Electron Backscatter Diffraction Pattern Simulation for Interaction Volume Containing Lattice Defects

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Contributors to the EMsoft Project: Patrick Callahan, Saransh Singh, Stuart Wright, Elena Pascal, Will Lenthe, Zihao Ding, Joseph Tessmer, Ke-Wei Jin, Michael Atkinson, Joao Fonseca, Michael Jackson, Joey Kleingers, Håkon Wiik Ånes, McLean Echlin.

Other people in the group: Marcus Ochsendorf, Christian Kurniawan, Maxwell Lee, Ke-wei Jin, Michael Kitcher, Clement Lafond

Github Link: https://github.com/EMsoft-org/EMsoft

Reference: C Zhu, M De Graef, 2020. EBSD pattern simulations for an interaction volume containing lattice defects, Ultramicroscopy 218, 113088

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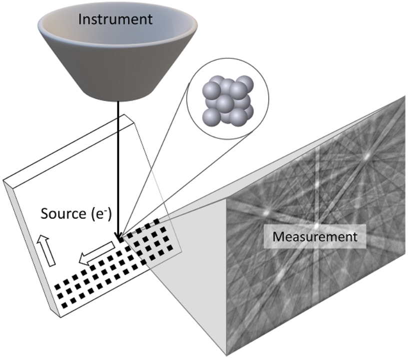
Electron Backscatter Diffractioin

Electron backscatter diffraction is a fully automated SEM-based characterization technique to extract **structural information** from materials to study their microstructure, texture, defects, etc.



Bragg Diffraction

$$2d_{hkl} \sin\theta_{hkl} = n\lambda$$

where d_{hkl} is the atomic plane distance, λ is the relativistic wavelength of electron beam, θ_{hkl} is the Bragg angle.
Kikuchi band width $= 2\theta_{hkl}$ between (h,k,l) and $(\bar{h}, \bar{k}, \bar{l})$.

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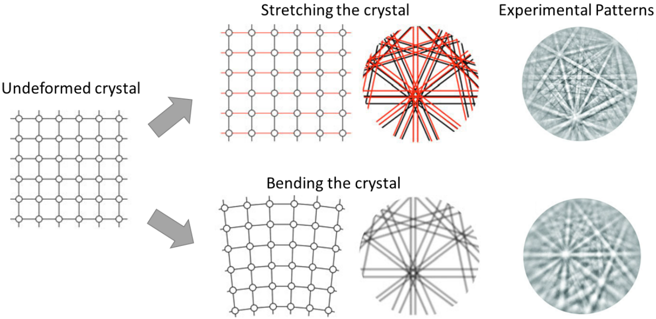
Elastic Strain: Stretching and Bending the Crystal

Elastic strain=Elastic Stretch + Lattice Rotation

Shifts in the zone-axis and changes in the inter-planar angles or blurring of Kikuchi pattern's edges.

Wright et al, Microsco. Microanal., 2011



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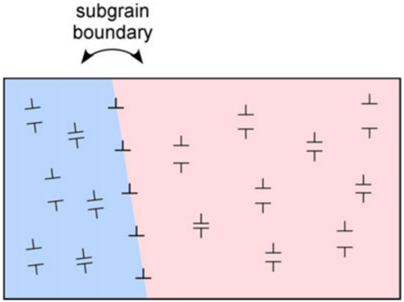
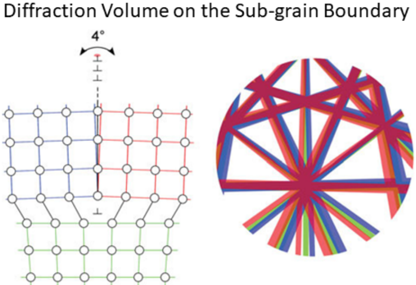
Plastic Strain: Creation and Motion of Dislocations

Local perturbations of diffracting planes leads to incoherent scattering i.e. pattern degradation.





Wright et al, Microsco. Microanal., 2011

Quantification of Deformation

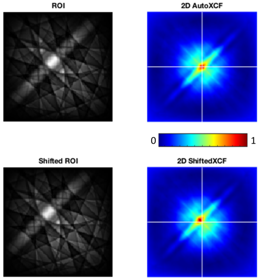
The quantification of deformation based on diffraction features is a complex inverse problem.

