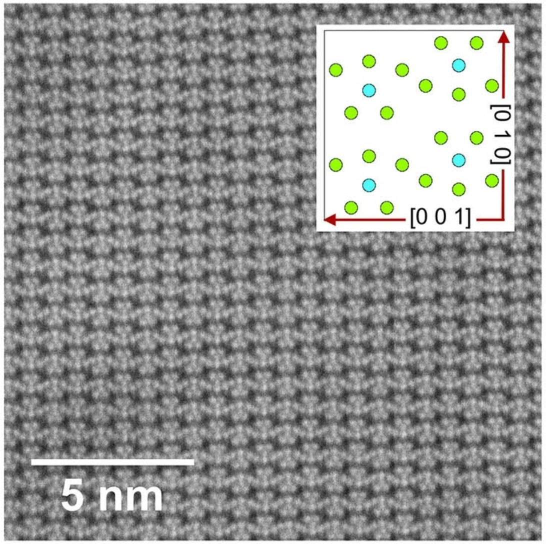
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

# Layered Chalcogenides: Diversity of compositional and structural arrangements

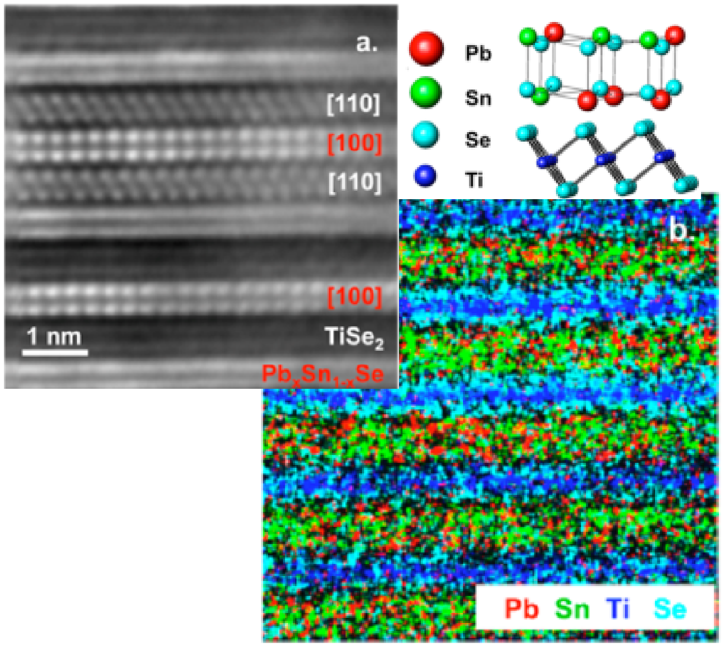
*Example: $Bi_2Te_3$*



*Example: $ZrTe_5$*




*Example: PbSnSe/$TiSe_2$ van der Waals Heterostructures*




**Anisotropic, layered structures contribute to useful and interesting thermal, electronic, optical properties,**

***Thermoelectrics, Photothermoelectric Effect, Topological Insulators, Dirac Semimetals***

# Interfaces and Dislocations are important

•**Impact on functional properties:**

-*Electron and phonon scattering*

➔ tradeoffs for thermoelectrics

-*Dislocations predicted to support novel quantum mechanical states.*

• **Processing:**

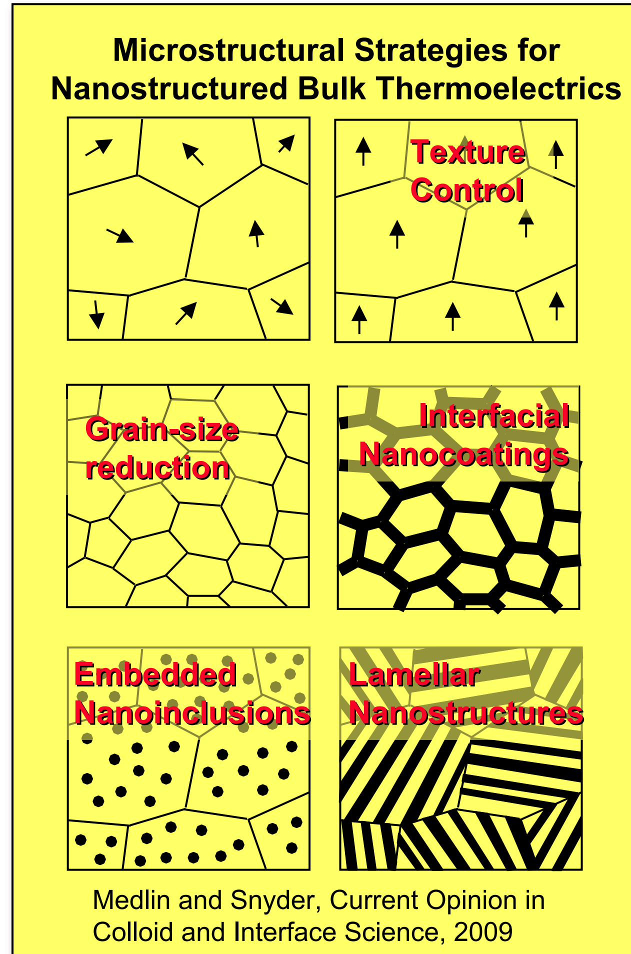
***Bulk approaches to thermoelectrics***

Hot pressing, Extrusion, nanostructuring, phase-transformations

***Thin film nanostructure growth, epitaxy,***

Accomodation of coherency strain

New layer nucleation

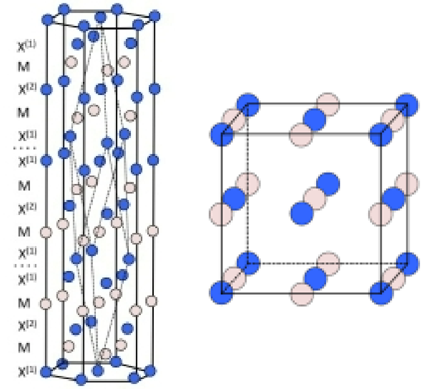



**Our focus: Atomic resolution electron microscopy to clarify structure of crystal defects in the layered chalcogenides**

Sandia National Laboratories

# Outline

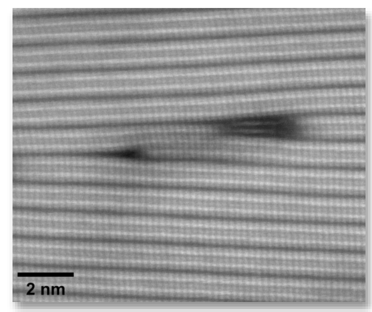
- ***Crystal structure in tetradymite-type compounds***
  - •Layered structure gives flexibility in accommodating compositional variations
  - •Close structural relationship to rock-salt chalcogenides

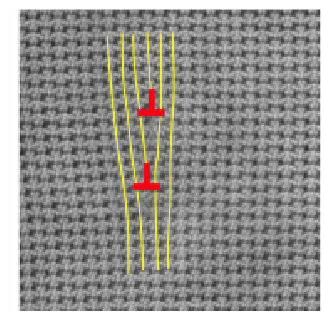


- ***Dislocation Structure:***

  Modes of core spreading and dissociation.

  $Bi_2Te_3$, $ZrTe_5$

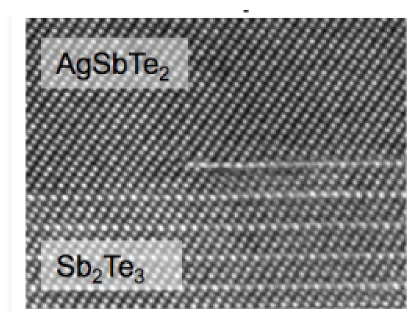


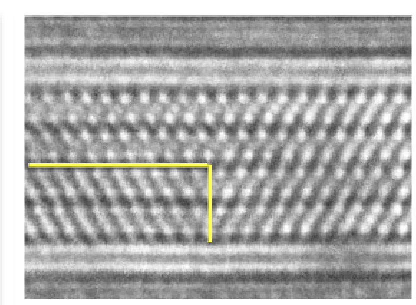



- ***Interfaces:***

  Interfacial line-defects in phase-transformation and strain accommodation

  Defects in van der Waals heterostructure thin films



Sandia National Laboratories

# Acknowledgements

**Sandia**

K. Erickson
J.D. Sugar
N.Y.C. Yang
C.Spataru
M. Siegal,
S. Limmer
W.G. Yelton
P. Sharma
W. Pan

**George Mason University**

Y. Mishin

**NIST**

L. Hale

**University of Oregon**

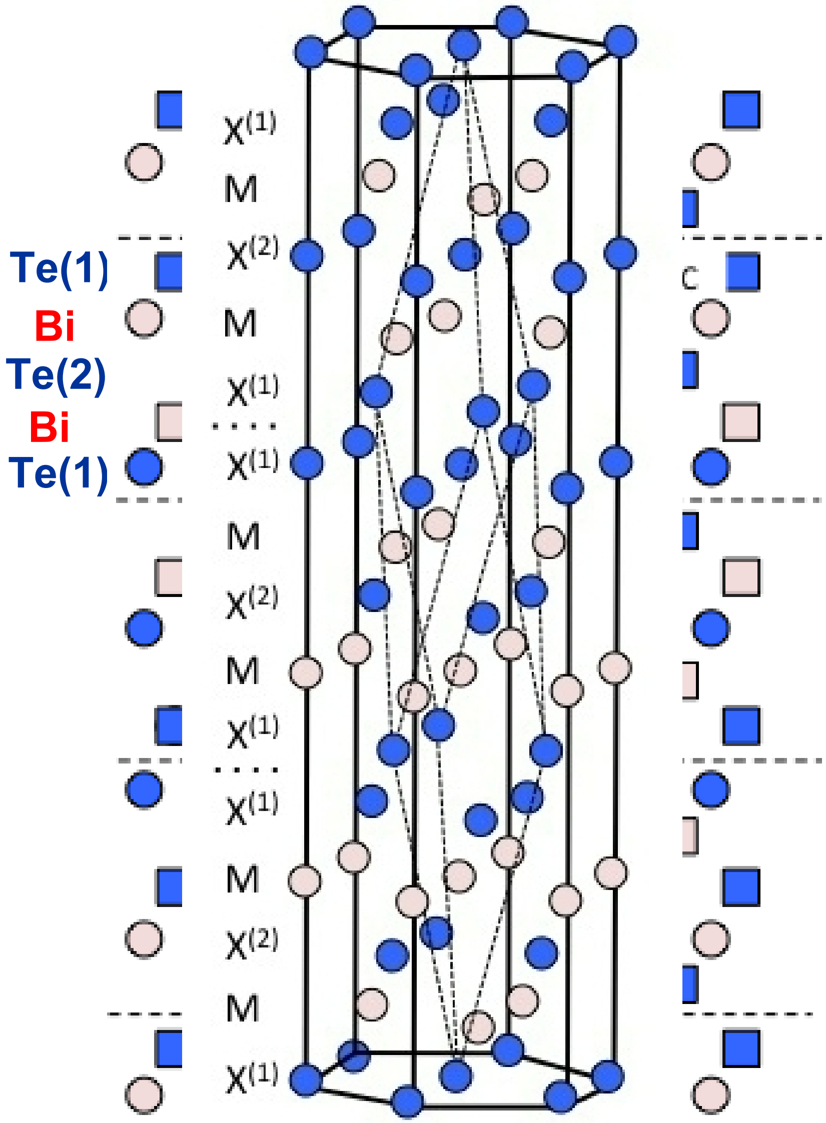
R. Gannon
D.Hamann
J. Ditto
D.Johnson

**California Institute of Technology**

N.A. Heinz,
T. Ikeda,
G.J. Snyder
(present address Northwestern Univ)

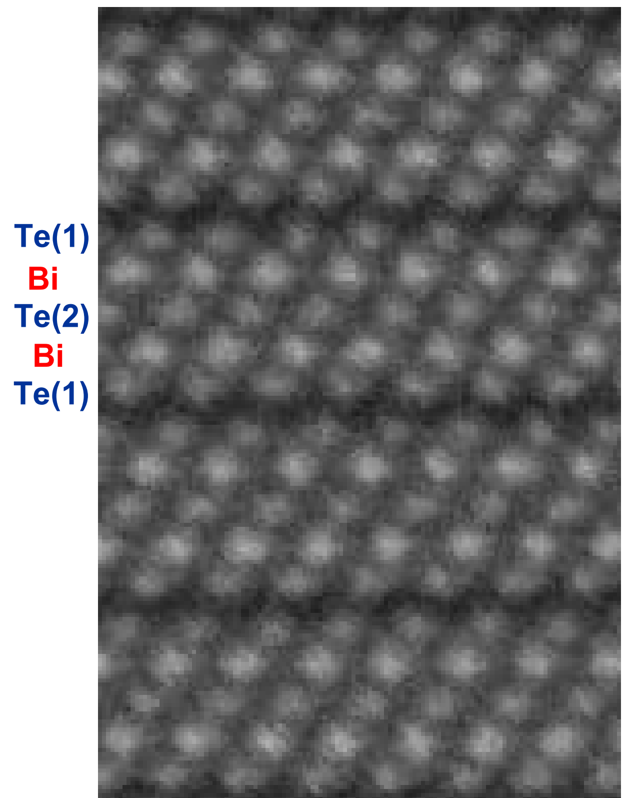
Sandia National Laboratories

# Bismuth Telluride ($Bi_2Te_3$): Crystal Structure




- Rhombohedral (R-3m) structure
- Based on tetradymite ($Bi_2STe_2$) prototype
- Isomorphous with $Bi_2Se_3$, $Sb_2Te_3$
- Three crystallographically distinct atomic sites
- $Te^{(1)}$-$Te^{(1)}$ layers: van der Waals bonding

HAADF-STEM



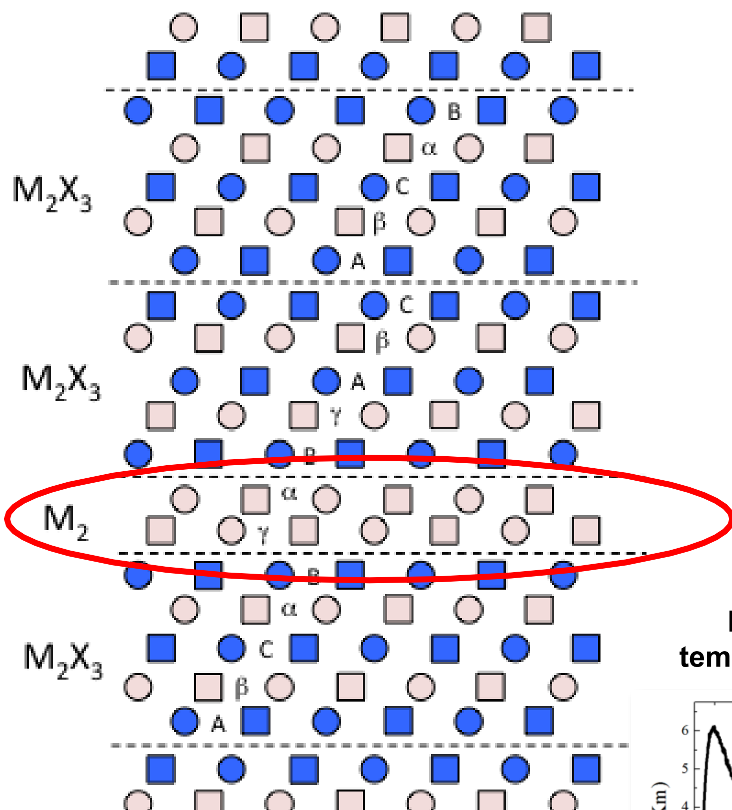

Bi: Z=83

Te: Z=52

Atomic number difference enables Bi and Te to be distinguished in HAADF-STEM

# Layered structure allows flexibility in accommodating variations in composition

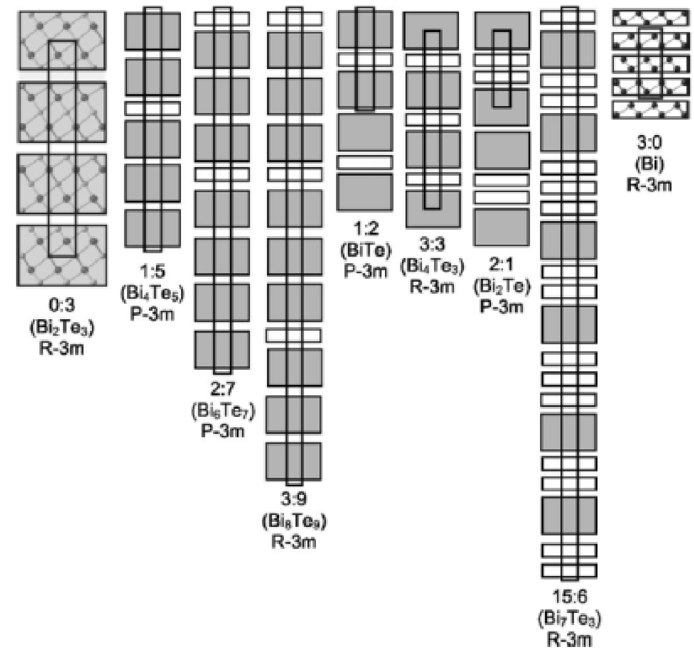
## Insertion of metal bi-layers



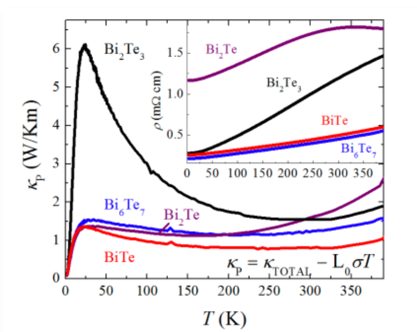

**Example:**
**$(Bi_2)_m(Bi_2Te_3)_n$ homologous series**

J.W.G. Bos, H.W. Zanderbergen, M.-H. Lee, N.P. Ong, R.J. Cava, Phys. Rev B 2007

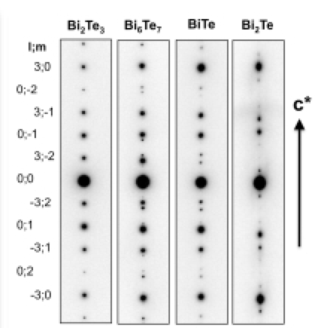
**Increasing Bismuth →**

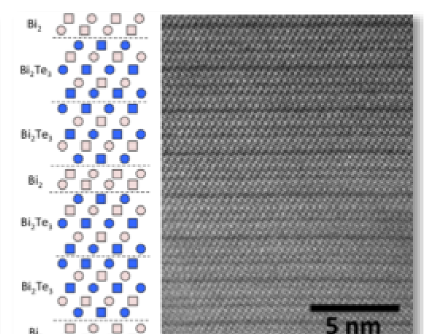



**Dramatic reduction in low-temperature thermal conductivity**




**Electron diffraction and HRSTEM show ordering in $(Bi_2)_m(Bi_2Te_3)_n$ series**



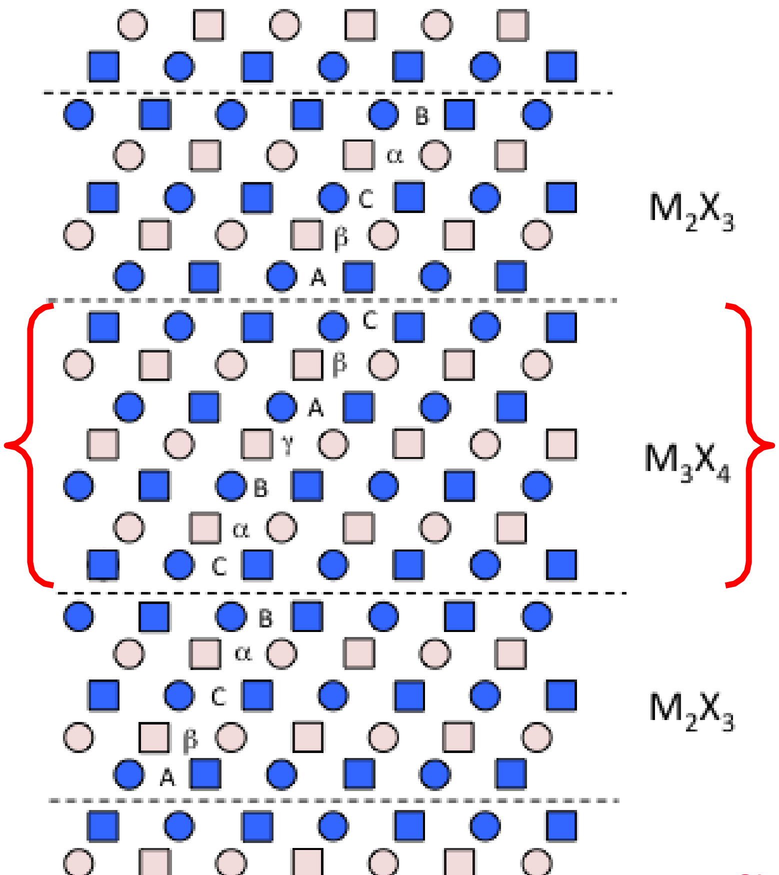




P. Sharma, D.Medlin, et al. PRB 83 (2011) 235209.

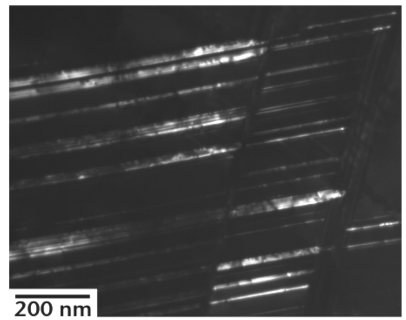
Medlin & Snyder, JOM 2013

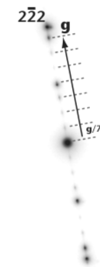
# Layered structure allows flexibility in accommodating variations in composition

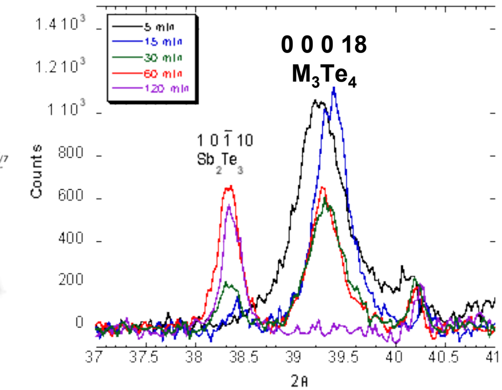
**Metal rich, 7-Layer $M_3X_4$ fault**

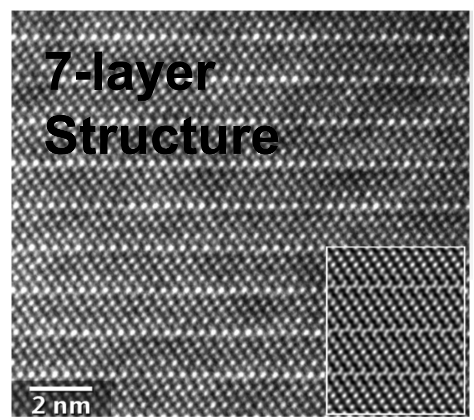


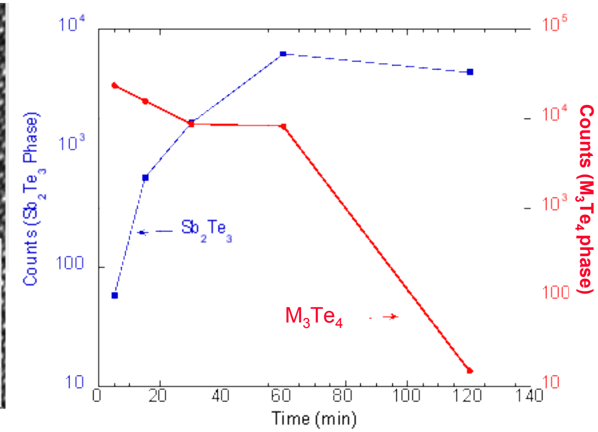

**Example: $(Ag,Sb)_3Te_4$ transition phase during nucleation of $Sb_2Te_3$ precipitates in $AgSbTe_2$**



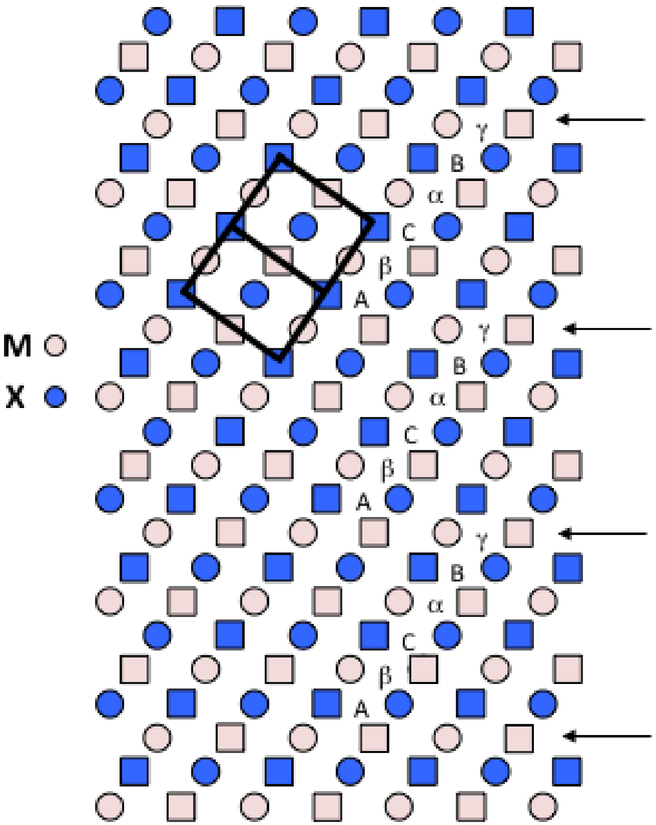

Sharma, Sugar, & Medlin, Journal of Applied Physics (2010).
Sugar and Medlin, Journal of Materials Science (2011)

