



Exploring the Interplay Between Grain Boundary Facet Junctions and Dislocations

D.L. Medlin¹, G. Lucadamo^{1,*}, J.A. Zimmerman¹

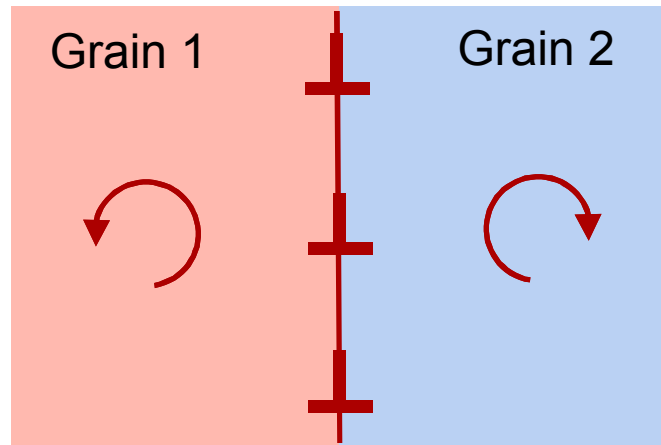
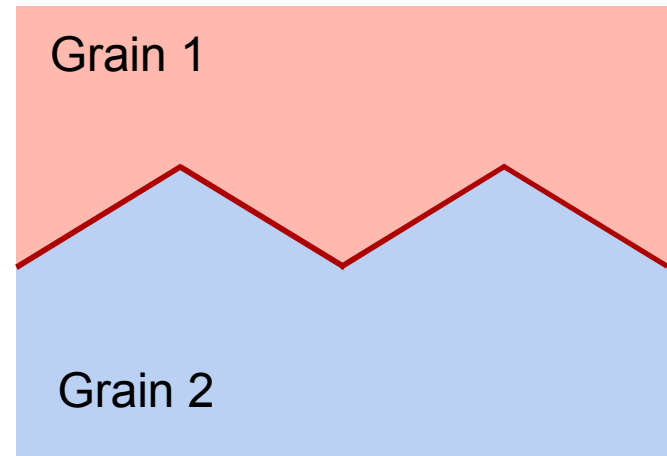
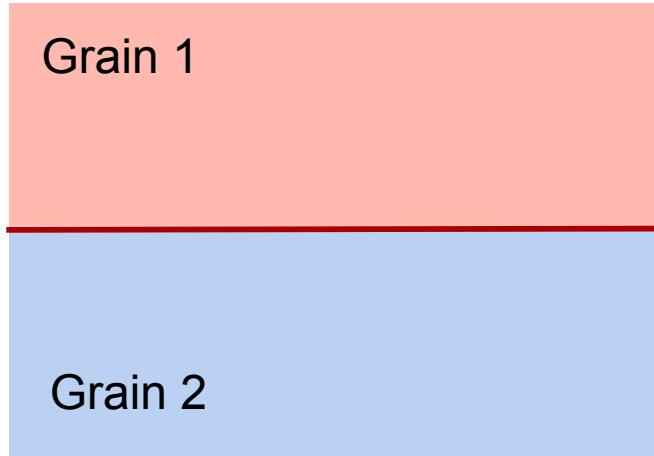
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*Present Address, Bettis Atomic Power Lab, PA, USA

Facet Junctions and Grain Boundary Dislocations: Accommodating deviations from low energy interfaces



- **Inclination:**

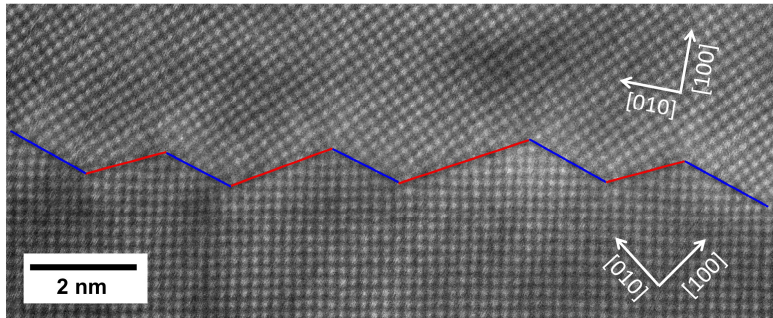
- Reduce energy by faceting on lower energy planes
- Facet junctions.

- **Misorientation:**

- Accomodate deviation with grain boundary dislocations

Focus for this talk: Interplay between facet junctions and grain boundary dislocations

$\Sigma=5$ {210}/{310} facets in BCC Fe

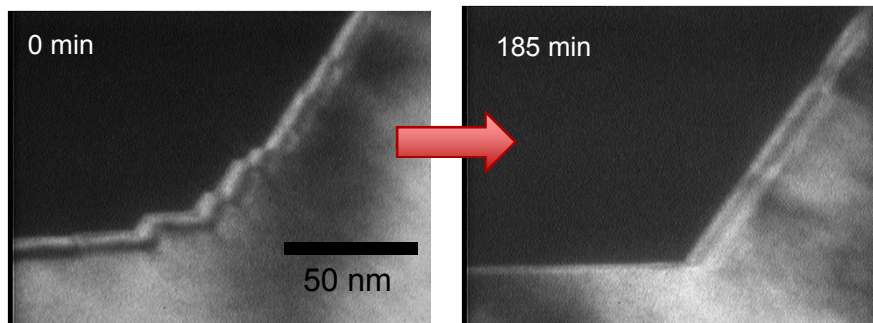


F. Abdeljawad, et al., in review, Journal of Applied Physics (2016)
D.L. Medlin, et al., in prep. for Acta Materialia (2016)

- Atomistic scale observations and calculations.

- Insights concerning facet junction structure and arrangement from consideration of grain boundary dislocation array

$\Sigma=3$ {112} facets in Au

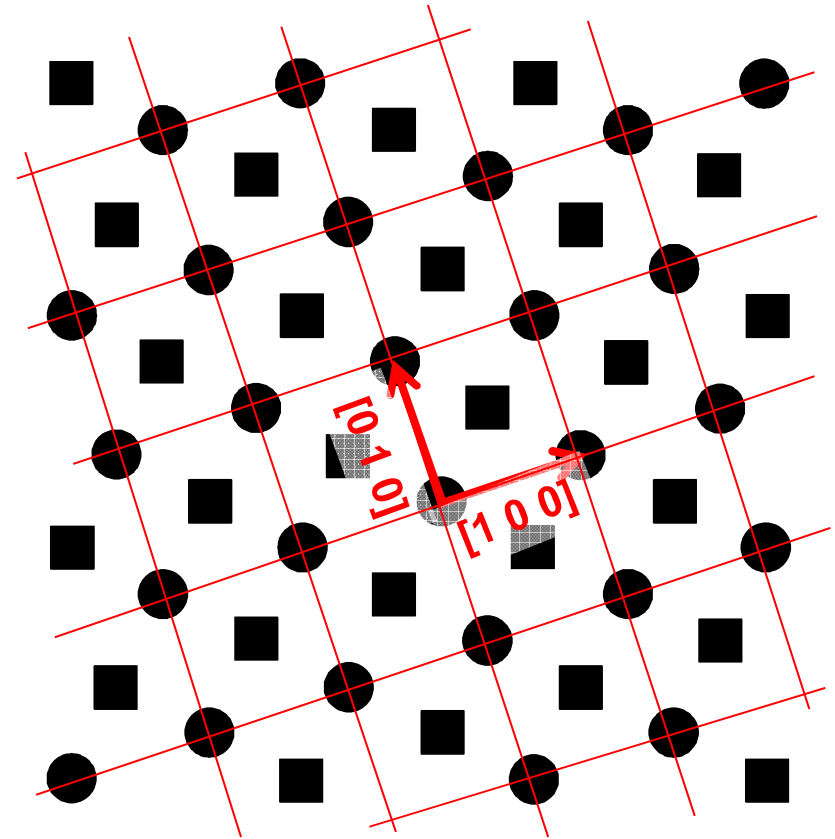
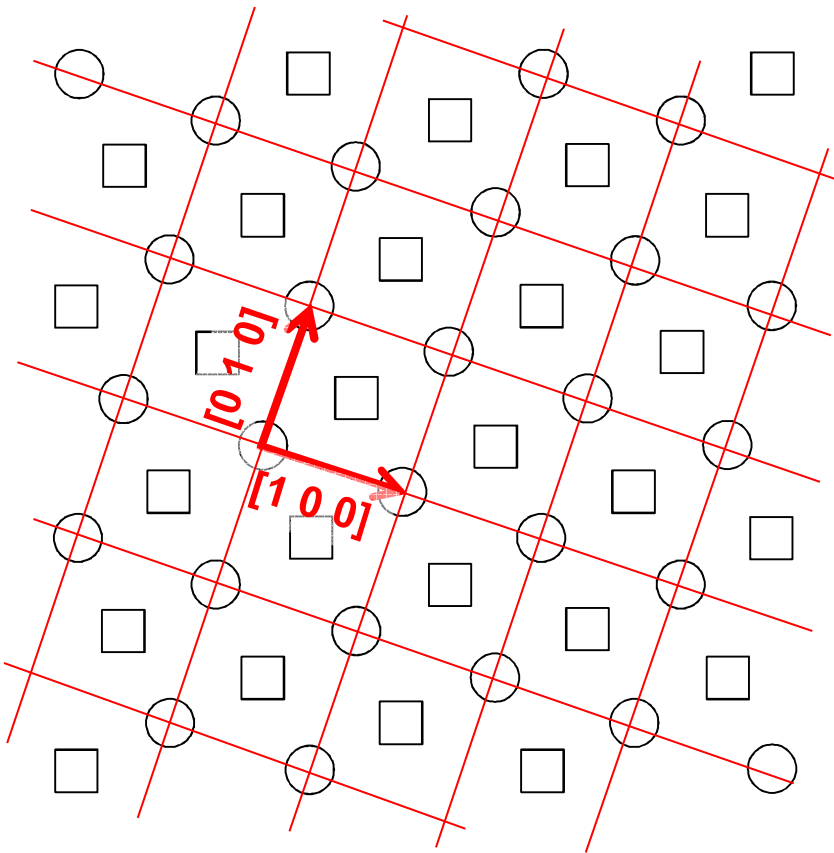


D.L. Medlin and G. Lucadamo, MRS Symp 252 (2001)
G. Lucadamo and D.L. Medlin, Acta Materialia 50 (2002) 3045.

- In situ TEM observations:

- Complex facet dynamics resulting from grain boundary dislocation, facet interactions.

BCC $\Sigma=5$ [001]: Interfacial Crystallography

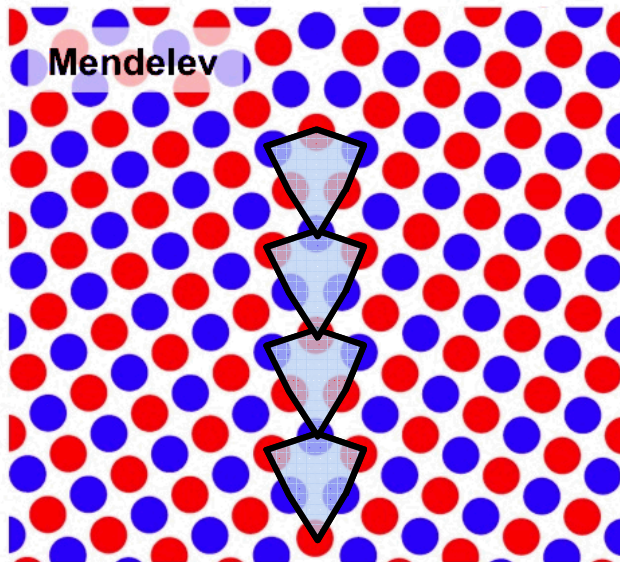


Misorientation: 36.87° Rotation about [001]

Inclination: Defines the boundary plane

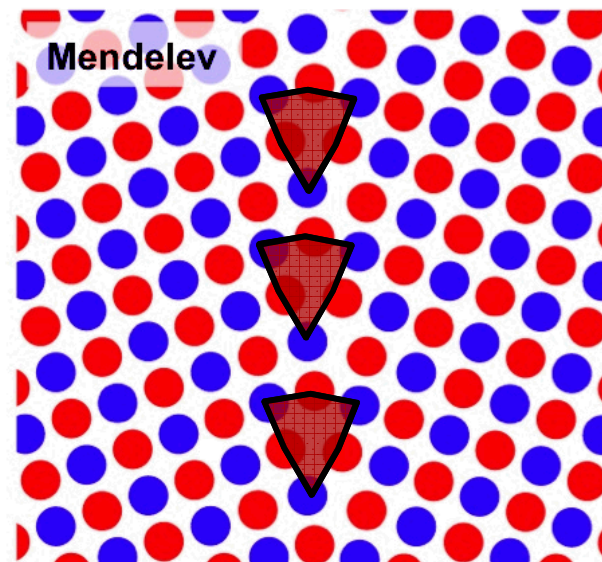
Two Symmetric inclinations: 310 and 210

{310}



Inclination: 0°

{210}

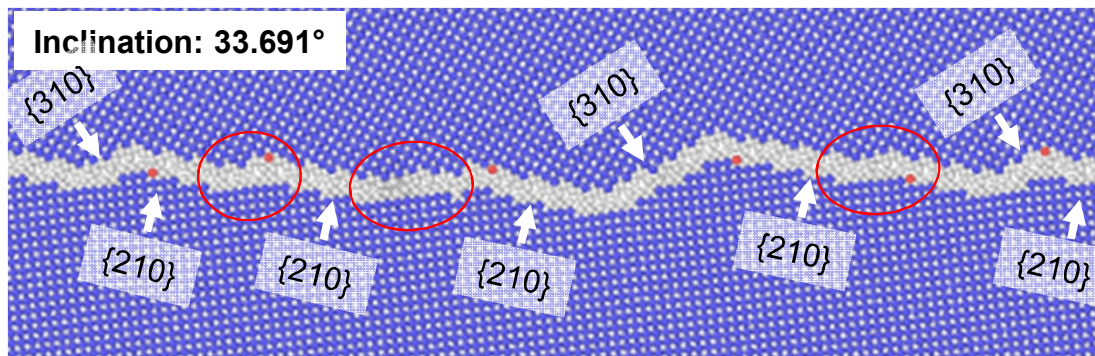
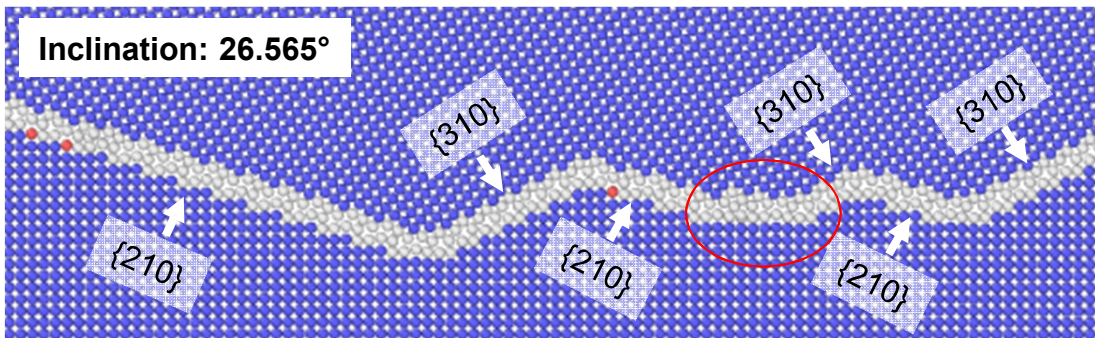
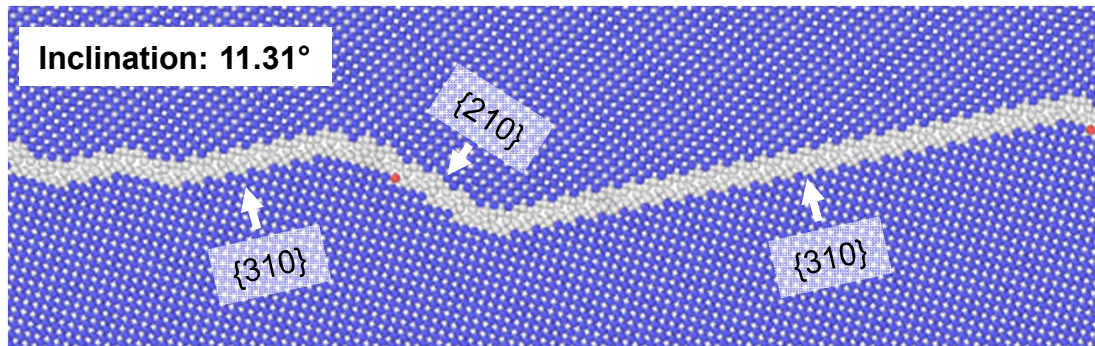


Inclination: 45°

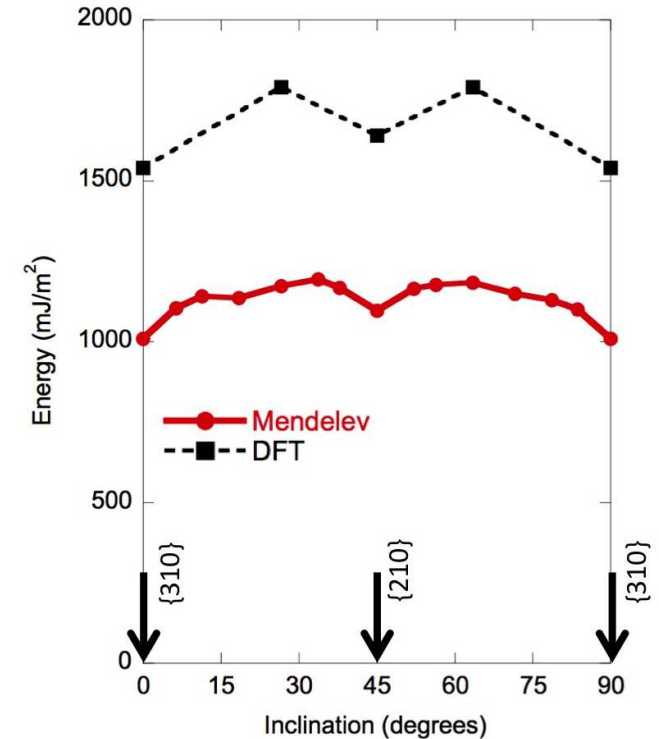
$\Sigma=5$ Misorientation: 36.87°

Atomistic calculations using the Mendelev Fe potential. Atoms shaded by depth.

Variation in Structure and Energy with Inclination: MD shows 310 and 210 faceting



Mendelev Fe Potential

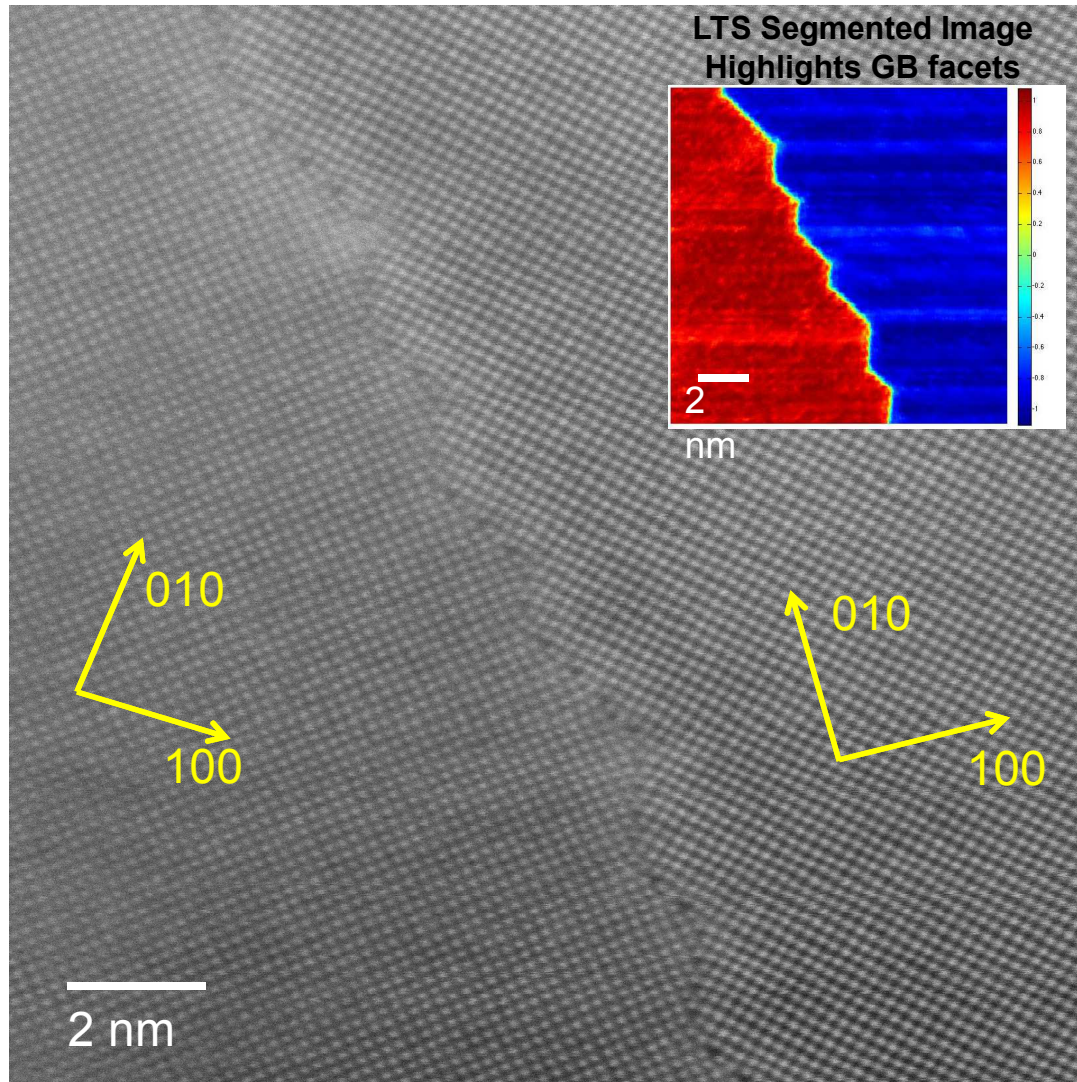


Additional faceting on {710}/{110} planes:

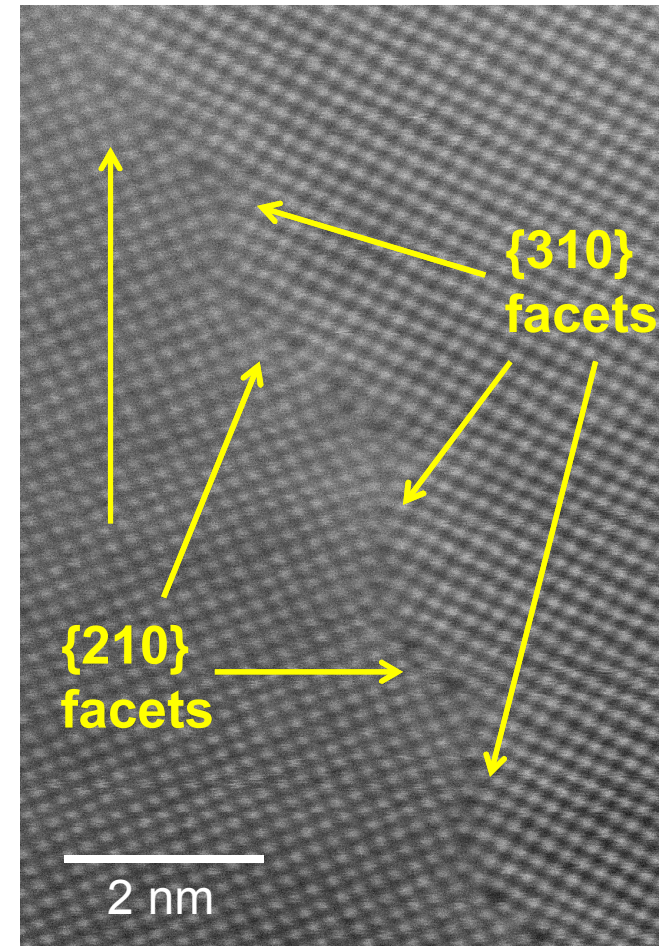
- 1:1 ratio of {310} and {210} units
- Not fully coarsened into lower energy {210}, {310} facets.