1. Image quality: average of peaks in Hough space (plastic strain)
2. Fourier spectrum: first moment of power spectra (plastic strain)
3. Kernel average misorientation (KAM): average misorientation between neighboring pixels (plastic strain)
4. **High-angular resolution EBSD (HR-EBSD): strain, lattice rotation, GNDs (elastic and plastic strain)**
5. etc

High-Angular Resolution EBSD

High-Angular Resolution EBSD (HR-EBSD) measures shifts from many regions of interests (ROIs) between a reference pattern and test pattern (deformed) to determine the elastic deformation gradient tensor F_e.

Cross-Correlation



$$f_{test} * f_{ref} = \Im^{-1}[\Im(f_{test}) \cdot conj(\Im(f_{ref}))]$$

Strain sensitivity 10^{-4} and rotation sensitivity 0.006°

Non-Linear Minimization

$$\min f(\mathrm{F_e}) = \frac{1}{2} \sum_{ROIs} \left\| \frac{Z^*}{(\mathrm{F_e} \cdot \mathrm{r}) \cdot \hat{\mathrm{k}}} \mathrm{F_e} \cdot \mathrm{r} - (\mathrm{r} + \mathrm{q}) \right\|^2$$

F_e: elastic deformation gradient tensor
r : position vector of ROI center
q : measured shifts
Z^*: detector distance.

Villert et al, Journal of Microscopy, 2009

Wilkinson et al, Ultramicroscopy, 2006

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Electron Scattering 101

The high energy (kVs) electron (wave-like particles) beam interaction with solid matter results in multitude of measurable signals. There are **three principally different scattering mechanisms**:

Bragg Scattering

Elastic (with conservation of electron energy) and coherent (with conservation of phase) scattering with small scattering angle ($\leq 5^\circ$) and large scattering cross-section.

Phonon Scattering or Thermal Diffuse Scattering

Quasi-elastic (low loss of energy 1 eV) and incoherent (out of phase) scattering with medium scattering angle ($\leq 10^\circ$).

Core-loss Scattering

Inelastic and incoherent scattering events close to the atomic nuclei with large energy loss from 10 eV to keV (secondary electrons , X-rays, etc).

Building a Forward Model for EBSD Pattern Simulation

Stochastic Component

Monte Carlo electron trajectory simulation using continuous slowing down approximation to predict spatial and energy distributions for BSEs.

Deterministic Component

Dynamical scattering model using scattering matrix or Bloch wave approaches to predict distribution of BSEs with respect to crystal reference frame.

Multiple Scattering

Elastic and inelastic scattering events combine to form a chain of scattering events with large scattering angles (up to 180°) and broad energy range (eV to keV).

The spatial and energy distribution of BSEs can be modeled with **Monte Carlo electron trajectory simulation**. For simplicity, constant rate of energy loss i.e. **continuous slowing down approximateion (CSDA)** can be assumed.

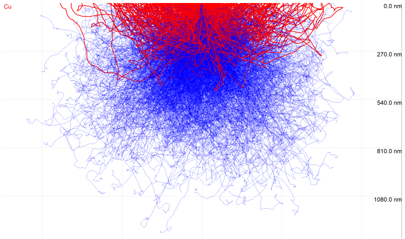

Figure: CASINO MC simulation of Cu at 20kV (Blue:BSE, Red:SE)

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Building a Forward Model for EBSD Pattern Simulation

Stochastic Component

Monte Carlo electron trajectory simulation using continuous slowing down approximation to predict spatial and energy distributions for BSEs.

Deterministic Component

Dynamical scattering model using scattering matrix or Bloch wave approaches to predict distribution of BSEs with respect to crystal reference frame.

Dynamical Simulation of Electron Diffraction

Dynamical theory of electron diffraction deals with diffraction phenomenon due to multiple scattering of existing BSEs based on **Bloch wave Ψ formalism**.
The back-scattered electron (BSE) yield as a function of direction **k** is integrated over energy E and subsurface depth z.

Back-scattered Electron Yield

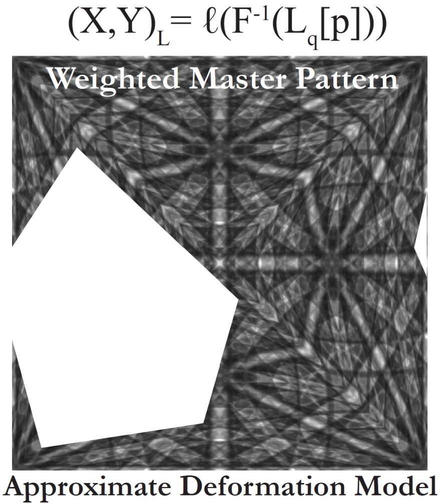
$$P(\mathrm{k}) = \sum_{n\in \mathrm{A.U.}} \sum_{j\in\mathcal{S}_n} \sigma_j \int_{E_{\mathrm{min}}}^{E_0} \mathrm{d}E \int_0^{z_0(E)} \mathrm{d}z \lambda_{\mathrm{k}}(E,z)|\Psi_{\mathrm{k}}(\mathrm{r}_j;E,z)|^2,$$

where σ_j the Rutherford scattering cross section for element j, E_0 the incident beam energy, E_{min} a suitably chosen minimum energy, and Ψ the electron wave function evaluated at the atomic position r_j.