Sandia National Laboratories

# Tetradymite and Rocksalt structures are closely related

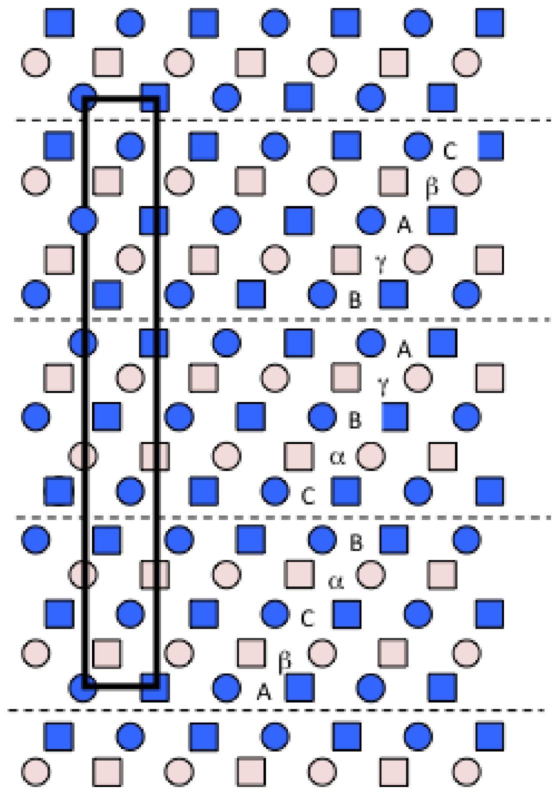
**Rocksalt (MX) Structure**

Fm-3m

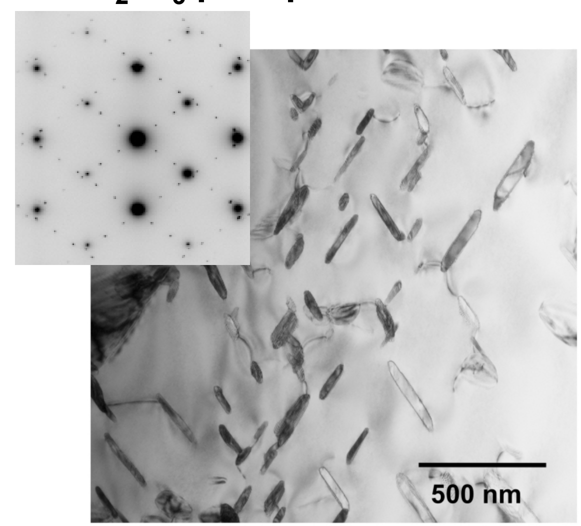



**Tetradymite ($M_2X_3$) Structure**

R-3m




**Example:**
**Crystallographically Aligned $Sb_2Te_3$ precipitates in PbTe**

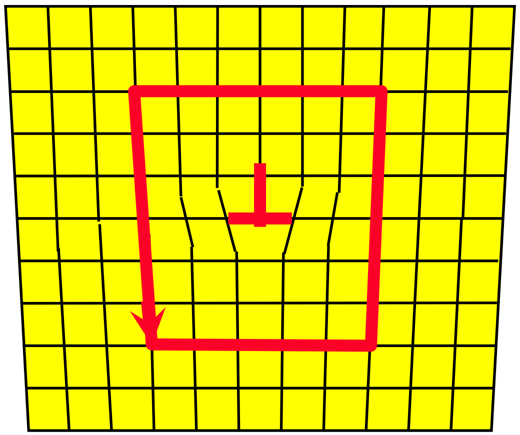



Heinz, Snyder, Ikeda, and Medlin, Acta Mat. 2011

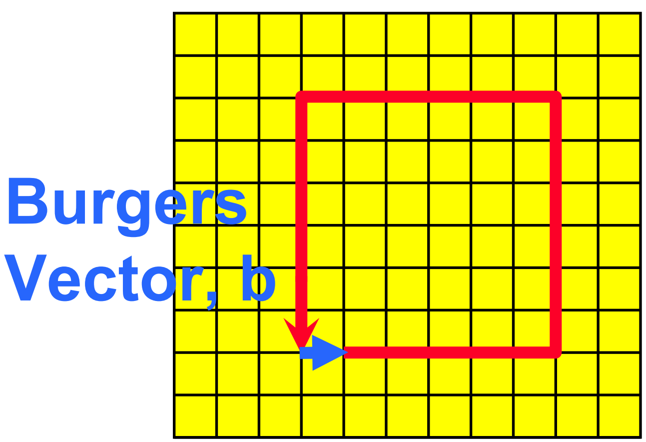
- **Solid-state phase transformations in chalcogenides**
- **Structural interpretation of extended defects**

# How do these structural considerations impact Dislocations and Interfaces?

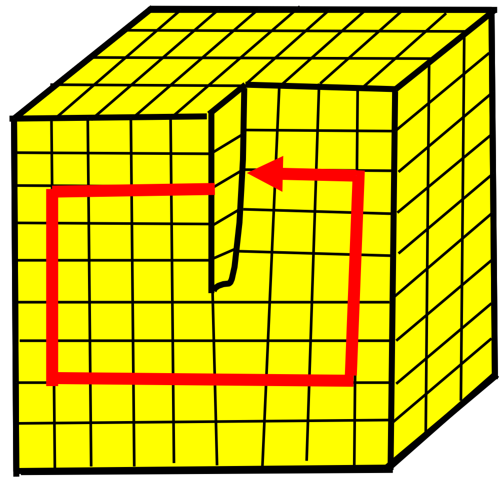
**Edge Dislocation**



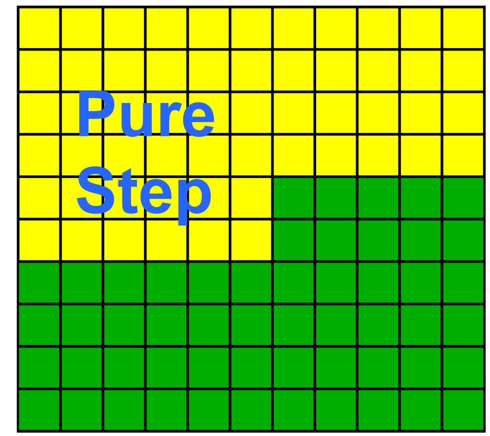
Reference Frame:
**Perfect Crystal**

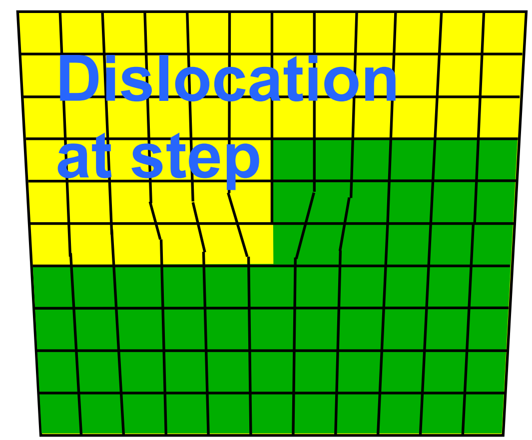



**Screw Dislocation**



**Interfacial Disconnection**



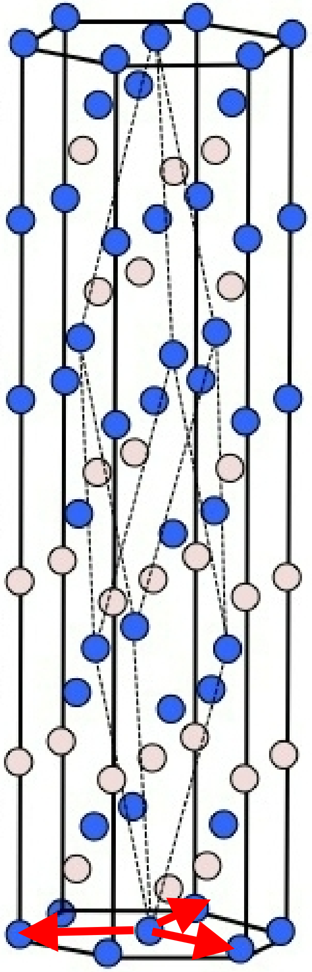




Sandia National Laboratories

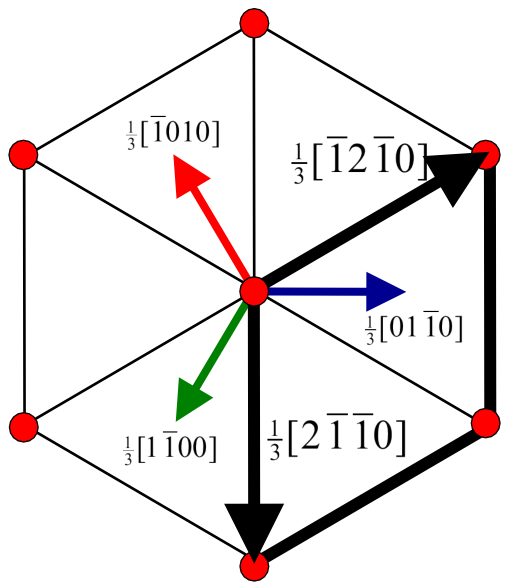
# How do these structural considerations impact Dislocations and Interfaces?

## *Dislocations in Bismuth Telluride*

**Burgers vectors lying in basal plane**




Looking down on basal plane




**Array of 1/3<2-1-1 0> Dislocations in $Bi_2Te_3$**

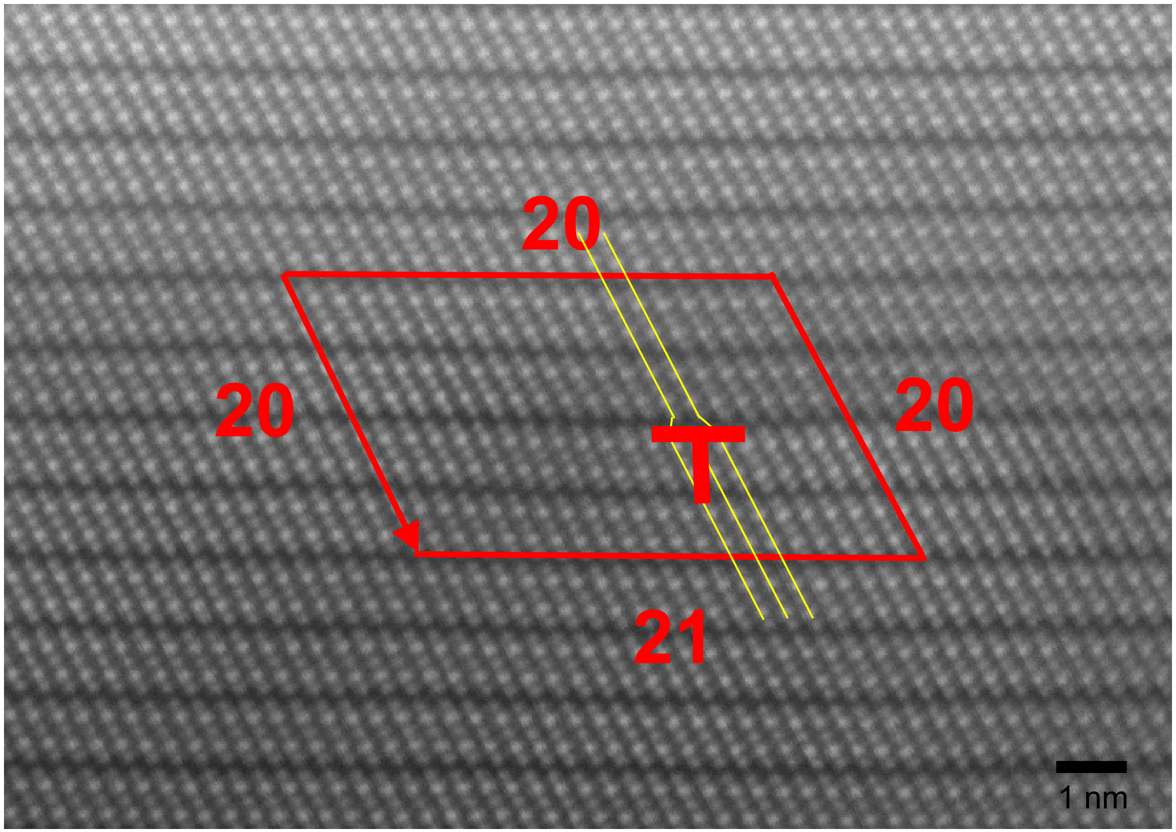

Fig. 1. Dislocation network in $Bi_2Te_3$. Note that certain segments of dislocations and certain node-points have left the foil

Sandia National Laboratories

# Dislocation Core structure: Termination at $Te^{(1)}$-$Te^{(1)}$ layer

5-layer $Bi_2Te_3$ "quintet"

[0 0 0 1]




<2 -1 -1 0 > projection

**b=1/3<2 -1 -1 0>**

**60° mixed dislocation**

**Stoichiometric Core**

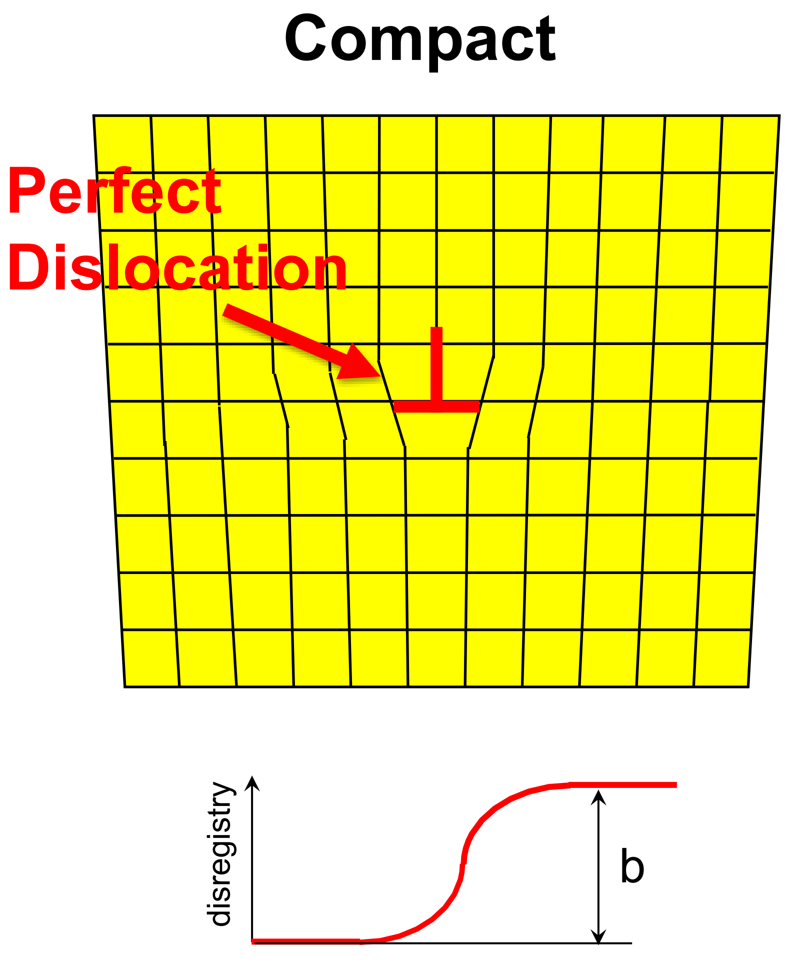
***Is dislocation compact or dissociated?***

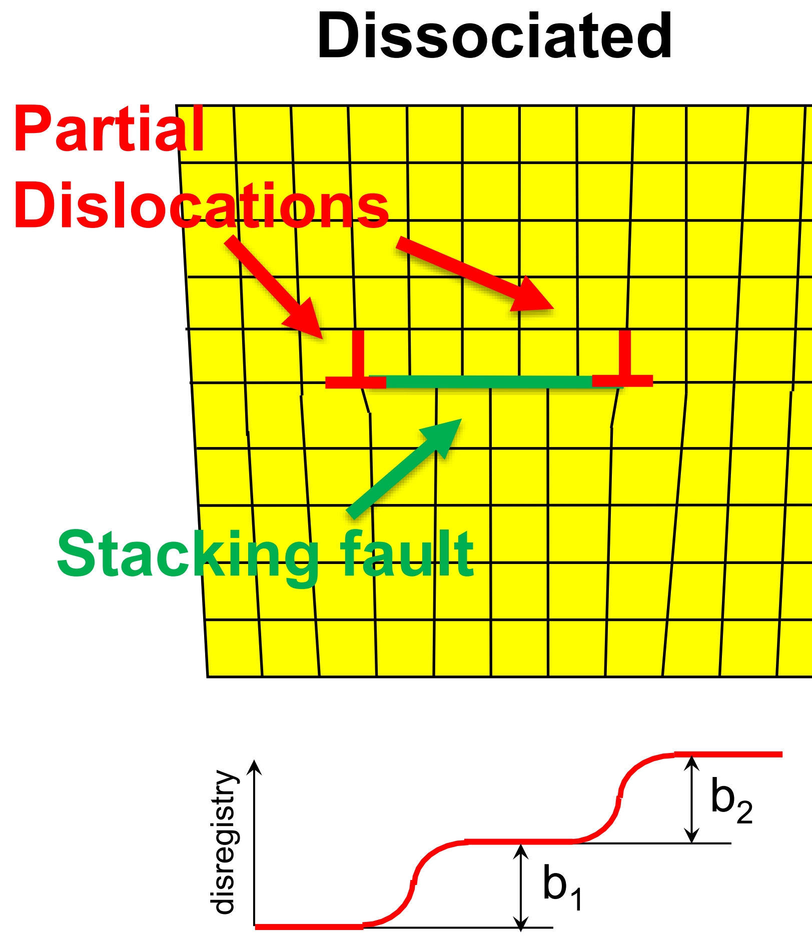
Medlin, Yang, Spataru, Hale, Mishin, Nature Communications 2019

Sandia National Laboratories

# Dissociated Dislocations

*Balance of strain energy and fault energy*



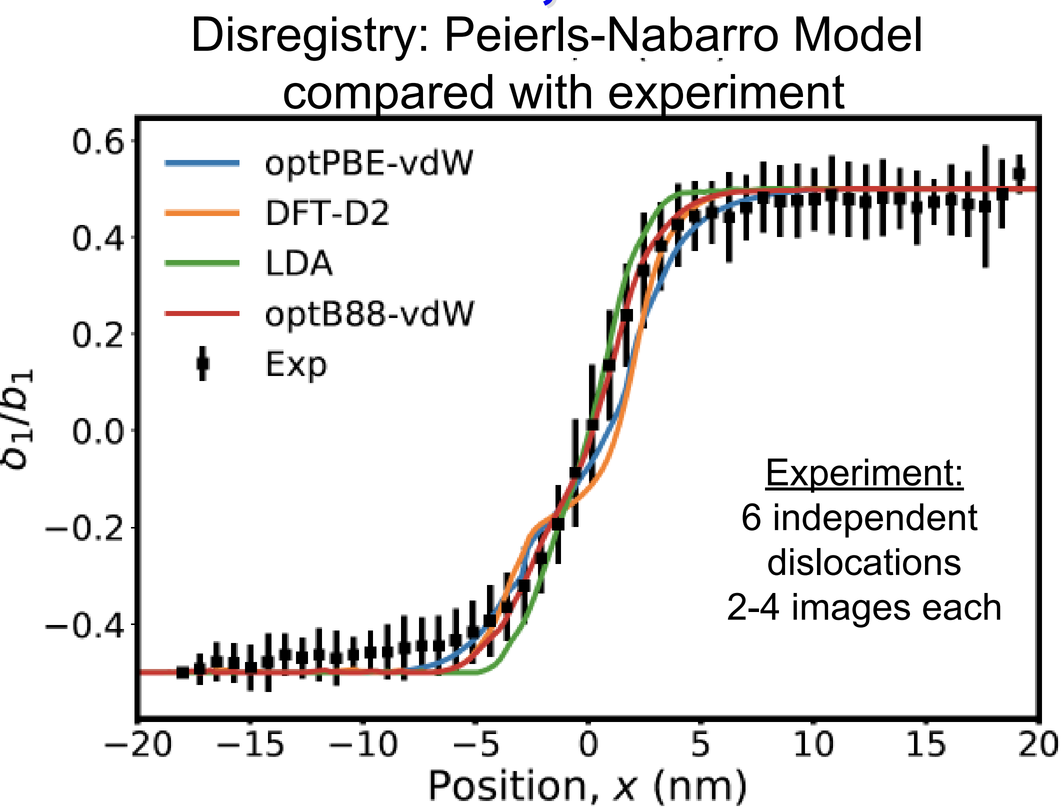



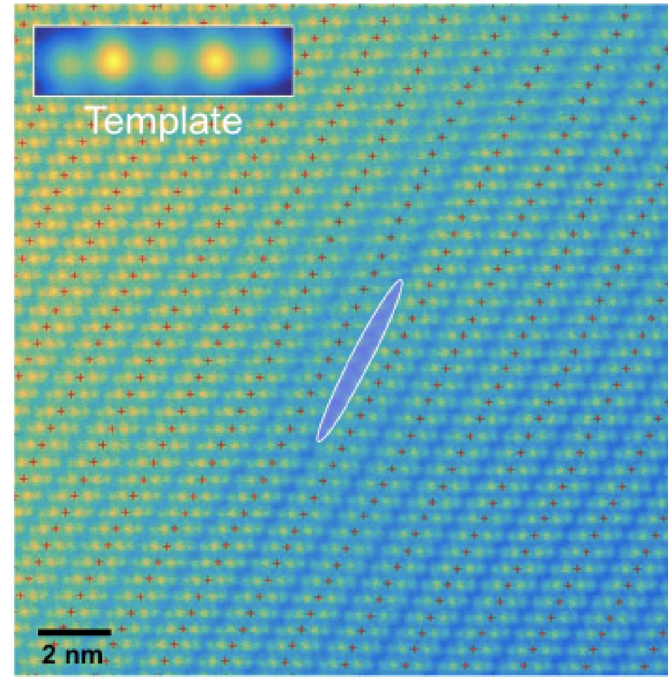

*What happens in bismuth telluride?*

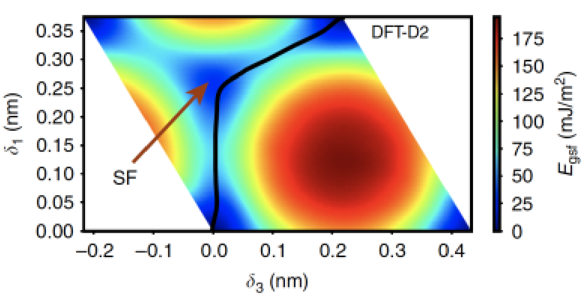


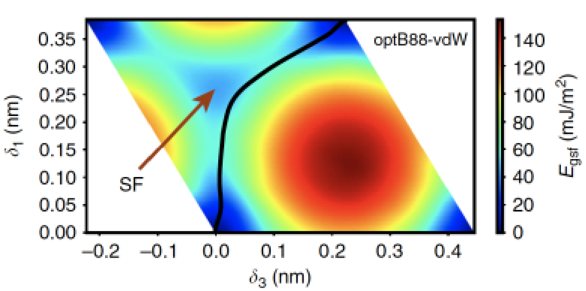
Sandia National Laboratories

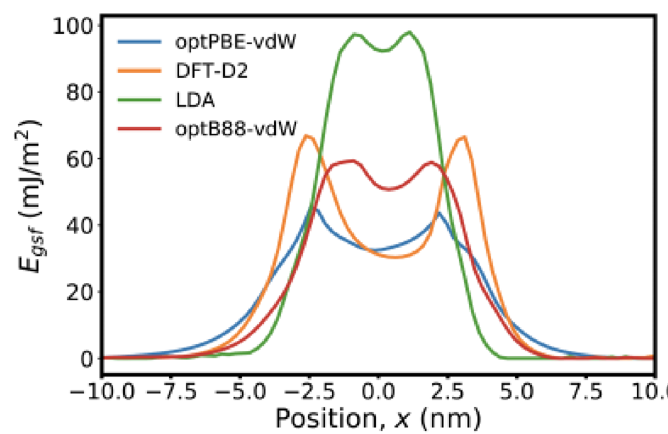
# Broad core, but not dissociated into partials



