Only thanks to the support of many people this paper has happened. Special thank you to T. Palmer (ANSTO), T. Nicholls (ANSTO), Z. Zhang (ANSTO), L. Edwards (ANSTO), and M.R. Hill (UC). I hope you find the paper useful.

/OM

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To maintain compatible deformation across variously oriented grains in a polycrystalline aggregate, the voids and overlaps between the individual grains, which would otherwise appear due to the crystallites (grains) anisotropy are corrected by the storing a portion of dislocations in the form of geometrically-necessary dislocations (GNDs). Plastically deformed material also stores so-called statistically-stored dislocations (SSDs), which are stored by mutual random trapping. Both GNDs and SSDs arrange themselves into energetically favourable configurations, forming geometrically-necessary boundaries (GNBs) and incidental dislocation boundaries (IDBs), respectively.
⇒ EBSD orientation map showing the overall equiaxed grain structure of our solution-annealed Ni201 before testing.

⇒ Interrupted tensile tests were performed to varying levels of imparted plastic strain. Samples were extracted from the gauge length for EBSD and HRSD measurement.

Ni-201

<table>
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<th>Ni</th>
<th>C</th>
<th>Si</th>
<th>P</th>
<th>Fe</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Cu</th>
<th>V</th>
<th>Nb</th>
<th>Ti</th>
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EBSD Measurements
Electron Back-Scatter Diffraction (EBSD)

⇒ EBSD, is a scanning electron microscope (SEM) based technique that gives crystallographic information about the microstructure of a sample.

⇒ The data collected with EBSD is spatially distributed and is visualised in so-called EBSD orientation maps.
GNDs have a geometrical consequence giving rise to a curvature of the crystal lattice, which can be measured by EBSD technique. The crystal orientation \((\phi_1, \Phi, \phi_2)\) changes only when the electron beam crosses an array of GNDs that has a net non-zero Burger’s vector.
A schematic representation of lattice curvature components calculation between two neighbouring crystals misoriented ($\Delta \theta$) by a rotation around the common crystallographic axis [100]$_c$ ([uvw]$_c$) and separated by pixel separation distance ($\Delta x_2$). Note, that in this example: $\kappa_{12} \approx \Delta \theta_1 / \Delta x_2$, and $\kappa_{22}, \kappa_{32} = 0$. 

$$\kappa_{ij} =  \frac{\partial \theta_i}{\partial x_j} \approx \frac{\Delta \theta_i}{\Delta x_j}$$

$L$attice $C$urvature $T$ensor Components

$$\begin{align*}
\kappa_{11} &\approx \frac{\Delta \theta_1}{\Delta x_1} ; \\
\kappa_{21} &\approx \frac{\Delta \theta_2}{\Delta x_1} ; \\
\kappa_{31} &\approx \frac{\Delta \theta_3}{\Delta x_1} \\
\kappa_{12} &\approx \frac{\Delta \theta_1}{\Delta x_2} ; \\
\kappa_{22} &\approx \frac{\Delta \theta_2}{\Delta x_2} ; \\
\kappa_{32} &\approx \frac{\Delta \theta_3}{\Delta x_2}
\end{align*}$$
Lattice Curvature & GND Density

\[ \kappa_{ij} = \sum_{t=1}^{N} \left( b_j^t l_i^t - \frac{1}{2} \delta_{ij} b_m^t l_m^t \right) \rho_G^t \]

N = 1 .. 36 – number of possible dislocation types

6 known lattice curvatures (measured)

36 possible dislocation types

1,947,792 possibilities!

\[ \begin{bmatrix} \kappa_{11} \\ \kappa_{21} \\ \kappa_{31} \\ \kappa_{12} \\ \kappa_{22} \\ \kappa_{32} \end{bmatrix} = \begin{bmatrix} \frac{1}{2} b_1^1 l_1^1 & \frac{1}{2} b_1^2 l_1^2 & \cdots & \frac{1}{2} b_1^{36} l_1^{36} \\ b_1^1 l_2^1 & b_1^2 l_2^2 & \cdots & b_1^{36} l_2^{36} \\ b_1^1 l_3^1 & b_1^2 l_3^2 & \cdots & b_1^{36} l_3^{36} \\ \frac{1}{2} b_2^1 l_1^1 & \frac{1}{2} b_2^2 l_1^2 & \cdots & \frac{1}{2} b_2^{36} l_1^{36} \\ b_2^1 l_2^1 & b_2^2 l_2^2 & \cdots & b_2^{36} l_2^{36} \\ b_2^1 l_3^1 & b_2^2 l_3^2 & \cdots & b_2^{36} l_3^{36} \end{bmatrix} \begin{bmatrix} \rho_G^1 \\ \rho_G^2 \\ \rho_G^3 \\ \vdots \\ \vdots \\ \rho_G^{36} \end{bmatrix} \]