Merging Monte Carlo Simulation with Dynamical Scattering

From the Monte Carlo simulation, $\lambda_k(E,z)$ weighting function describing the fraction of incident electrons of energy E at exist depth z traveling in the direction k can be determined.

Approximate Model for the Inclusion of Deformation



Rotated and Undeformed Pattern Coordinates

$$(X,Y)_L = \mathcal{L}(L_q[\hat{\mathrm{p}}])$$

where $\hat{\mathrm{p}}$ is the detector pixel coordinates with respect to the illumination point, L_q is the quaternion rotation operator, q is the (passive) rotation required to rotation the sample frame to crystal frame, $\mathcal{L}$ is the square Lambert transformation.

Rotated and Deformed Pattern Coordinates

$$(X,Y)'_L = \mathcal{L}\left(F^{-1}(L_q[\hat{\mathrm{p}}])\right)$$

where F is the deformation gradient tensor.

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Computation of the Deformed Pattern from the Interaction Volume

Depth-Dependent Back-scattered Electron Yield

$P(\mathrm{k}) = \sum_{p=1}^{N_z} P(\mathrm{k}, z_p)$

3D Deformation Field

$\mathrm{F}(\mathrm{x},\mathrm{y},\mathrm{z}) = \nabla \mathrm{u}(\mathrm{x},\mathrm{y},\mathrm{z}) + \mathrm{I}$

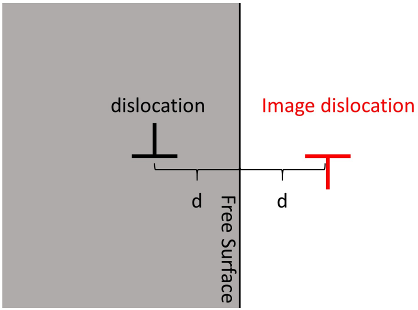
Intensity of Deformed Pattern from the Interaction Volume

- Gaussian beam spread of the Monte Carlo e- yield data
- Determine the region of interaction volume to be used and the weight factor
- Calculate all the deformed patterns (within the ivol) from depth dependent master patterns
- Summation of all the deformed patterns weighted by the Monte Carlo e- yield weight factor

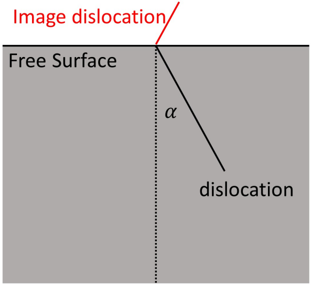
Yoffe-Shabaini-Hazzeldine Model

In a finite body, the traction free boundary condition must be satisfied at the free surfaces. The resulting surface relaxations are important and have a strong local effect on the BSE yield.

Dislocation Parallel to the Free Surface

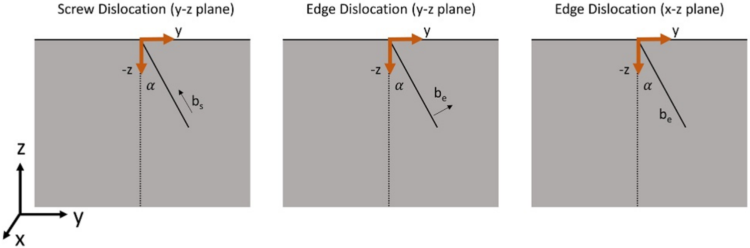


Surface Threading Dislocation



Yoffe-Shabaini-Hazzeldine Model

From the YSH model, the near surface displacement field, u(x, y, z), of an inclined threading dislocation can be determined for any position in the $-z$ half space of the sample, based on a few input parameters $(b_x, b_y, b_z, \alpha, E, \nu)$ (i.e., the Burgers vector components, the inclination angle of the dislocation line, and the Young's modulus and Poisson ratio of the isotropic material).



E.H. Yoffe, Philos. Mag., 1961.