HRSTEM shows nanoscale faceting at Grain boundary Sandia National Laboratories

HAADF-STEM $\Sigma=5$ $\langle 001 \rangle$ Boundary in Fe

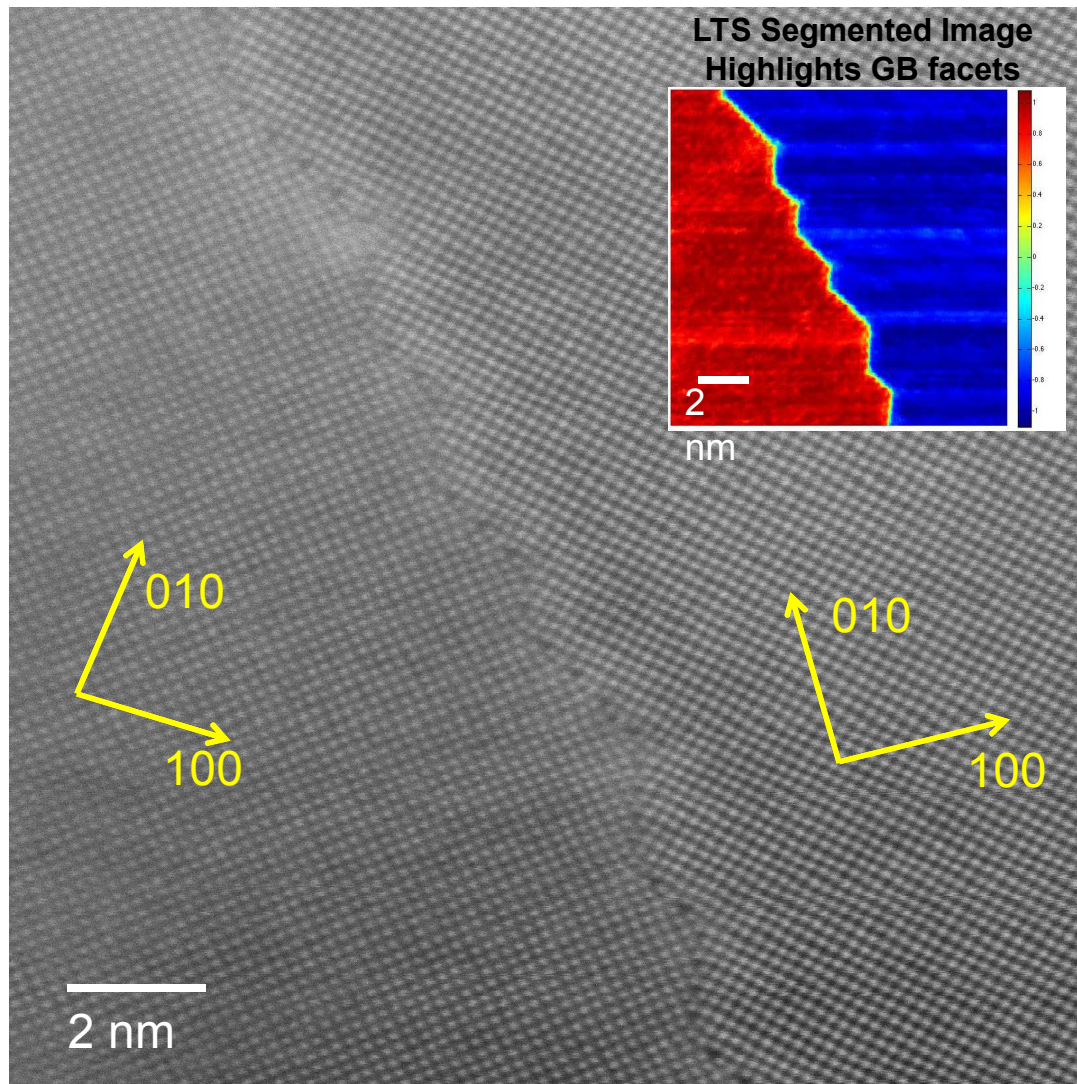


Boundary is faceted on $\{210\}$ and $\{310\}$ type inclinations

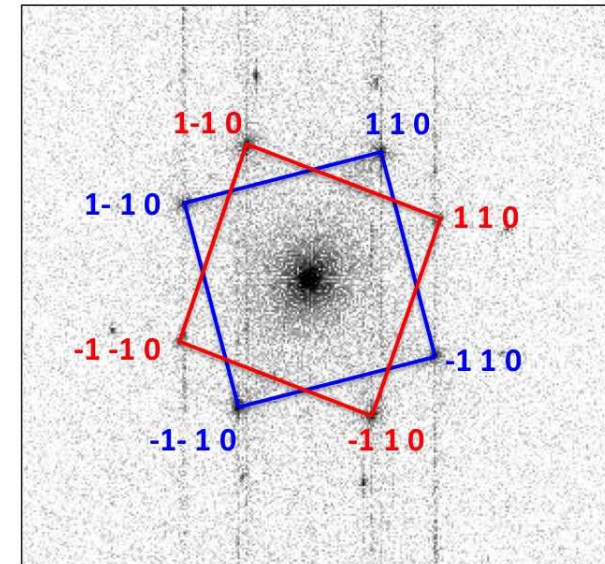


HRSTEM shows nanoscale faceting at Grain boundary Sandia National Laboratories

HAADF-STEM $\Sigma=5$ $\langle 001 \rangle$ Boundary in Fe



Boundary Geometry

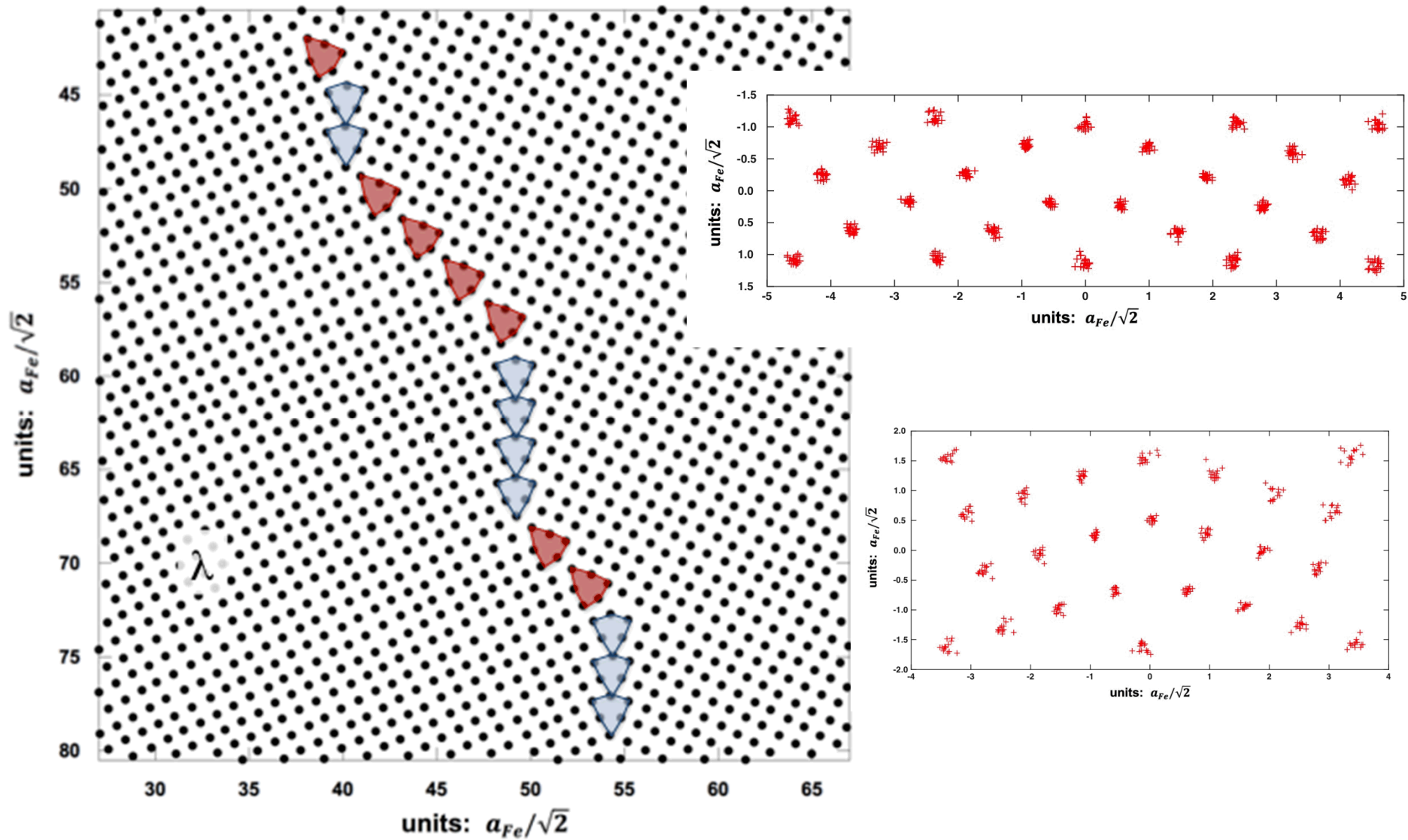


Inclination from $\{310\}$:
 $\phi = 26.3^\circ \pm 1^\circ$

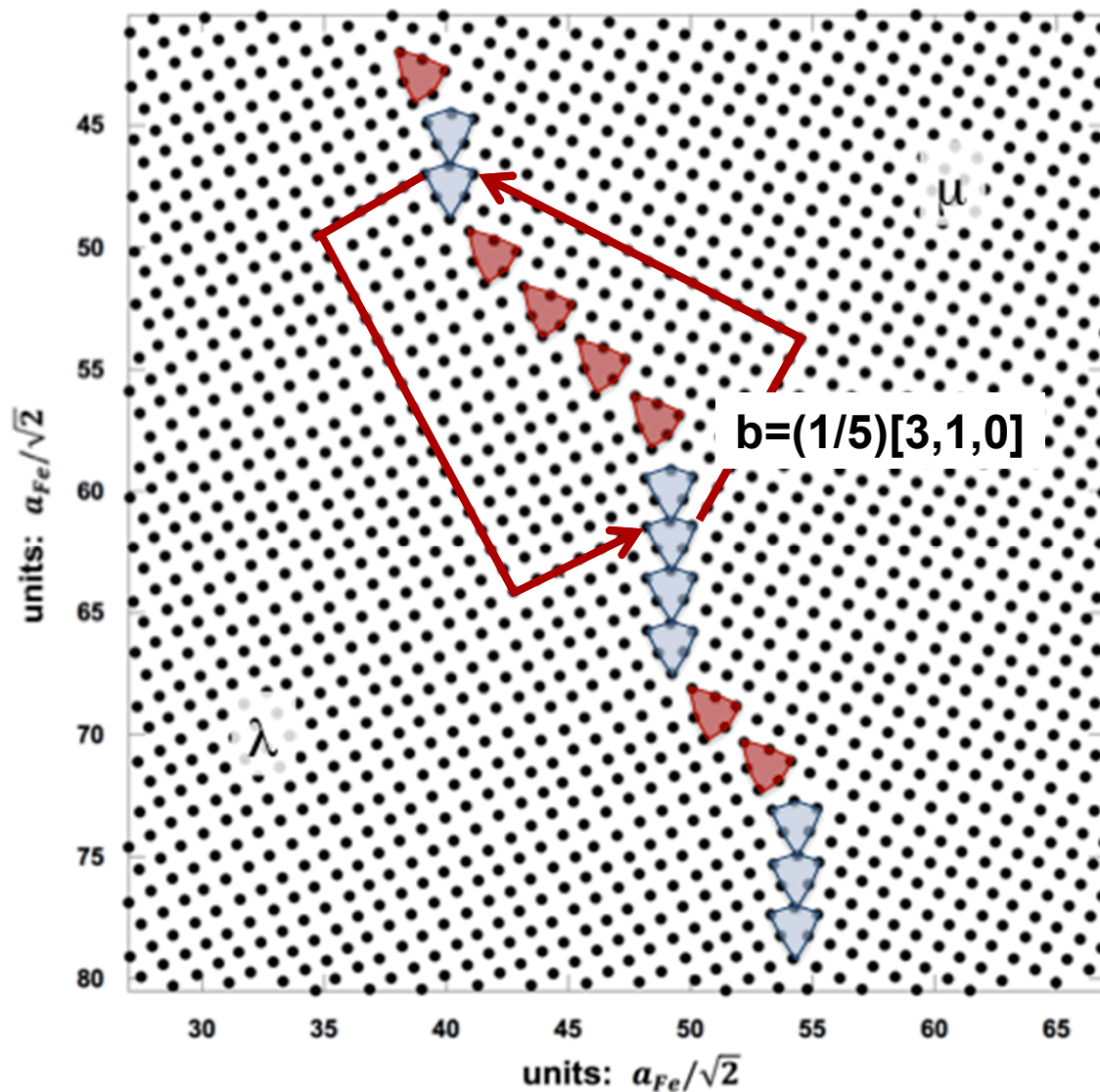
Misorientation:
 $\theta = 34.49 \pm 0.75^\circ$

$\Delta\theta = -2.38 \pm 0.75^\circ$
from exact $\Sigma=5$

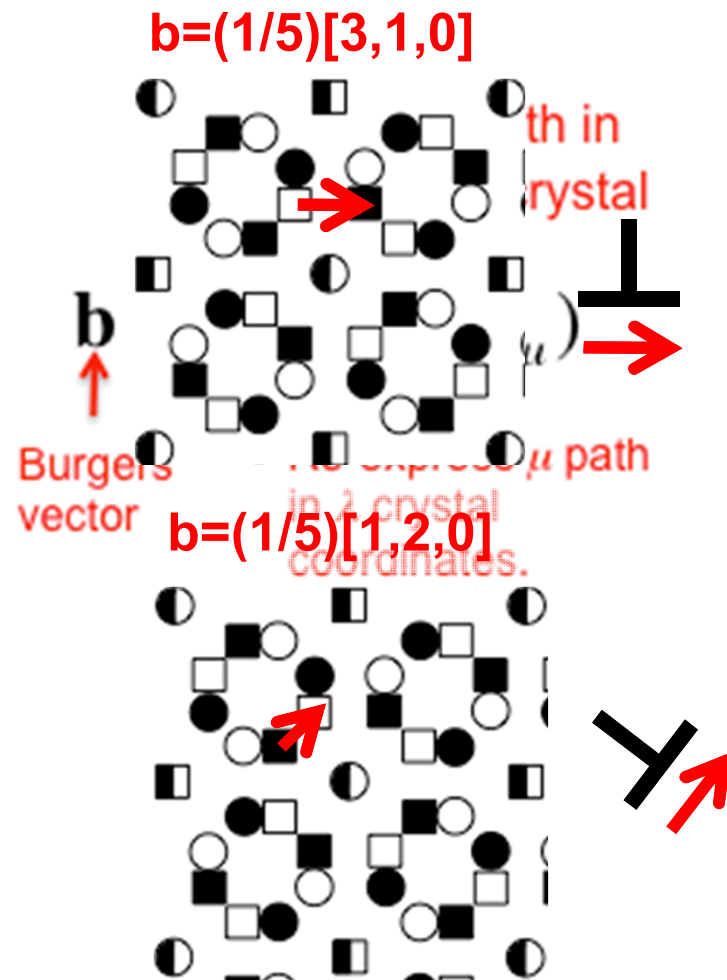
Experimental Intensity peak positions from HAADF-STEM



Circuit Mapping: Secondary GB Dislocations at all facet Junction Pairs



Two types of defect observed:



Observed SGBD Density Accommodates Deviation from Exact $\Sigma=5$ Misorientation

Dislocation Content Required is function of both Misorientation *and* Inclination

Frank-Bilby Equation:

$$\mathbf{b}\text{-vector density} \rightarrow \mathbf{B} = (\mathbf{I} - \mathbf{P}^{-1}) \mathbf{v} \leftarrow \text{Interface vector, defining inclination}$$

Matrix defining misorientation from reference

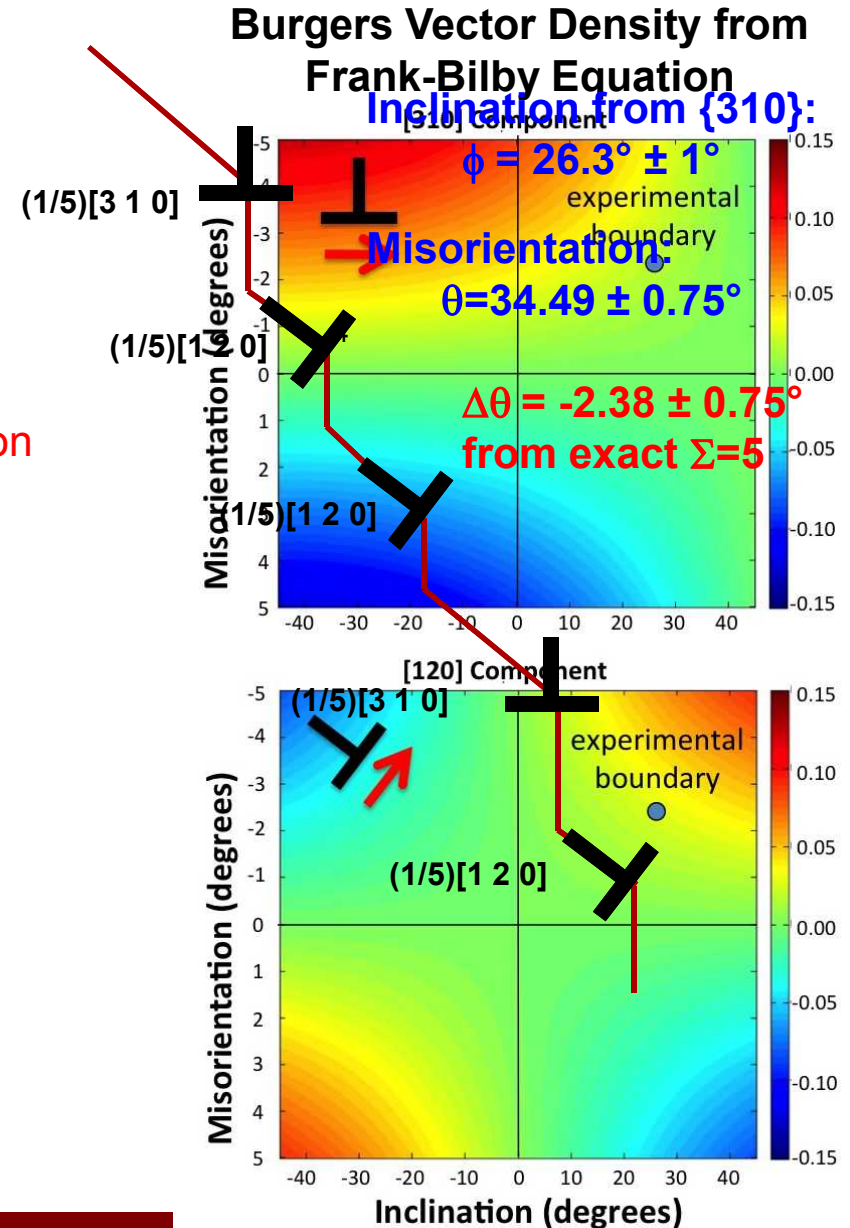
Predicted Burgers Vector Density:

$$\mathbf{B} = [0.029 \pm 0.010, 0.030 \pm 0.010, 0]$$

Observed Burgers Vector Density:

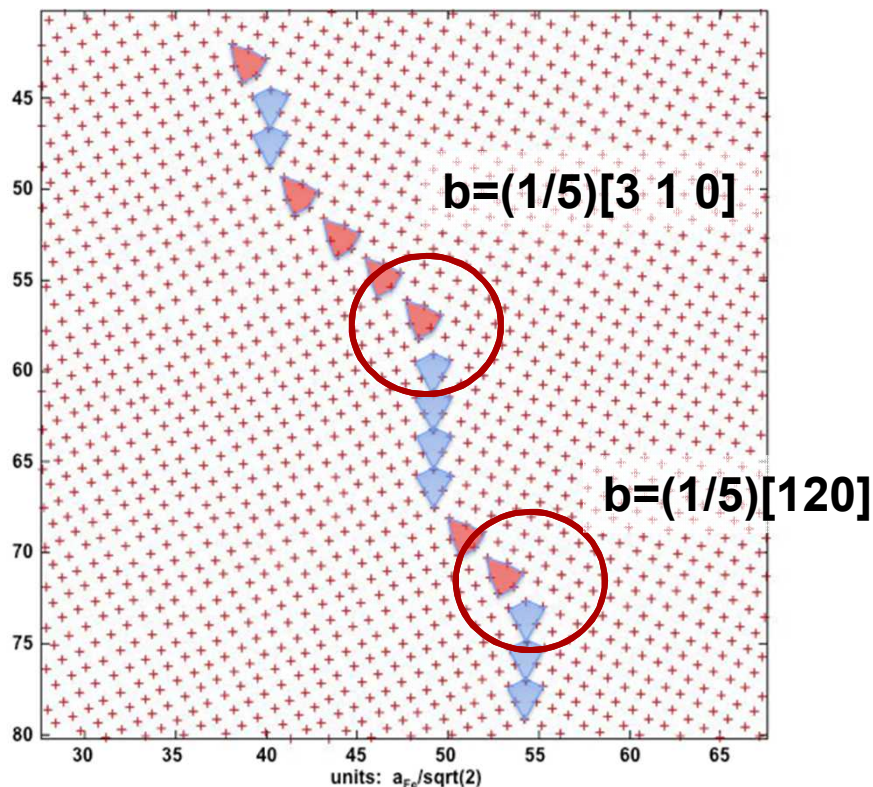
$$\mathbf{B} = [0.030 \pm 0.002, 0.027 \pm 0.002, 0]$$

(b per unit length of boundary)

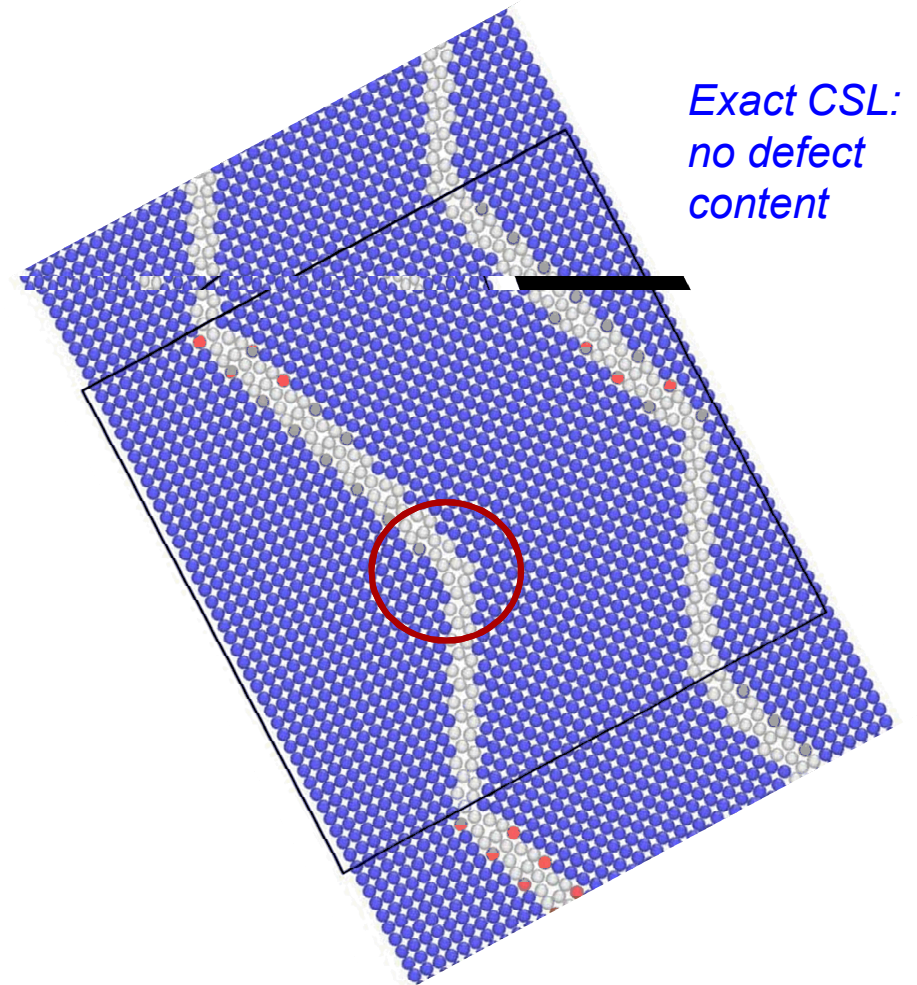


How are the grain boundary dislocations manifested in the junction structure?

Experimental Junctions $b=(1/5)(120)$ and $(1/5)(310)$

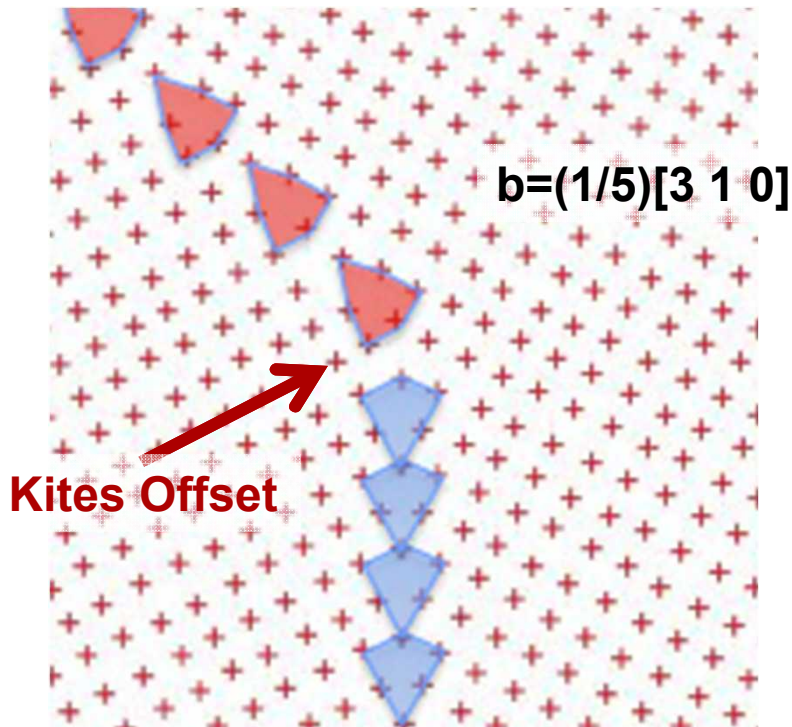


Relaxed Periodic Atomistic Structure

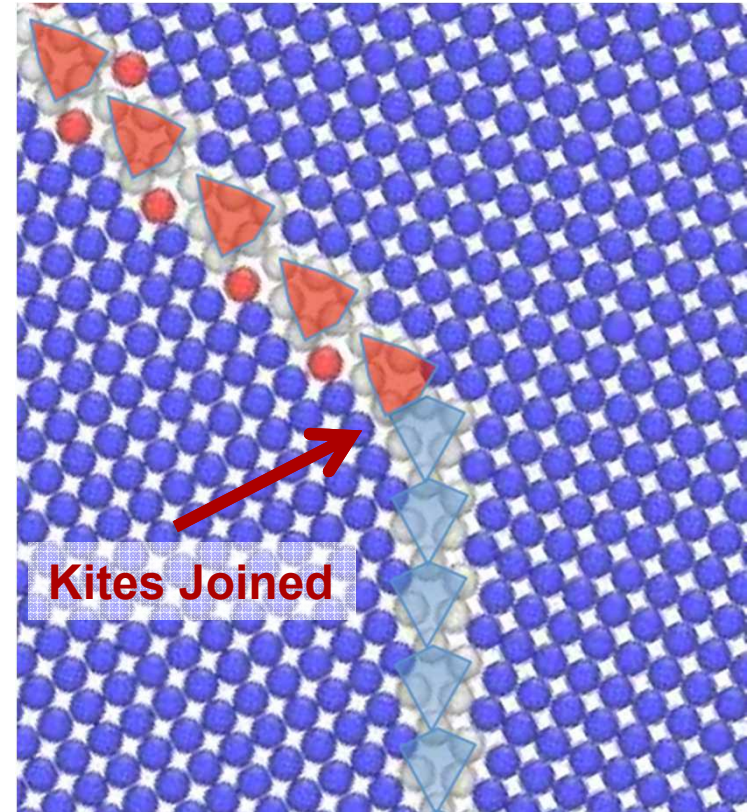


How are the grain boundary dislocations manifested in the junction structure?