GND Density

Burgers Vector

Line Vector

Edge Dislocations: 12
Screw Dislocations: 6
Dislocation Types: 2 x 18 = 36

dislocations of opposite sign needs to be distinguished

Dislocation Types (fcc)

**12x Deformation Modes**

- (111)\langle 0\bar{1}1\rangle
- (\bar{1}11)\langle 0\bar{1}1\rangle
- (\bar{1}11)\langle 101\rangle
- (1\bar{1}1)\langle 101\rangle
- (1\bar{1}1)\langle 011\rangle
- (111)\langle 011\rangle
- (1\bar{1}1)\langle \bar{1}01\rangle
- (\bar{1}11)\langle \bar{1}01\rangle
- (1\bar{1}1)\langle 110\rangle
- (111)\langle 110\rangle
- (\bar{1}11)\langle 0\bar{1}0\rangle
- (\bar{1}11)\langle 0\bar{1}0\rangle

**6x Burger’s Vectors**

- \(\bar{b}_1 = \langle 0\bar{1}1\rangle a / 2\)
- \(\bar{b}_2 = \langle 101\rangle a / 2\)
- \(\bar{b}_3 = \langle 011\rangle a / 2\)
- \(\bar{b}_4 = \langle \bar{1}01\rangle a / 2\)
- \(\bar{b}_5 = \langle 110\rangle a / 2\)
- \(\bar{b}_6 = \langle 0\bar{1}0\rangle a / 2\)

**12x Line Vectors**

- \(\vec{t}_1 = \bar{b}_1 \times \bar{n}_1\)
- \(\vec{t}_2 = \bar{b}_1 \times \bar{n}_2\)
- \(\vec{t}_3 = \bar{b}_2 \times \bar{n}_3\)
- \(\vec{t}_4 = \bar{b}_2 \times \bar{n}_4\)
- \(\vec{t}_5 = \bar{b}_3 \times \bar{n}_5\)
- \(\vec{t}_6 = \bar{b}_3 \times \bar{n}_6\)
- \(\vec{t}_7 = \bar{b}_4 \times \bar{n}_7\)
- \(\vec{t}_8 = \bar{b}_4 \times \bar{n}_8\)
- \(\vec{t}_9 = \bar{b}_5 \times \bar{n}_9\)
- \(\vec{t}_{10} = \bar{b}_5 \times \bar{n}_{10}\)
- \(\vec{t}_{11} = \bar{b}_6 \times \bar{n}_{11}\)
- \(\vec{t}_{12} = \bar{b}_6 \times \bar{n}_{12}\)

**6x Line Vectors**

- \(\vec{t}_{13} = \bar{b}_1\)
- \(\vec{t}_{14} = \bar{b}_2\)
- \(\vec{t}_{15} = \bar{b}_3\)
- \(\vec{t}_{16} = \bar{b}_4\)
- \(\vec{t}_{17} = \bar{b}_5\)
- \(\vec{t}_{18} = \bar{b}_6\)

**Number of Dislocation Types**

- Edge = 12
- Screw = 6

**18 x 2 = 36**

Dislocations of opposite sign needs to be distinguished
Lower-Bound GND Density

\[ \kappa_{ij} = \sum_{t=1}^{N} \left( b_i^t l_i^t - \frac{1}{2} \delta_{ij} b_m^t l_m^t \right) \rho_G^t \]

N = 1 .. 36 – number of possible dislocation types

Burgers Vector

\[ w^t = \| \vec{b}^t \| \| \vec{l}^t \| \]

\[ E_{\text{Screw}} = (1 - \nu) E_{\text{Edge}} \]

6 known lattice curvatures (measured)

36 possible dislocation types

1,947,792 possibilities!

Not all dislocation types are equally energetically favourable.

\[ \rho_G = \sum_{t=1}^{36} \rho_G^t \approx \min \sum_{t=1}^{6} \rho_G^t \]

\[ \sum_{t=1}^{6} w^t \rho_G^t = \min \]

With a set of 6 linear equations and 36 unknowns, a large number of possible solutions exists whereby a unique solution cannot be obtained. It is therefore necessary to constrain the solution using physically-based constraints.