S.J. Shaibani, P.M. Hazzledine, Philos. Mag., 1981

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Beam interaction volume (Ni 10kV)

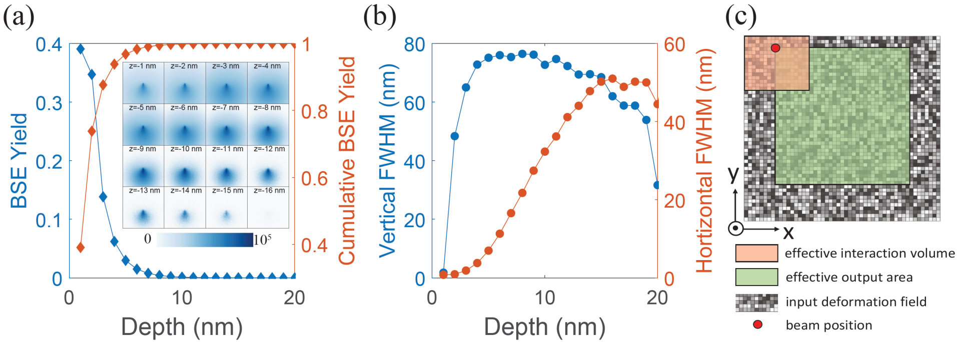

Figure: (a) BSE yield and cumulative BSE yield as a function of exit depth, including visualization of the shape of the interaction volume representing the number of electrons from each exit depth bin, (b) vertical/horizontal FWHM of the shape of interaction volume as a function of exit depth, (c) schematic of the effective area (green) probed by an effective interaction volume (red) on a randomly generated grid of deformation field (gray).

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Validation of YSH model: Stress tensor

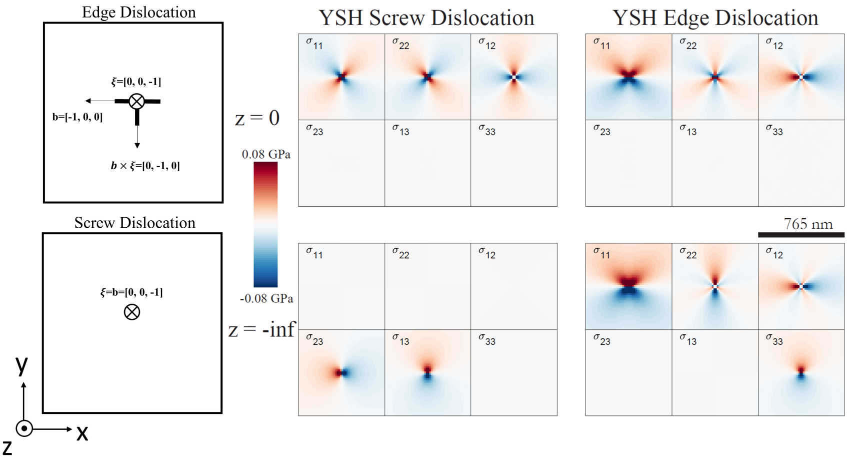

Figure: Stress tensor components derived from the YSH model for pure screw and edge dislocations at $z = 0$ and $z = -\infty$. Each box represents a map of a particular tensor component with a box dimension of 765×765 nm^2 and 3 nm pixel size.

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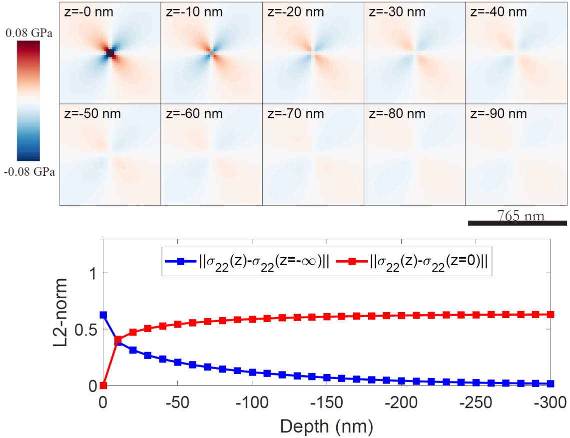
Validation of YSH model: Stress Tensor



Attenuation of screw dislocation's σ_{22} image stress in the depth $(-z)$ direction. Stress maps are plotted from $z = [0, -90]$ nm. $L2$-norms between $\sigma_{22}(z)$ and $\sigma_{22}(z = 0)/\sigma_{22}(z = -\infty)$ are plotted from $z = [0, -300]$ nm. Stress map color scale range: $[-0.1, 0.1]$ GPa.