Experimental Junctions
 $b=(1/5)(120)$ and $(1/5)(310)$

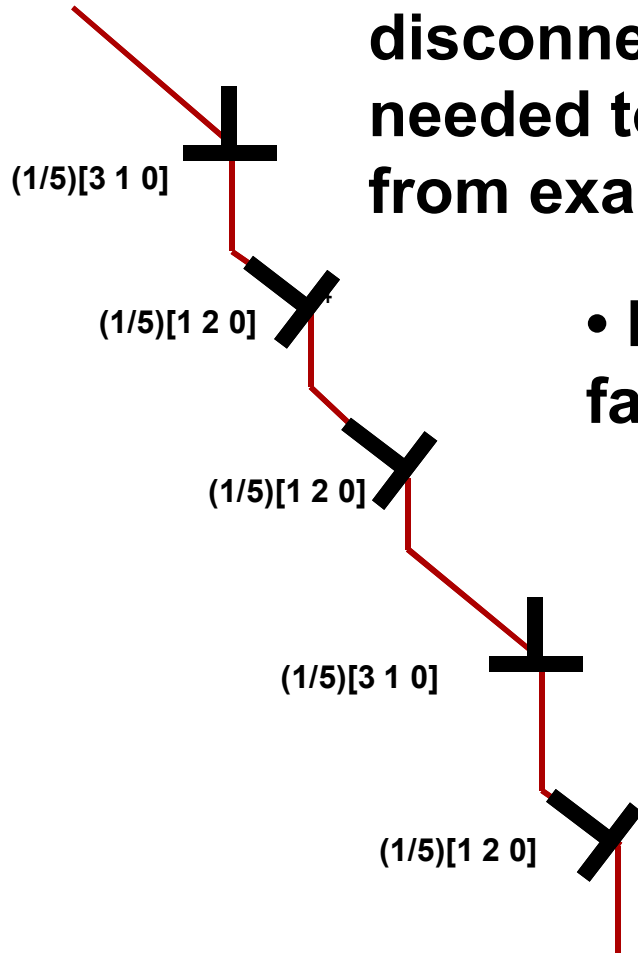


Relaxed Periodic Atomistic Structure



Impact on facet length scale

- Distribution of grain boundary disconnections consistent with that needed to accommodate misorientation from exact $\Sigma=5$ misorientation.

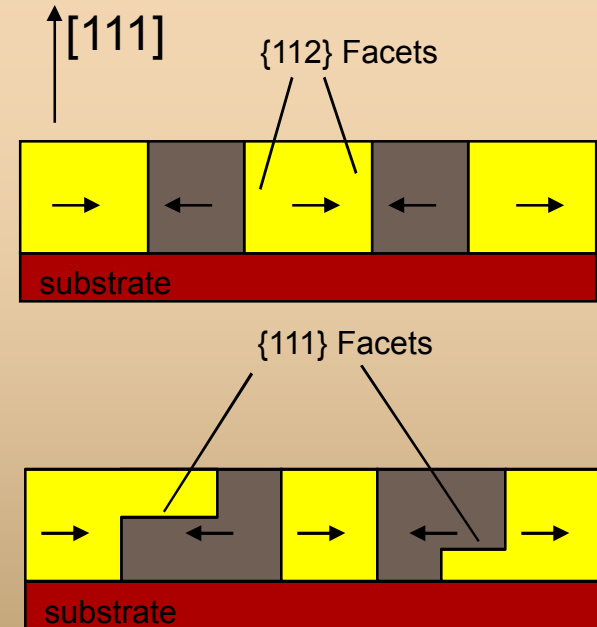
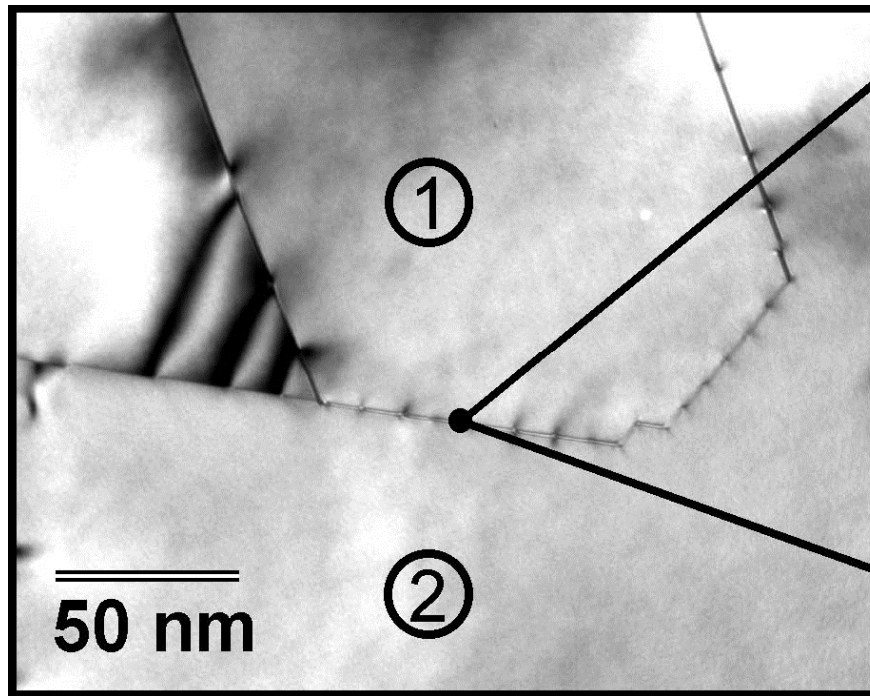


- Disconnection cores located at facet junctions.

- Suggests that facet length scale here is tied to misorientation/inclination via dislocation content.

- What are the implications for evolution of morphology?

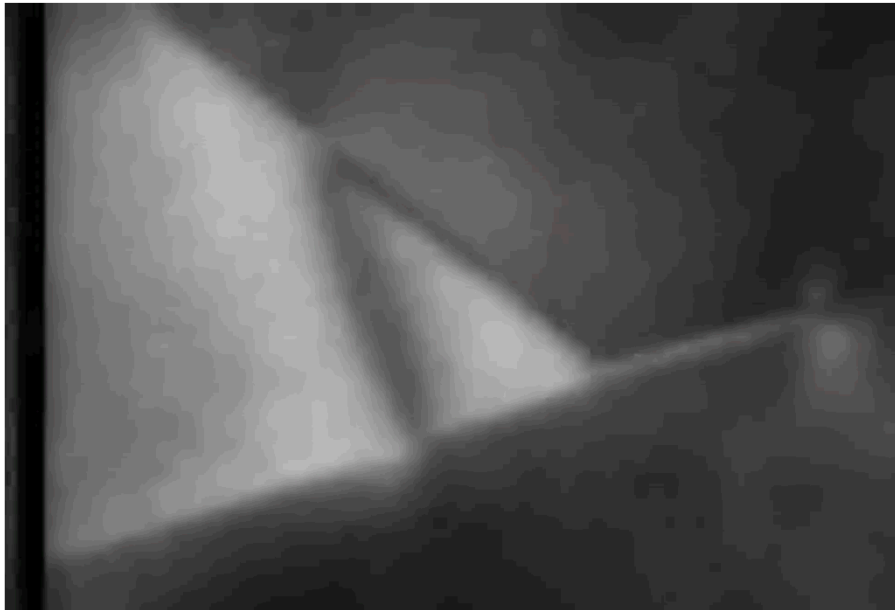
Secondary Grain Boundary Dislocations at $\Sigma=3$ boundary facets in Au



- Epitaxial film growth on Ge gives two 180° related orientation variants.
- Substrate removed to produce electron transparent membrane.

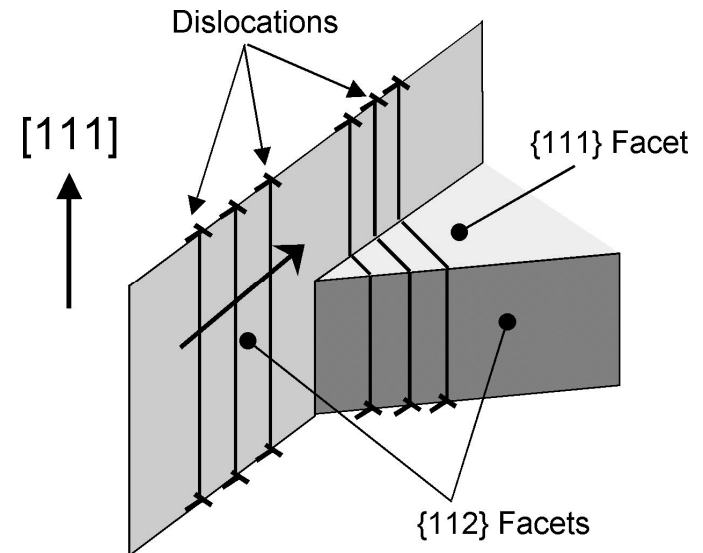
G. Lucadamo and D.L. Medlin, Acta Materialia 50 (2002) 3045

Climb of SGBDs during annealing



$T=550^{\circ}\Delta t=400$ sec
Movie is repeated

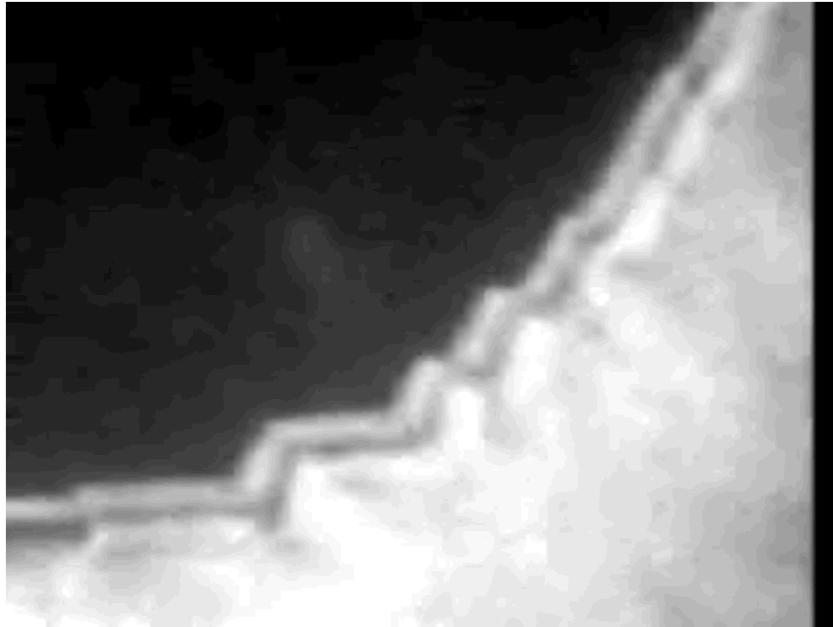
20 nm



- $(1/6)\langle 112 \rangle$ dislocations climb on $\Sigma=3$ {112} facets
 - Segments on horizontal {111} facets move by glide.
- Climb is driven by repulsive elastic interactions between the dislocations
 - Finite tilt wall, un-relaxed long-range stresses

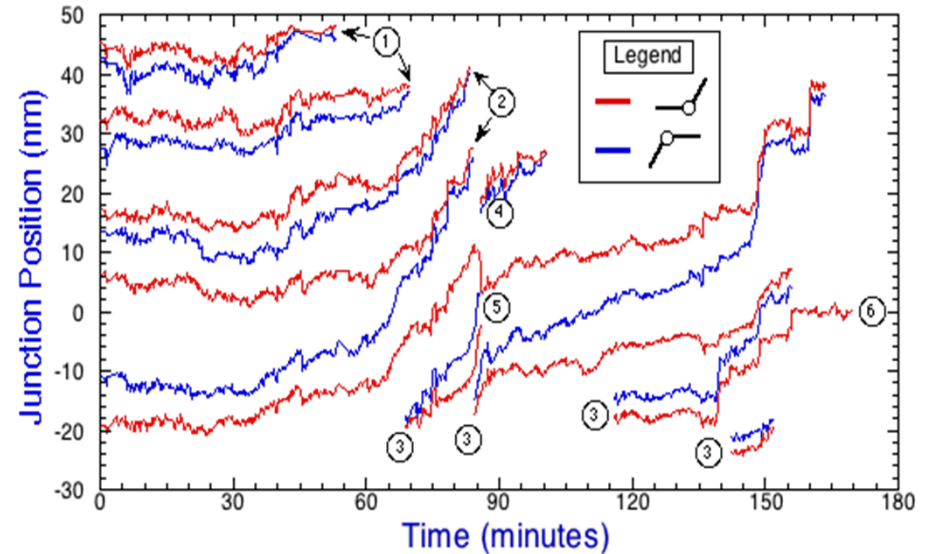
SGBD motion couples with the facets to give complex evolution dynamics

$\Sigma=3$ {112} facets in Au (Dark Field TEM)



50 nm

T=490°C
185 minutes



- Coordinated, biased motion
- Facets grow as they traverse the macroscopic corner.

D.L. Medlin and G. Lucadamo, MRS Symp 252 (2001)

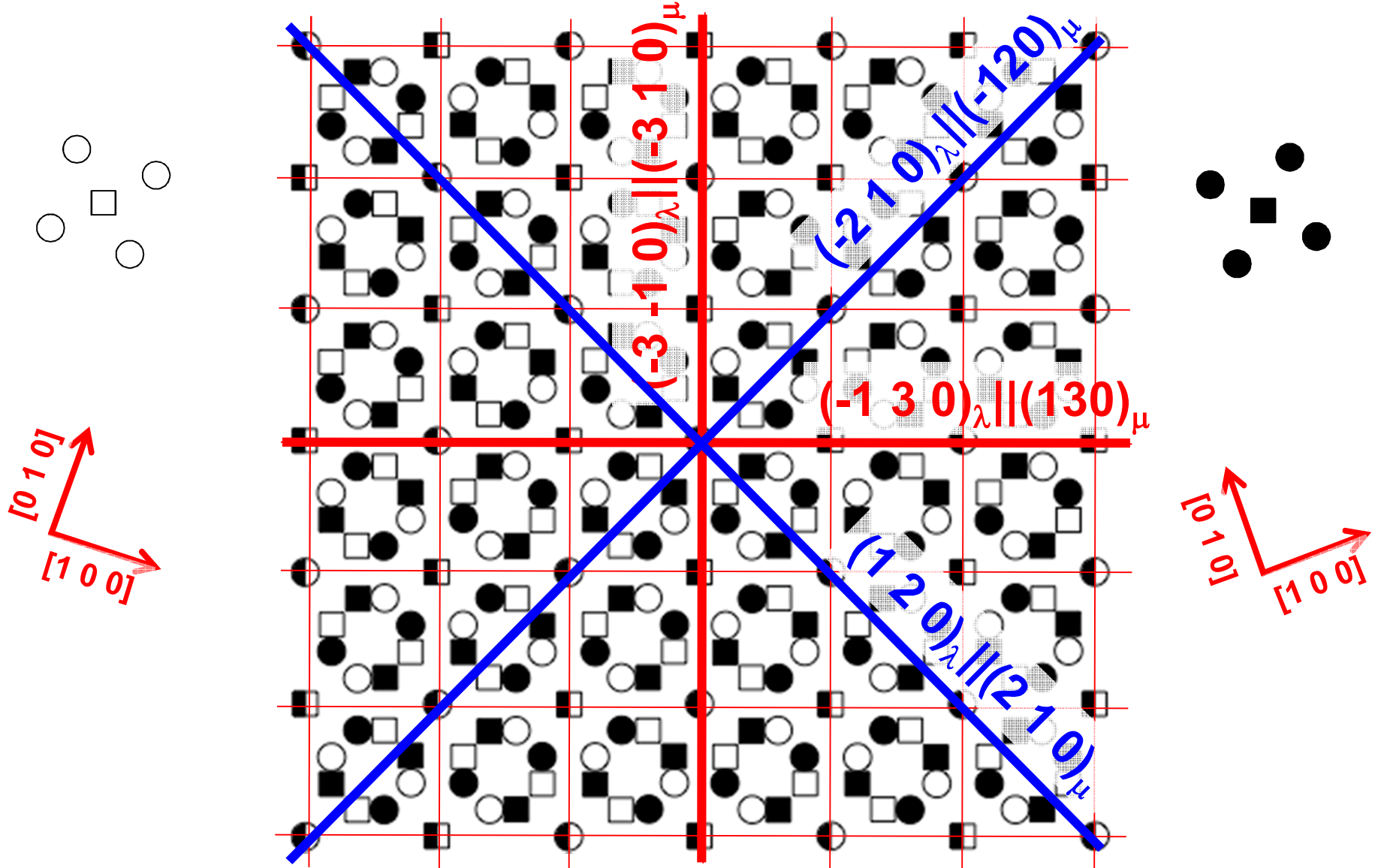
Conclusions.

- **Interactions between grain boundary dislocations and facet junctions are important to understanding grain boundary morphology and dynamics.**
 - **Examples from this presentation:**
 - Fe $\Sigma=5$ $\{210\}$ and $\{310\}$ facets
 - Au $\Sigma=3$ $\{112\}$ facets.
- **Accommodation of misorientation strains will impact faceting length scale if the necessary grain boundary dislocations are pinned to the facet junctions.**
- **Elastic interactions between the grain boundary dislocations will affect the morphological evolution in non-equilibrated configurations.**

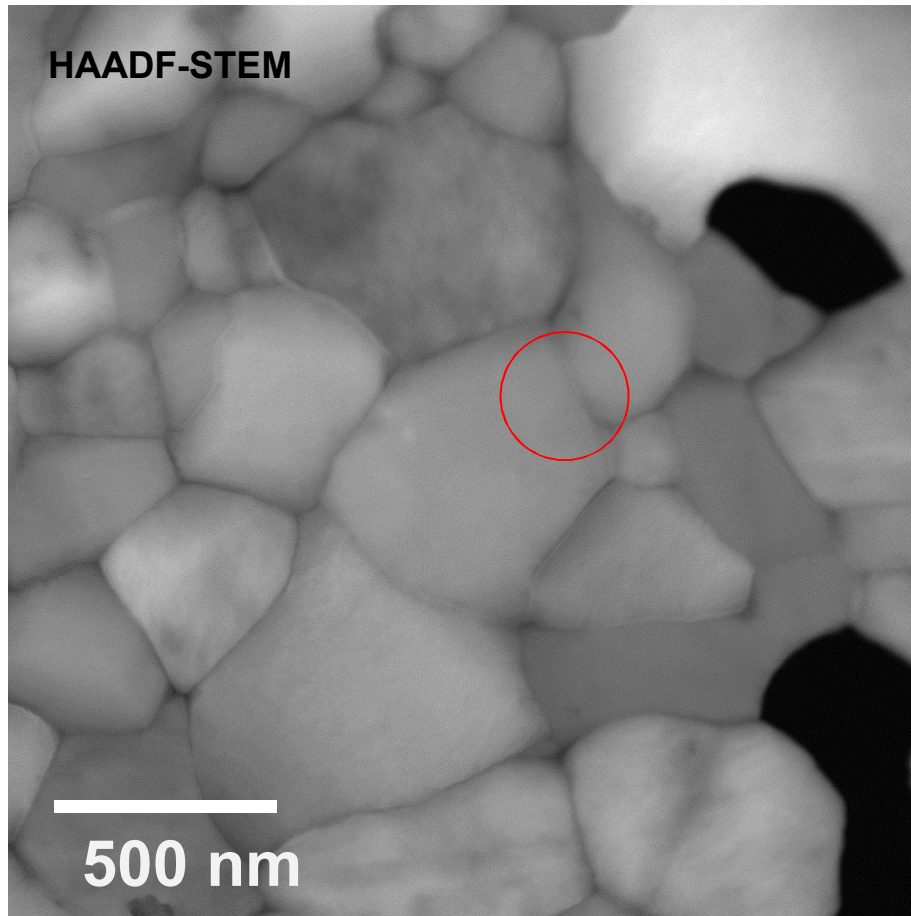
extra

BCC $\Sigma=5$ [001]: Interfacial Crystallography

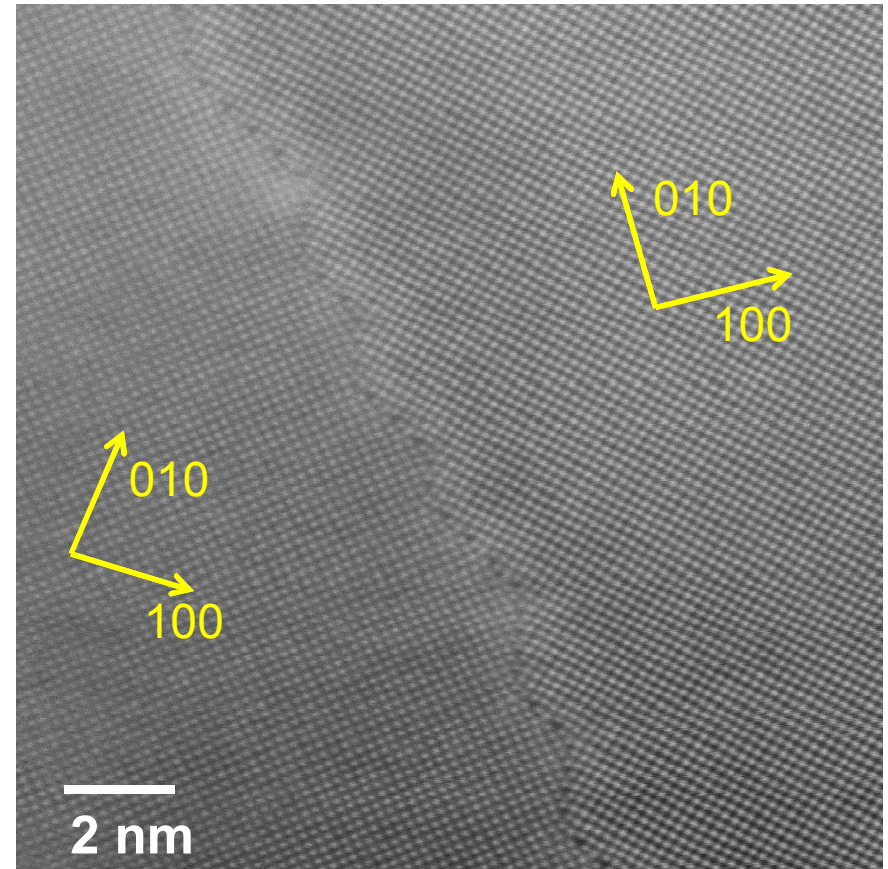
Dichromatic Pattern



Observations: Polycrystalline BCC Fe film

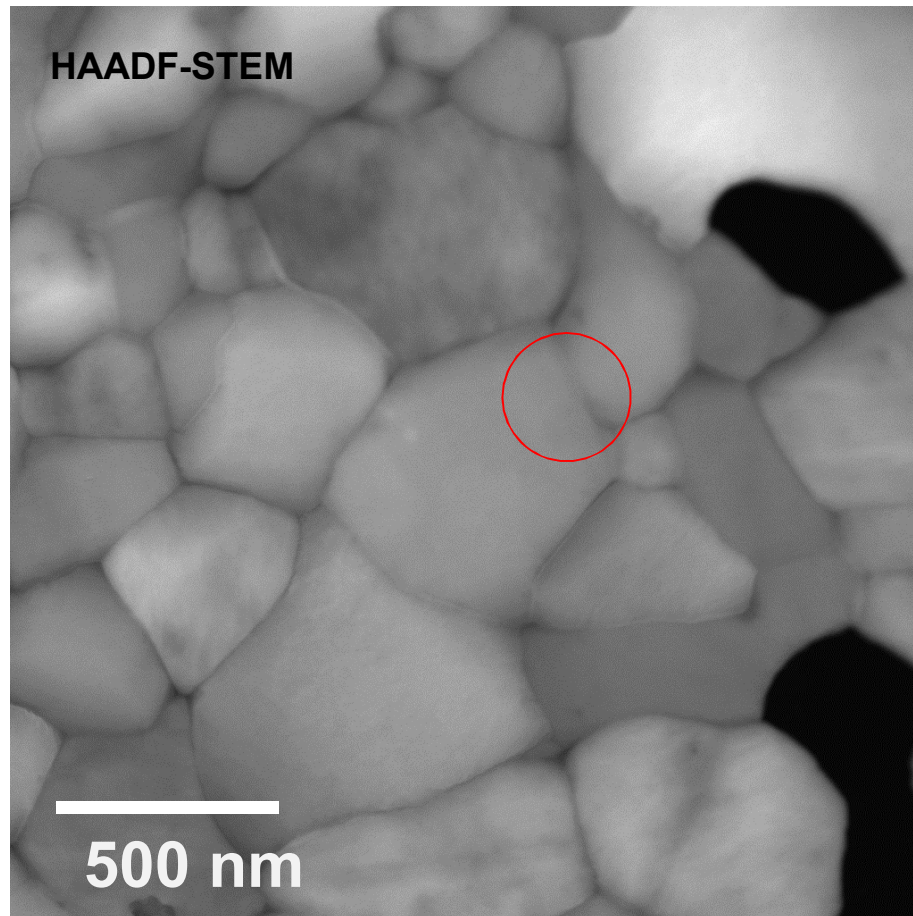


Pulsed Laser Deposited Fe on Rocksalt (NaCl). 36 nm thickness.
Specimen released and annealed on Mo grid 675°C, 2 hours.
under vacuum