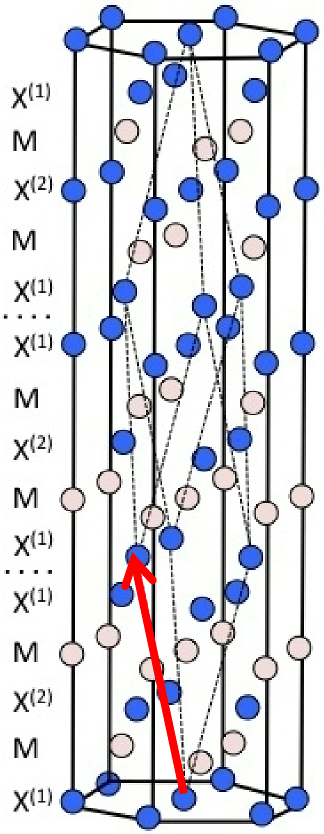




**Shallow minimum relative to unstable SFE prevents localization into discrete partial dislocations**

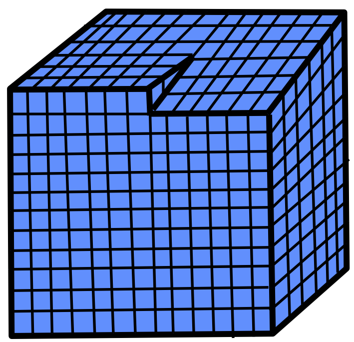
Medlin, Yang, Spataru, Hale, Mishin, Nature Communications 2019

Sandia National Laboratories

# Non-basal dislocations:
## *Screw dislocations important to crystal growth*

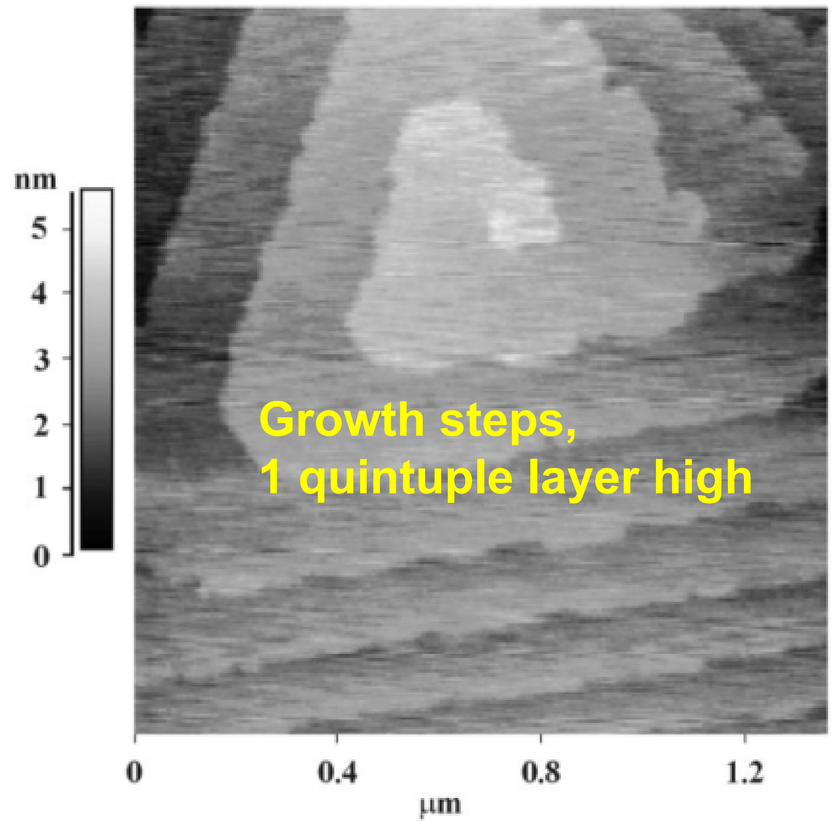



Screw Dislocation



b = (1/3)[0-111)$_H$
= [001]$_R$

**Example: spiral growth steps at screw dislocation in $Bi_2Te_3$ thin film**

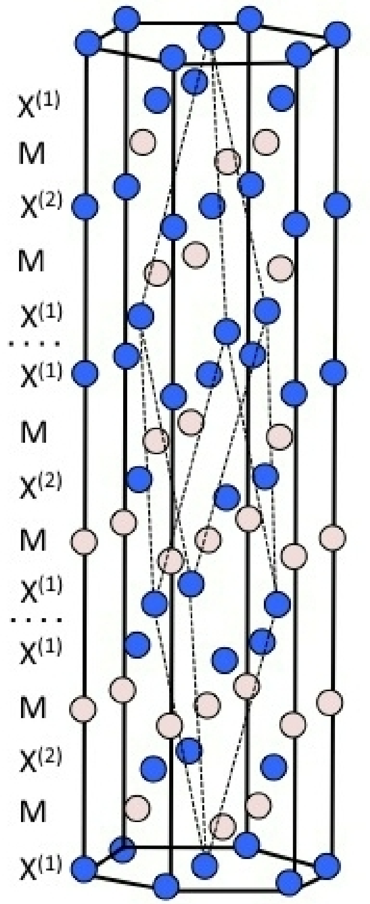


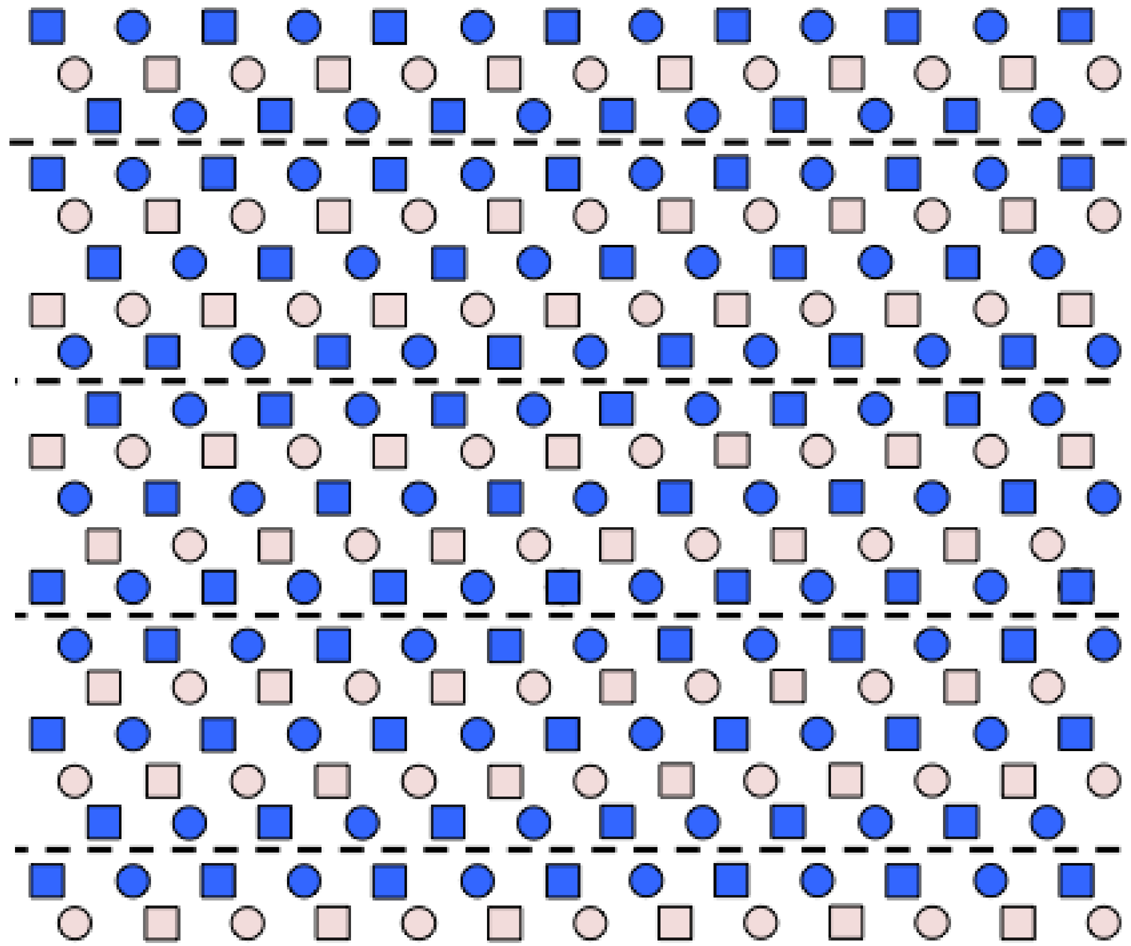

M. Ferhat, J.C. Tedenac, J. Nagao, J. Crystal Growth (2000)

**What about edge dislocations with non-basal Burgers vectors?**

Sandia National Laboratories

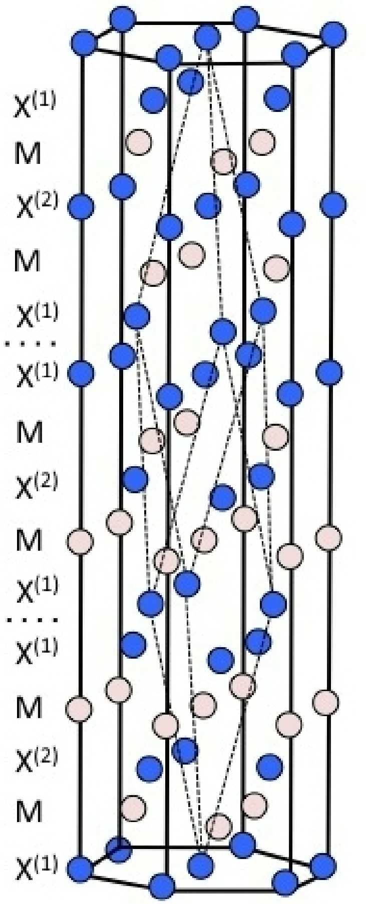
# Non-basal edge dislocations: What happens if we pull out a quintuple unit?

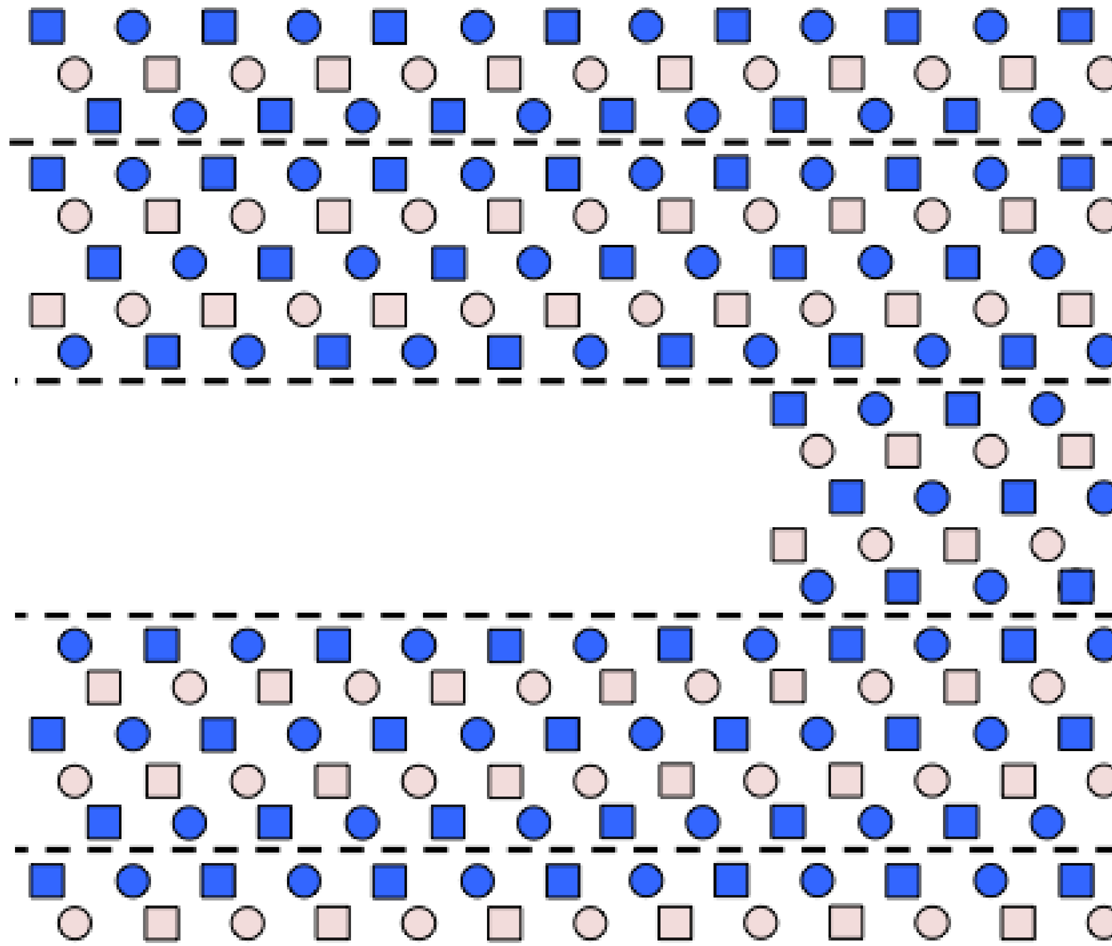


Sandia National Laboratories

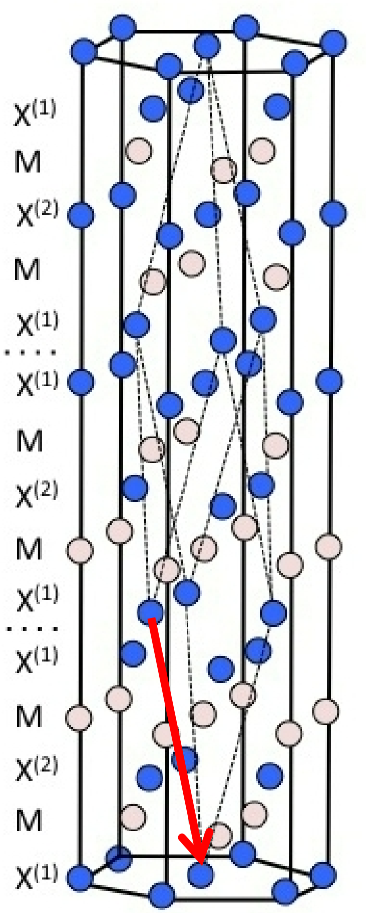
# Non-basal edge dislocations:
# What happens if we pull out a quintuple unit?