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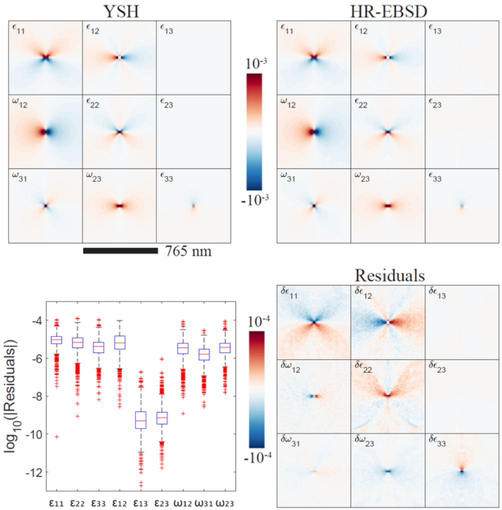
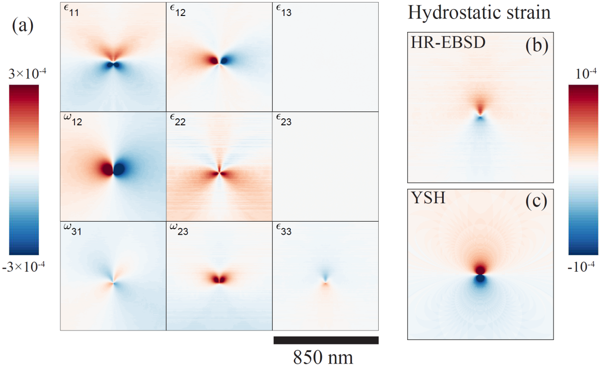
YSH free surface edge dislocation: Beam as delta function



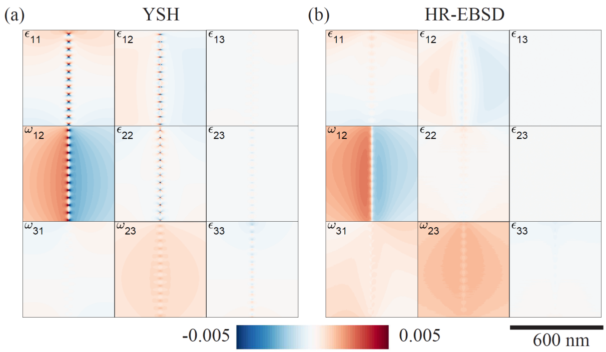
- No interaction volume effect taken into account (delta beam)
- YSH input for the approximate deformation inclusion approach (z=0)
- Deformation calculated from simulated patterns using HR-EBSD (accuracy=10^{-5})

YSH surface threading edge dislocation: Interaction volume



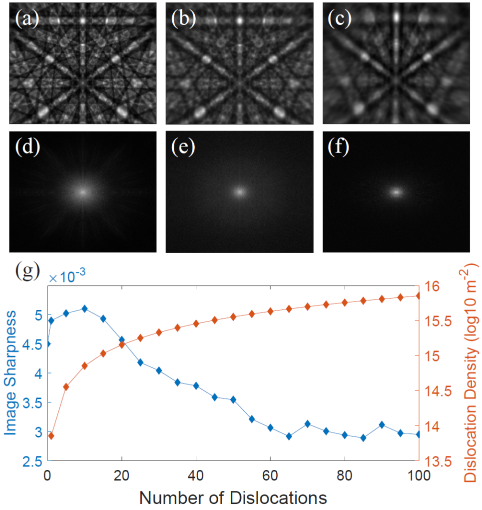
- Interaction volume taken into account
- volumetric YSH deformation field data (0-10 nm)
- Deformation calculated from simulated patterns using HR-EBSD
- Deformation field is a weighted field from the within interaction volume

YSH Low-angle grain boundary: Interaction volume



- Interaction volume taken into account
- volumetric YSH deformation field data (0-10 nm)
- Deformation calculated from simulated patterns using HR-EBSD
- Deformation field is a weighted field from the within interaction volume

Dislocation density and pattern sharpness



- randomly position pure edge STD in the interaction volume
- sharpness is evaluated using high-frequency components of the power spectrum
- pattern sharpness reduces with increasing number of dislocations
- pattern sharpness stays unchanged after reaching dislocation density $10^{15.5}$ $lines/m^2$

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Summary (Zhu and DeGraef, Ultramicroscopy, 2020)

- Developed and validated the approximate model for deformation inclusion in diffraction pattern
- Developed a forward model to simulate a diffraction pattern from an interaction volume containing lattice defects
- Validated the YSH analytical model for STD at free surface and far away from the surface
- Interaction volume approach distorts and smooths out the deformation field.
- Interaction volume approach also reveals that the deformation information is mostly from the material very close to the surface.
- Pattern sharpness reduces with increasing dislocation density until $10^{15.5}\, lines/m^2$