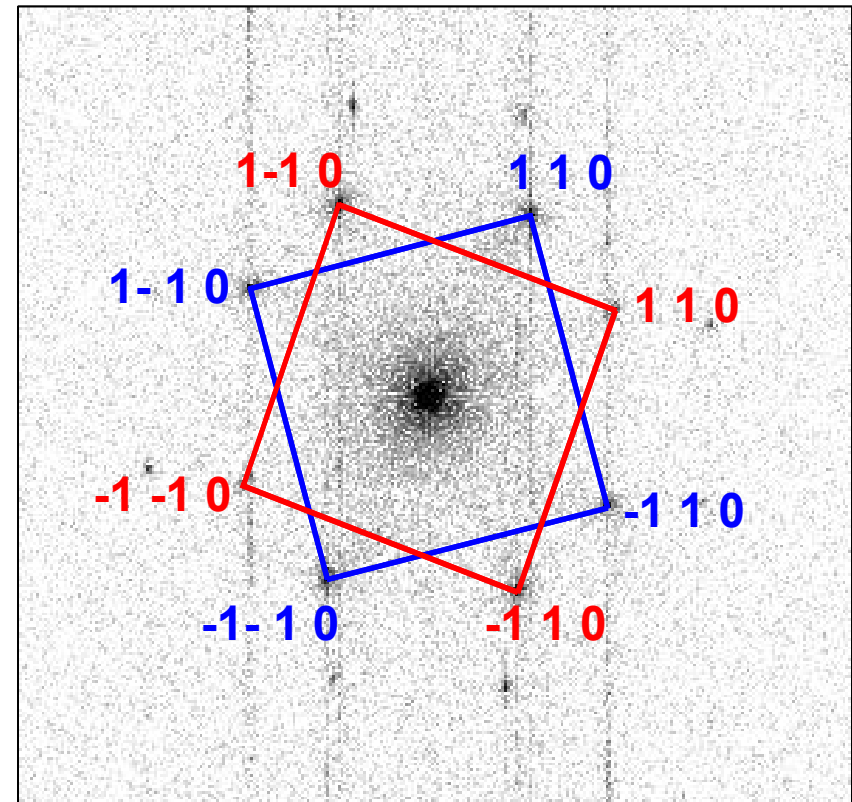


HAADF-STEM
FEI-200 keV probe corrected Titan

Observations: polycrystalline Fe thin film



Pulsed Laser Deposited Fe on Rocksalt (NaCl). 36 nm thickness.
Specimen released and annealed on Mo grid 675°C, 2 hours.
under vacuum



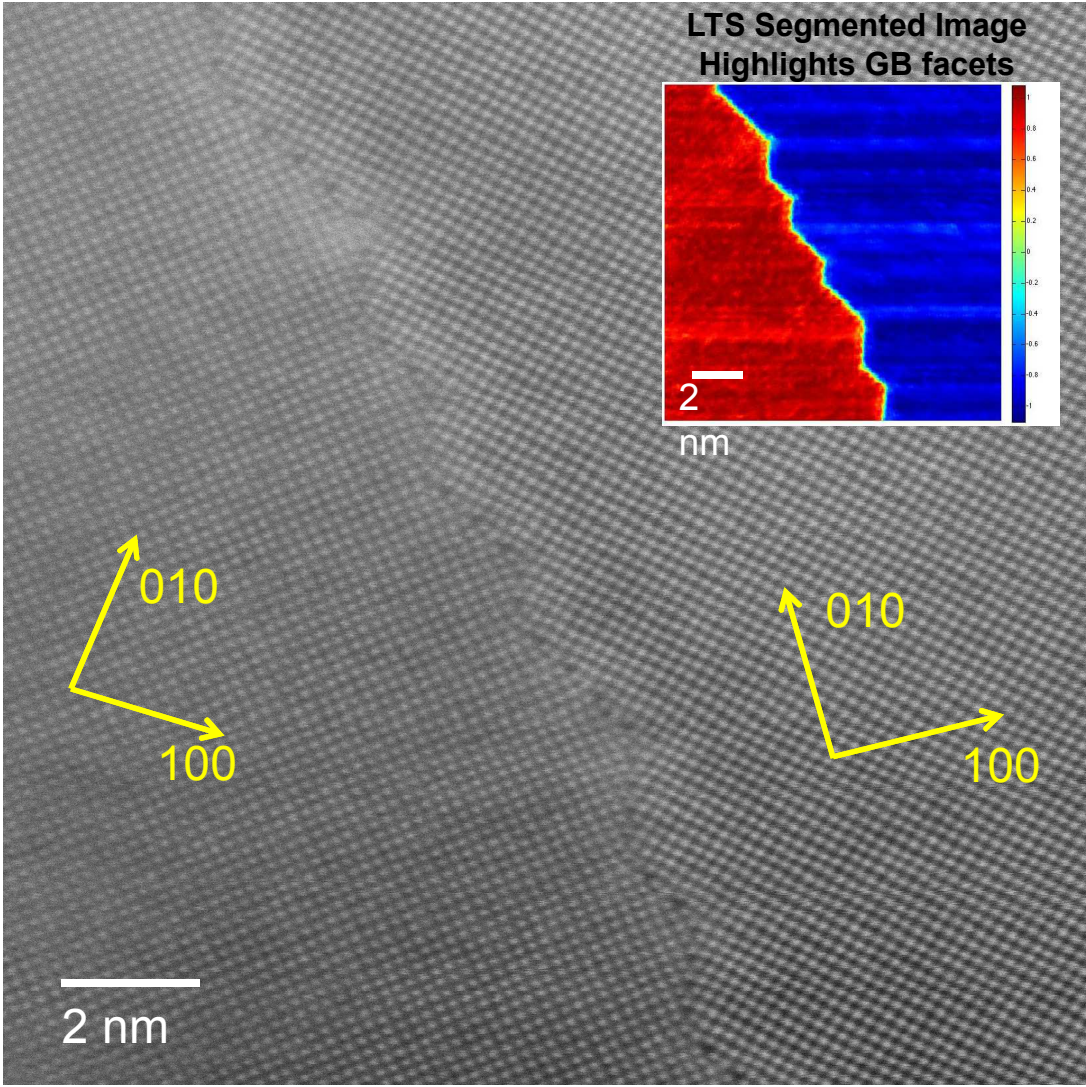
Measured misorientation: $34.49^\circ \pm 0.7^\circ$

Very close to $\Sigma=5$: $\theta_{\Sigma=5}=36.87^\circ$

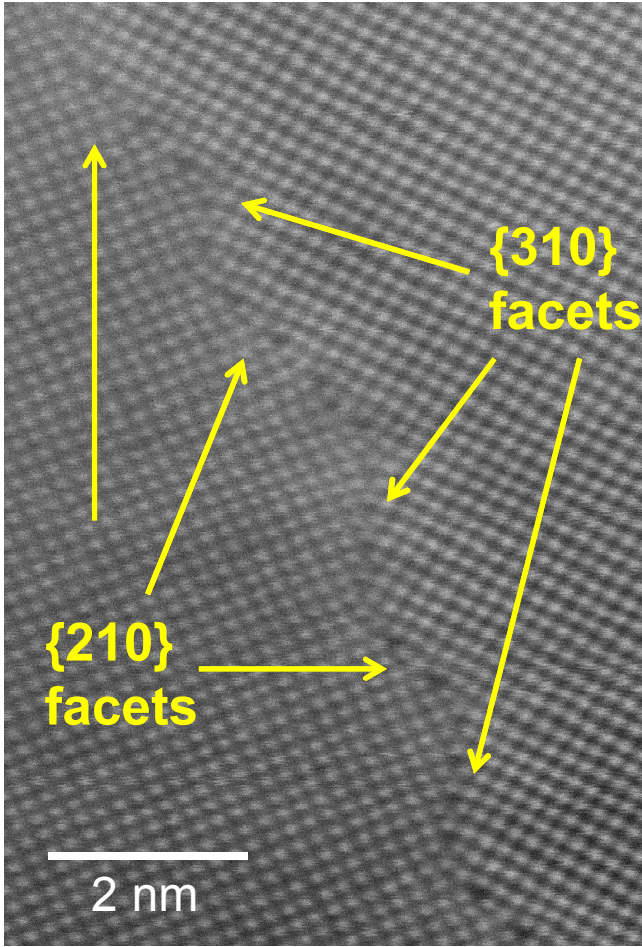
$\Delta\theta = -2.38^\circ$

HRSTEM shows nanoscale faceting at Grain boundary

HAADF-STEM $\Sigma=5$ $\langle 001 \rangle$ Boundary in Fe



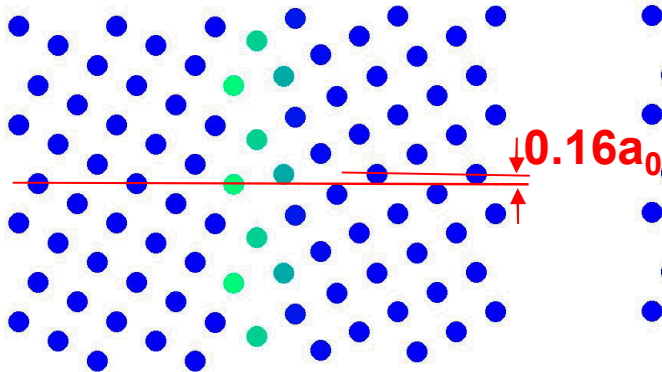
Boundary is faceted on $\{210\}$ and $\{310\}$ type inclinations



Inclination from $\{310\}$: 26.3°

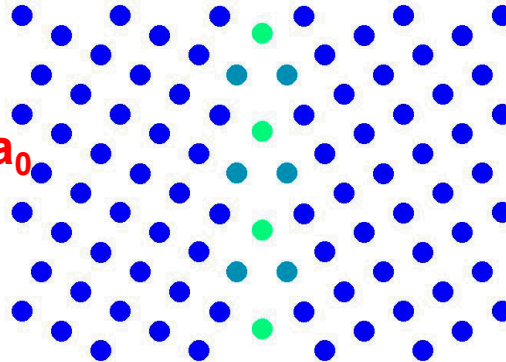
$\Sigma=5$ {310} Structures with different Potentials

Asymmetric



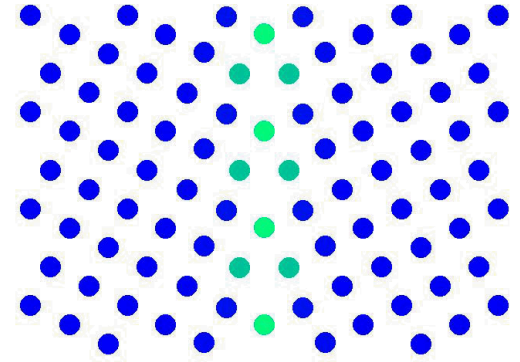
Potential: Chamati, 2006

Symmetric



Potential: Mendelev, 2003

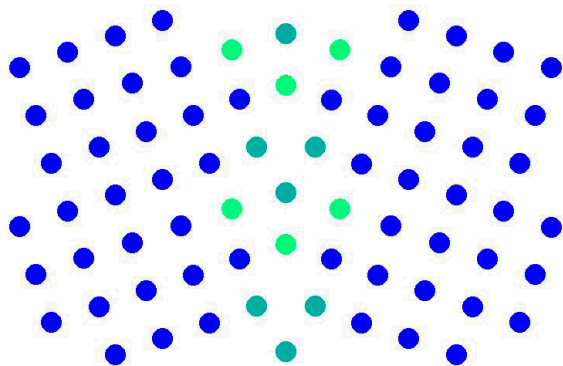
Symmetric



Potential: Proville, 2012

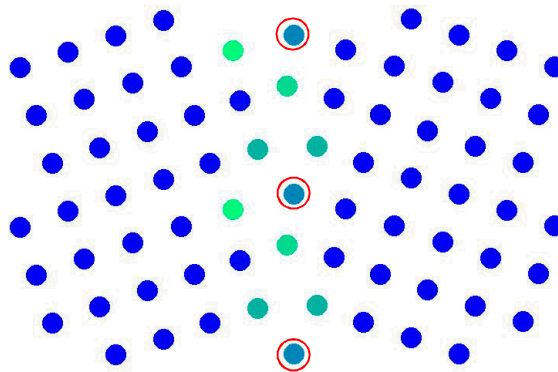
$\Sigma=5$ {210} Structures with different Potentials

Symmetric



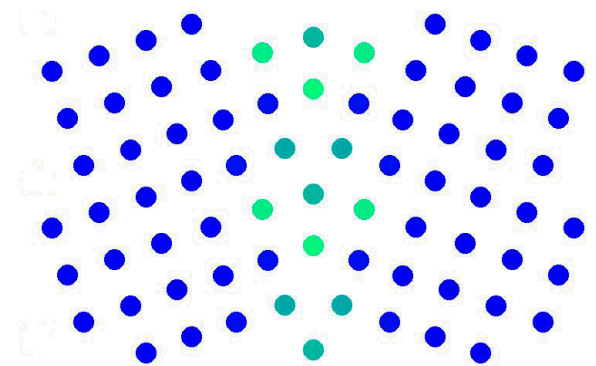
Potential: Chamati, 2006

Asymmetric



Potential: Mendelev, 2003

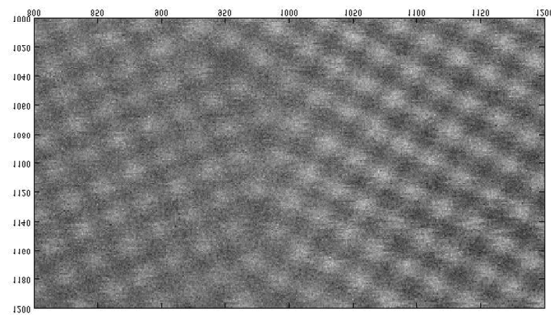
Symmetric



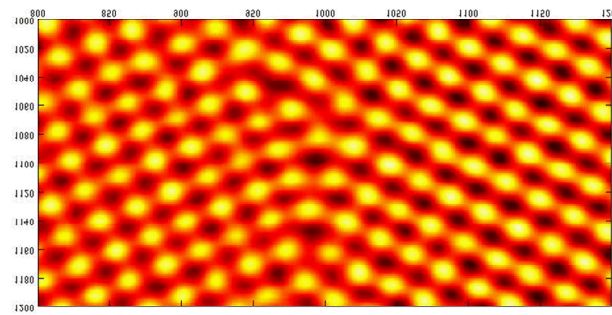
Potential: Proville, 2012

Quantifying the GB Images: Peak Location

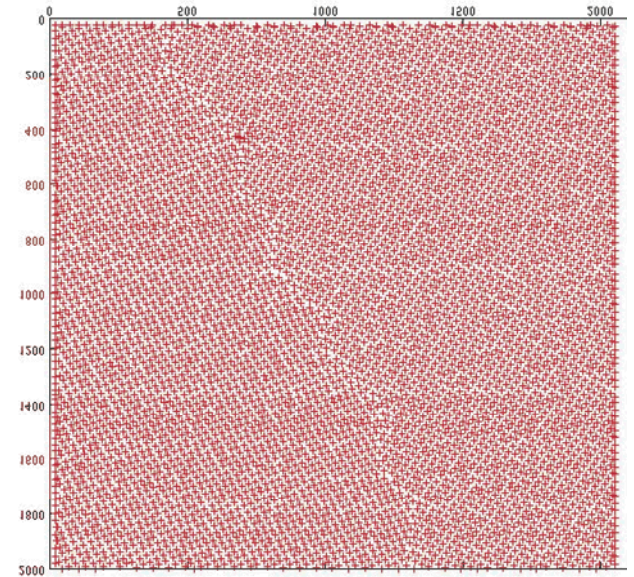
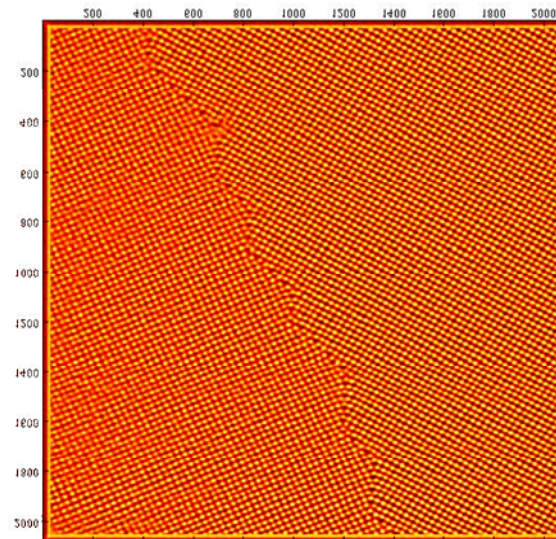
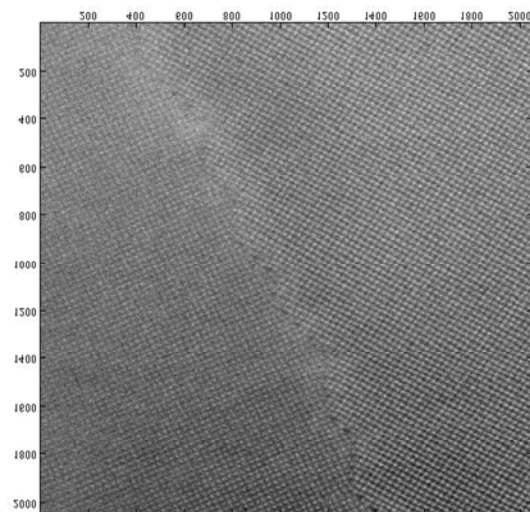
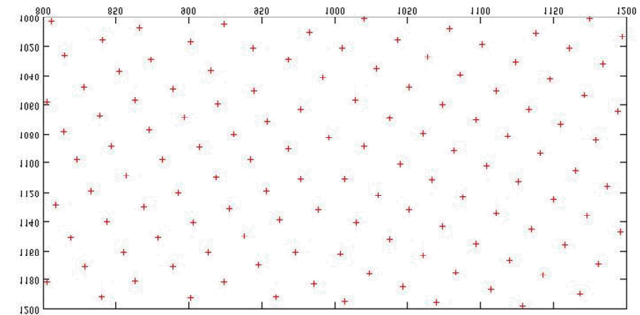
Raw HAADF STEM Image



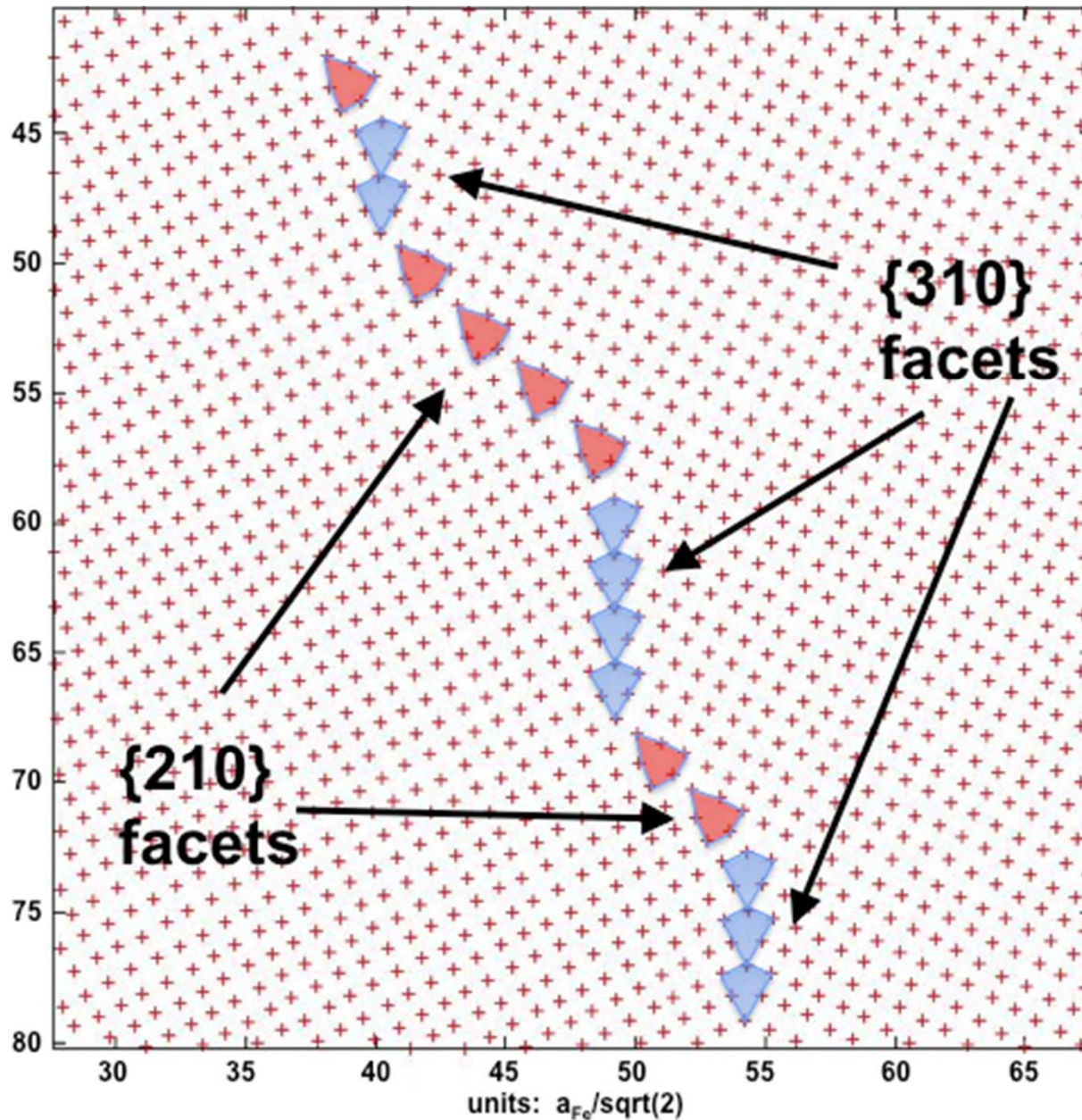
Correlation Image-Gaussian



Peak Positions



**Shear distortion due to specimen drift during image acquisition.
Corrected by affine transformation to peak position array.**

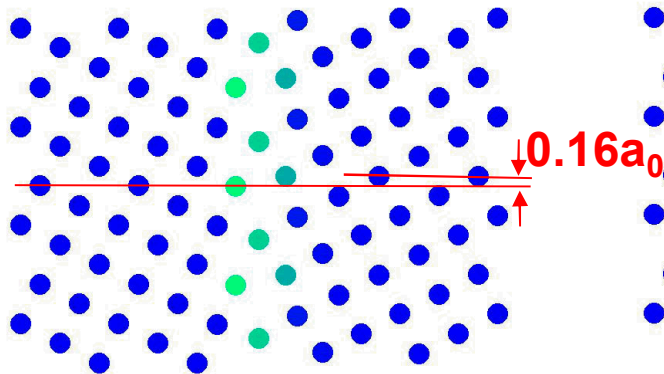


Intensity peak
positions from
HAADF-STEM
of Fe $\Sigma=5$
grain boundary

*How do the {310}
and {210}
structural units
compare with
atomistic
predictions?*

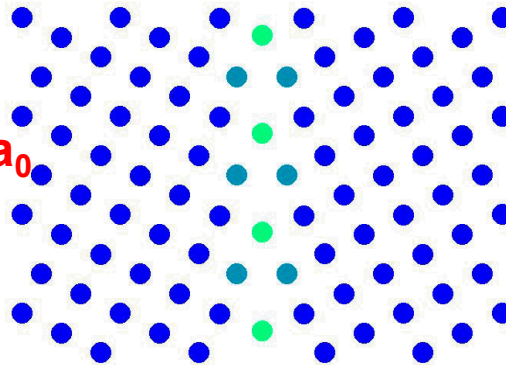
$\Sigma=5$ {310} Structures with different Potentials