Edge Dislocations: 12
Screw Dislocations: 6
Dislocation Types: 2 x 18 = 36
dislocations of opposite sign needs to be distinguished

Simplex Optimisation Algorithm

all possible solutions

optimal solution along the edge

optimal solution

initial guess
Density of geometrically-necessary dislocations (GND, $\rho_G$) maps calculated from the EBSD-measured Euler Angles ($\phi_1, \Phi, \phi_2$) for specimens with 0% (as-received), 7.8% and 13.9% of imparted plastic strain.

- Step size (h) = 200 nm
- Magnification = 153x
- Discrete measurements provide information on spatial distribution of GND across the microstructure.
- GNDs arrange themselves into energetically favourable configurations forming geometrically-necessary boundaries (GNBs) subdividing grains into the sub-grains.
GND Spacing

⇒ Spacing between geometrically-necessary dislocations (GND, $d_G$) recalculated from the GND density ($\rho_G$) for specimens with 0% (as-received), 7.8% and 13.9% of imparted plastic strain.

⇒ Non-uniform distribution of GNDs in the microstructure as GNDs arrange themselves into energetically favourable configurations subdividing grains into the sub-grains.

$$d_G = \frac{1}{\sqrt{\rho_G}}$$

Dislocation spacing

GND Density

$\rho_G$
Density of geometrically-necessary dislocations (GND, $\rho_G$) calculated from the EBSD-measured Euler Angles ($\phi_1, \Phi, \phi_2$).

Spacing between geometrically-necessary dislocations (GND, $d_G$) recalculated from the GND density ($\rho_G$).

⇒ Step size (h) = 20 nm
⇒ Magnification = 1000x
GND Density

Density of geometrically-necessary dislocations (GND, $\rho_G$) maps calculated from the EBSD-measured Euler Angles ($\phi_1, \Phi, \phi_2$).
⇒ Distribution (histogram) of discrete GND density ($\rho_G$) measurements for specimen with 0% (as-received), 7.8% and 13.9% of imparted plastic strain ($\varepsilon_p$).

Log-Normal Distribution

$$f(\rho_G | \mu, \sigma) = \frac{1}{x\sigma\sqrt{2\pi}} \exp\left(-\frac{(\ln(\rho_G) - \mu)^2}{2\sigma^2}\right)$$

Mean & Variance

$$m(\rho_G) = \exp\left(\mu + \frac{\sigma^2}{2}\right)$$

$$v(\rho_G) = \exp(2\mu + \sigma^2)(\exp(\sigma^2))$$

⇒ The variance of the GND density distribution describes the heterogeneity of the GND distribution across variously oriented grains within the microstructure, the mean can be then taken as the microstructure-averaged (bulk) GND density.
The development of the mean GND density as a function of number of analysed grains in GND density maps for specimen with 0% (as-received), 7.8% and 13.9% of imparted plastic strain ($\varepsilon_p$).
The development of the mean GND density distribution as a function of imparted plastic strain ($\varepsilon_p$) for all tested specimens.

The development of the variance of GND density distribution as a function of imparted plastic strain ($\varepsilon_p$) for all tested specimens.
GND Types in Solution

⇒ Map showing the ratio of screw dislocations to the total number of dislocations in the solution (6) for the specimen with 13.9% imparted plastic strain.

⇒ Screw dislocation ratio as a function of imparted plastic strain for all tested specimens.

⇒ The uniqueness of the solution is not guaranteed.
⇒ Only pure edge and pure screw dislocations have been considered in the calculation.
HRSD Measurements
HRSD Set-Up

- Detector: 2048 × 2048 pixels
- Pixel Size: 200 × 200 μm
- Distance: 1873 mm

⇒ High-resolution synchrotron diffraction (HRSD) set-up at 1-ID high-energy beamline at the Advanced Photon Source (APS), Argonne National Laboratory (ANL).
Diffraction Peak Broadening

⇒ The total diffraction peak shape (which includes peak broadening) $I_{\text{TOTAL}}$ of a is the convolution of the shape contribution caused by the size of coherently scattering domains (sub-grains) $I_{\text{SIZE}}$ and the contribution caused by strain fields of present dislocations $I_{\text{STRAIN}}$.