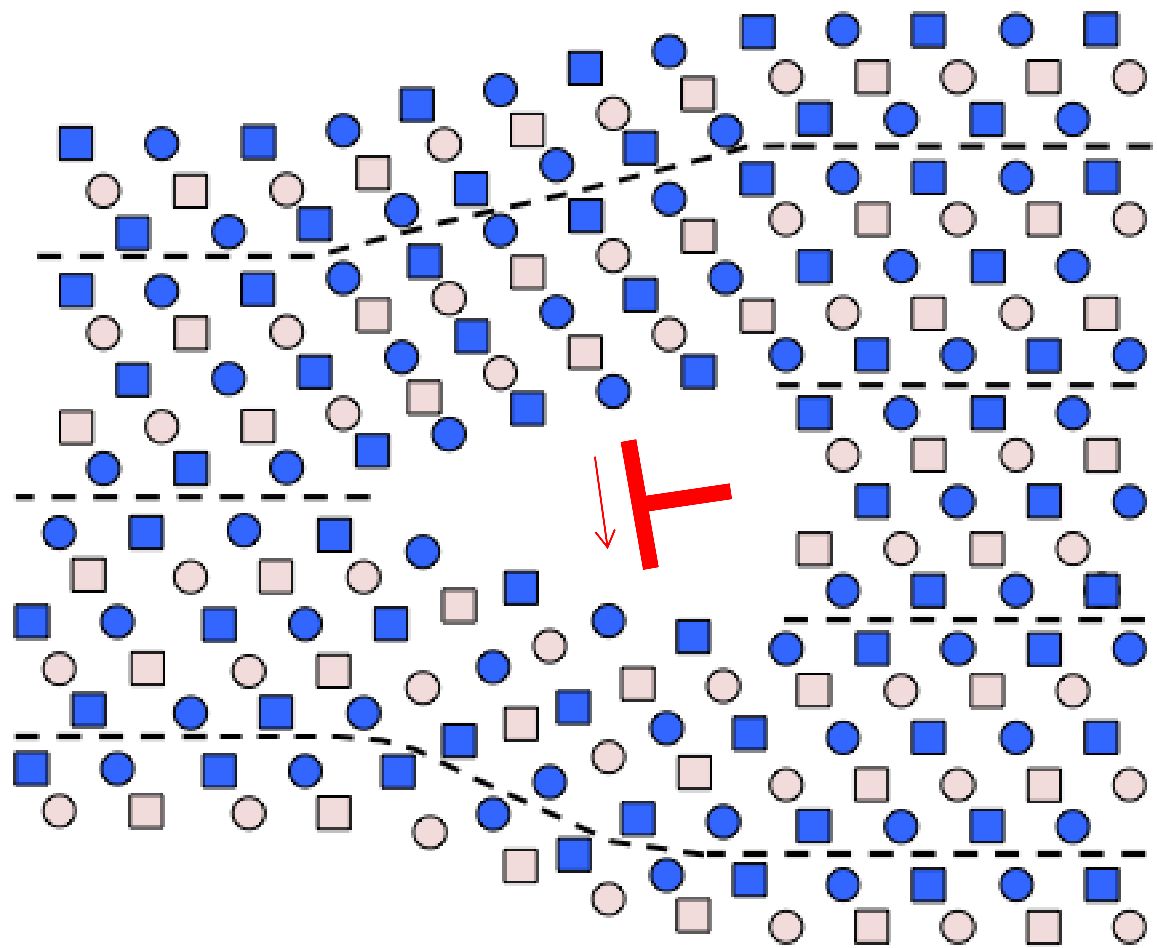


Sandia National Laboratories

# Non-basal edge dislocations:
# What happens if we pull out a quintuple unit?

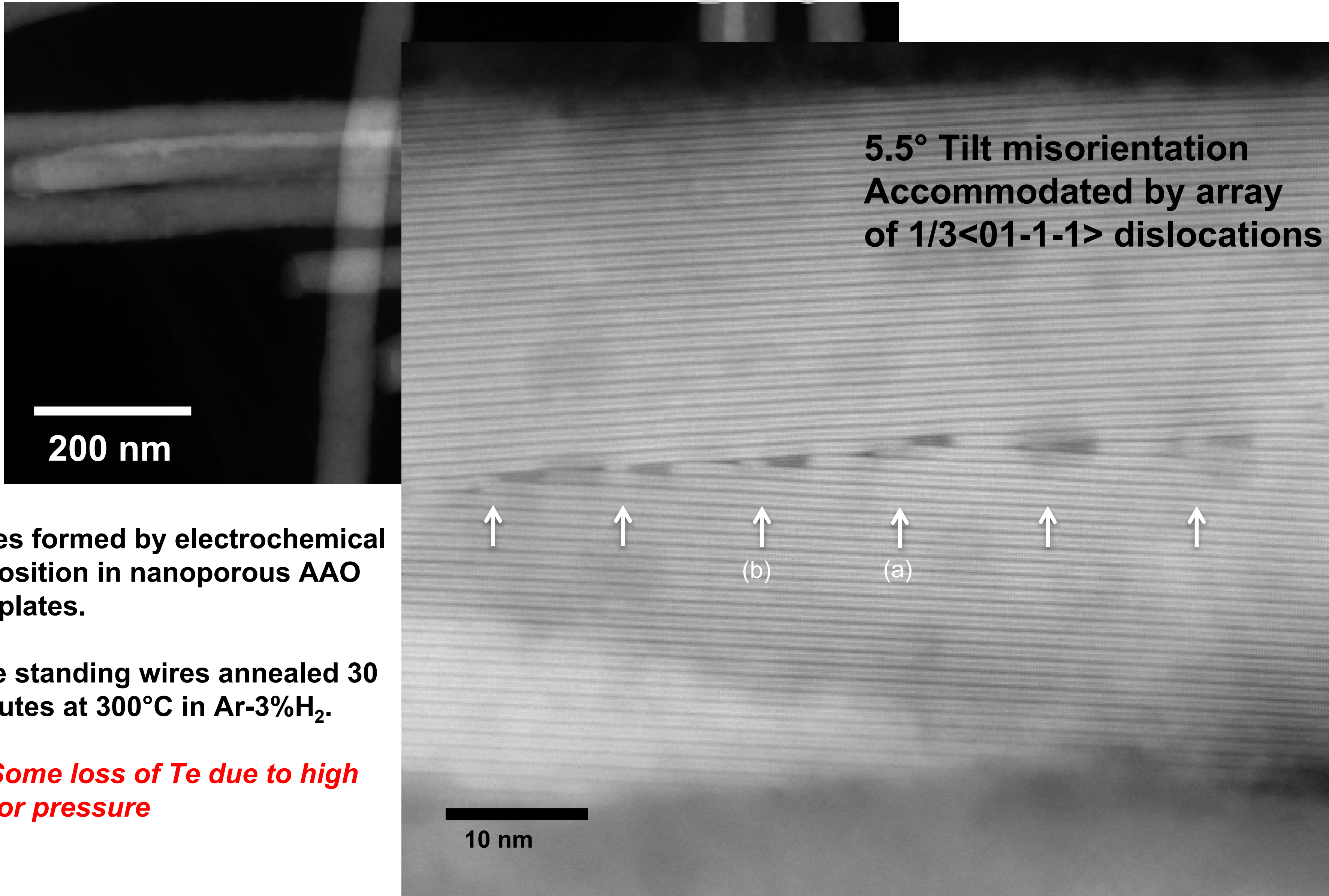


$$b = (1/3)[01\text{-}1\text{-}1)_H = [00\text{-}1]_R$$

Sandia National Laboratories

# Dislocations in $Bi_2Te_3$ Nanowires



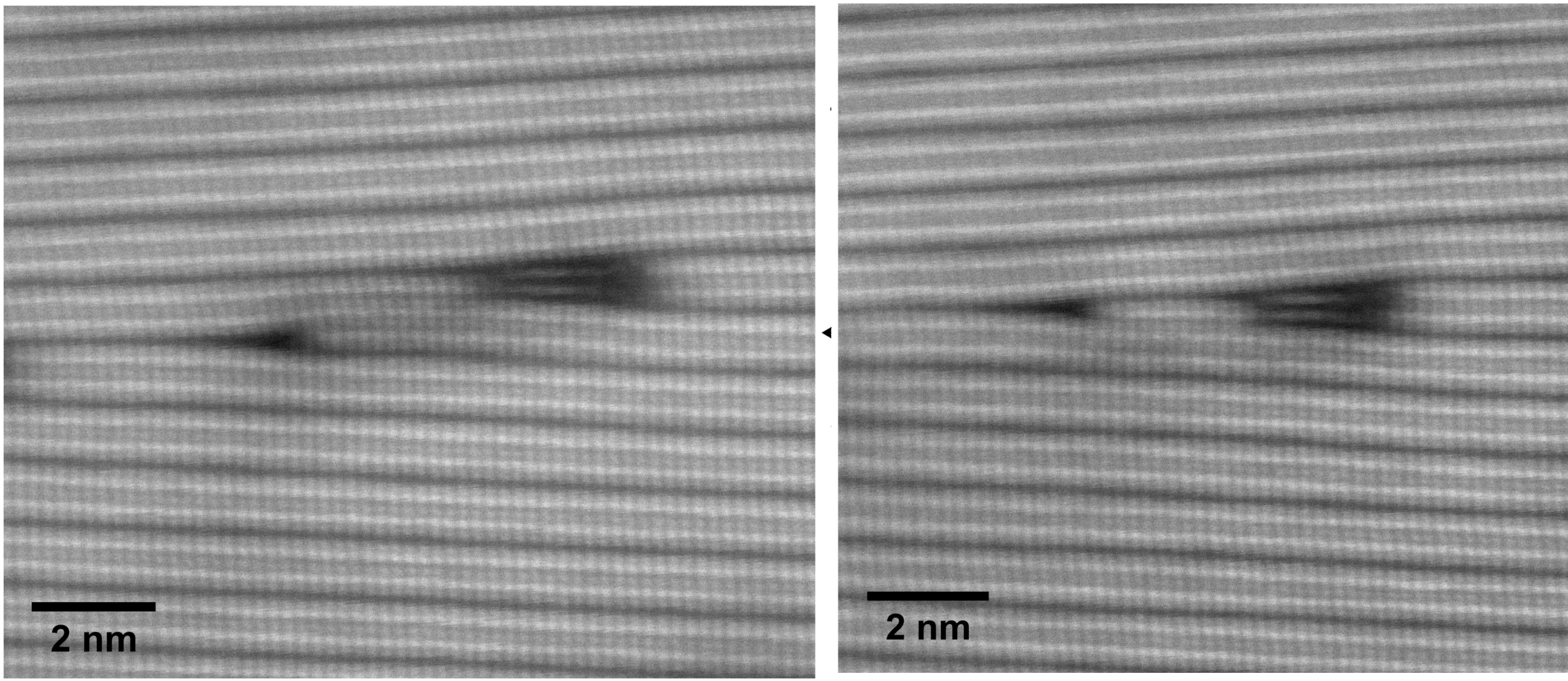

**Wires formed by electrochemical deposition in nanoporous AAO templates.**

**Free standing wires annealed 30 minutes at 300°C in Ar-3%$H_2$.**

***➔ Some loss of Te due to high vapor pressure***

Medlin, Erickson, Limmer, Yelton, Siegal, J. Mat. Sci. 2014

# Dislocations have dissociated core: two configurations

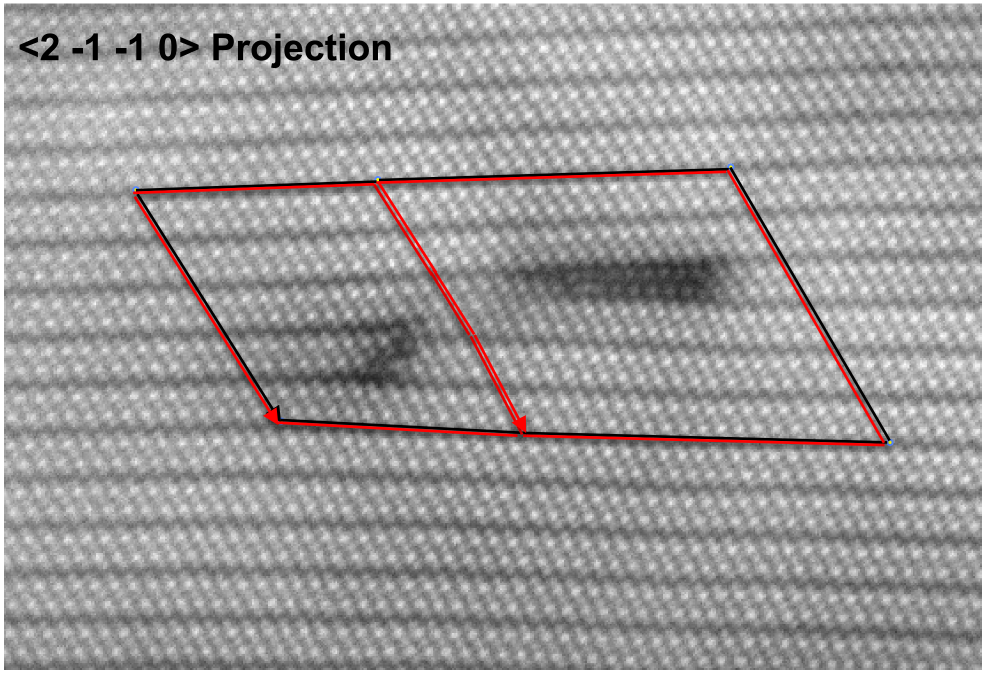



**<1 0 -1 0> Projection**

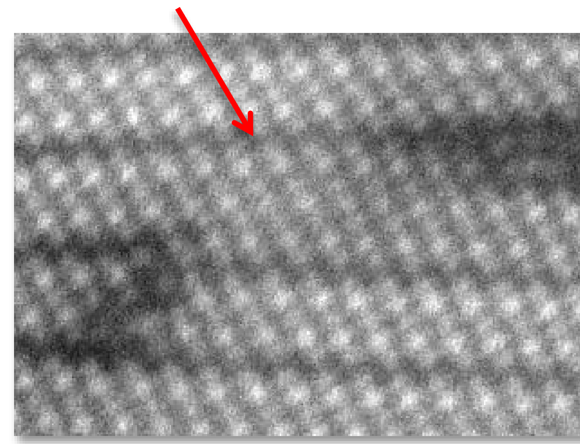
Medlin, Erickson, Limmer, Yelton, Siegal, J. Mat. Sci. 2014

Sandia National Laboratories

# (1/3)[0 1 -1 -1] Dislocation in $Bi_2Te_3$:




Core structure:
$Bi_3Te_4$ 7-layer fault



**b = (1/3) [0 1 -1 -1] → (1/15) [0 5 -5 -2] + (3/15) [ 0 0 0 -1]**

$$b^2 > b_1^2 + b_2^2$$

109.8 Å² > 22.9 Å² + 37.2 Å² =60.2 Å²

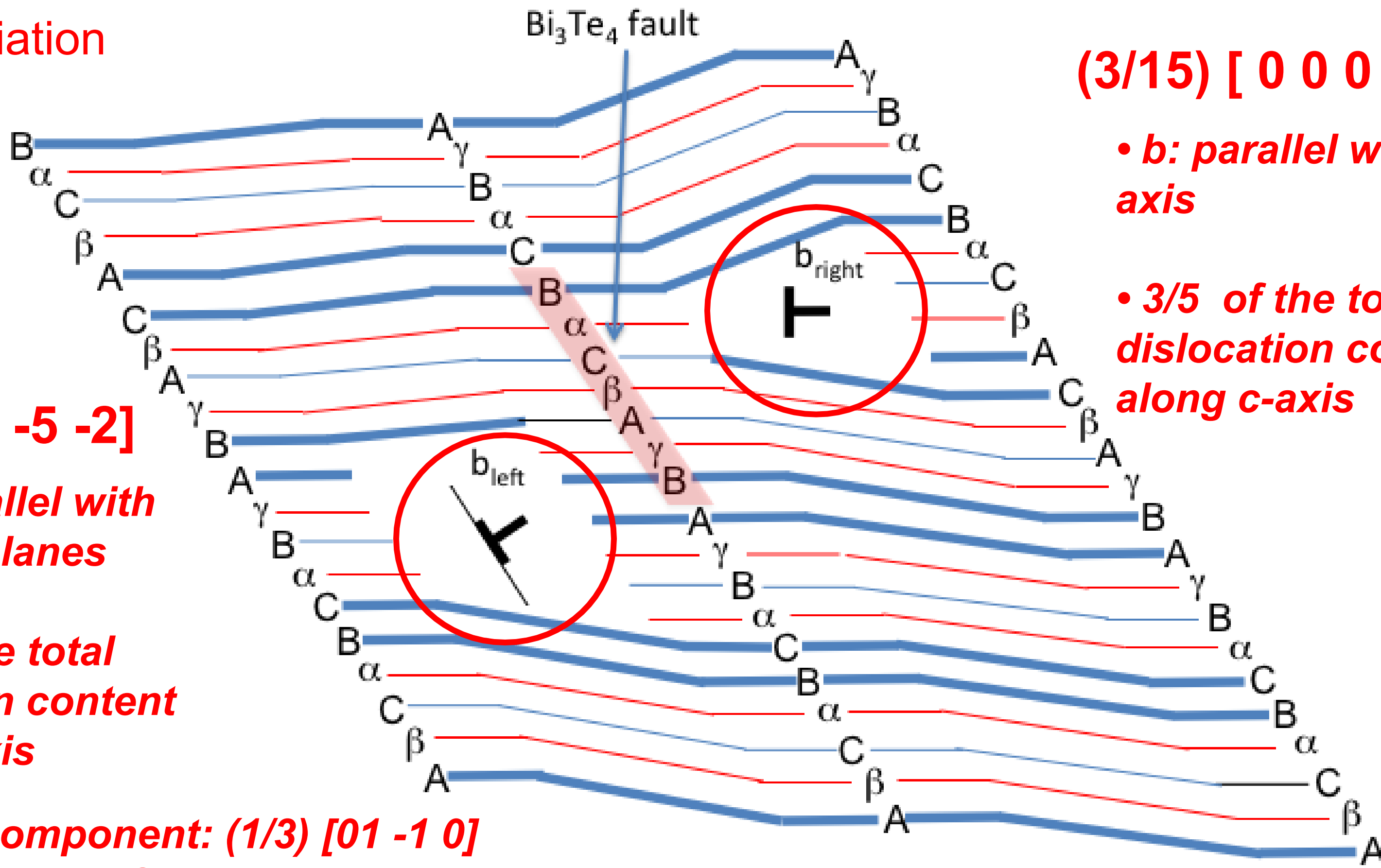
***Reduced strain energy with dissociation***

Medlin, Erickson, Limmer, Yelton, Siegal, J. Mat. Sci. 2014

Sandia National Laboratories

# *7-Layer $Bi_3Te_4$ faults:* Mechanism to accommodate Te loss during annealing

Climb dissociation




**(3/15) [ 0 0 0 -1]**

- ***b: parallel with c-axis***
- ***3/5 of the total dislocation content along c-axis***

**(1/15) [0 5 -5 -2]**

- ***b is parallel with (0 1 -1 5) planes***
- ***2/5 of the total dislocation content along c-axis***

***Horizontal component: (1/3) [01 -1 0]***

- ***analogous to Shockley partial.***
- ***avoids fault in stacking resulting from additional 2 planes at $Bi_3Te_4$ fault***

Medlin, Erickson, Limmer, Yelton, Siegal, J. Mat. Sci. 2014

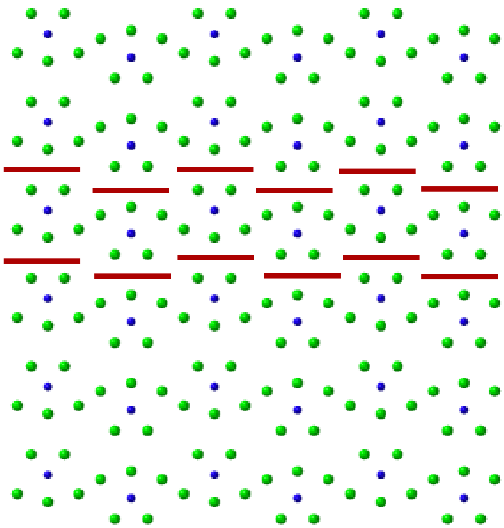
Sandia National Laboratories

# $ZrTe_5$: A complex, layered chalcogenide

**Material of interest as a Dirac Semimetal**

## Corrugated layering structure

Orthorhombic, *Cmcm*

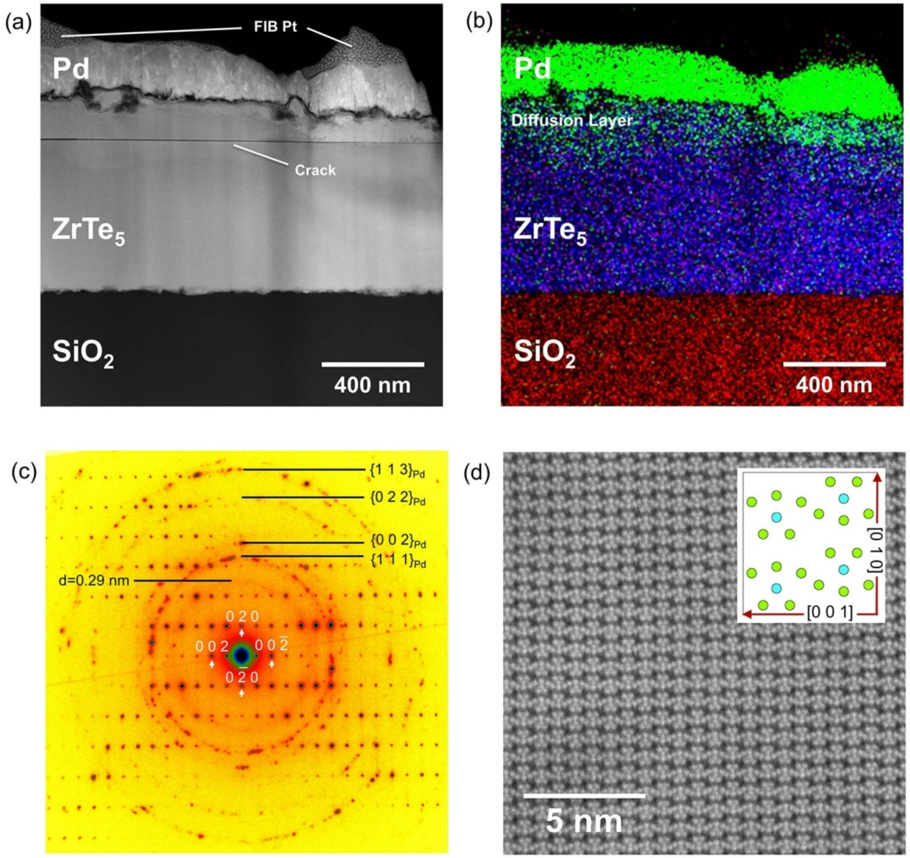


[ 1 0 0] projection

a=3.9875Å, b=14.530Å, c=13.724Å

H. Fjellvåg, A. Kjekshus, Solid State Communications, 60 (2) (1986) 91-93

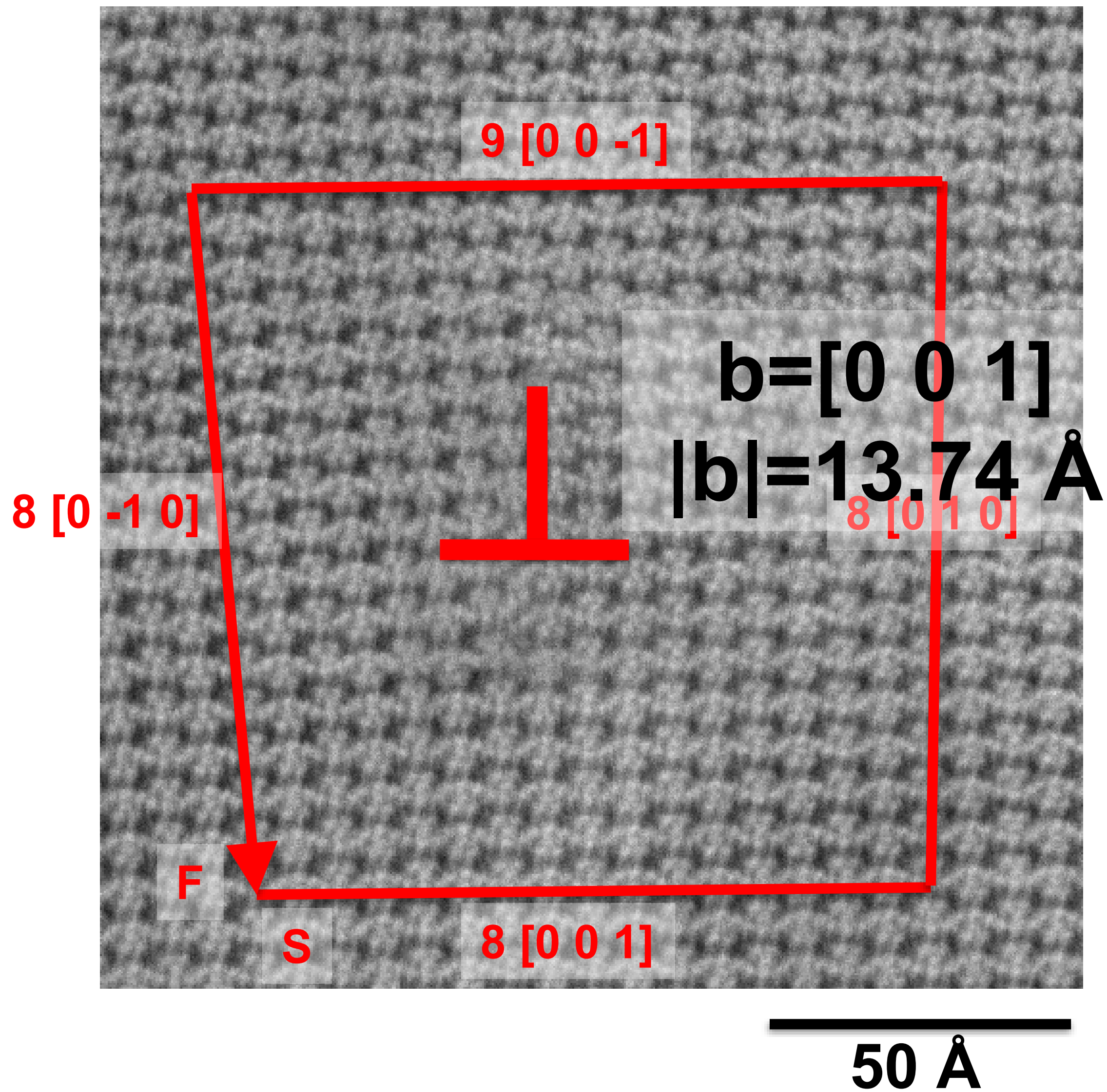
## Interdiffusion of contact metallization causes large strains




F. Léonard, W.L. Yu, K.C. Collins, D.L. Medlin, J.D. Sugar, A.A. Talin, W. Pan *ACS Applied Materials and Interfaces 9 (42) (2017) 37041-37047*

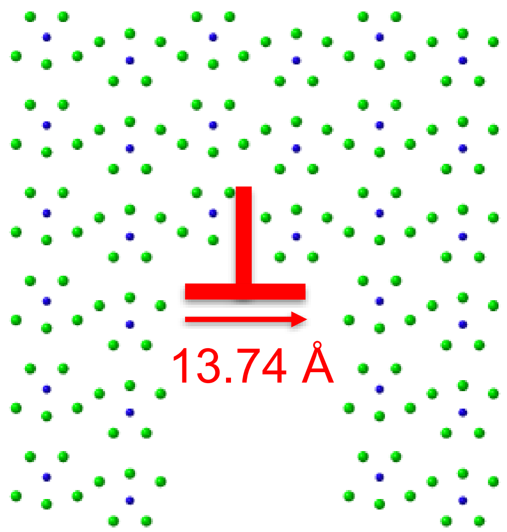
# Dislocation Structure in Zirconium Telluride

**HAADF-STEM**




**$ZrTe_5$ Crystal structure**

Orthorhombic, *Cmcm*



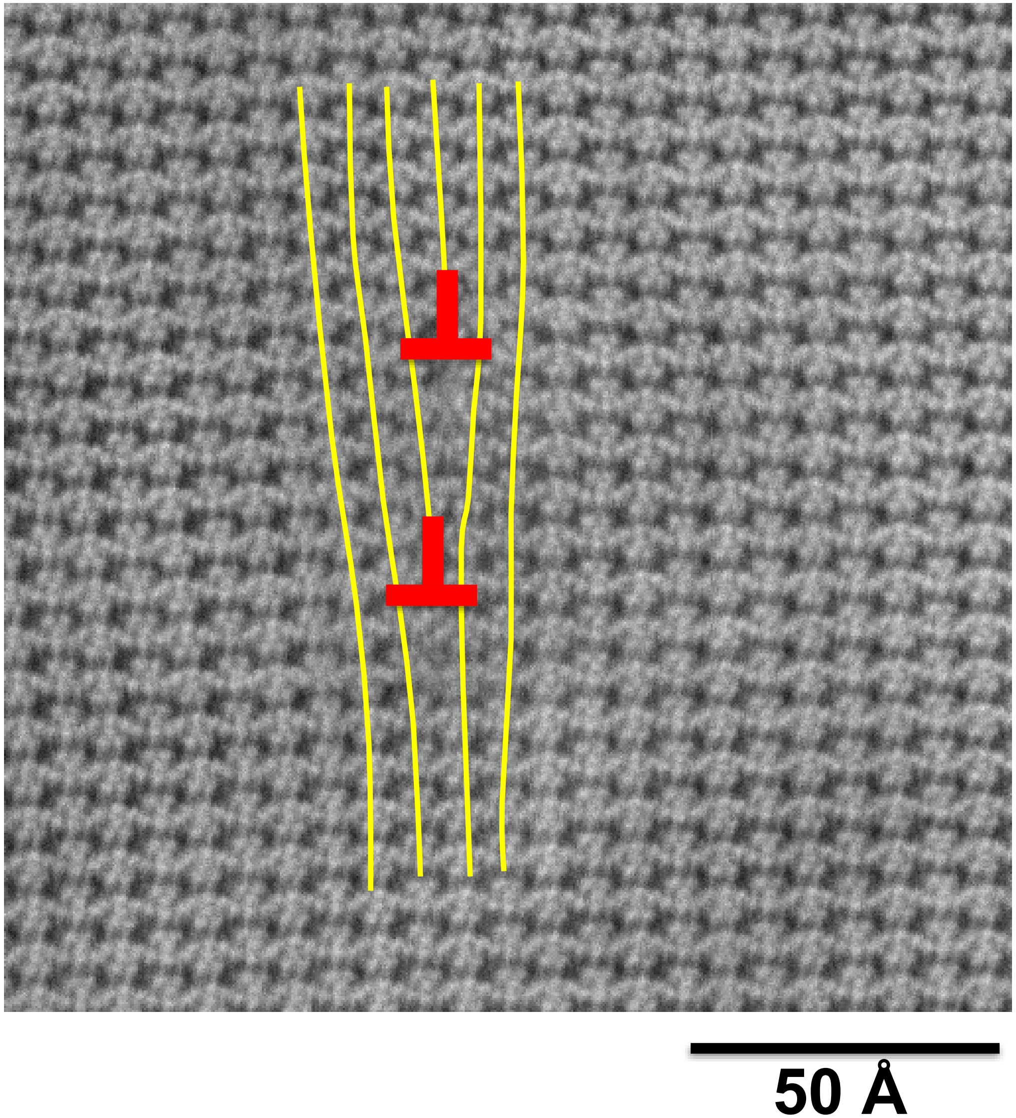

[ 1 0 0] projection

a=3.9875Å, b=14.530Å, c=13.724Å

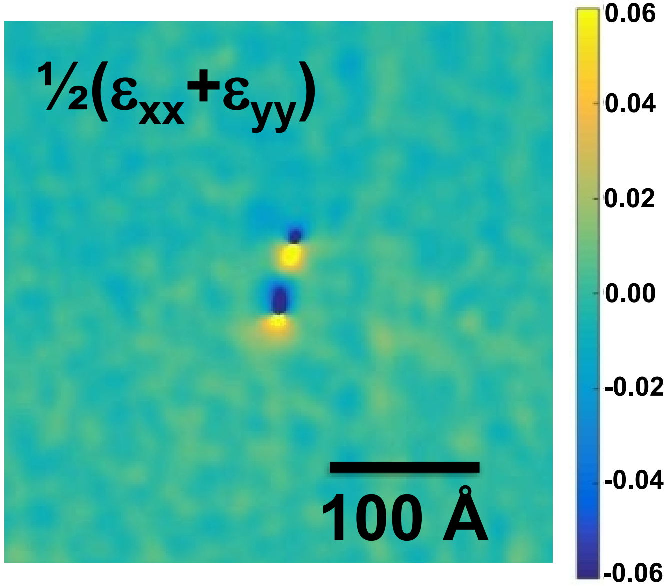
H. Fjellvåg, A. Kjekshus, Solid State Communications, 60 (2) (1986) 91-93