Asymmetric



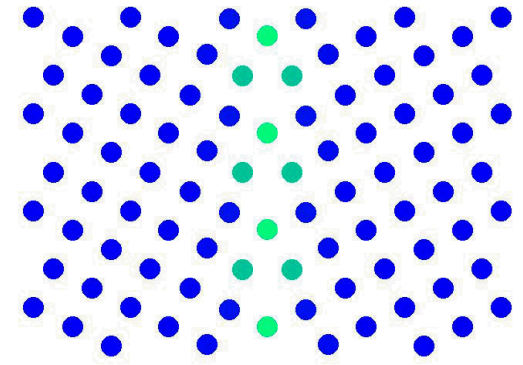
Potential: Chamati, 2006

Symmetric



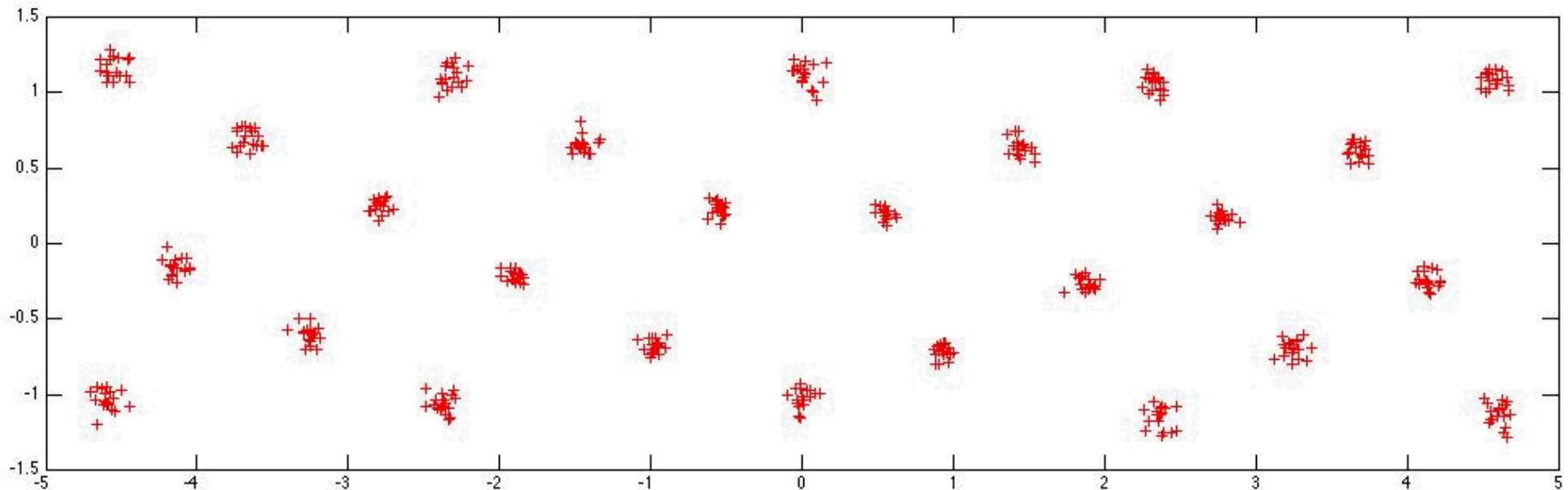
Potential: Mendelev, 2003

Symmetric



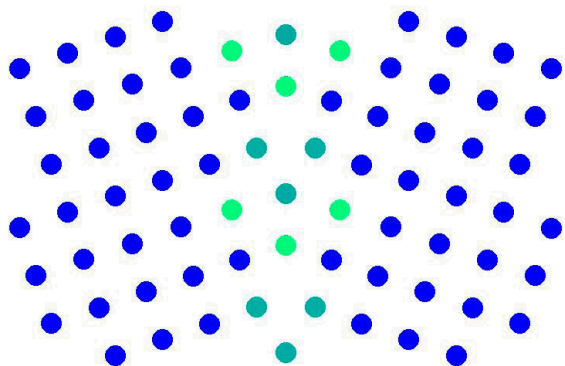
Potential: Proville, 2012

Experimental Peak Positions (HAADF STEM) $\Delta y = -0.015 \pm 0.036 a_0$



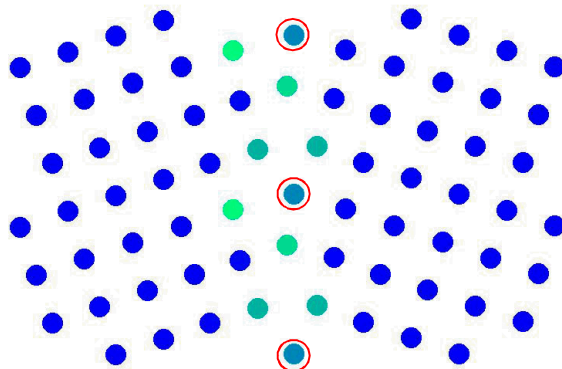
$\Sigma=5$ {210} Structures with different Potentials

Symmetric



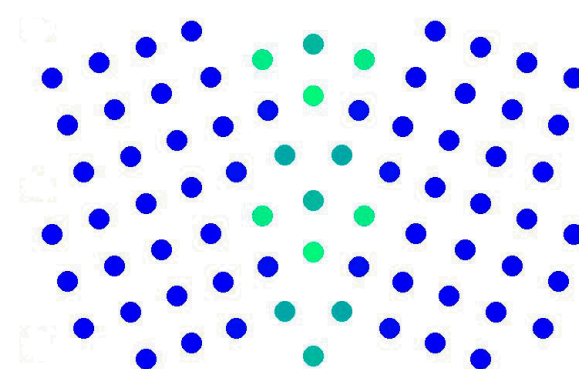
Potential: Chamati, 2006

Asymmetric



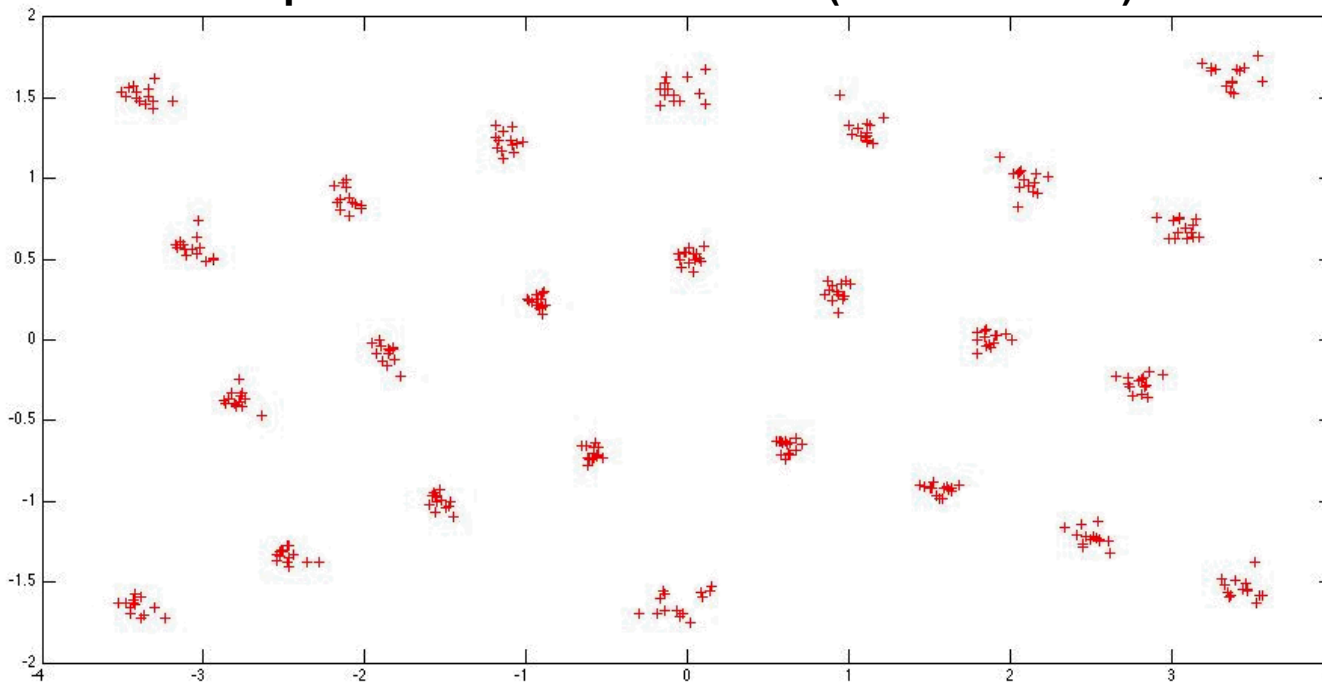
Potential: Mendelev, 2003

Symmetric



Potential: Provile, 2012

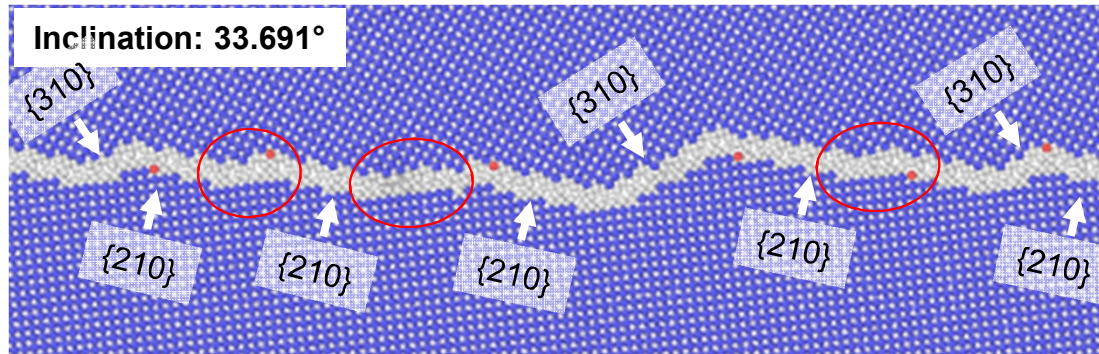
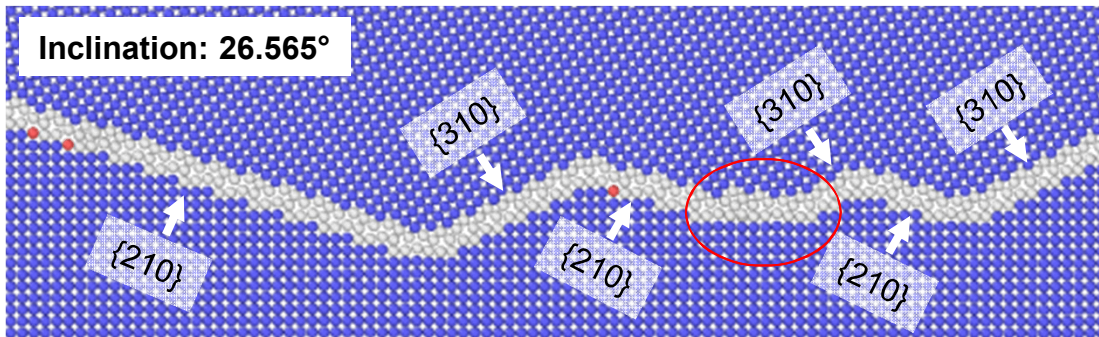
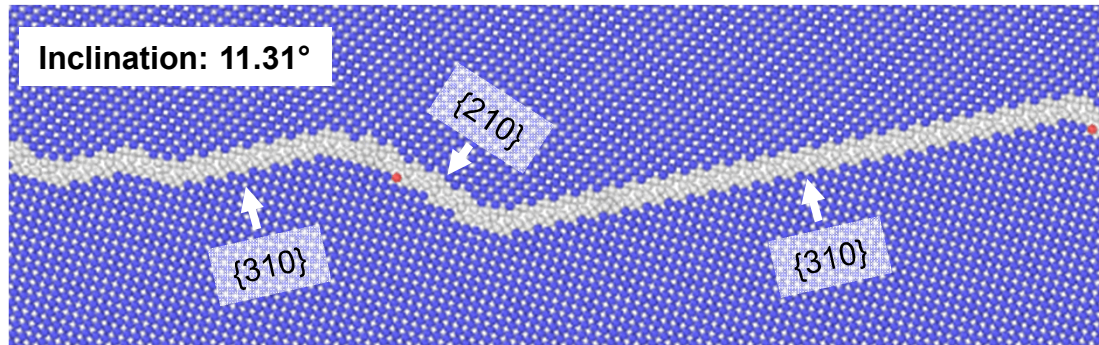
Experimental Peak Positions (HAADF STEM)



$$\Delta y = 0.035 \pm 0.015 a_0$$

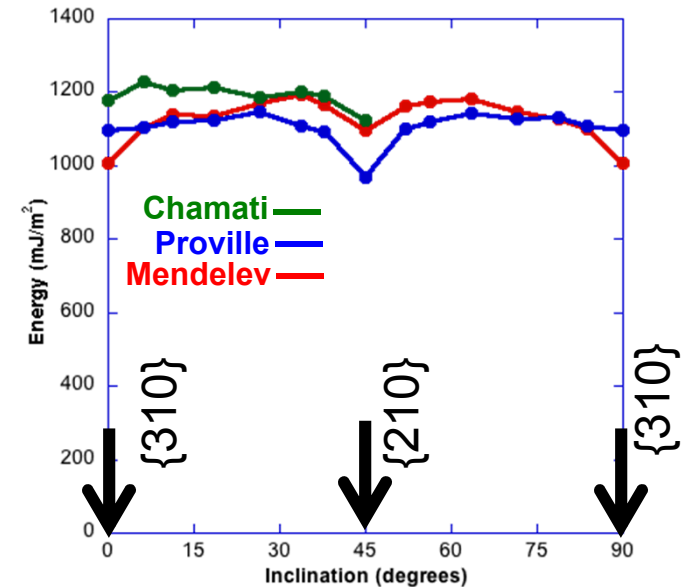
Variation in Structure and Energy with inclination:

MD shows 310 and 210 faceting



Mendelev Potential

GB Energy vs. Inclination

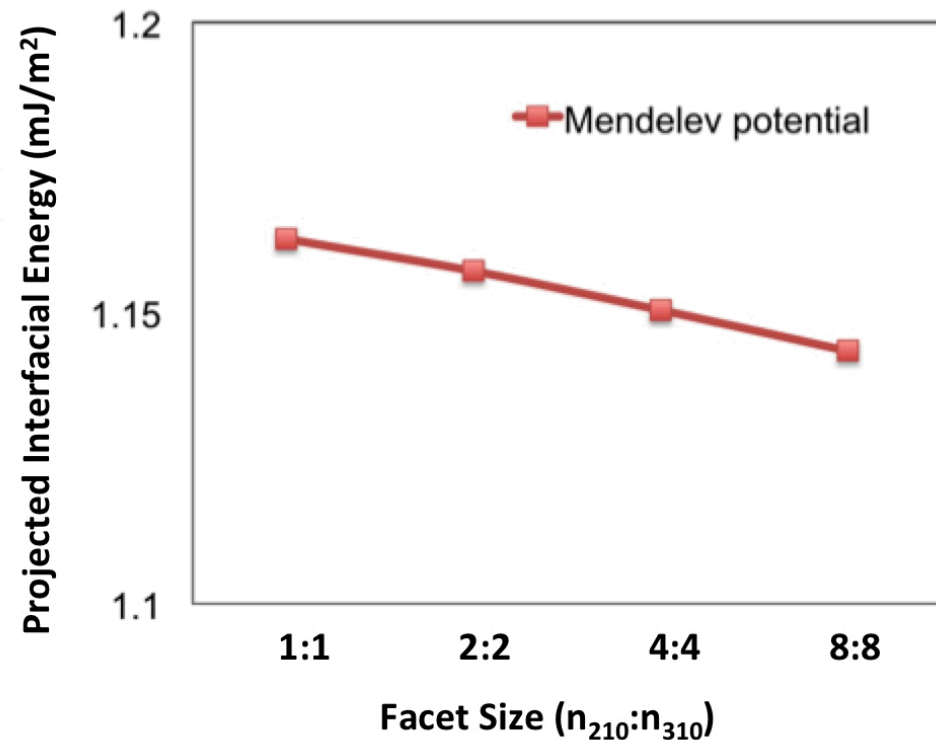
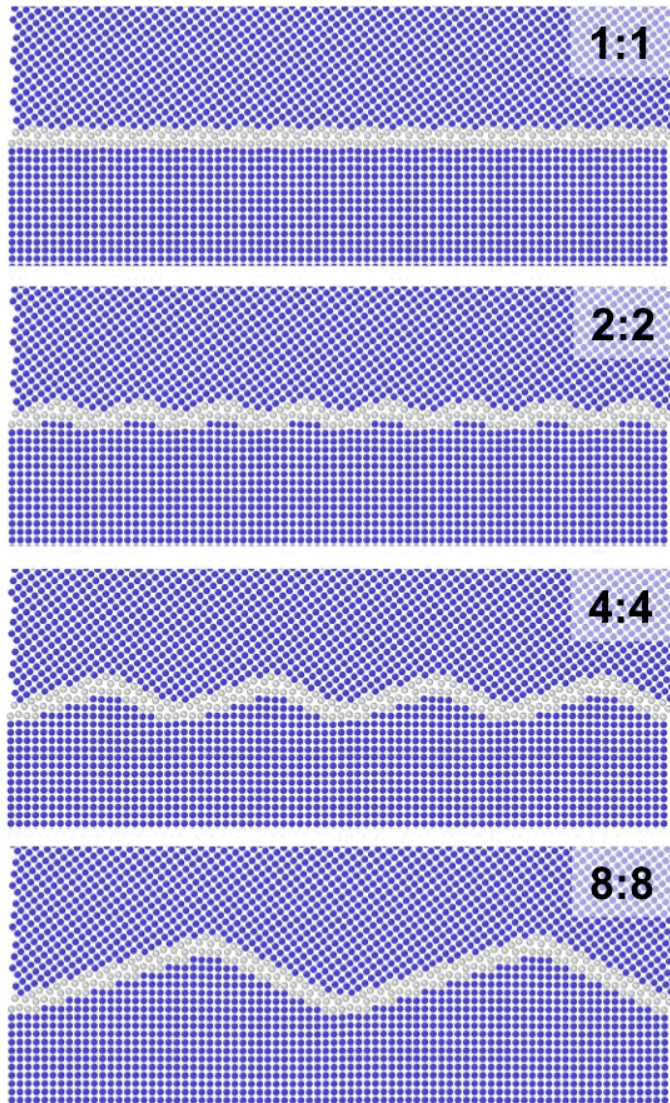


Atomistics show dissociation into coexisting {310} and {210} facets.

Additional faceting on {710}/{110} planes:

- 1:1 ratio of {310} and {210} units
- Not fully coarsened into lower energy {210}, {310} facets.

Reduction of energy with facet coarsening



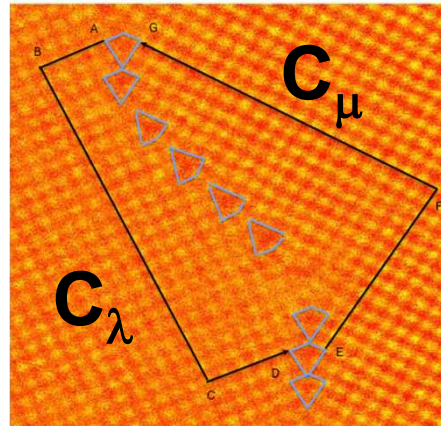
Inclination: 26.565°
 $\{710\}/\{110\}$

Are Grain Boundary Dislocations Present?

Boundary is misoriented from exact $\Sigma=5$ ($\Delta\theta=-2.38^\circ$)

Determine defect content by Circuit Mapping over all facet junctions

Two types of defect observed:

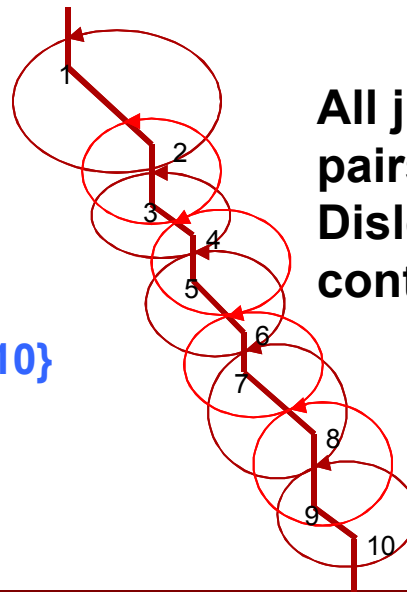


Path in μ crystal Path in λ crystal

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

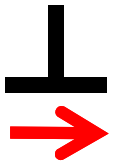
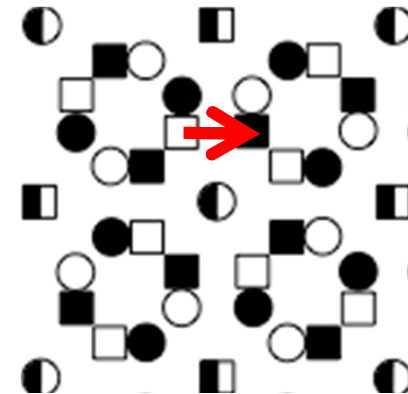
Burgers vector Re-express μ path in λ crystal coordinates.

- Circuits must cross at equivalent GB sites
- Every circuit then includes 2 junctions.
- Alternate between circuits on $\{210\}$ and $\{310\}$ inclinations

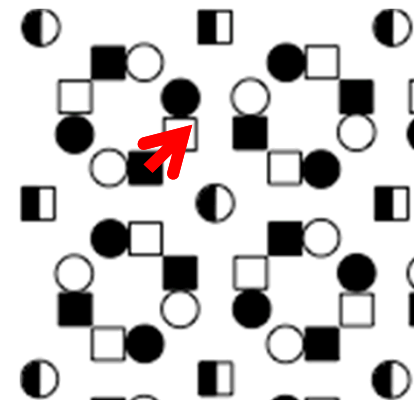


All junction pairs exhibited Dislocation content

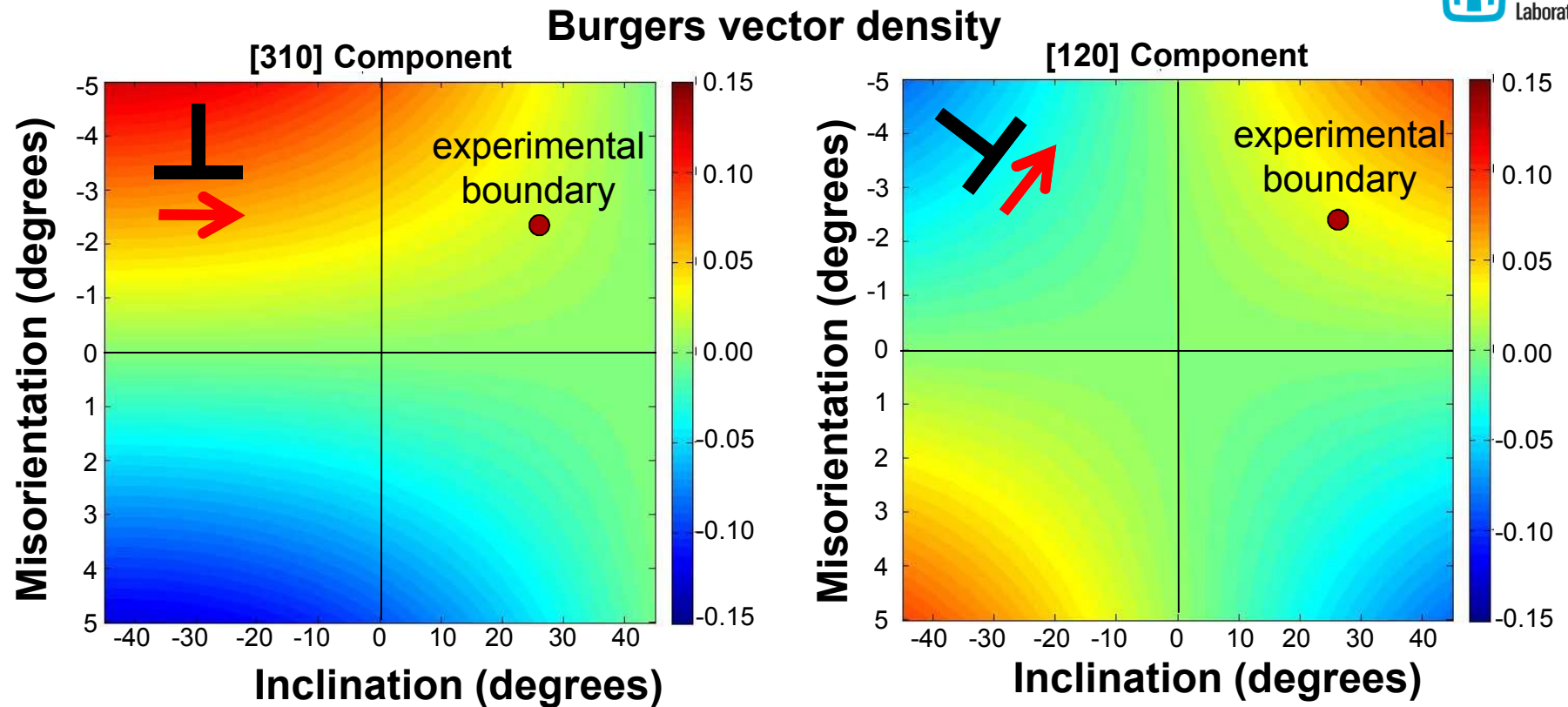
$$\mathbf{b} = (1/5)[3, 1, 0]$$



$$\mathbf{b} = (1/5)[1, 2, 0]$$



Defect content tied to misorientation and inclination



- Burgers vector density related to misorientation and inclination through Frank-Bilby Equation: $\mathbf{B} = (\mathbf{I} - \mathbf{P}^{-1}) \mathbf{v}$

Experimental

<310> component: 0.021 ± 0.002

<120> component: 0.022 ± 0.002

Frank-Bilby equation ($\theta = -2.38^\circ \pm 0.75^\circ$, $\phi = 26.3 \pm 1.0^\circ$)

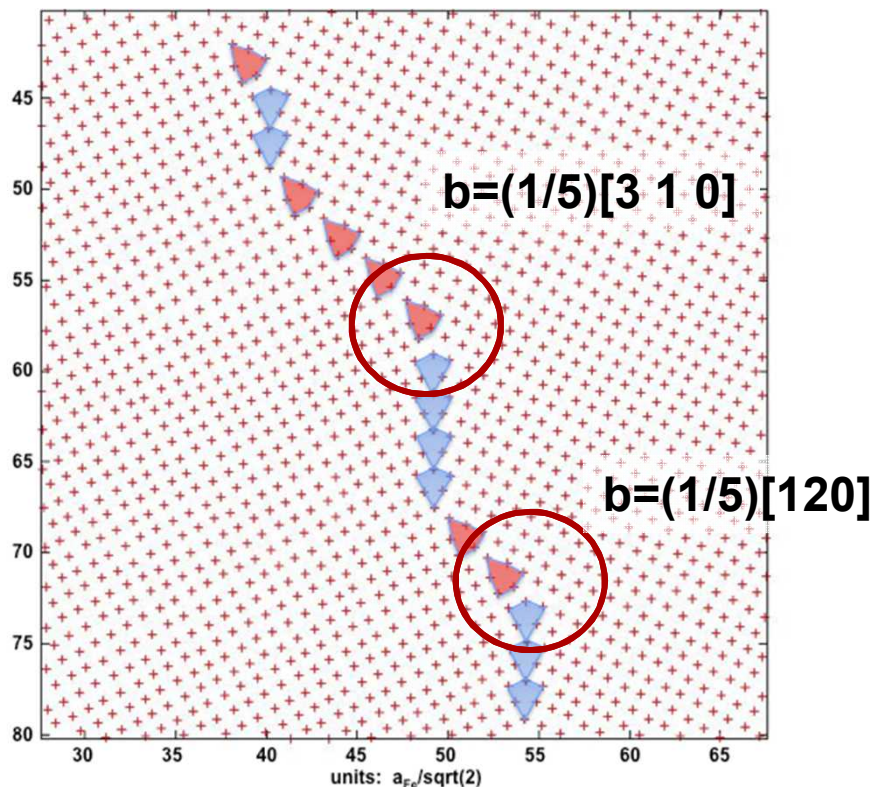
<310> component: 0.018 ± 0.006

<120> component: 0.027 ± 0.010

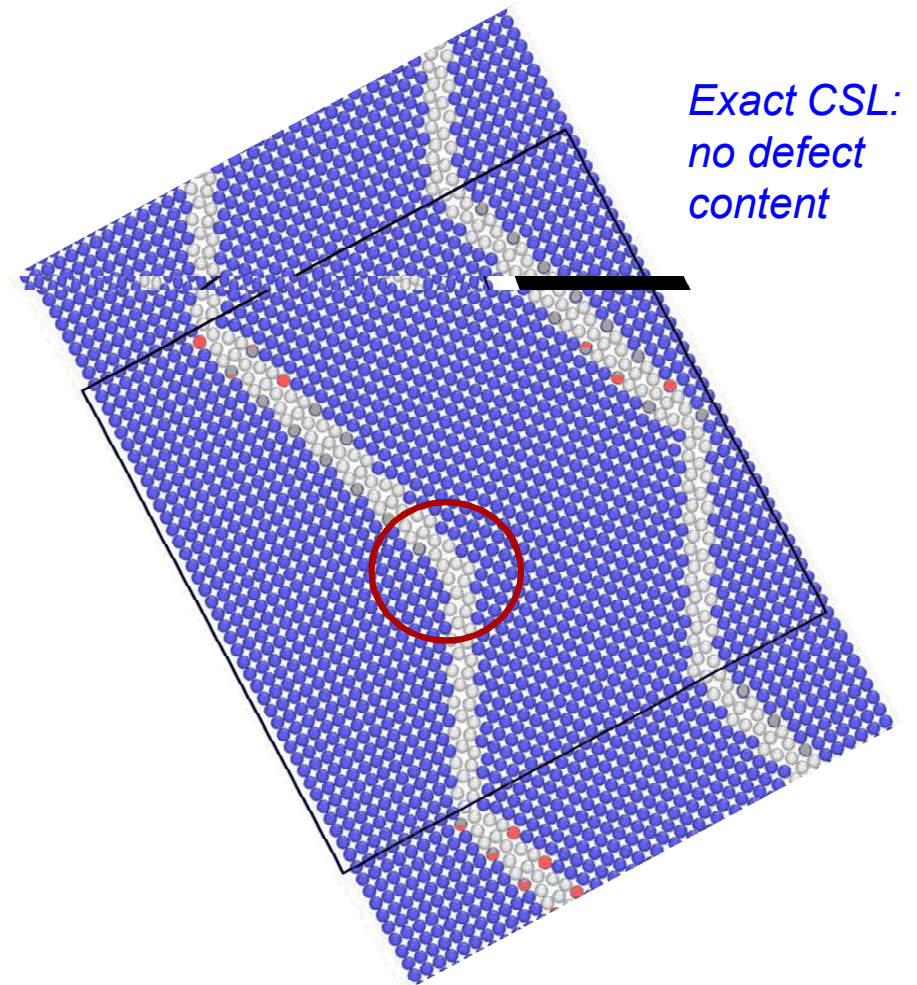
- For inclinations away from {310}, b_{120} component required to accommodate interfacial coherency strains.