⇒ Convolution is defined as the inverse Fourier transform of the product of the individual Fourier transform of the components.

$$I_{\text{TOTAL}} = I_{\text{SIZE}} \ast I_{\text{STRAIN}} = \mathcal{F}^{-1}(A_{\text{Size}} A_{\text{Strain}})$$

$$A_{\text{Size}} = \mathcal{F}(I_{\text{SIZE}}) \quad A_{\text{Strain}} = \mathcal{F}(I_{\text{STRAIN}})$$

Size (sub-grain) contribution to the peak shape.

Strain (dislocation) contribution to the peak shape.
Diffraction Peak Broadening

⇒ The broadening due to the size of the coherently diffracting domains (sub-grains) is the same for all hkl diffraction peaks, while the broadening component due to the strain field of present dislocations varies between diffraction peaks. This variation in the strain (dislocation) broadening is not monotonous due to the anisotropic behaviour described by the dislocation contrast factors.

- $\langle X \rangle_A = 60 \text{ nm}$
- $\rho_T = 21E+14 \text{ m}^{-2}$
- $q = 2$
- $M = 1.6$

Williamson-Hall Plot

- Simulation
- Size (Sub-Grain) Peak Broadening
- Strain (Dislocation) Peak Broadening
- Combined Size & Strain Peak Broadening

Comparison of full diffraction patterns for specimens with 0% (as-received) and 13.9% of imparted plastic strain. The different behavior of size (sub-grain) and strain (dislocation) peak broadening can be resolved if many peaks are available.

The diffraction peak broadening was analysed using the eCMWP (extended Convolutional Multiple Whole Profile) LPA software.
Total dislocation density ($\rho_T$) and size of the coherently scattering domains (SCDs) obtained by line profile analysis (LPA) of HRSD patterns as a function of imparted plastic strain ($\varepsilon_p$) - open symbols represents individual measurements along the sample loading axis, and solid symbol represents the mean values.
EBSD + HRSD Measurements
EBSD- & HRSD- Measured Dislocation Density

⇒ Comparison of the HRND-measured total dislocation density (\( \rho_T \)) and the EBSD-measured density of GNDs (\( \rho_G \)), together with expected dislocation densities calculated using the modified Taylor’s model, and single-slip Ashby’s model.

⇒ Both GNDs and SSDs contribute to the work-hardening of the material.
⇒ SSDs represent more than 80% of all the present dislocations.
Comparison of the HRSD-measured size of CSDs (red circles) with EBSD-measured spacing of GNDs ($d_G$) (blue squares), and the estimated minimum size of CSDs (green triangles) from EBSD-measured density of GNDs ($\rho_G$).

$\langle X \rangle_A \geq \langle X \rangle_{A,\text{min}} = \frac{2}{3} \frac{1}{\Delta x \rho_G} = \sqrt{\frac{2}{3} \frac{d_G^2}{\Delta x}}$

$\rho_G \geq \frac{2}{3} \frac{1}{\Delta x \langle X \rangle_A}$

This defines the connection between EBSD-measured $\rho_G$ and HRSD-measured $\langle X \rangle_A$, one can then estimate $\rho_G$ from $\langle X \rangle_A$. 

GNDs spacing

EBSD step size

GNDs density

Size of SSDs

EBSD step size

Size of SSDs
Conclusions

⇒ EBSD measures the lower-bound $\rho_G$, while HRSD measures $\rho_T$.
⇒ The minimum detected $\rho_T$ measured by HRSD is about $1E13 \text{ m}^{-2}$, while the minimum $\rho_G$ measured by EBSD is about $2E12 \text{ m}^{-2}$.
⇒ EBSD is more sensitivity to the small amount of plastic deformation in the material, while HRSD gets more accurate with higher amount of plastic deformation.
⇒ There is a connection between EBSD-measured $\rho_G$ and HRSD-measured size of CSDs ($\langle X \rangle_A$).
⇒ EBSD = Density of GNDs ($\rho_G$), + estimate the minimum Size of CSDs
⇒ HRSD = Total Dislocation Density ($\rho_T$), size of CSDs ($\langle X \rangle_A$), + estimate of minimum density of GNDs ($\rho_G$)

$$\langle X \rangle_A \geq \langle X \rangle_{A,\text{min}} = \sqrt[3]{\frac{2}{3}} \frac{1}{\Delta x \rho_G} = \sqrt[3]{\frac{2 d_G^2}{3 \Delta x}}$$
Thank you for your time and interest in this work. We hope you will find it useful.