# Dislocation Structure in Zirconium Telluride

**HAADF-STEM**




**GPA Strain Map**

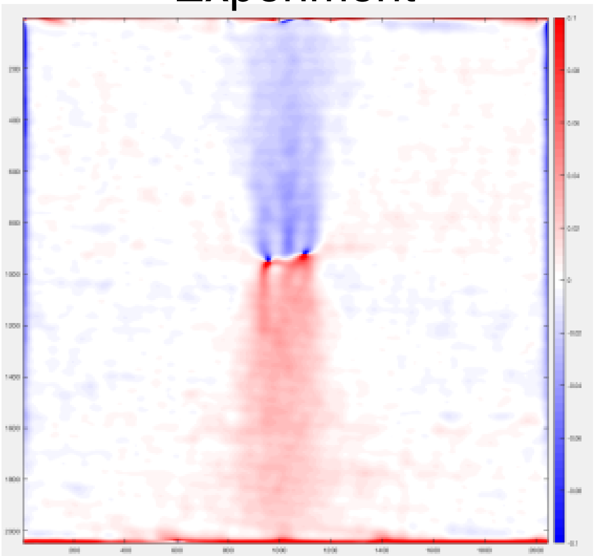



**Climb dissociation into two ½[0 0 1] partial dislocations**

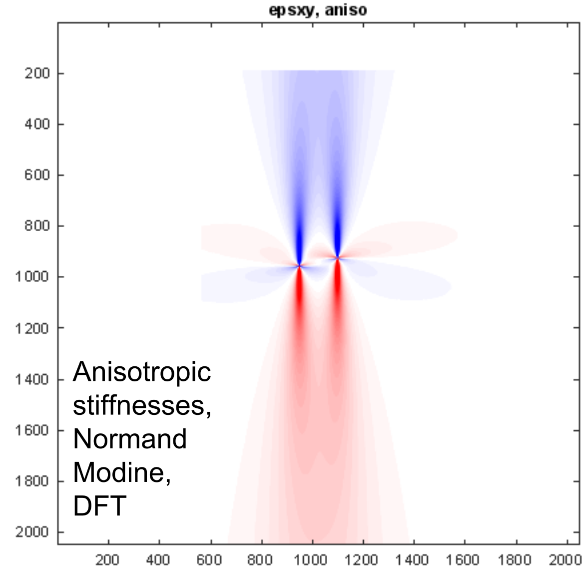
# Shear strain, parallel with (0 1 0) planes

Sandia National Laboratories

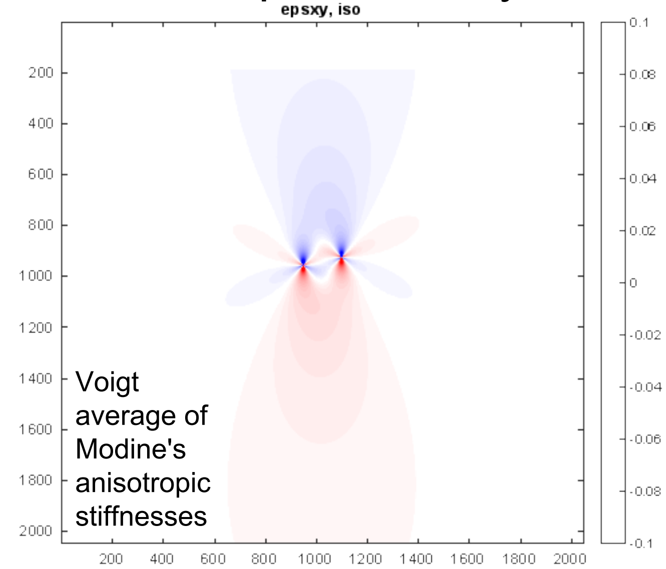
Experiment

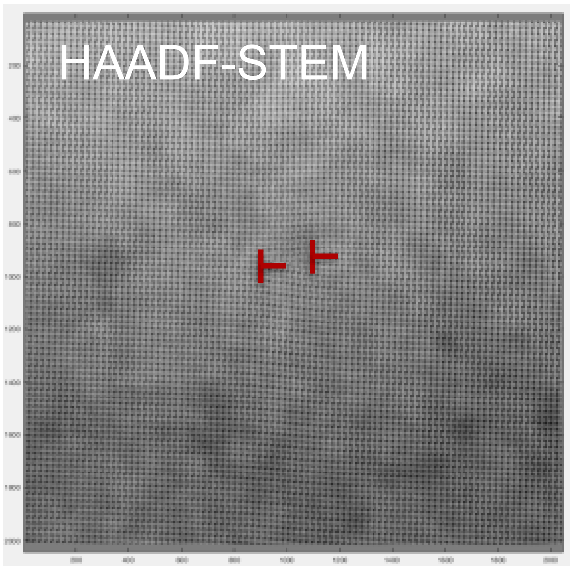


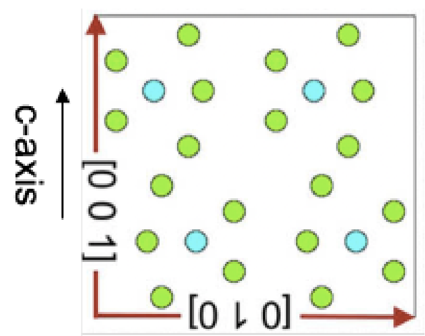
Anisotropic Elasticity

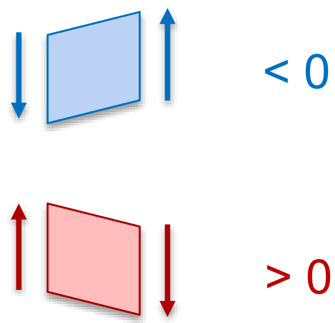



Isotropic Elasticity




Anisotropic solution from J.W. Steeds, "Introduction to Anisotropic Elasticity Theory of Dislocations," Clarendon Press, Oxford, 1973

Solved for 2 dislocations, each with b= 1/2(001).
placed at the experimentally measured core positions.

# Interfaces

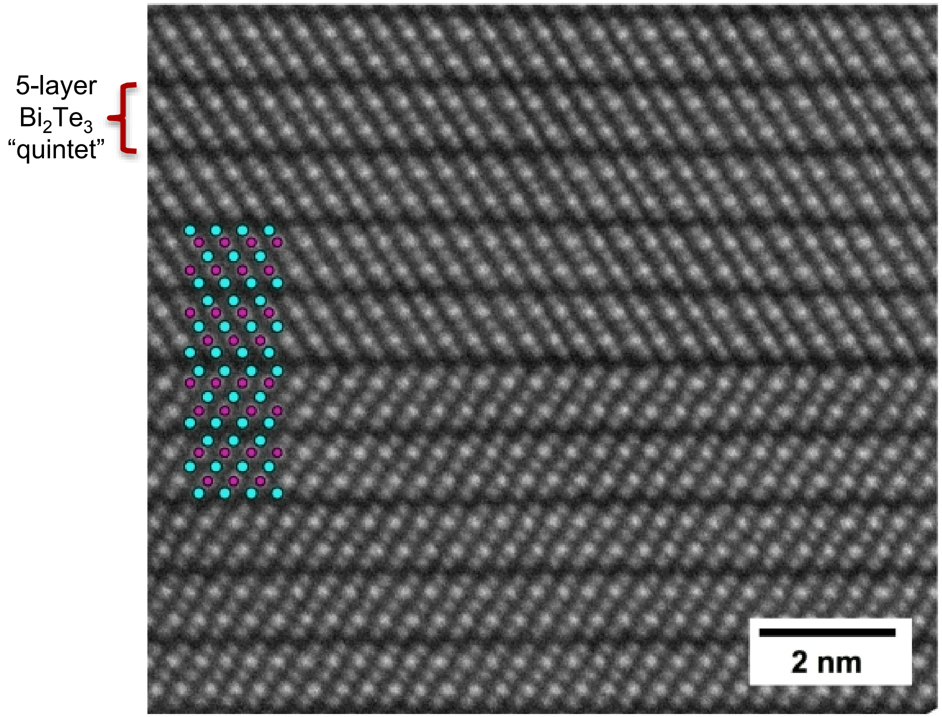
# Atomic Structure of the $Bi_2Te_3$ Basal Twin:

Energetic preference for termination at $Te^{(1)}$ sites

Sandia National Laboratories

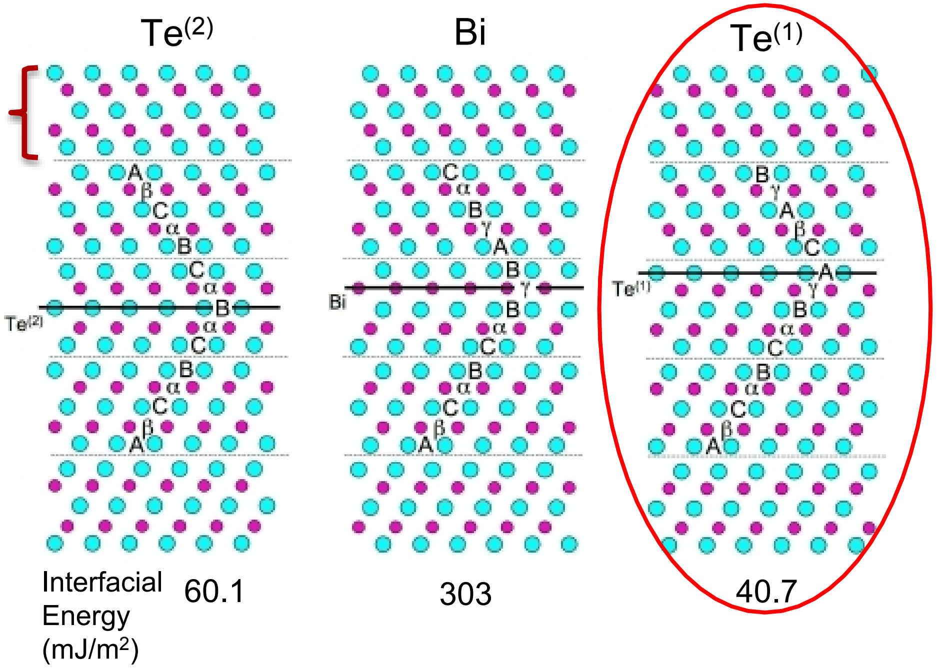
**HAADF-STEM Imaging:**

***Twin Boundary Terminated at $Te^{(1)}$ layer***




**DFT Calculations:**

***Three Possible Compositional Terminations***



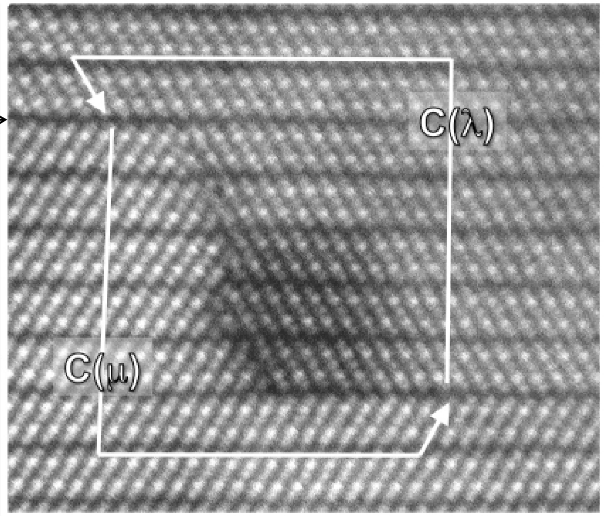

**D.L. Medlin, Q.M. Ramasse, C. D. Spataru, N.C. Yang, J. Appl. Phys. (2010)**

# Preferential termination at Te(1) layers:

## Impact on boundary morphology

Example: step in $Bi_2Te_3$ Basal Twin:
25 planes high (5 quintets)

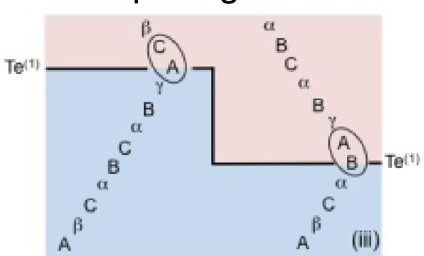
Twin



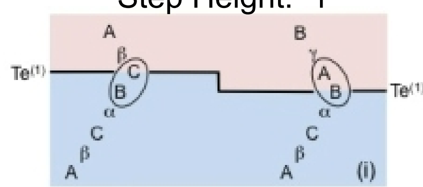

**b**=1/3<10-10>

Twin

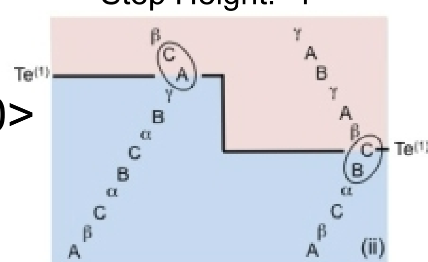
Step Height: 5




Step Height: 1



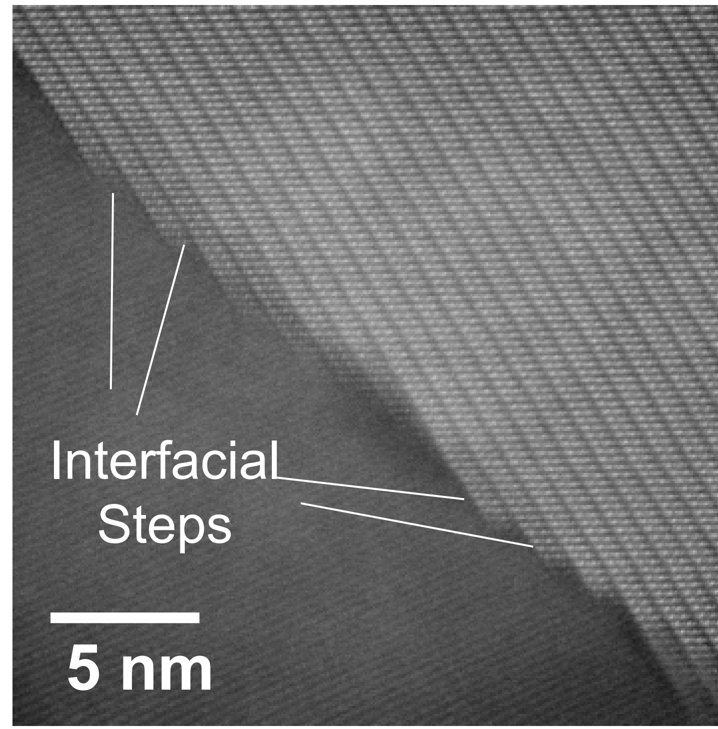

Step Height: 4




**b**=1/3<10-10>

**Medlin and Yang, Journal of Electronic Materials, (2012)**

Grain boundary vicinal to (0001):
Steps of integral 5-plane $Bi_2Te_3$ Quintets

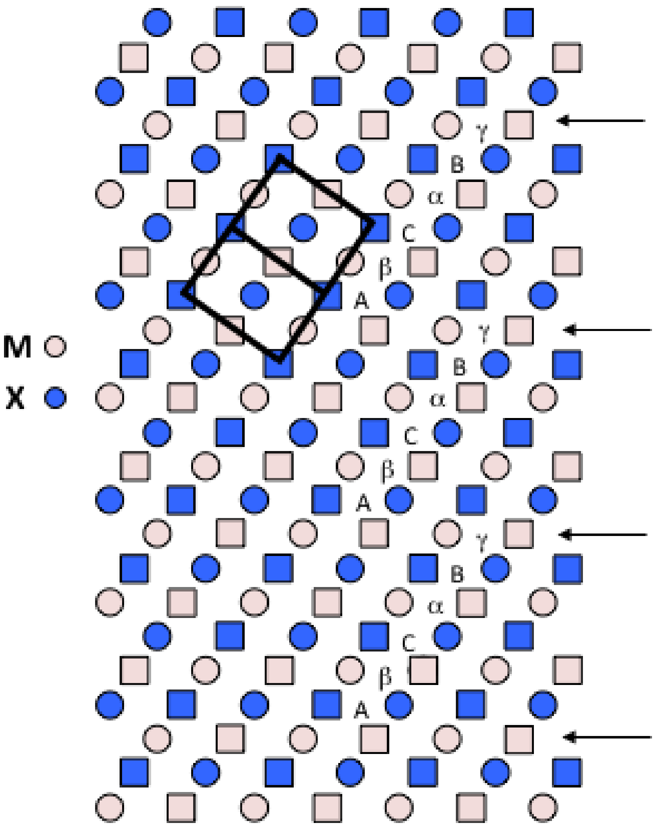



**Medlin and Snyder, JOM (2013)**

# Heterophase Rocksalt/Tetradymite interfaces

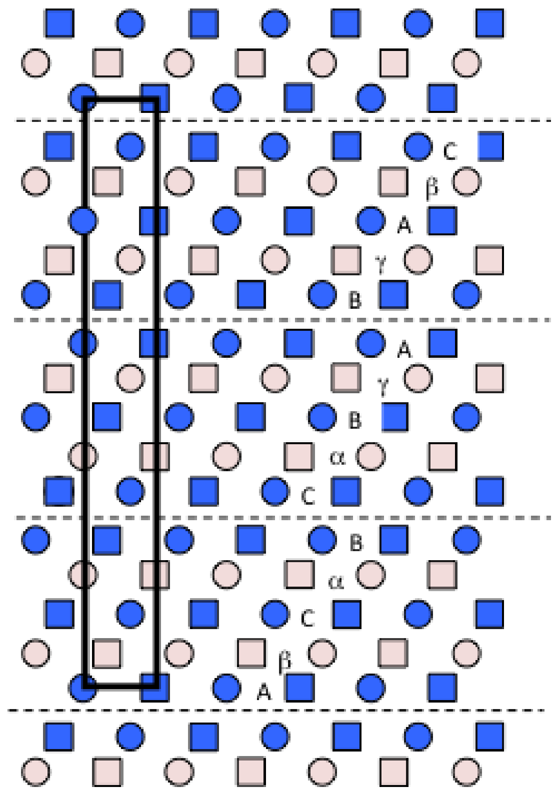
**Rocksalt (MX) Structure**

Fm-3m

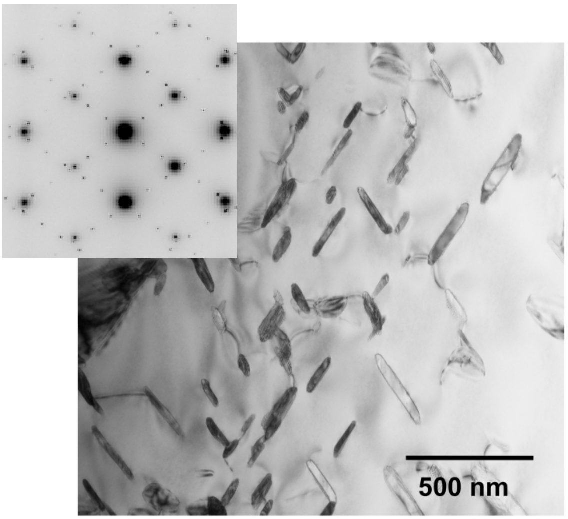



**Tetradymite ($M_2X_3$) Structure**

R-3m




**Example:**
**Crystallographically Aligned $Sb_2Te_3$ precipitates in PbTe**



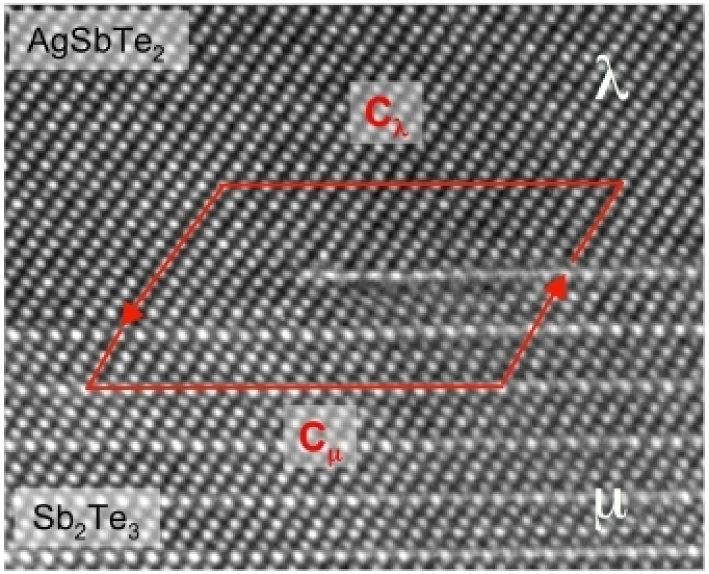

**Heinz, Snyder, Ikeda, and Medlin, Acta Mat. 2011**

***Line defects help us understand atomic mechanisms for precipitate growth and strain accommodation***

# Disconnections in Phase Transformations

Example: $Sb_2Te_3$ precipitate in $AgSbTe_2$ thermoelectric

**Burgers vector:**




**Upper crystal circuit** **Lower crystal circuit**

$$\mathbf{b} = -(\mathbf{C}_\lambda + \mathbf{P}\mathbf{C}_\mu)$$

**-Coordinate Transformation from Tetradymite to Rock-salt.**

**-Coherently strained reference Frame (0.79% misfit)**

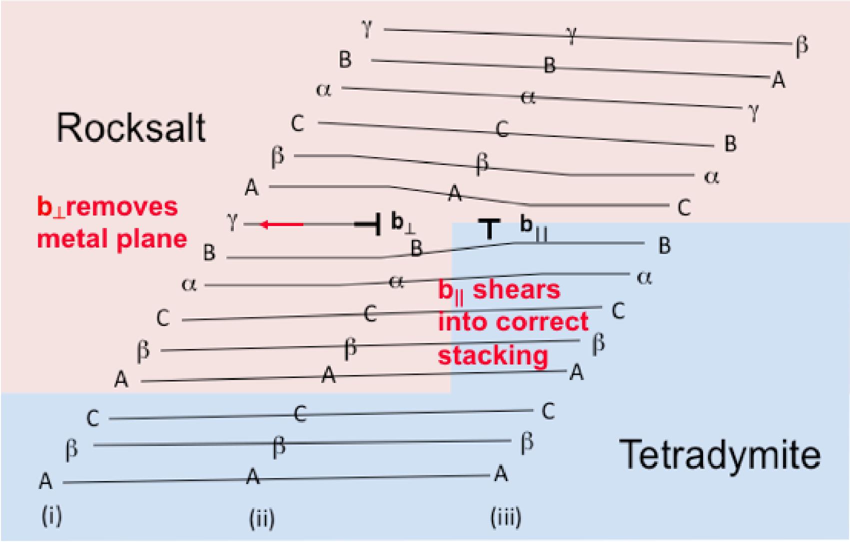
**Resolve b into components normal and parallel to interface**

$$\mathbf{b}_n = (a_{cub} - c_{hex}/3\sqrt{3})[111]$$

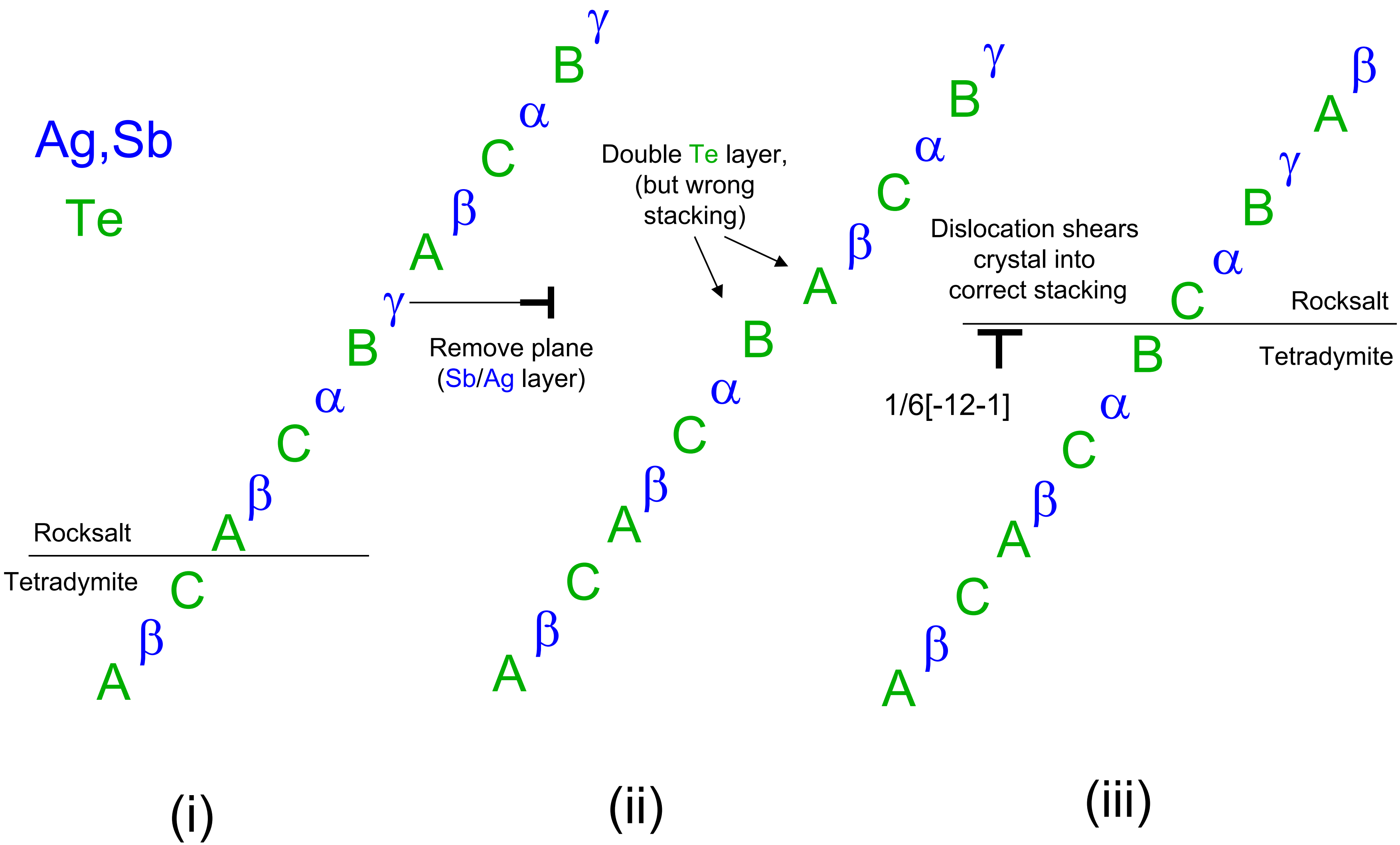
- ***mismatch of step heights.***
- ***$|b_n|$=0.3747Å***

$$\mathbf{b}_{\parallel} = \frac{a_{cub}}{6}[\bar{1}2\bar{1}]$$

- ***Analogous to Shockley partial Dislocation***
- ***$|b_{\parallel}|$=2.48Å***