How are the grain boundary dislocations manifested in the junction structure?

Experimental Junctions $b=(1/5)(120)$ and $(1/5)(310)$



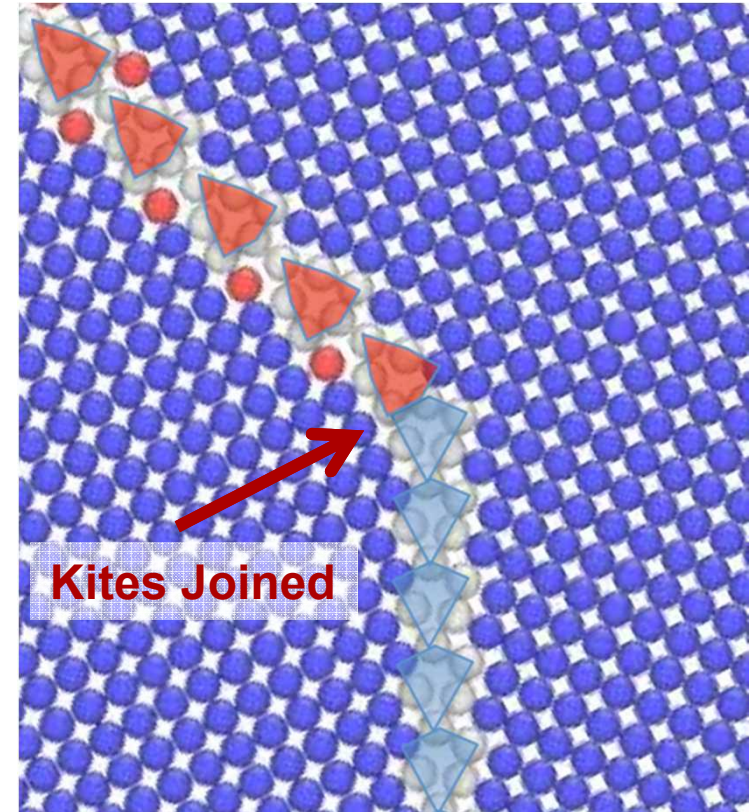
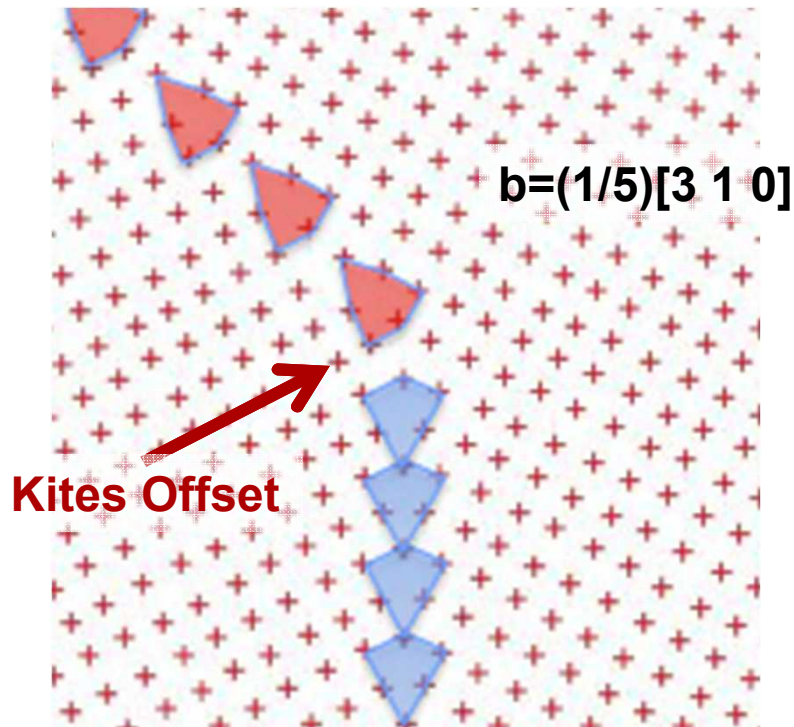
Relaxed Periodic Atomistic Structure



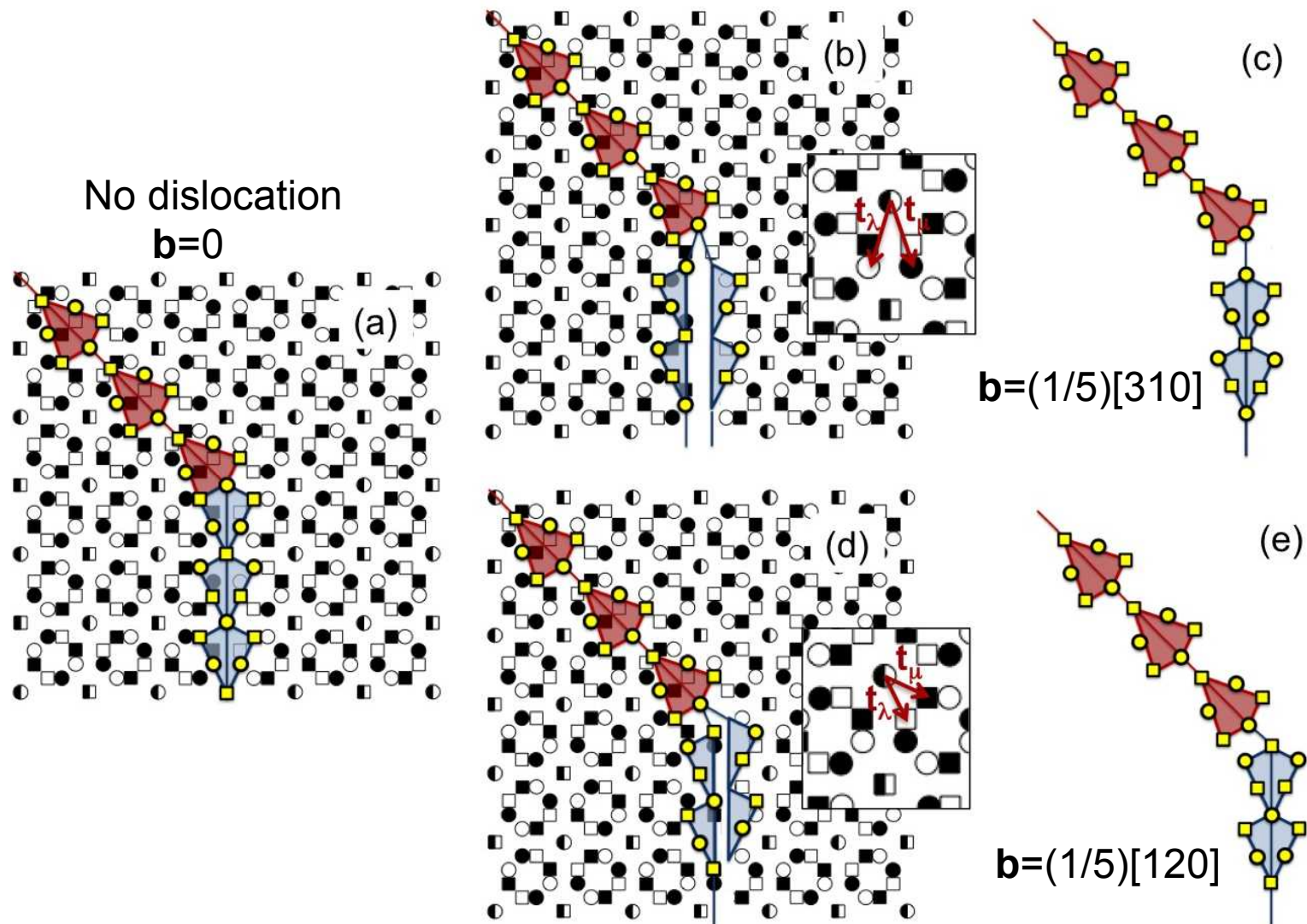
How are the grain boundary dislocations manifested in the junction structure?

Experimental Junctions
 $b=(1/5)(120)$ and $(1/5)(310)$

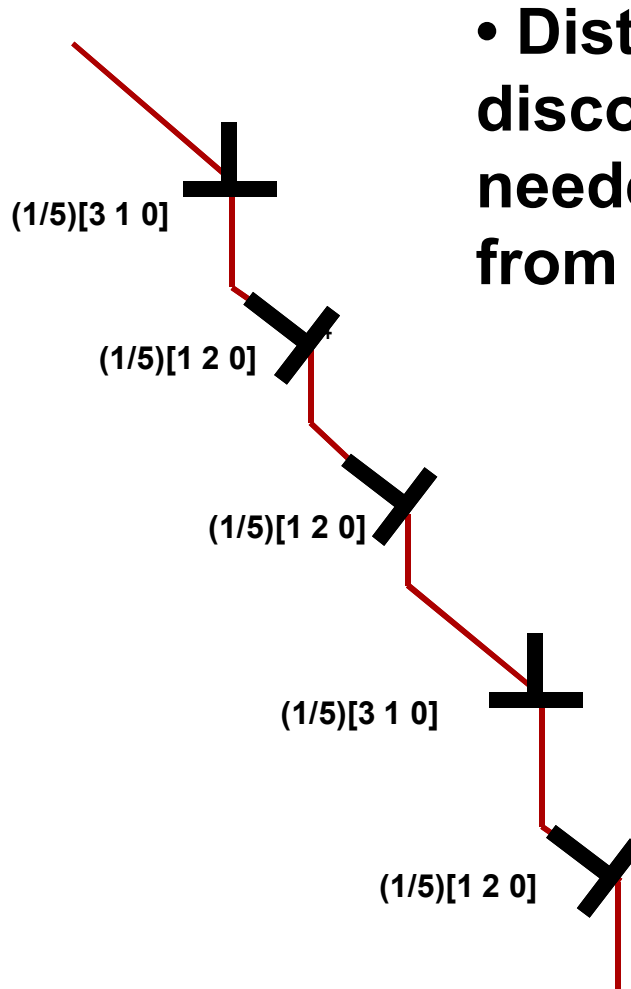
Relaxed Periodic Atomistic Structure



Geometric construction links junction core structure to defect content



Impact on facet length scale

- 
- Distribution of grain boundary disconnections consistent with that needed to accommodate misorientation from exact $\Sigma=5$ misorientation.
 - Disconnection cores located at facet junctions.
 - Suggests that facet length scale here is tied to misorientation/inclination via dislocation content.

Conclusions.

- HRSTEM observations of a $\Sigma=5$ $\langle 001 \rangle$ Boundary in Fe shows nanoscale faceting
 - Facets are on $\{310\}$ and $\{210\}$ planes, which correspond to the mirror symmetry planes for the $\Sigma=5$ dichromatic pattern.
- The atomic structures observed along the $\{310\}$ and $\{210\}$ facets are consistent with predictions of atomistic calculations.
- Circuit analysis shows presence of grain boundary dislocations at all facet junction pairs.
 - two types of defect observed:
 $b=(1/5)(3,1,0)$ and $b=(1/5)(1,2,0)$.
 - Defect density accommodates misorientation/inclination.
- Localization of defects to facet junctions suggests mechanistic interplay between misorientation and facet length scale

EXTRA

Local Translational Symmetry Segmentation Algorithm:

Approach:

(1) At each pixel, compute cross-correlation of local image region with its immediate surroundings (within ~ 2 nearest neighbor distance)

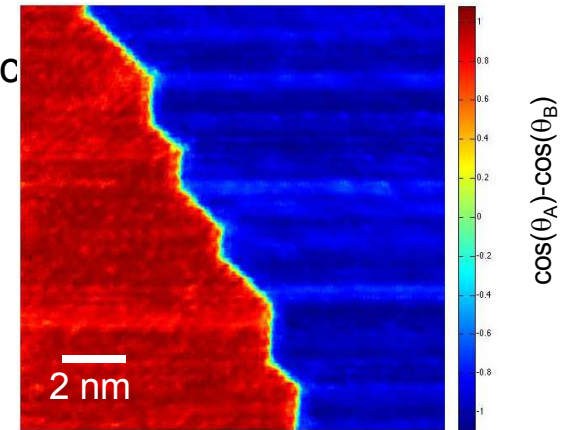
Why? This provides a measure of the local translational symmetry and orientation that is invariant at all points within an undistorted crystal (i.e., constant regardless of whether pixel is on or off an atomic column)

(2) Compare the cross-correlation image with templates obtained from cross-correlation images averaged over region of bulk crystal on either side of interface.

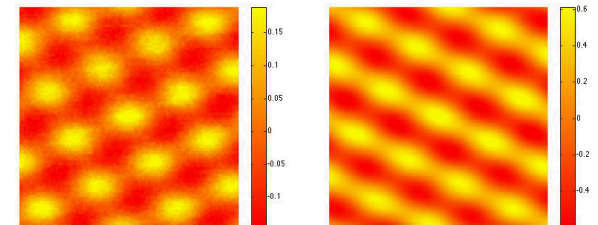
(3) Determine similarity, pixel-by-pixel, by computing angle, θ , between n-dimensional vectors for the reference correlation image templates and the correlation image of the raw image.

$$\cos(\theta_A) = \frac{P \bullet P_A}{\|P\| \|P_A\|} \quad \cos(\theta_B) = \frac{P \bullet P_B}{\|P\| \|P_B\|}$$

Facets at $\Sigma=5$ Fe GB



Reference
Cross-Correlation Templates



-cross-correlate central 32x32 pixel region with outer 128x128 region
-average over reference regions in left and right grains.

Faceting: Signature of anisotropic interfacial energy

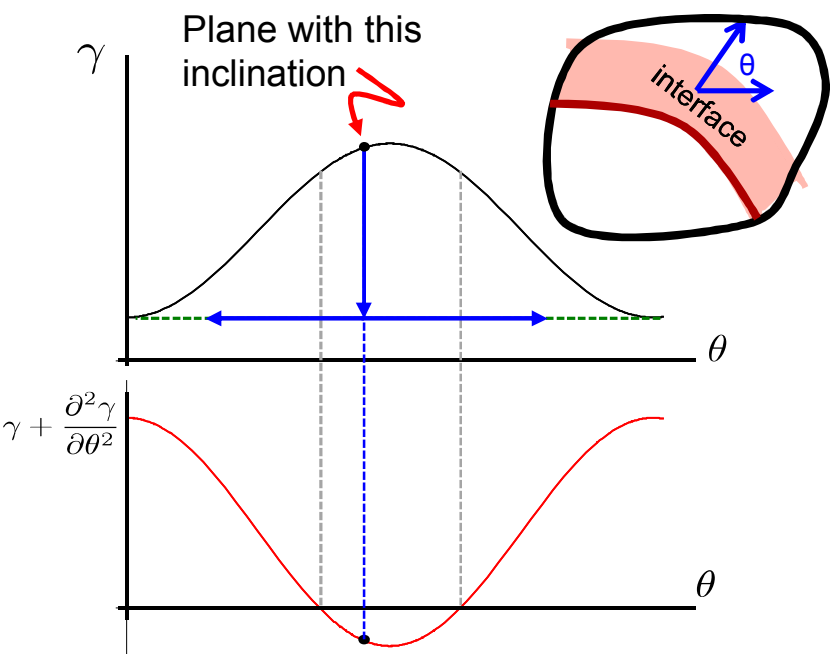
Driving force (μ) for interface evolution:

H: mean curvature
 V_m : molar volume
 γ : interface energy

$$\mu \sim v_m \left(\underbrace{\gamma + \frac{\partial^2 \gamma}{\partial \theta^2}}_{\text{Interface stiffness}} \right) H$$

W. W. Mullins (1963)

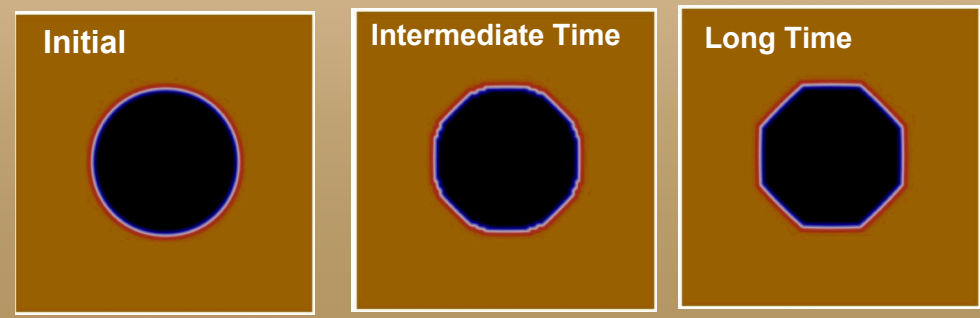
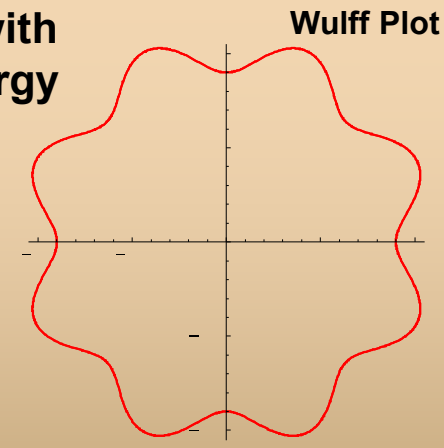
- Inclinations with negative interface stiffness break into facets with minimum energy orientations.
- "interface spinodals": analogous to phase separation in bulk materials.



Frank (1963), Cabrera (1964),
Stewart (1992), Liu (1993)

Example: GB Evolution with anisotropic interfacial energy

- Wulff surface with minima every 45°
- Phase-field simulation.



Faceting: Signature of anisotropic interfacial energy

Driving force (μ) for interface evolution:

H: mean curvature
 V_m : molar volume
 γ : interface energy

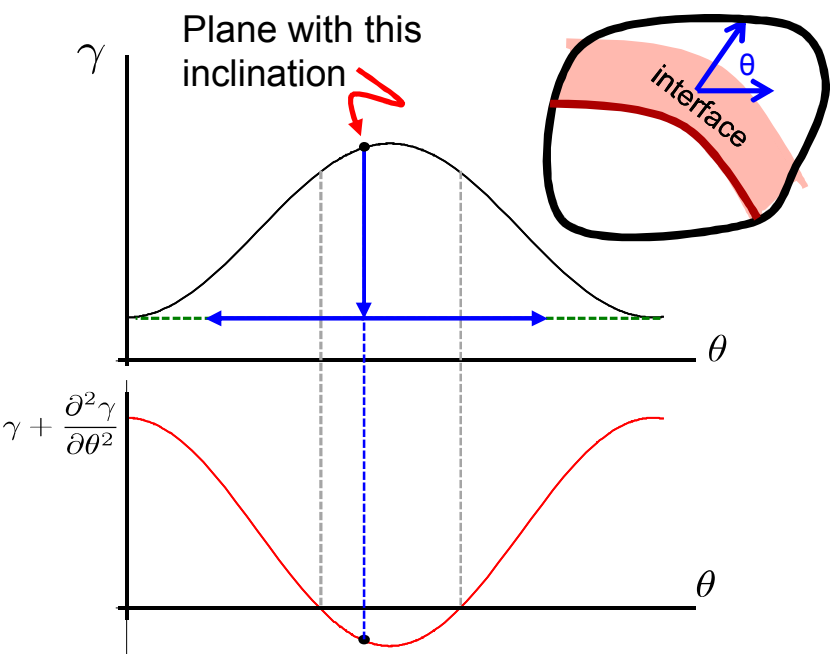
$$\mu \sim v_m \left(\gamma + \frac{\partial^2 \gamma}{\partial \theta^2} \right) H$$

W. W. Mullins (1963)

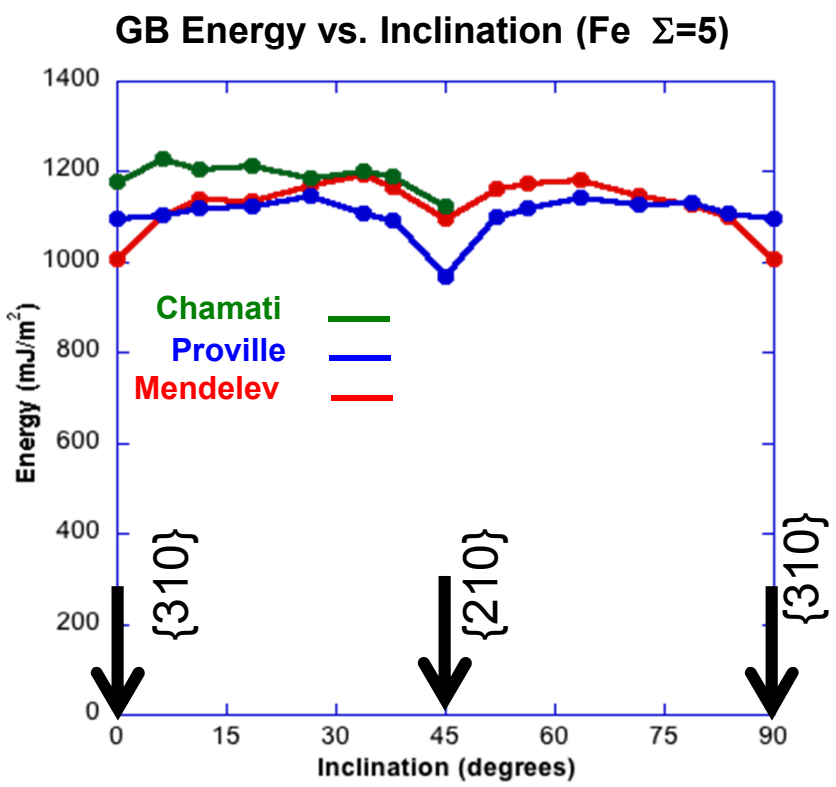
Interface stiffness

-Inclinations with negative interface stiffness break into facets with minimum energy orientations.

-"interface spinodals": analogous to phase separation in bulk materials.

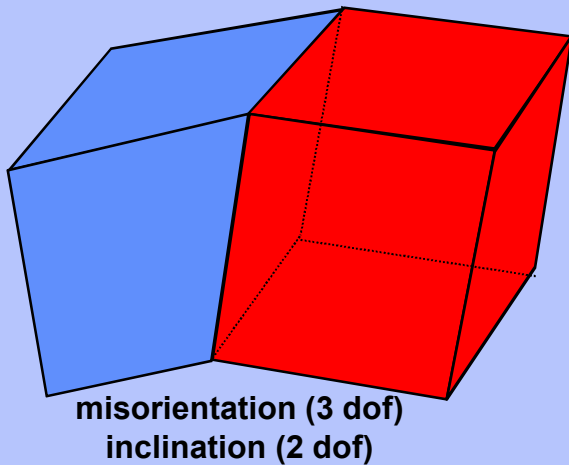


Frank (1963), Cabrera (1964),
Stewart (1992), Liu (1993)



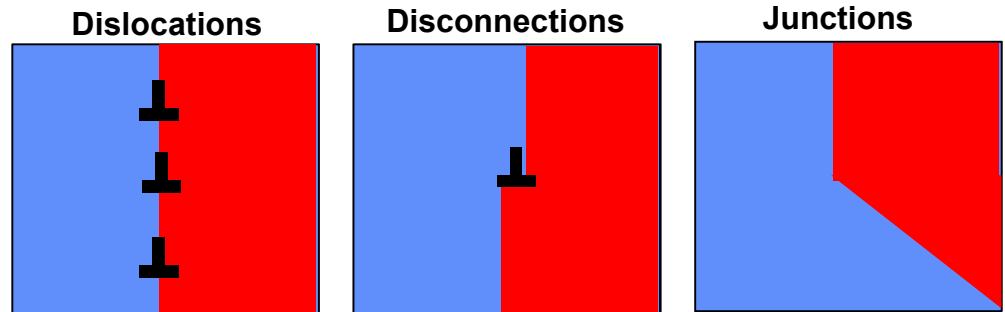
How to connect between atomistic and continuum descriptions of grain boundaries?

Grain boundary geometry characterized by 5 "macroscopic" degrees of freedom



Our approach:

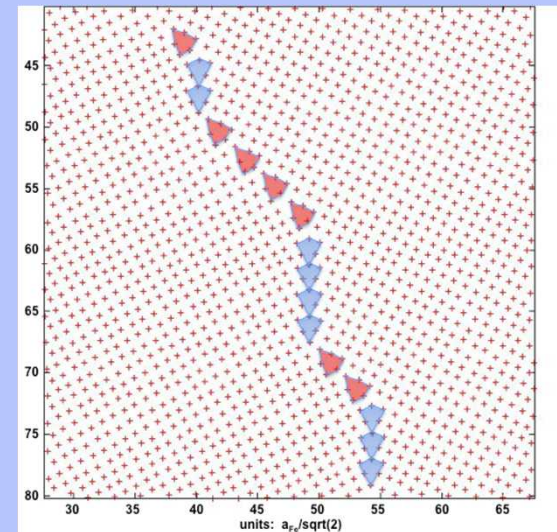
Focus on arrangements and interactions of elementary interfacial line defects



Atomistic scale microscopy and modeling

Focus for Today's talk

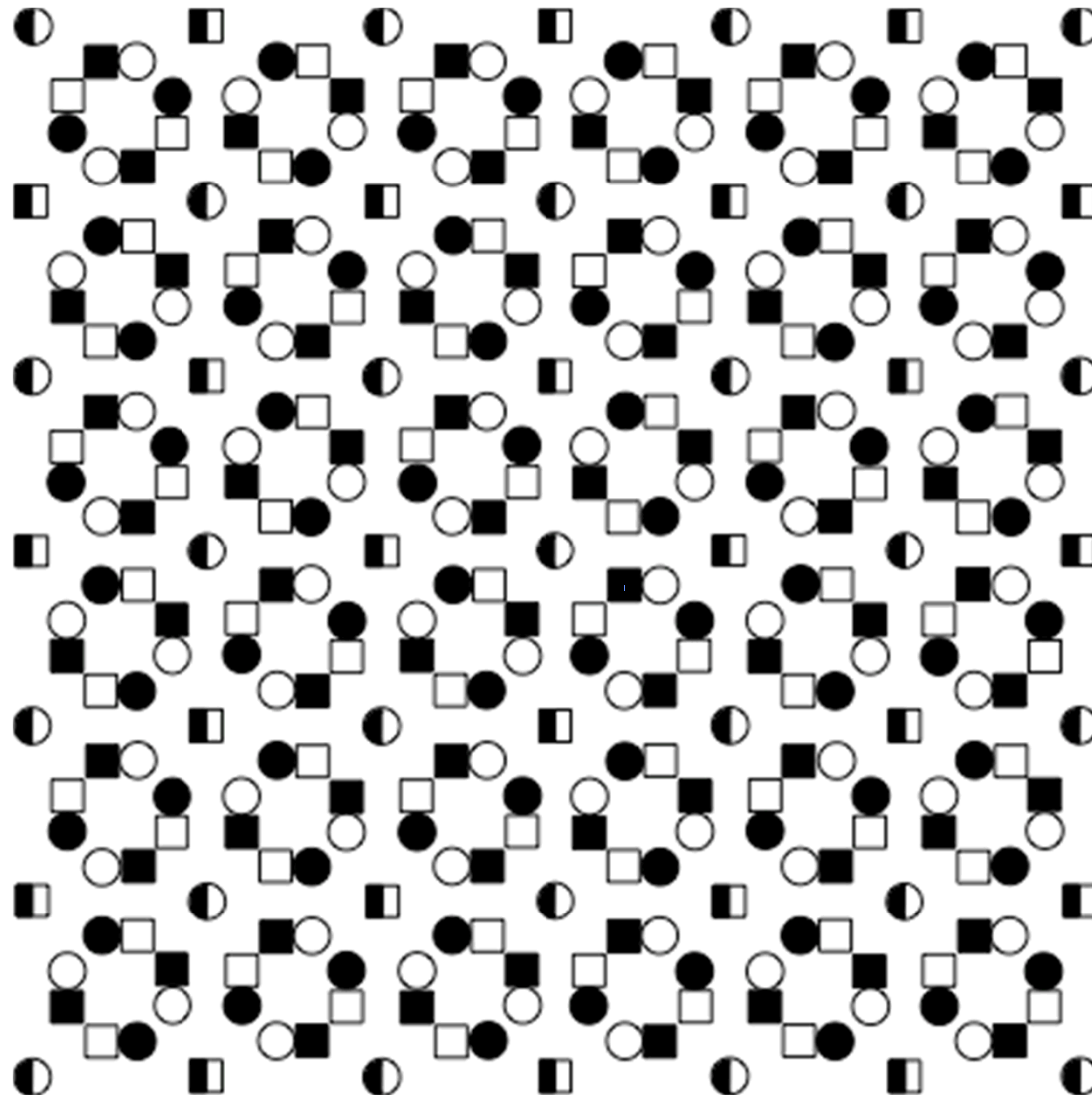
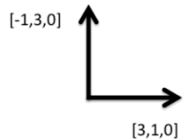
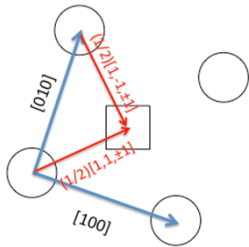
- Observations and calculations of a $\Sigma=5$ Grain boundary in BCC Fe
- Deviation from symmetric inclination and ideal misorientation:
 - nanoscale faceting
 - interfacial dislocations



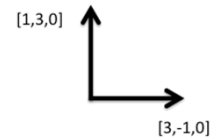
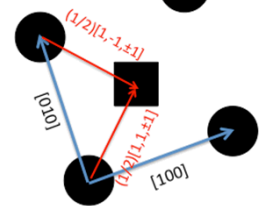
Geometric origin of the junction structure

$\Sigma=5$
Dichromatic
Pattern

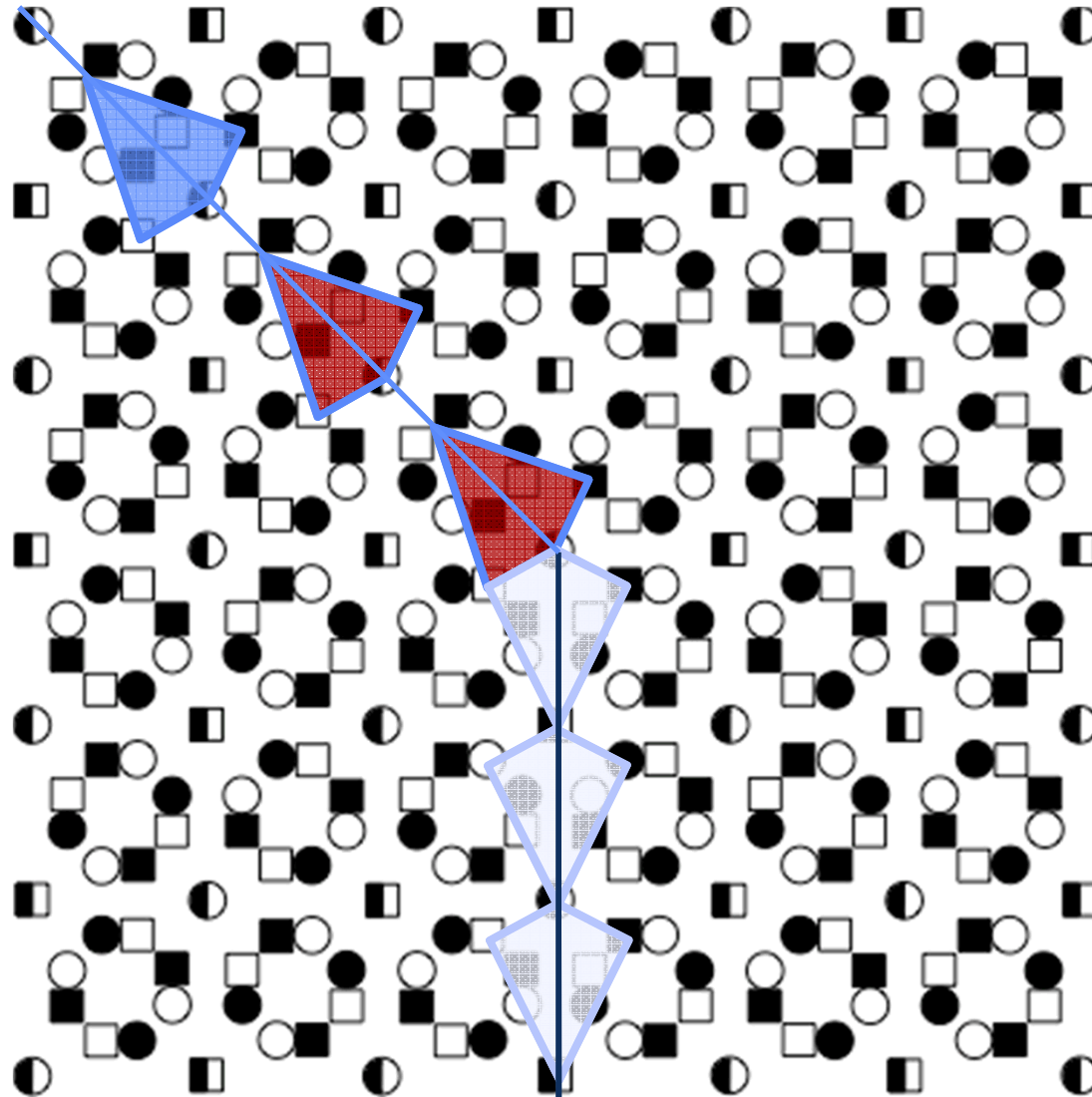
$\lambda=\text{white}$



$\mu=\text{black}$

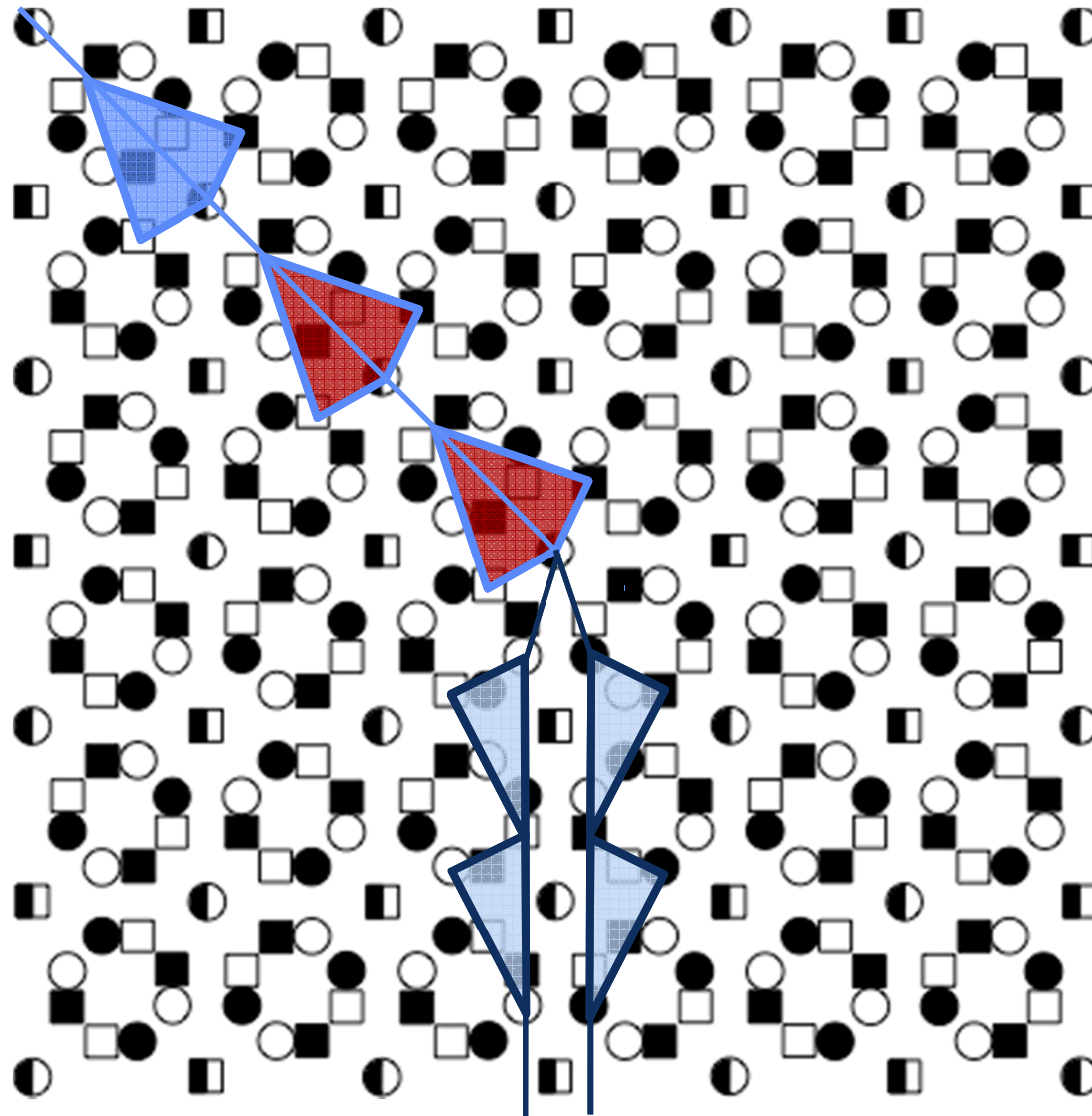


Geometric origin of the junction structure



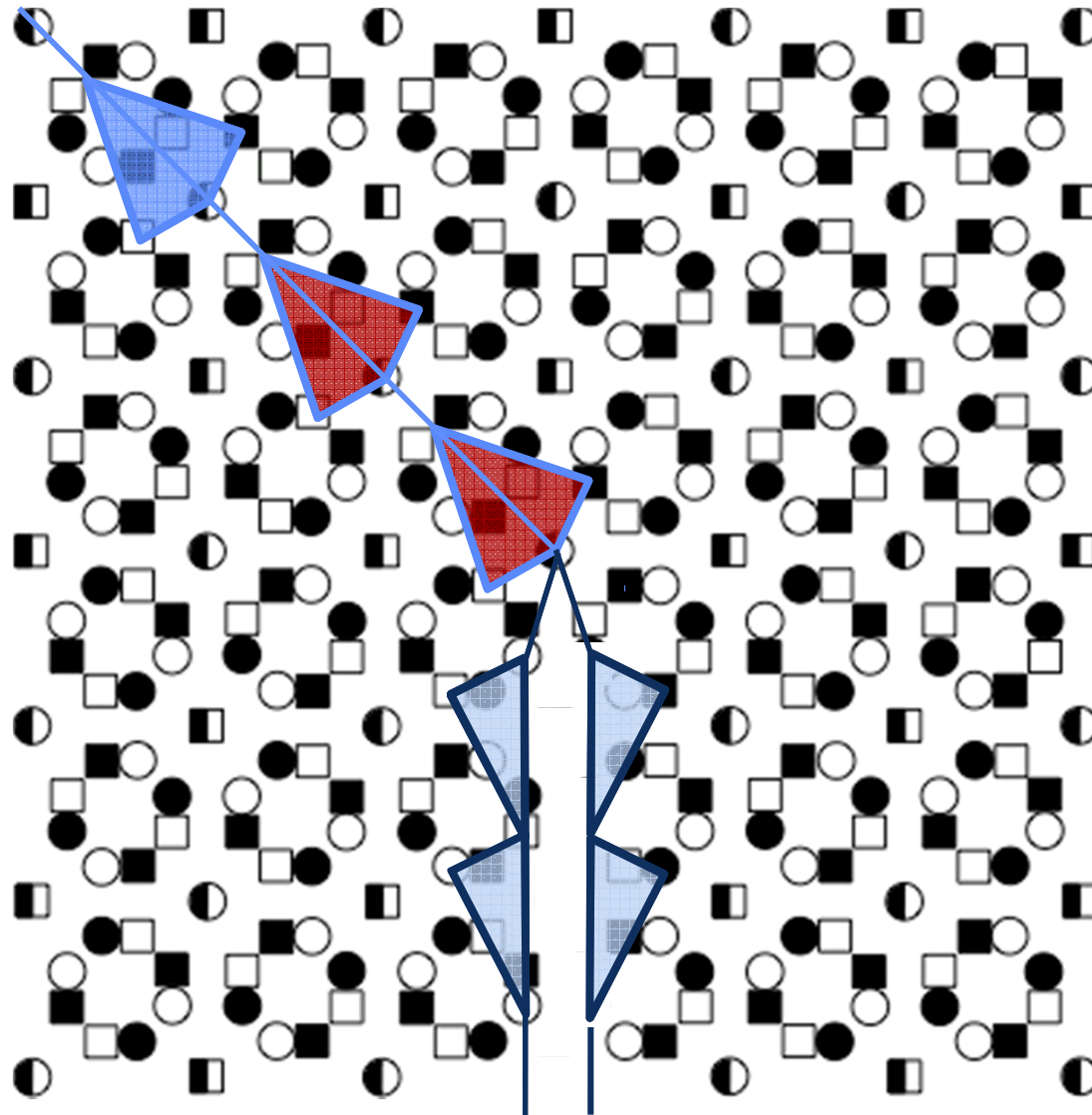
Geometric origin of the junction structure

Remove
material from
half-plane
of width
 $(1/5)[310]$

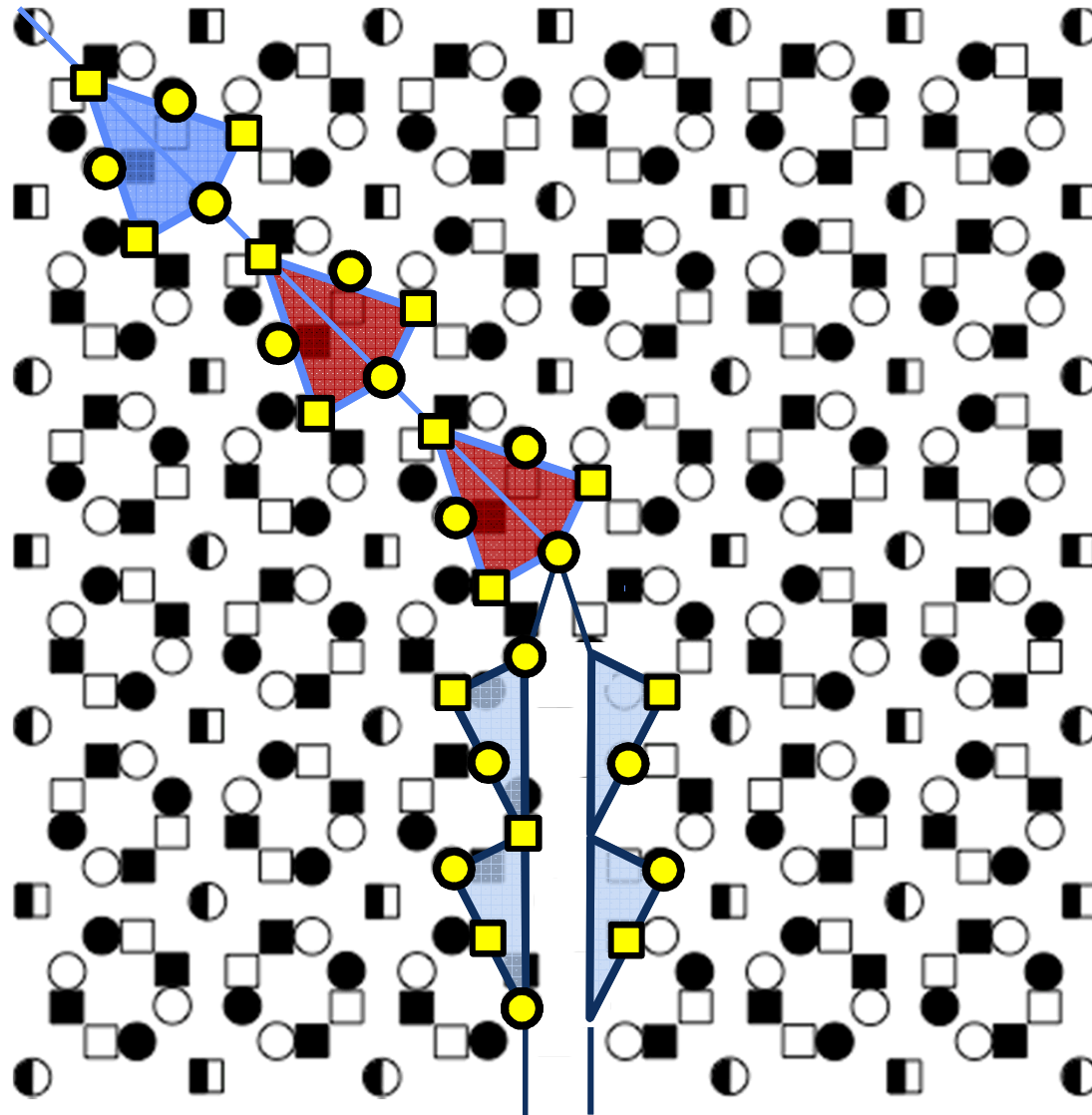


Geometric origin of the junction structure

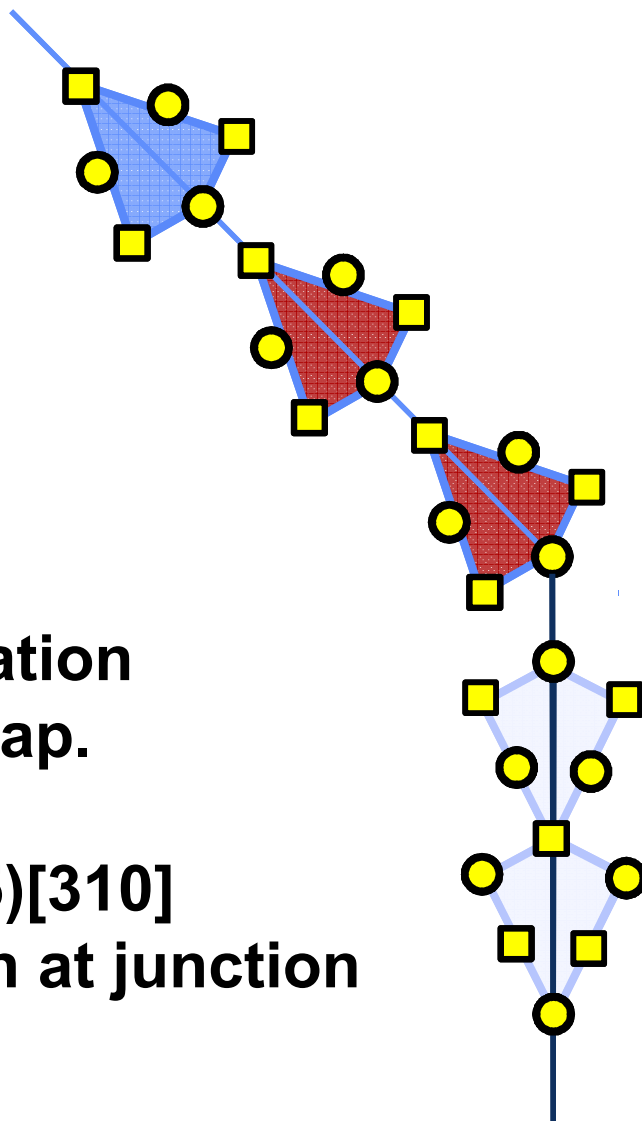
Remove
material from
half-plane
of width
 $(1/5)[310]$



Geometric origin of the junction structure



Geometric origin of the junction structure

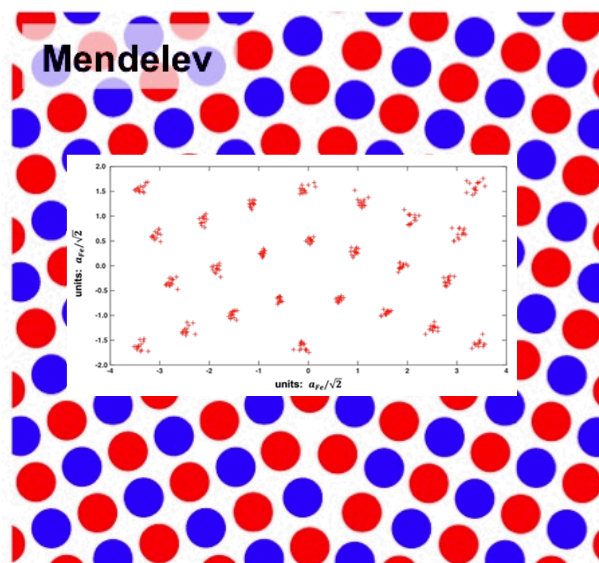
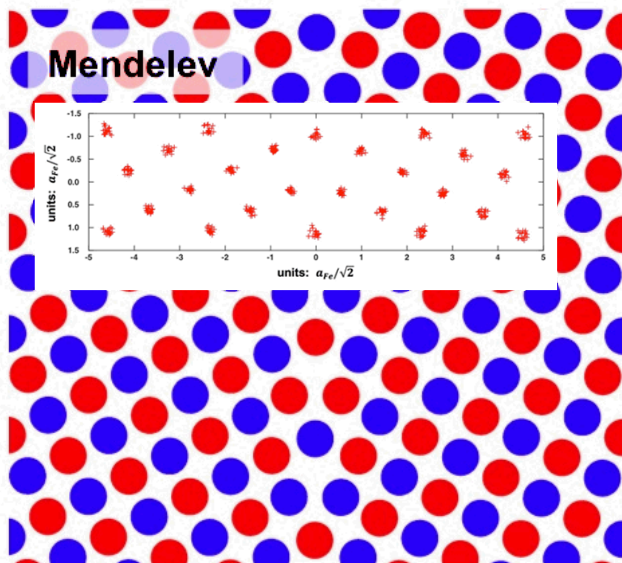