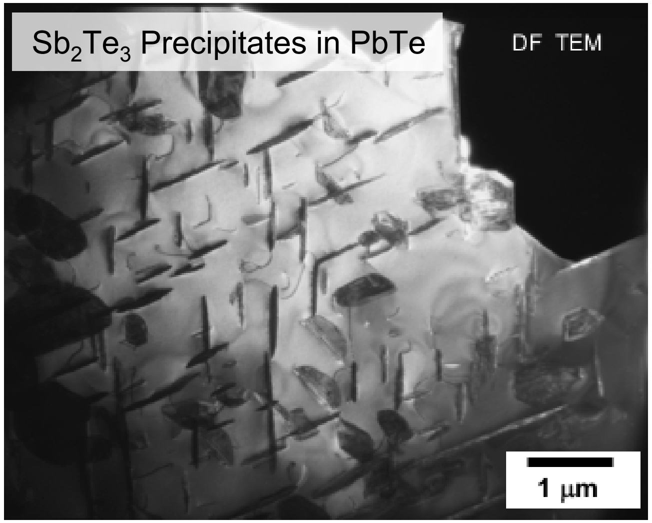


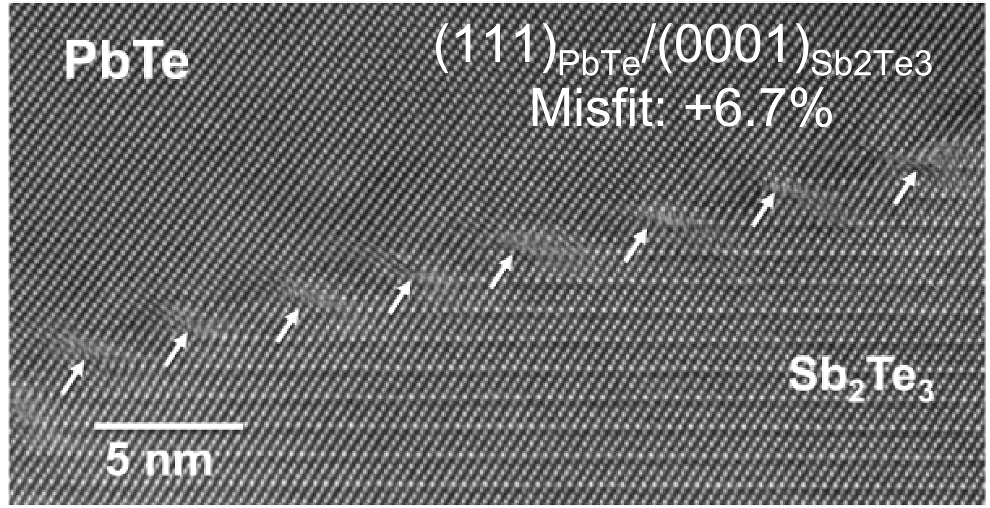
# Schematic of Transformation Sequence

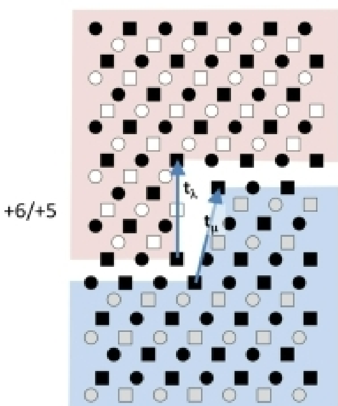


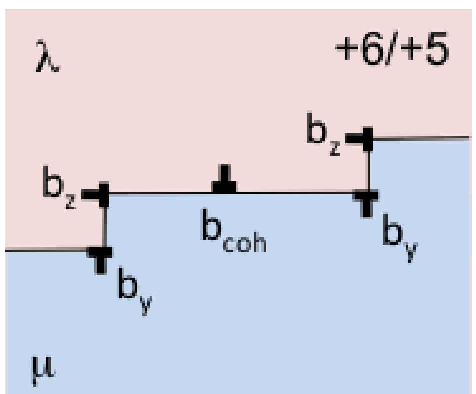

Medlin and Sugar, Scripta Materialia, 2010.

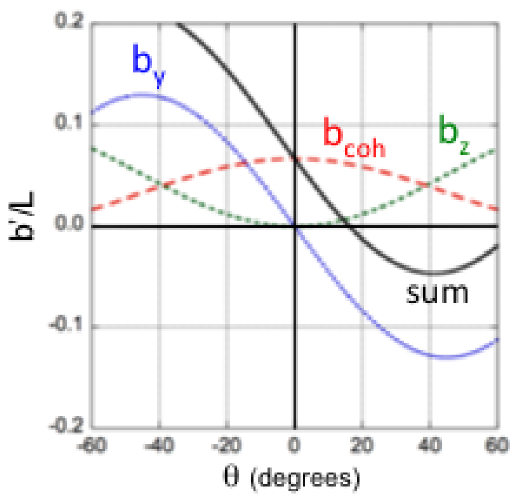
# A Rocksalt/Tetradymite Interface with Large Misfit




<u>Habit plane inclination:</u>

Measured: 14.8°
Predicted: 16.2°

***Defect spacing accommodates the (111)/(0001) coherency strain.***

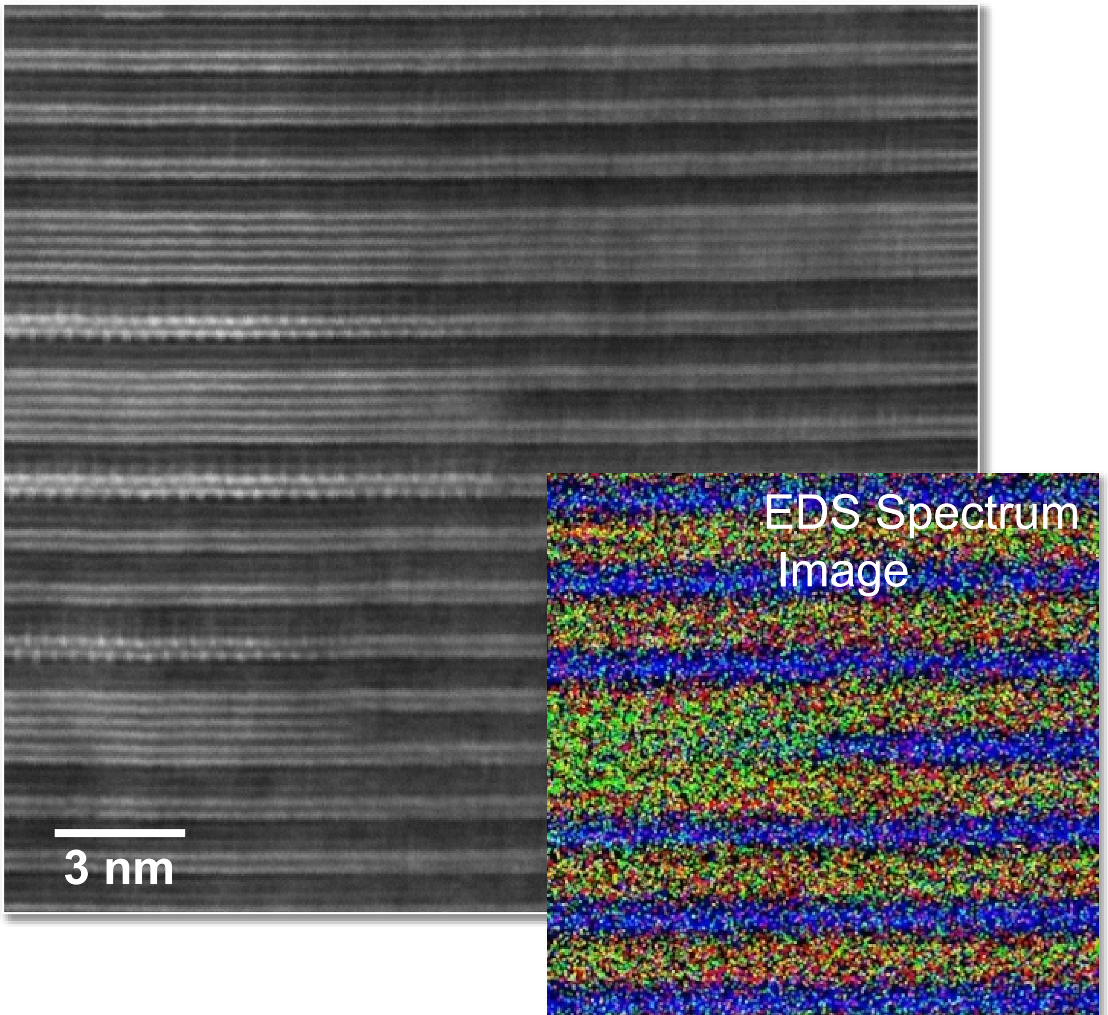
**Heinz, Ikeda, Snyder, Medlin, Acta Materialia (2011)**

Sandia National Laboratories

# Defects in van der Waals Heterostructures

Collaboration with David Johnson. University of Oregon

**Example: Compositional Defects in $(Pb_xSn_{1-x}Se)_{1+\delta}TiSe_2$ Multi-layer thin film**



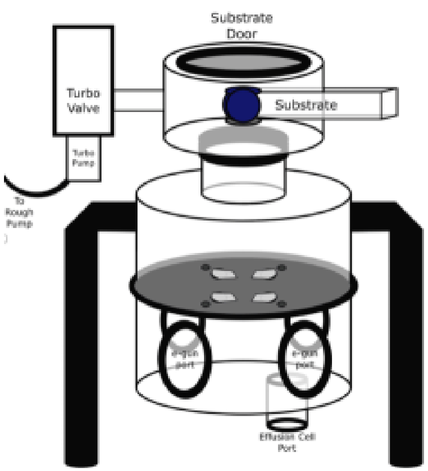

- **Linking defect structure to local composition.**
  - -Connection of kinetics and thermodynamics to mechanisms underpinning growth and stability.
- **Extending defect analysis formalisms to systems with lower dimensional periodicities**
  - -"turbostratic" disorder in layered systems;

Sandia National Laboratories

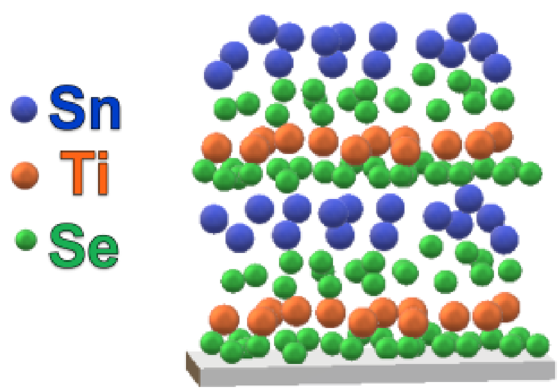
# The MER method: flexible approach for tailoring atomic-sequence of layered chalcogenides

Modulated Elemental Reactants (MER) method:

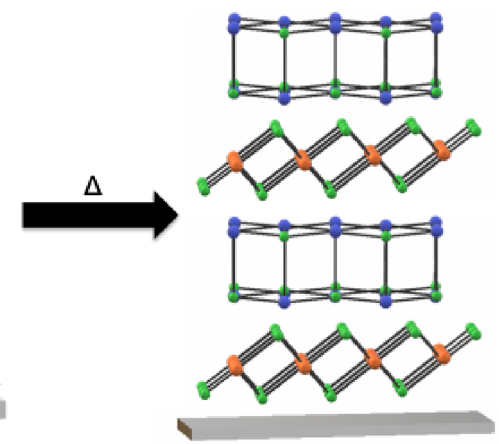
- Precursor structure designed to mimic the final product
- Deposit a target number of atoms of each element per layer
- Gentle annealing to crystallize, typically 5 - 30 minutes at 250 – 450$^{o}$C




Amorphous




Crystalline




In-plane rotational ("turbostratic") disorder

$SiO_2$/Si substrate (NOT epitaxial)

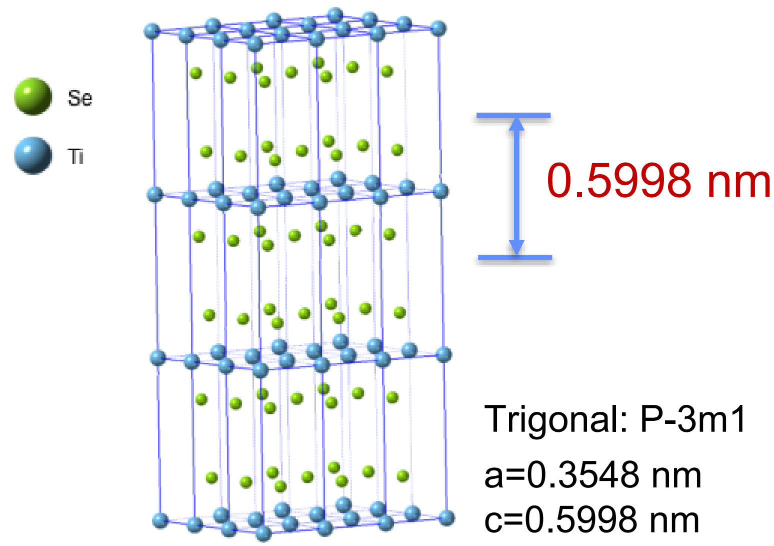
David Johnson et al., University of Oregon

**What types of crystal and layer defects arise?**

Sandia National Laboratories

# System: Multilayers of $TiSe_2$ & SnSe:

**$TiSe_2$**

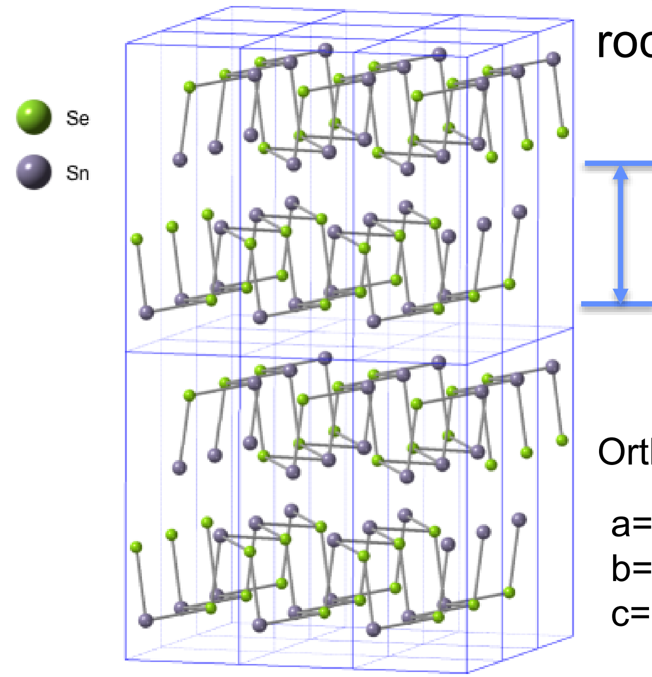



Trigonal: P-3m1
a=0.3548 nm
c=0.5998 nm

P. Erhlich, Angewandte Chemie (1948)

**SnSe**

Distortion from rocksalt structure




Orthorhombic: Pnma

a=1.14942 nm
b=0.4151 nm
c=0.4442 nm

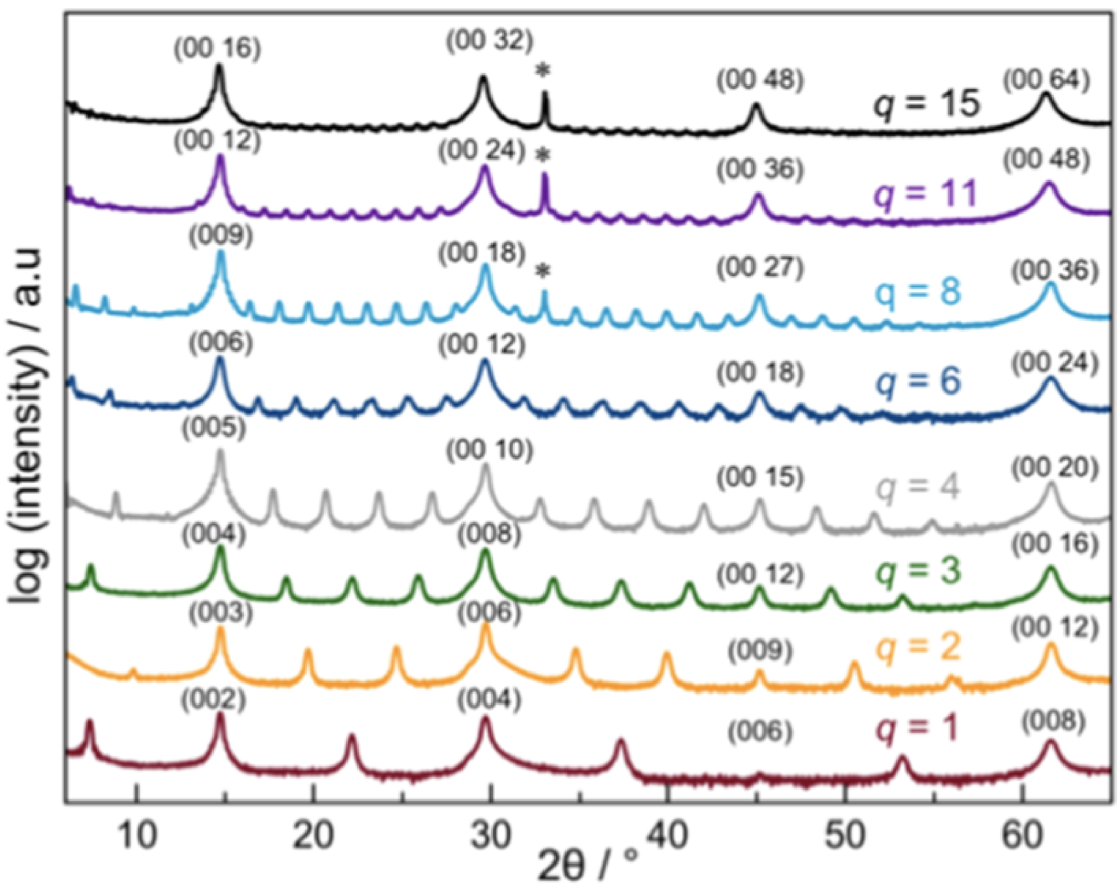
M. Sist et al., Acta Cryst. B (2016)

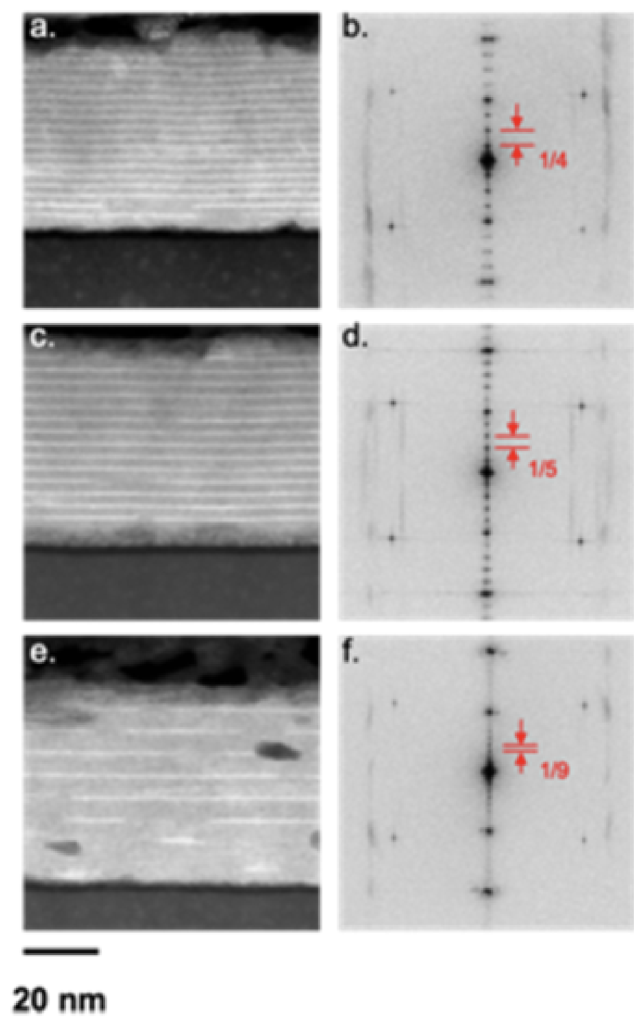
## Layer thicknesses are closely matched

-Bulk: Phase transition to Cmcm ~810 K)
-Monolayers: rectangular unit cell,
2D space group: p2gg
(Hamann et al., Inorganic Chemistry, 2017)