Kites are now off-set

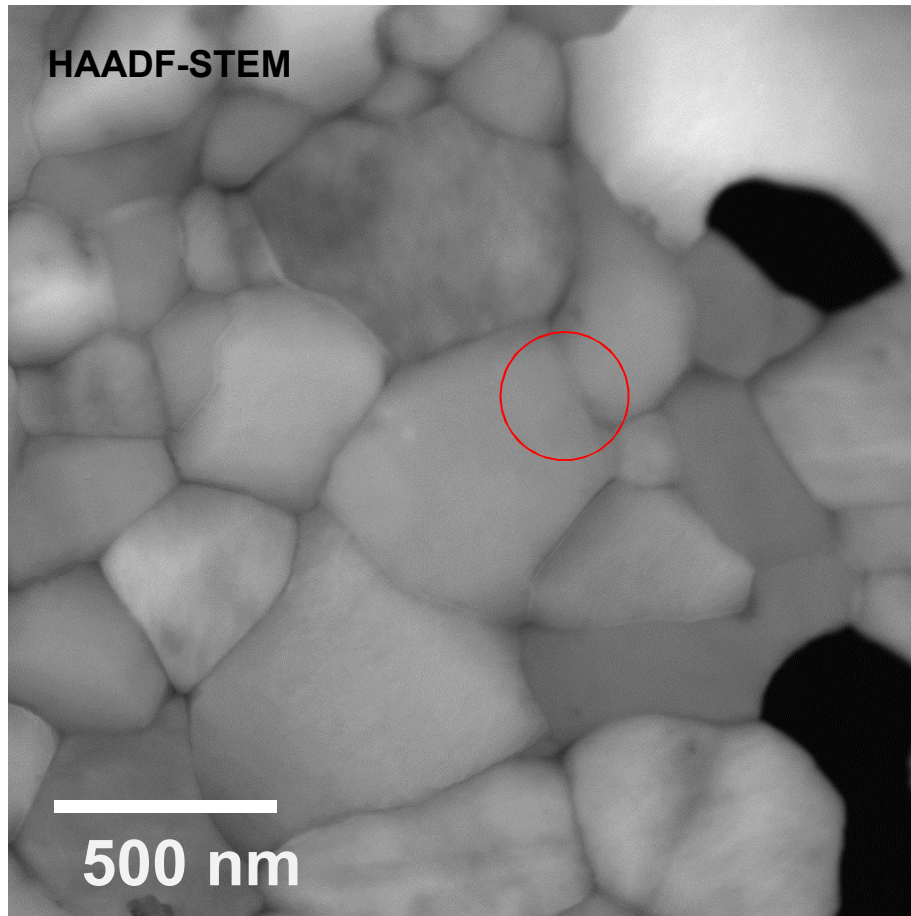
**Volterra operation
to close the gap.**

**Creates a $(1/5)[310]$
disconnection at junction**

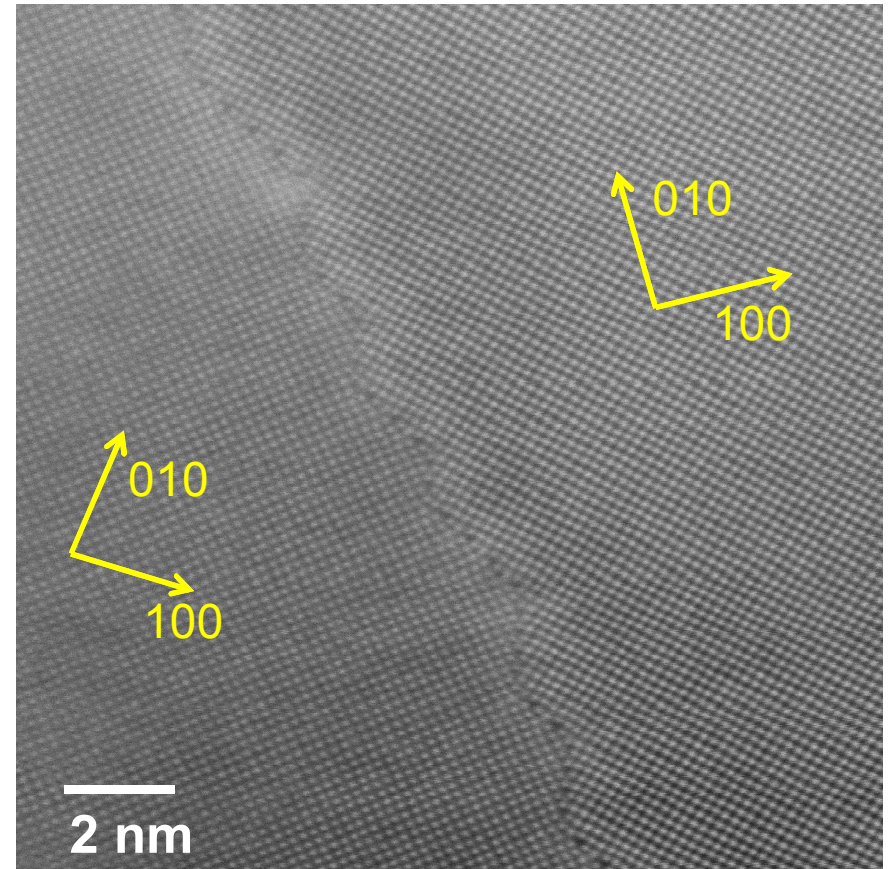
**Similar
construction for
 $(1/5)[120]$
disconnection**



Observations: Polycrystalline BCC Fe film

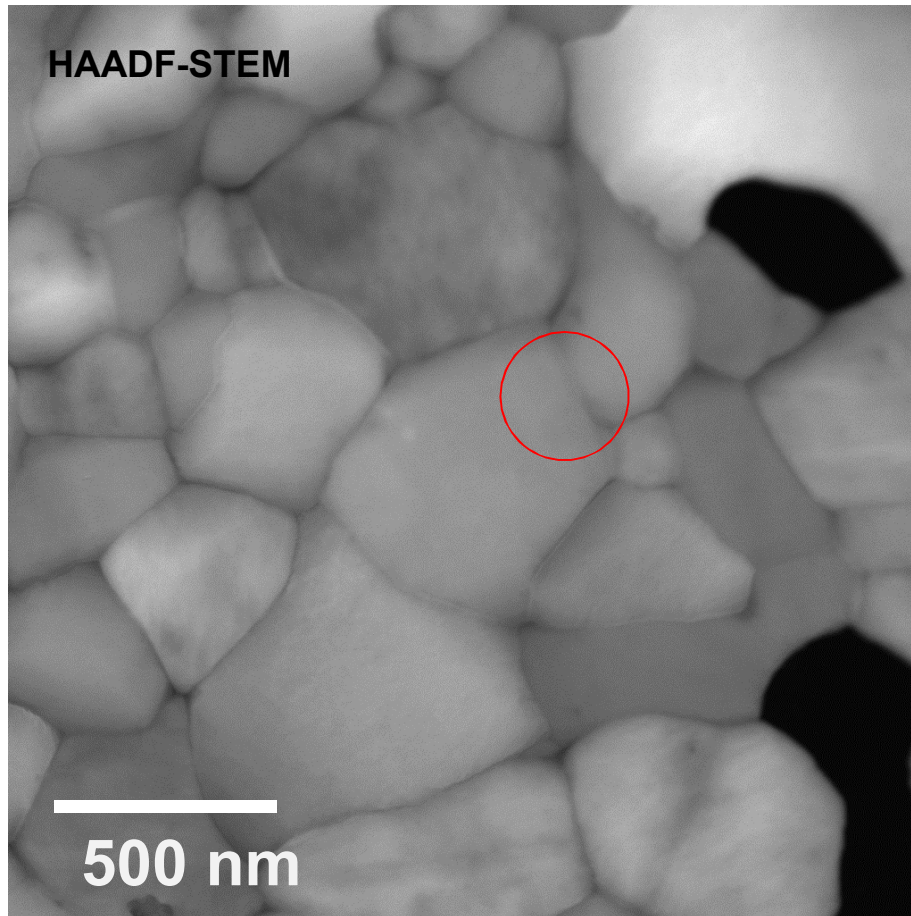


Pulsed Laser Deposited Fe on Rocksalt (NaCl). 36 nm thickness.
Specimen released and annealed on Mo grid 675°C, 2 hours.
under vacuum

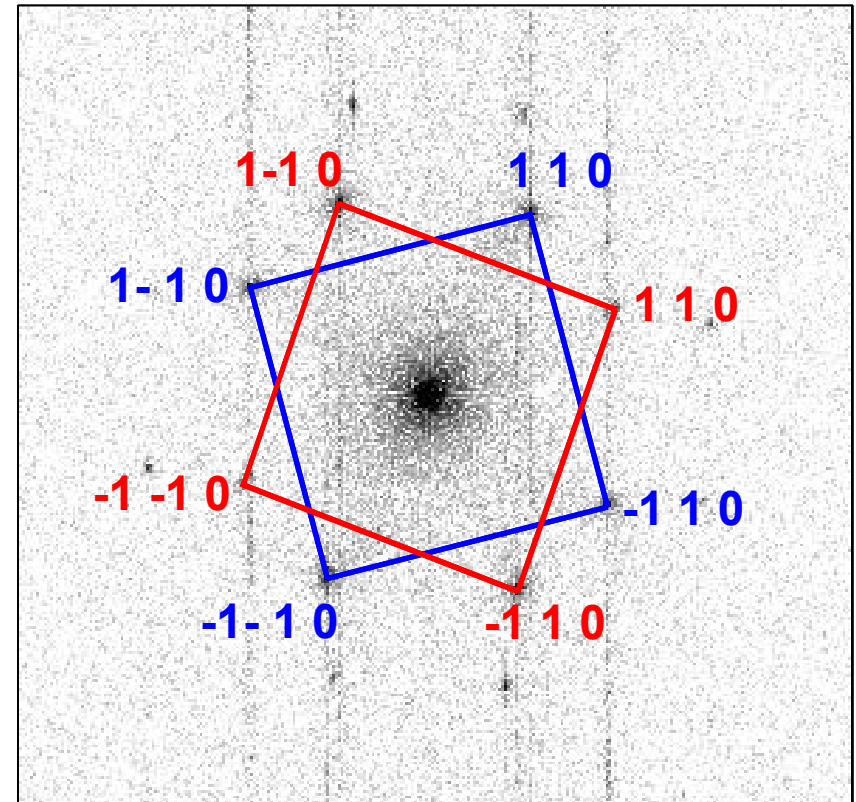


HAADF-STEM
FEI-200 keV probe corrected Titan

Observations: polycrystalline Fe thin film



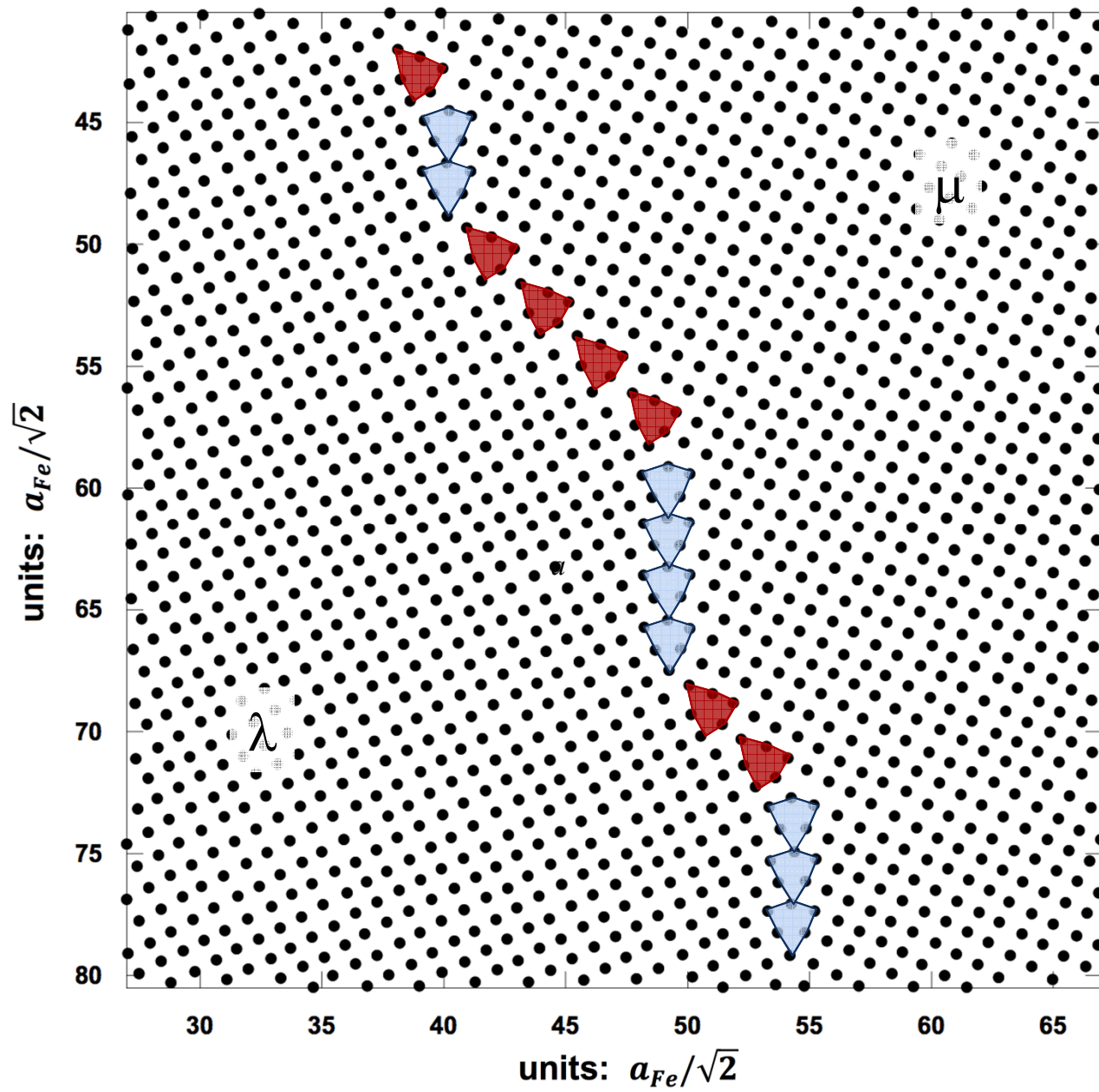
Pulsed Laser Deposited Fe on Rocksalt (NaCl). 36 nm thickness.
Specimen released and annealed on Mo grid 675°C, 2 hours.
under vacuum



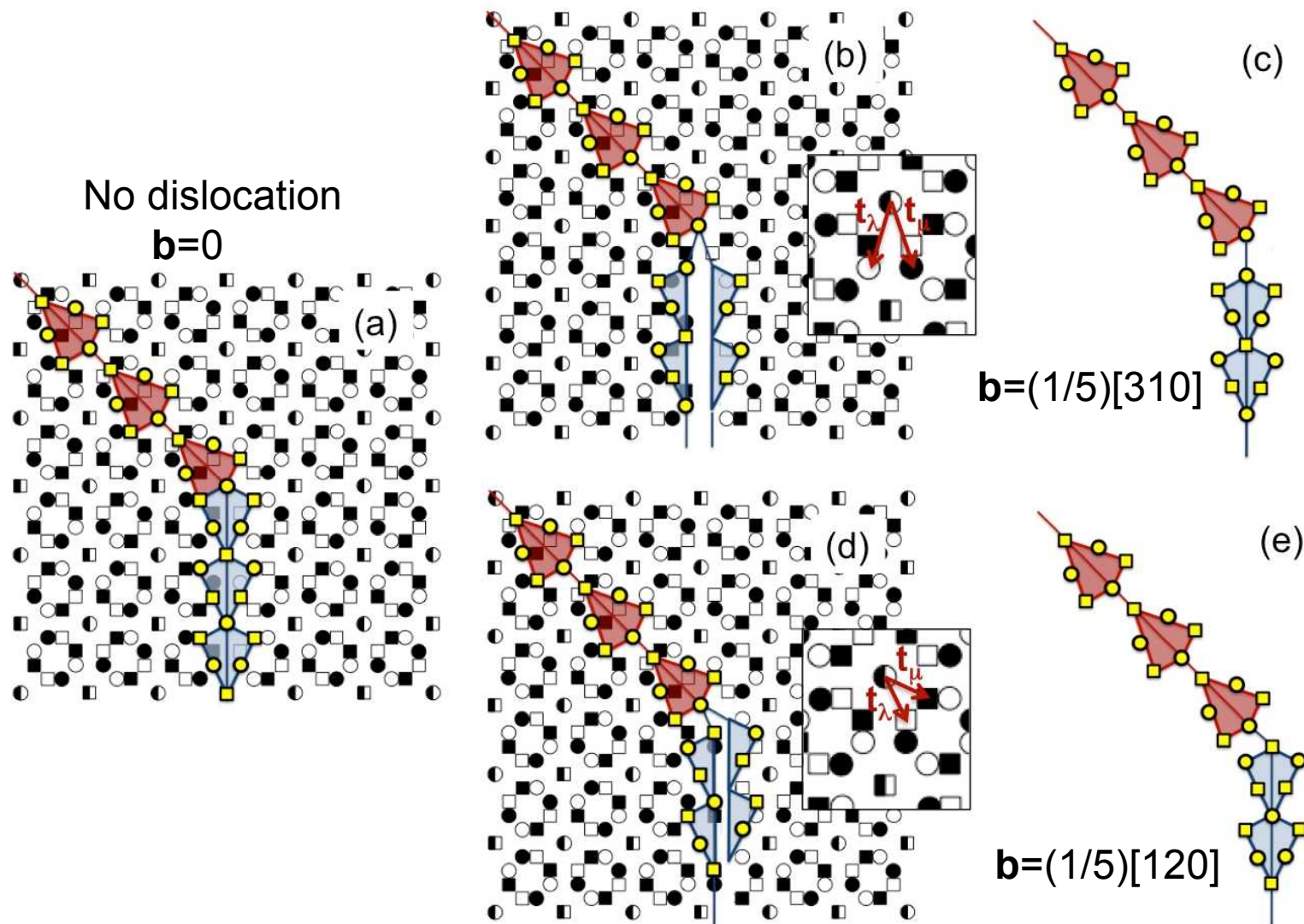
Measured misorientation: $34.49^\circ \pm 0.7^\circ$

Very close to $\Sigma=5$: $\theta_{\Sigma=5}=36.87^\circ$

$\Delta\theta = -2.38^\circ$

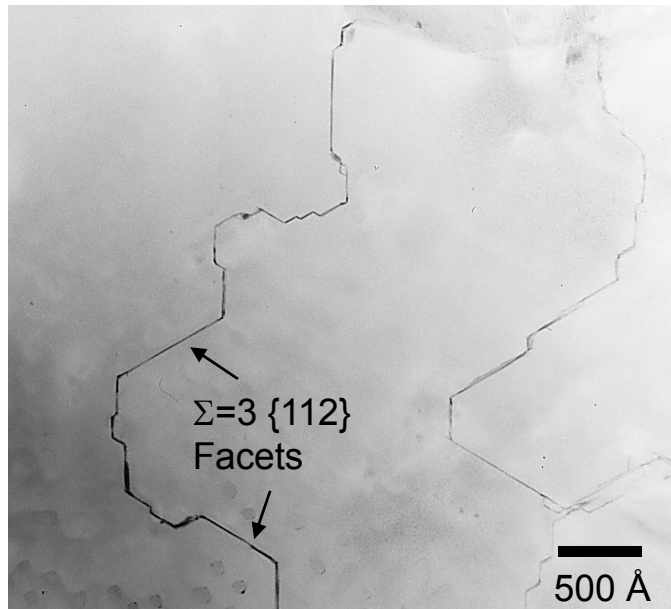


Geometric construction links junction core structure to defect content



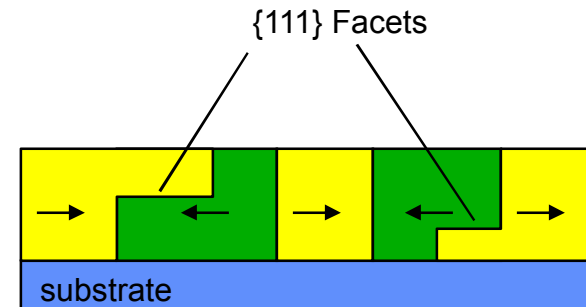
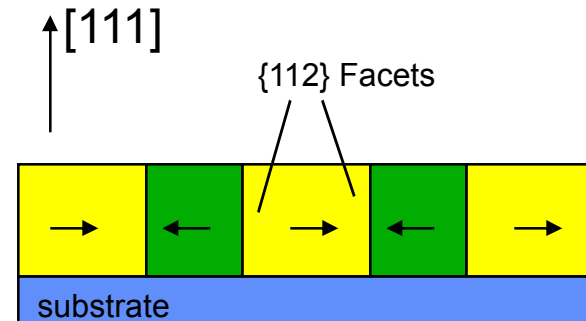
$\Sigma 3$ boundary in Au film

Plan View TEM Image of
Gold Thin Film $\Sigma=3$ Bicrystal

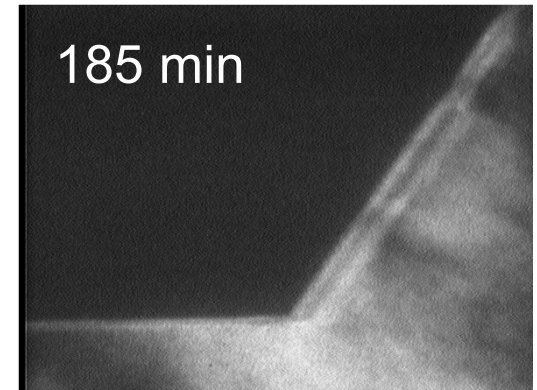
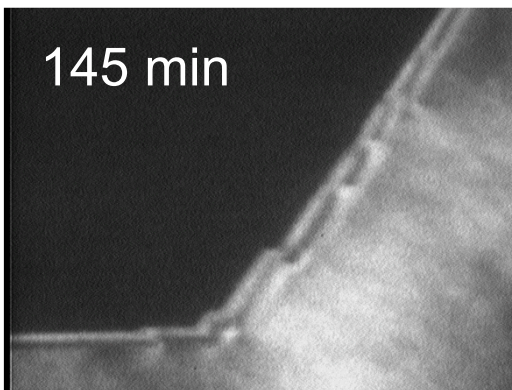
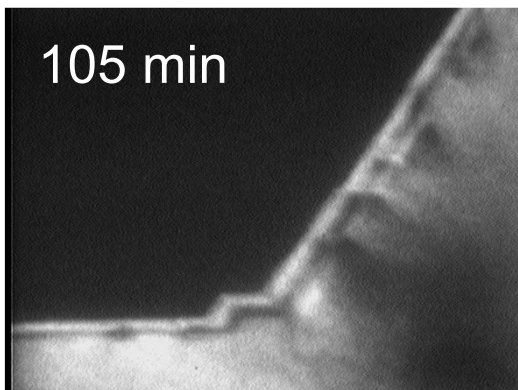
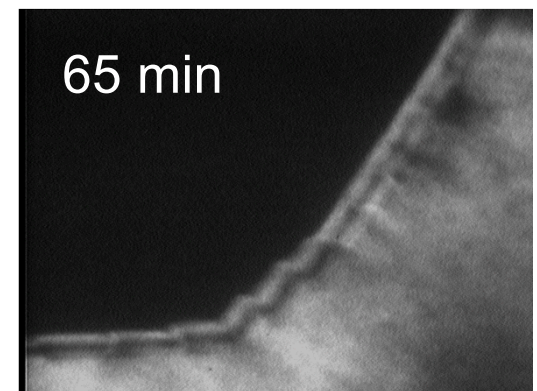
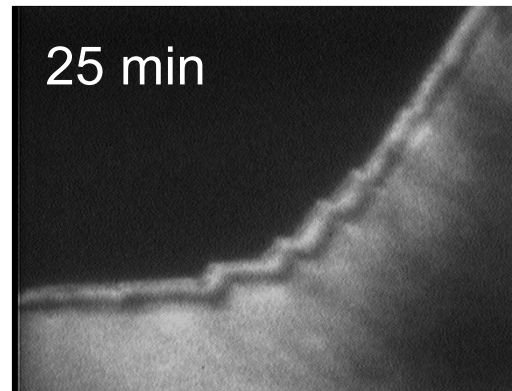
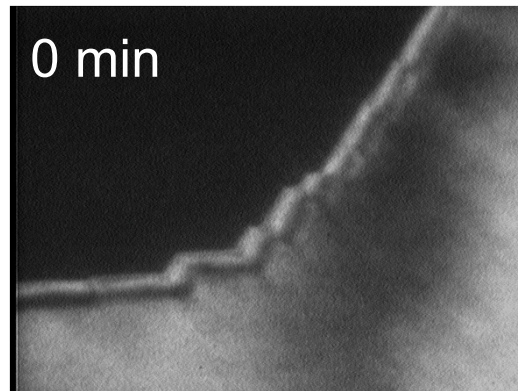


Epitaxial film growth gives two 180° related orientation variants.

Substrate removed to produce electron transparent membrane.



Step motion occurs during annealing as sharp corner develops




50 nm

T=490°C

D.L. Medlin and G. Lucadamo, MRS Symp 252 (2001)