Sandia National Laboratories

# Good control of layer periodicities: $TiSe_2$/SnSe



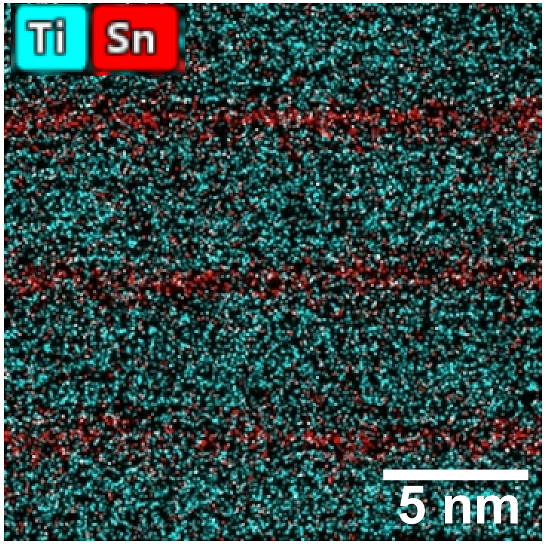




D.M. Hammon, D. Bardgett, S.R. Bauers, T.W. Kasel, A.M. Mroz, C.H. Hendon, D.L. Medlin, D.C. Johnson, Chem Materials, 2020

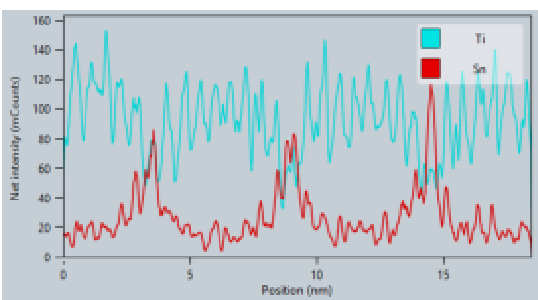
Sandia National Laboratories

# Example layered structure: 1 SnSe /8 $TiSe_2$

**EDS Spectrum Image**

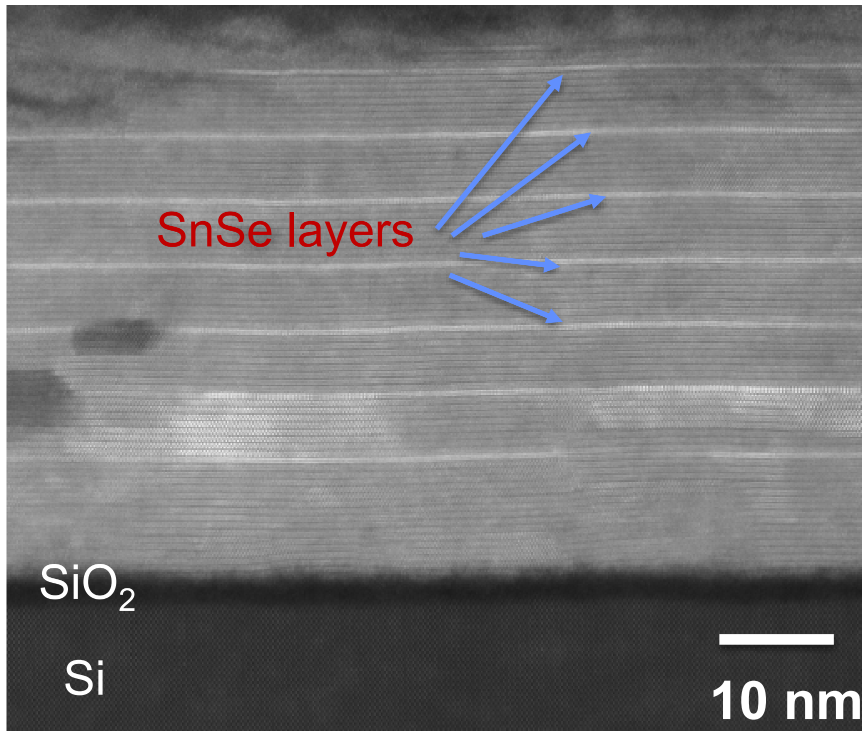



20180518_UO_Fischer/DH1-8/003)EDSmap_2p55mx/Si-EDS-HAADF 0908.emd,
net, postfilter 3 pix average

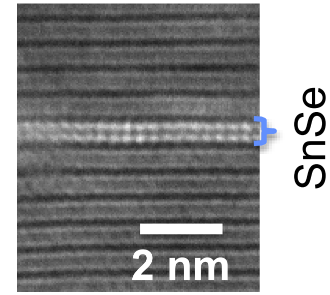



20180518_UO_Fischer/DH1-8/003)EDSmap_2p55mx/Si-EDS-HAADF 0908.emd,
net, prefiltering 3 pix average, postfilter 3 pix average

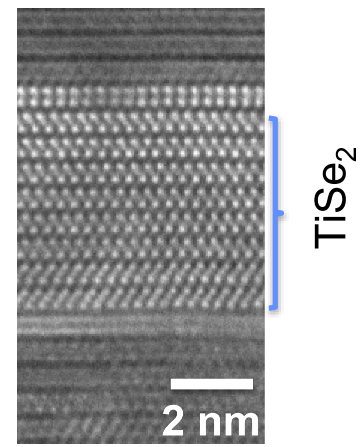
**HAADF-STEM**




20180518_UO_Fischer/DH1-8/002_area2/08.45.20_dfstem_fov73p81nm.jpg




08.48.48_dfstem_fov18p45nm.jpg



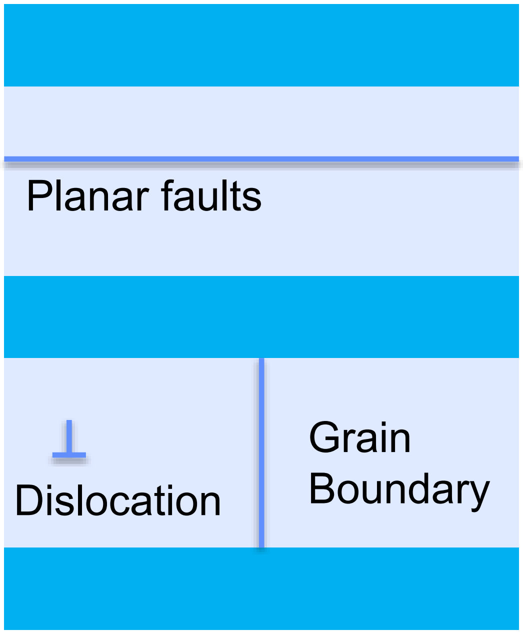

08.49.36_dfstem_fov18p45nm.jpg

*What about local defects?*

Sandia National Laboratories

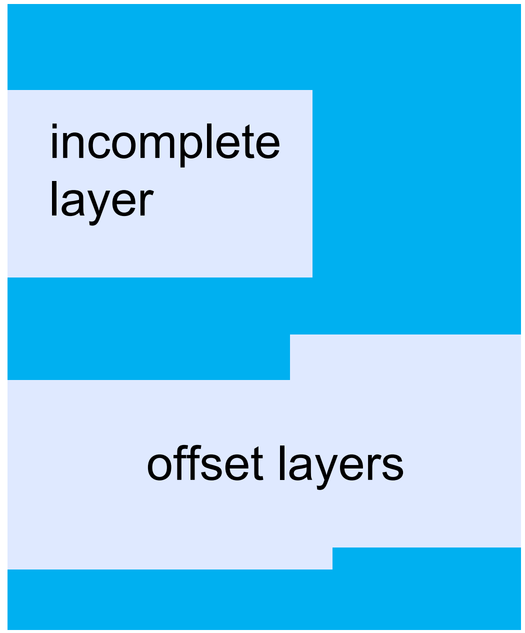
# Defects: Broad Classifications

## Intralayer defects



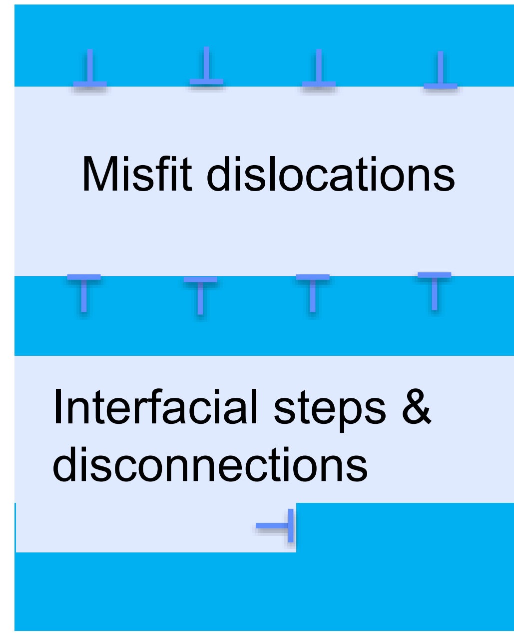

Crystalline defects within the confines of an individual layer

## Interlayer defects




Morphological defects in the layer arrangements

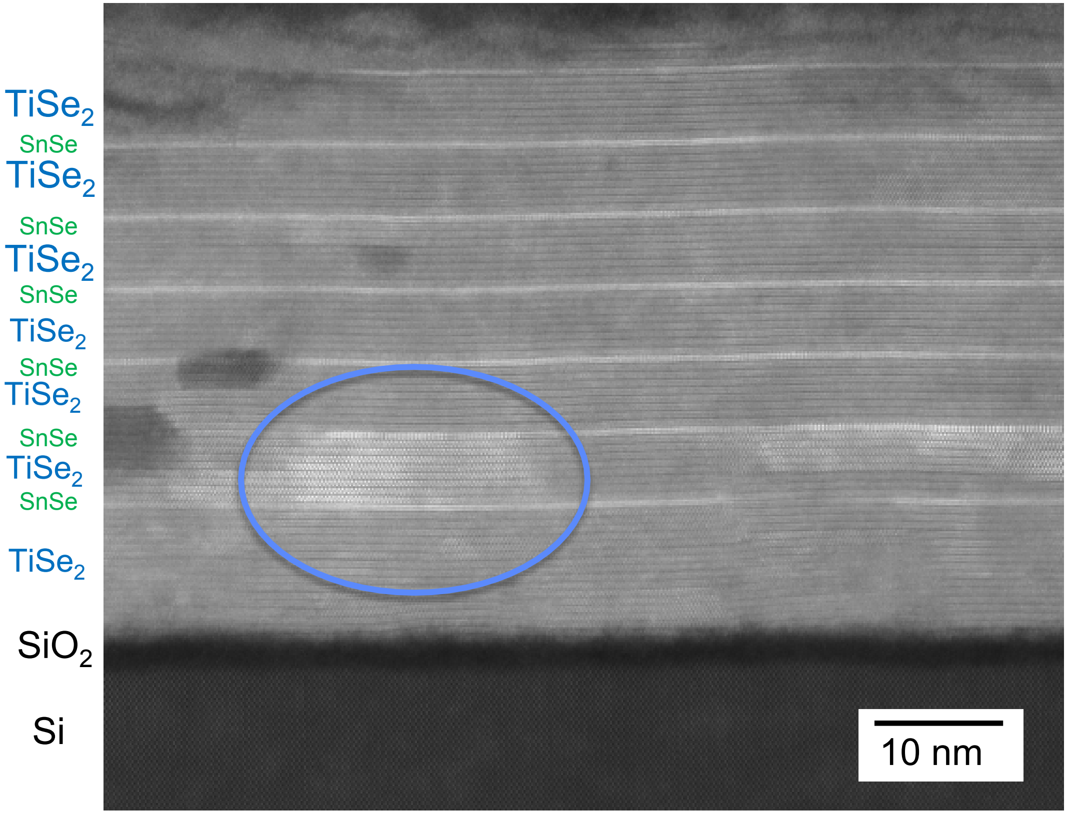
## Interfacial defects



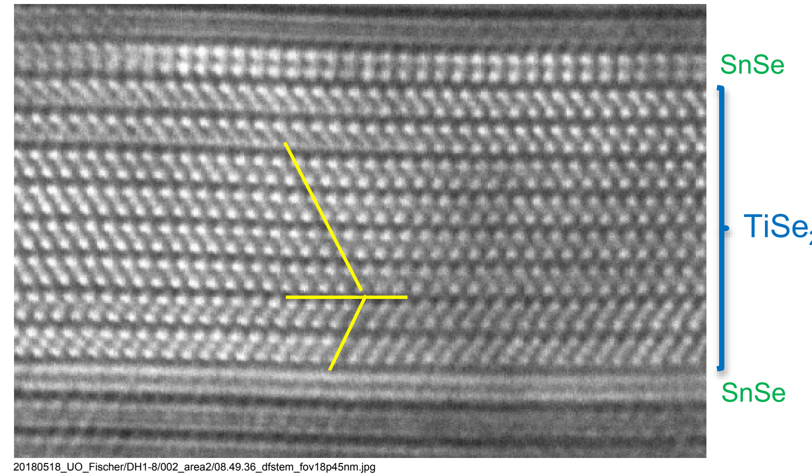

Crystalline defects at the interfaces between layers

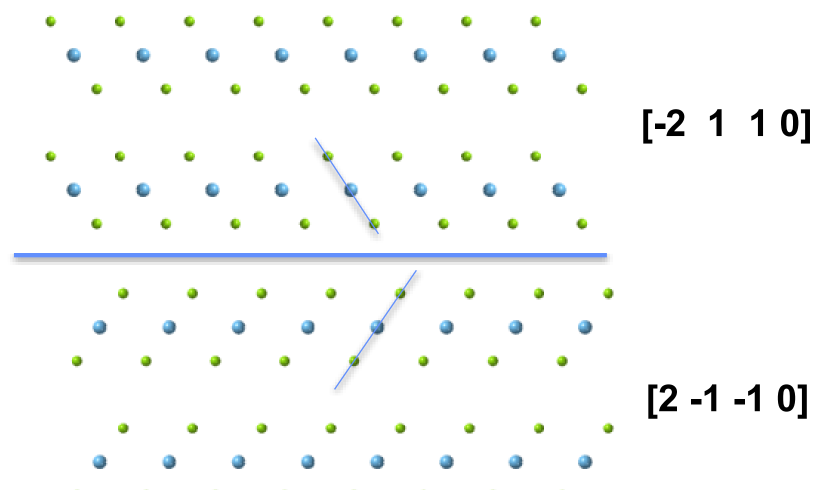
# Intralayer defects: GBs and twinning

1 SnSe : 8 $TiSe_2$




Twin boundary in $TiSe_2$ layer




$TiSe_2$ has rhombohedral symmetry
180° rotation about [0001] axis gives twin

Crystal nucleation during annealing

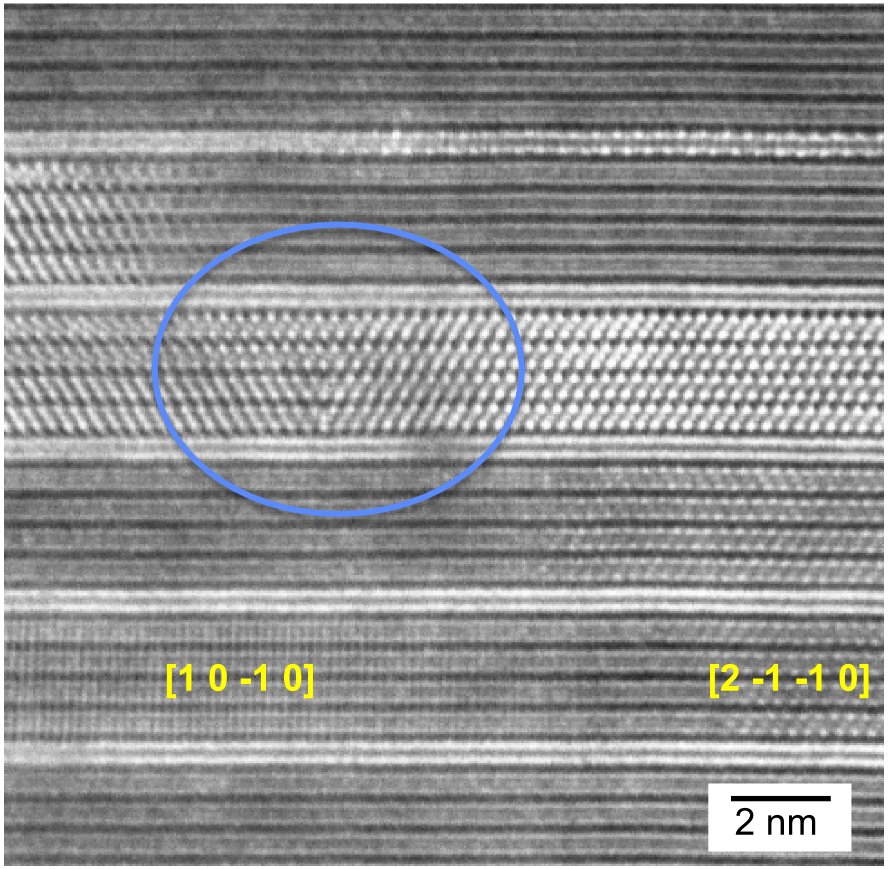
R. Gannon, D. Hamann, J. Ditto, G. Mitchson, S. Bauers; D. Merrill, D. Medlin; D. Johnson,
ACS Applied Nano Materials, submitted 2021

Sandia National Laboratories

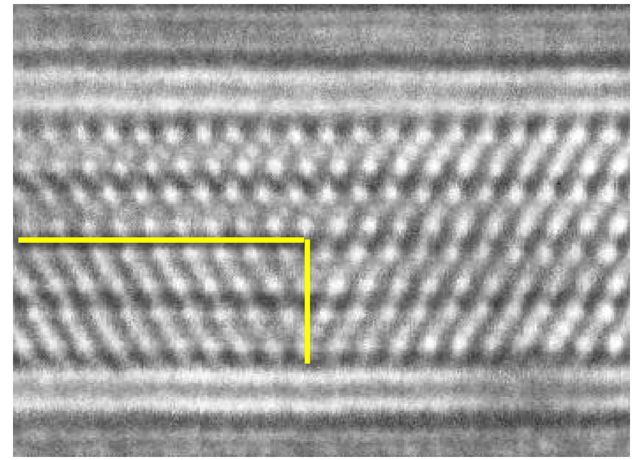
# Termination of a twin within a $TiSe_2$ layer

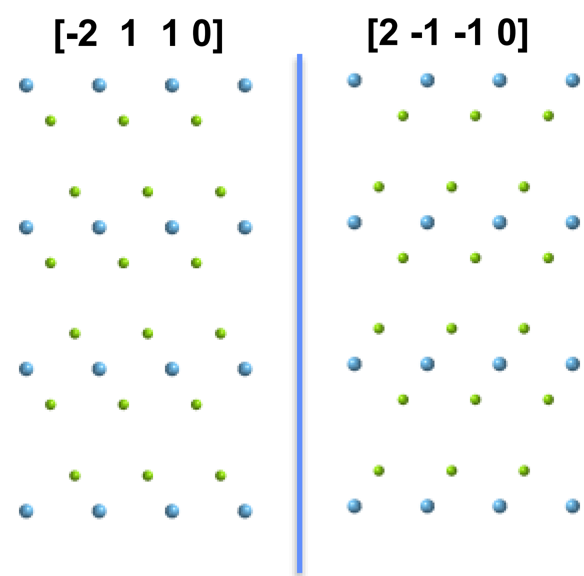
1 SnSe: 4 $TiSe_2$

$TiSe_2$
SnSe
$TiSe_2$
SnSe
$TiSe_2$
SnSe
$TiSe_2$
SnSe
$TiSe_2$
SnSe




20180518_UO_Fischer/DH1-4/10.40.55_dfstem_fov19p14nm_rotm87p843deg.jpg





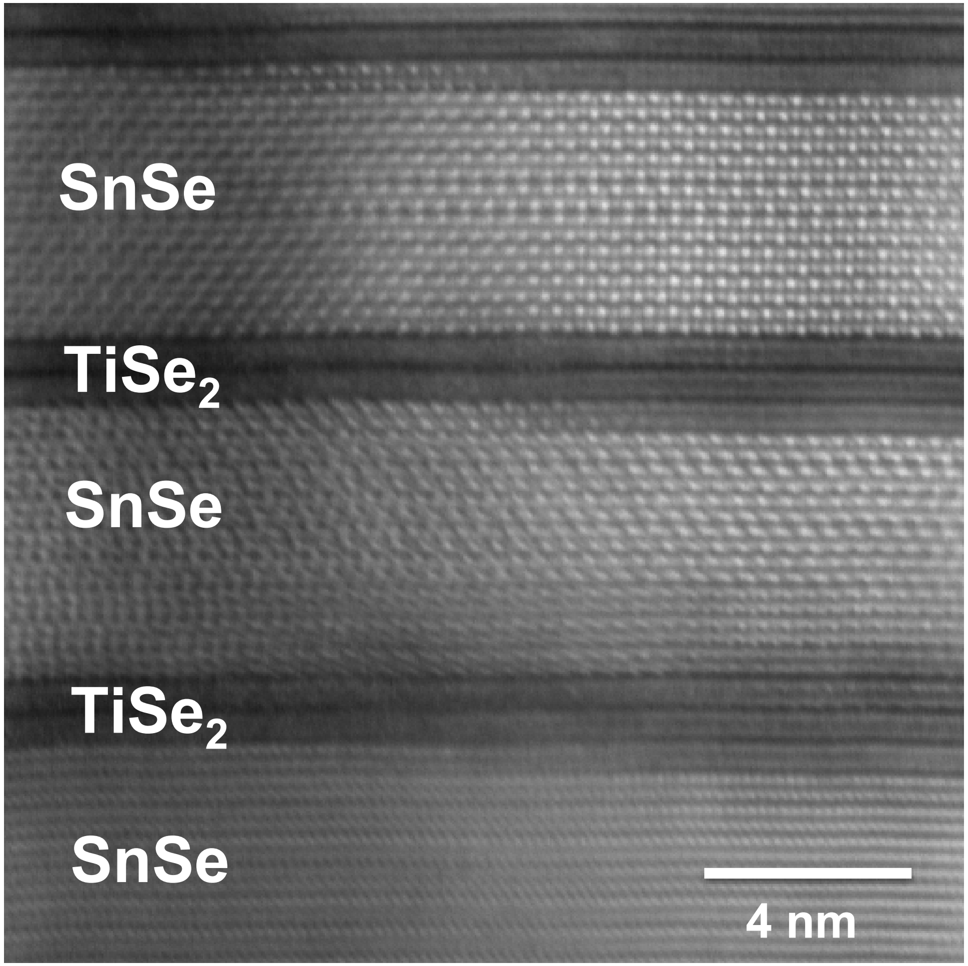

Analogous to "incoherent twin" in FCC materials

"Double-positioning" boundary

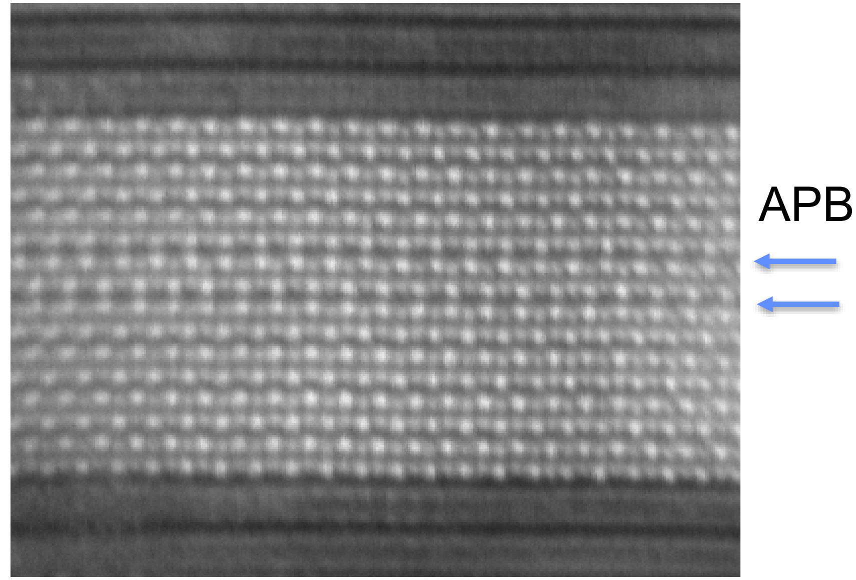

R. Gannon, D. Hamann, J. Ditto, G. Mitchson, S. Bauers; D. Merrill, D. Medlin; D. Johnson,
ACS Applied Nano Materials, submitted 2021

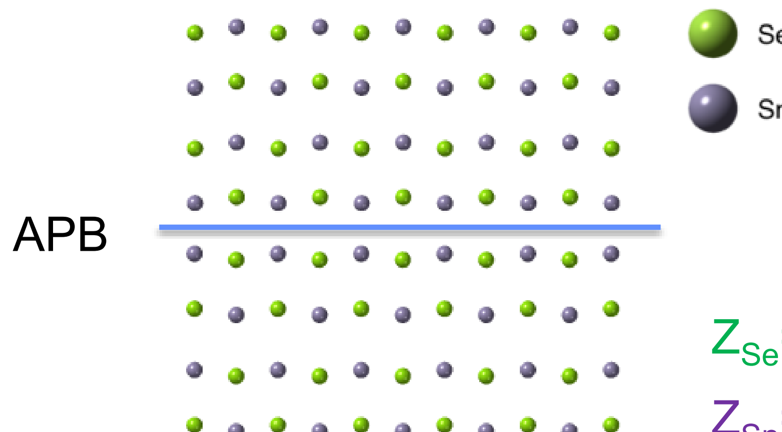
# Intralayer defect: Anti-Phase Boundaries in SnSe

9 SnSe : 2 $TiSe_2$




20190322_UO_SnSe_TiSe2/9_2/001_area1/14.55.58_dfstem_fov18p45nm.jpg




$Z_{Se}=34$

$Z_{Sn}=50$

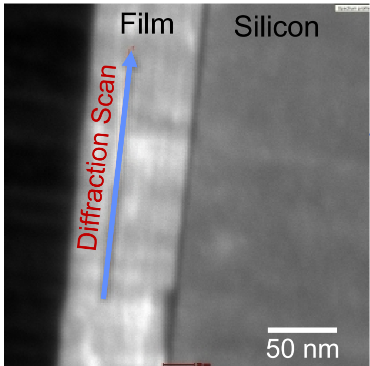
[001] direction

Orthorhombic Pnma structure

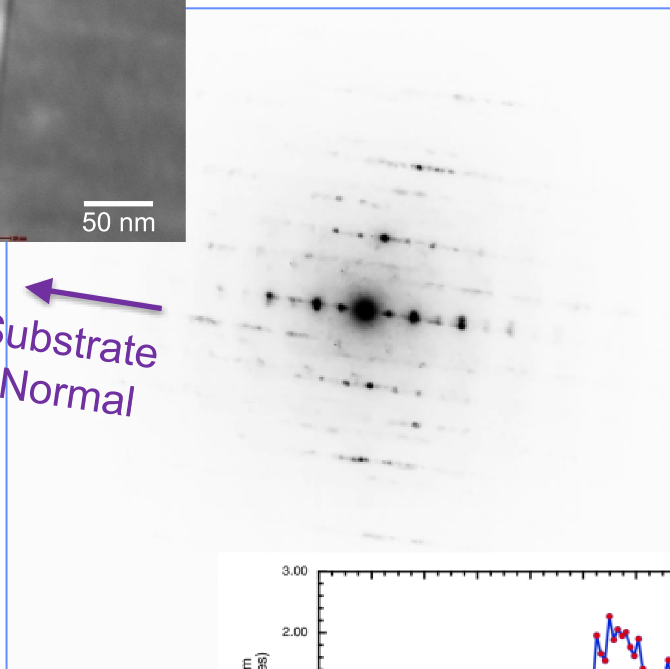
R. Gannon, D. Hamann, J. Ditto, G. Mitchson, S. Bauers; D. Merrill, D. Medlin; D. Johnson,
ACS Applied Nano Materials, submitted 2021

Sandia National Laboratories

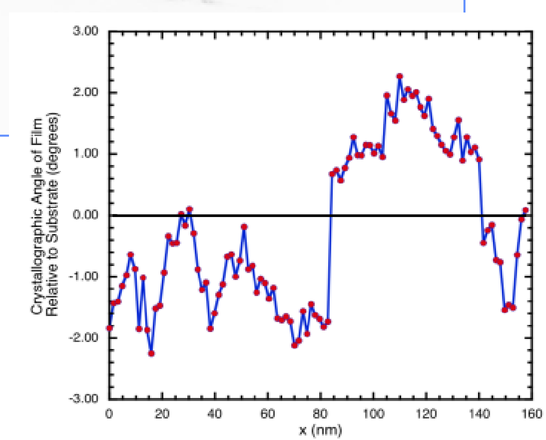
# Spread in out-of-plane orientation



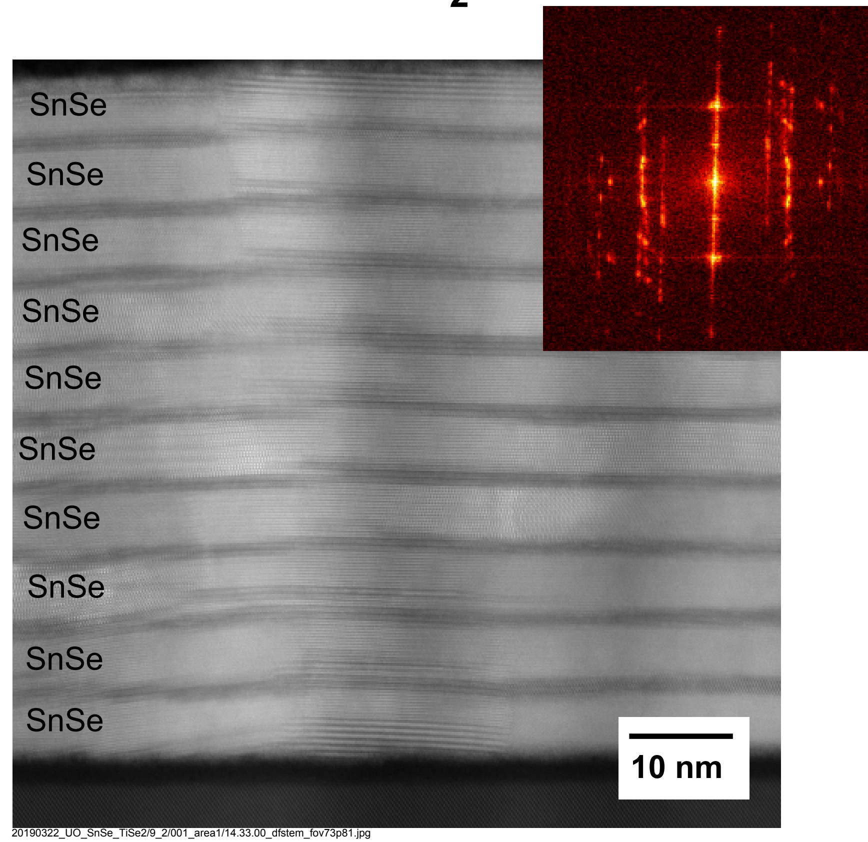

Scanning Microbeam Diffraction




9 SnSe : 2 $TiSe_2$



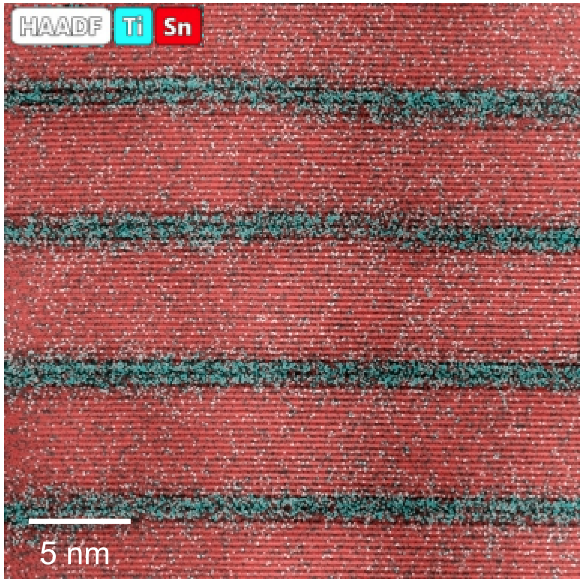

9 SnSe : 2 $TiSe_2$



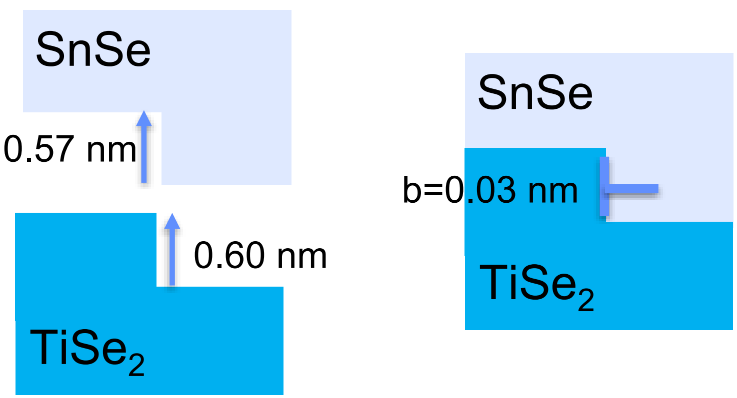

**Key signature of interlayer defects**

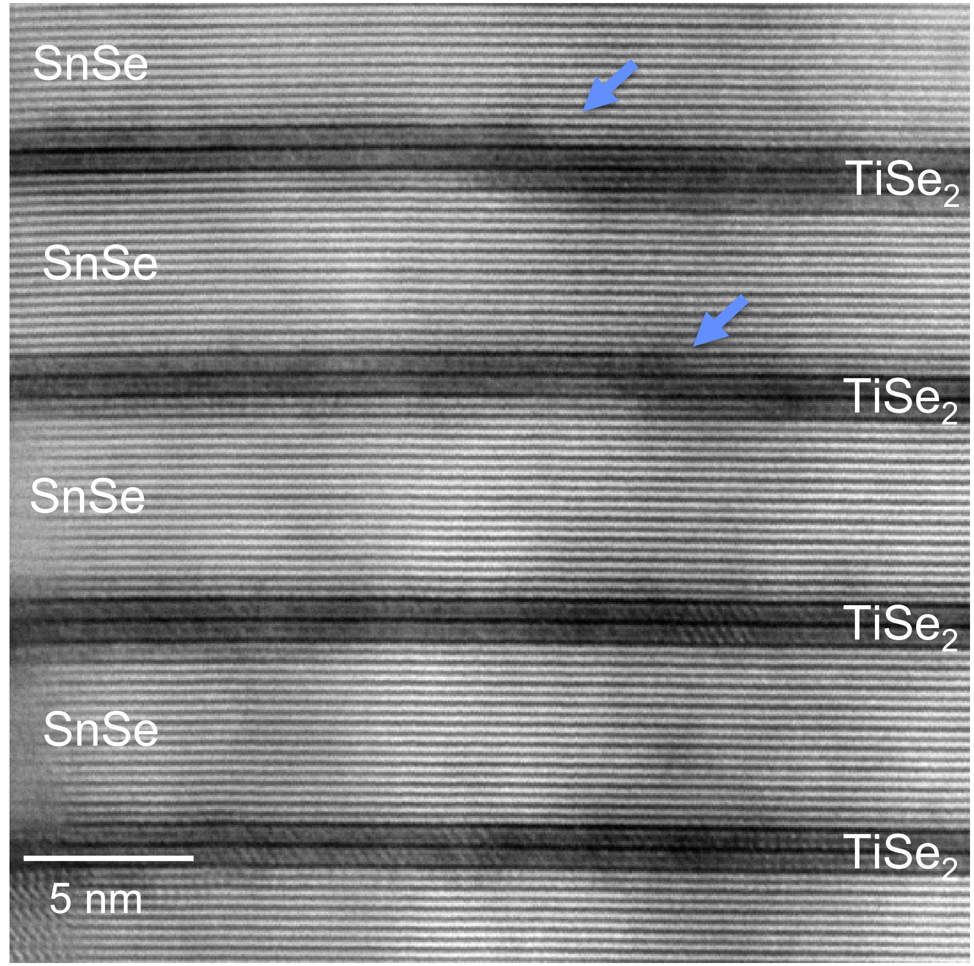
Sandia National Laboratories

# Interlayer Defects: Joining of two incomplete layers




20190407_UO_SnSe_TiSe2/9_2/007_EDSSI_2p55mx/SI EDS-HAADF 0329.emd



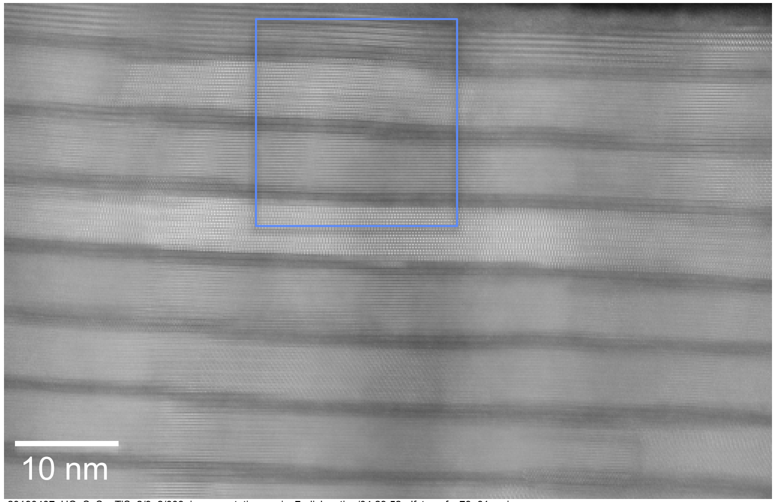

**"Disconnection" == defect with both step and dislocation content**

**Because the step-heights are similar, the dislocation content is small**

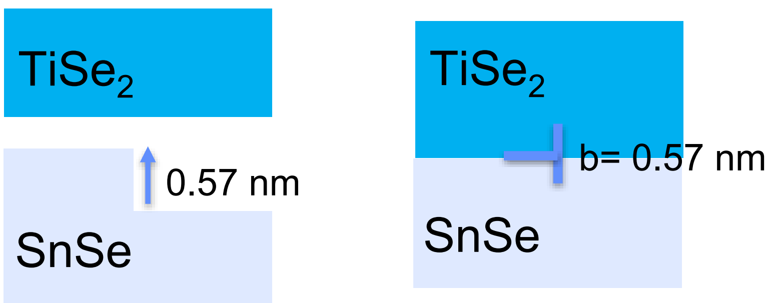
R. Gannon, D. Hamann, J. Ditto, G. Mitchson, S. Bauers; D. Merrill, D. Medlin; D. Johnson,
ACS Applied Nano Materials, submitted 2021

Sandia National Laboratories

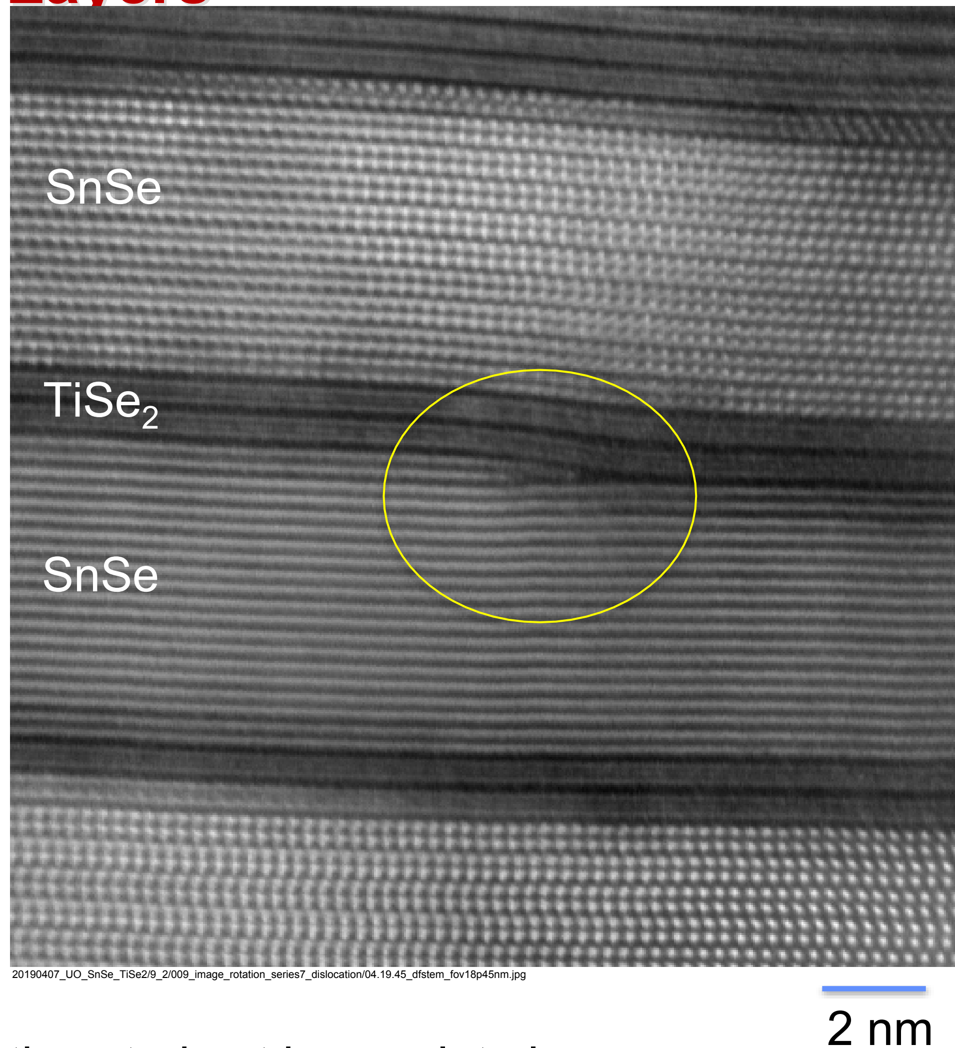
# Interlayer Defects: Incomplete Layer between Full Layers




20190407_UO_SnSe_TiSe2/9_2/009_image_rotation_series7_dislocation/04.29.52_dfstem_fov73p81nm.jpg




Much larger Burgers vector




20190407_UO_SnSe_TiSe2/9_2/009_image_rotation_series7_dislocation/04.19.45_dfstem_fov18p45nm.jpg

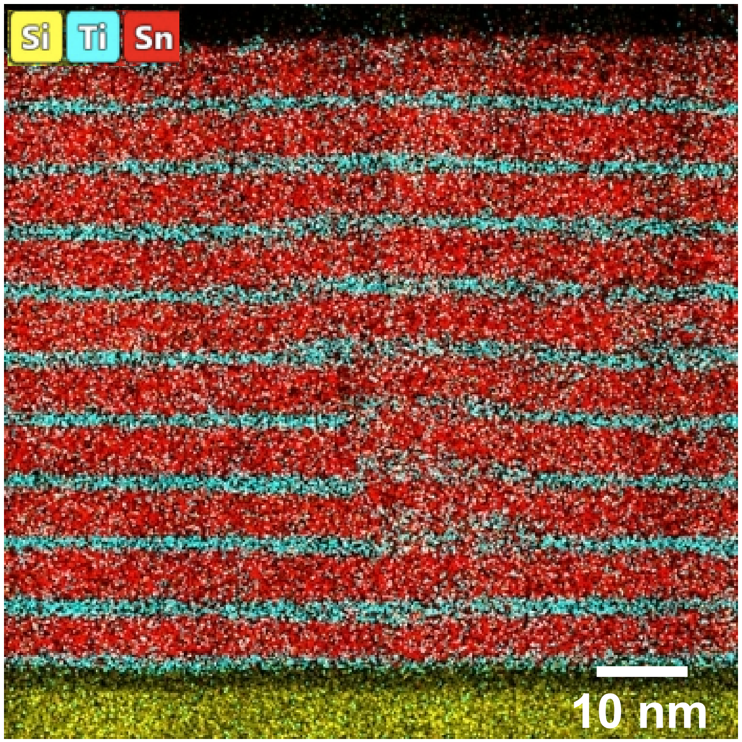
Bending associated with the dislocation strain at incomplete layers is the likely origin of spread in out-of-plane orientation.

R. Gannon, D. Hamann, J. Ditto, G. Mitchson, S. Bauers; D. Merrill, D. Medlin; D. Johnson, ACS Applied Nano Materials, submitted 2021

Sandia National Laboratories

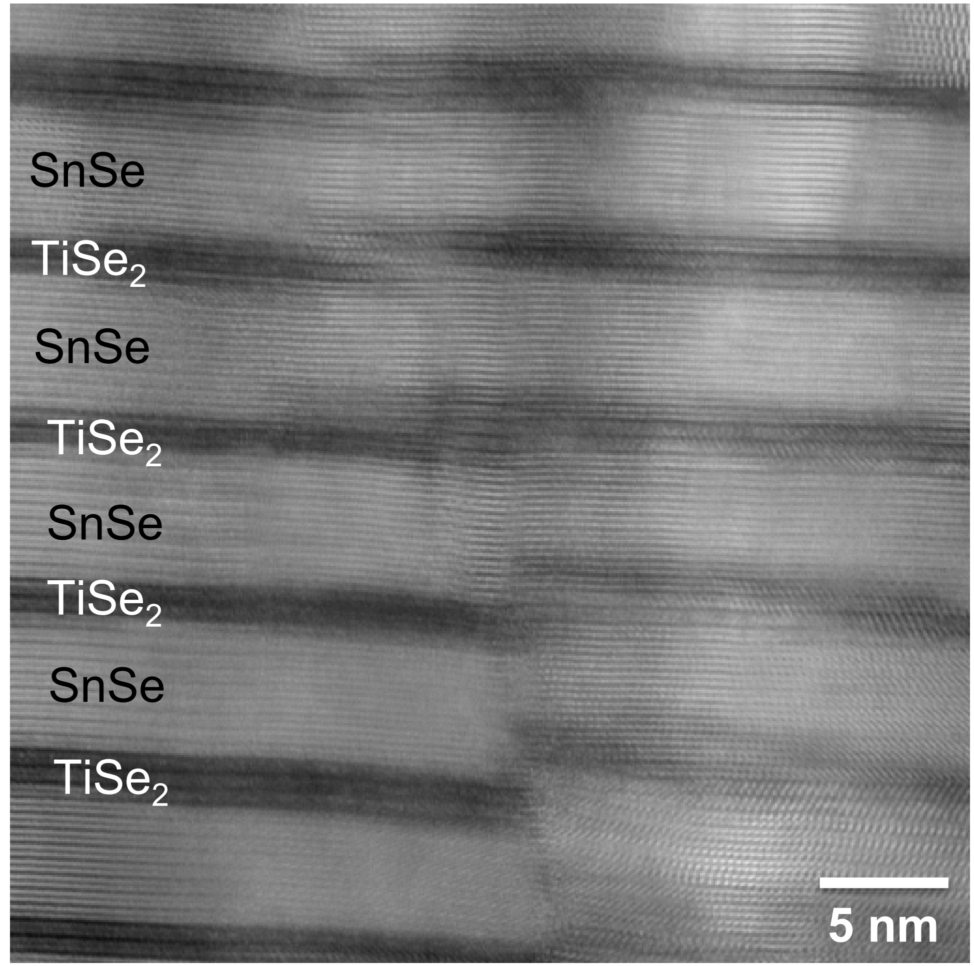
# Interlayer Defects: Offset layers

EDS Spectrum Image




20190407_UO_SnSe_TiSe2/9_2/006_EDSSI_910kx/SI EDS-HAADF 0305.emd

HAADF-STEM




20190407_UO_SnSe_TiSe2/9_2/005_image_rotation_series5/02.48.38_dfstem_fov36p91nm.jpg

# Synopsis

**Key structural aspects layered chalcogenides are manifested in the detailed structures of extended defects in these materials.**

- **Weak, van der Waals bonding across double chalcogenide layers**
- **Ability to accommodate non-stoichiometry through altering the layer stacking**
- **Close inter-relationship between the rocksalt and tetradymite structural types.**

**Atomic resolution microscopy is clarifying the character of dislocations, interfaces, and interfacial line defects in the chalcogenides**

- **Complex, dissociated core structures observed in $Bi_2Te_3$ and $ZrTe_5$**
- **Role in phase transformations and accommodating coherency strain**
- **Insight to defect formation in novel synthesis strategies.**

Sandia National Laboratories

# Acknowledgements

**Sandia**

K. Erickson
J.D. Sugar
N.Y.C. Yang
C.Spataru
M. Siegal,
S. Limmer
W.G. Yelton
P. Sharma
W. Pan

**George Mason University**

Y. Mishin

**NIST**

L. Hale

**University of Oregon**

R. Gannon
D.Hamann
J. Ditto
D.Johnson

**California Institute of Technology**

N.A. Heinz,
T. Ikeda,
G.J. Snyder
(present address Northwestern Univ)


Sandia National Laboratories