MSA Short Course
Neutron Scattering in Earth Sciences

Acknowledgements:
Mineralogical Society of America
DOE-BES
Lujan Center, LANSCE
Spallation Neutron Source
COMPRES-NSF
Thursday
7:30 a.m. Registration and continental breakfast
10:25 Coffee break
12:00 – 1:00 Buffet lunch
3:00 Coffee break
6:30 Reception with cash bar
7:30 Banquet

Friday
7:30 a.m. Continental breakfast
9:50 Coffee break
12:00 – 1:00 Buffet lunch
3:00-3:15 Coffee break
5:00 Adjourn
Dedicated to James D. Jorgensen 1948-2006
Texture Analysis with Neutron Diffraction

Rudy Wenk
Dept. Earth and Planetary Science, UC Berkeley
• What are textures?
• Representation of textures.
• How do textures form?
• Texture measurements with neutron diffraction
  – Monochromatic
  – TOF
  – HIPPO
• Texture calculations
  – Pole figures
  – Rietveld
Applications

• Phase transformations / Variant selection
  – Iron (bcc – fcc)
  – Ice
  – Quartz (trigonal – hexagonal)

• Geological applications
  – Mechanical twinning in quartz: a paleopiezometer
Representation of Preferred Orientation

- Orientation Distribution Function (ODF)
- Pole Figures
- Inverse Pole Figures
(100) indicated by location of boats
(010) indicated by color and heading

Equal Area Projection
Orientation sphere to define three Euler angles
How do textures form?

- Growth (Topotaxy, epitaxy, temperature gradient, stress field, magnetic field etc.)
- Deformation (Slip, twinning, grain shape)
- Recrystallization
- Phase transformations
Rigid particles in viscous matrix: Jeffery 1923, March 1932

Compression $\varepsilon_i = \rho_i^{-1/3} - 1$

Compaction $\varepsilon_c = \rho_{max}^{-1/2} - 1$
Crystal rotations during deformation by slip
Cold-rolled titanium
Upper Bound Theories (compatibility)

Homogeneous deformation of a microstructure with originally square-shaped grains

Lower Bound Theories (equilibrium)

Favorably oriented grains deform first: grains overlap, gaps form

Homogeneous deformation: grain boundaries remain intact
Recrystallization: Modification of Texture

Strain energy is reduced by:

- **Growth** of relatively undeformed grains by grain boundary migration.
- **Nucleation** of new domains in highly deformed regions.
Texture measurements
Neutron scattering
Neutron
Monochromatic
Bragg’s Law:

1) \( 2d \sin \theta = n \lambda \),

2) reflection on lattice planes
GPPD-IPNS: Kappa Goniometer
0001 Pole figure of calcite measured at GKSS
Quartzite 0001: U-stage – Neutron diffraction
Calcite marble: X-ray – Neutron diffraction
ILL D1B
ILL D1B, stack of spectra, limestone
Neutron
TOF
N1-2;qz;400C;s=-3
Bank no. = 1 Two-theta = 89.53

Mouse (keyboard): Left(H) – Height, Right(W) – Location Both(X) – exit
Dubna
UCMRD (University of California Materials Research Diffractometer) or HIPPO (High Pressure Preferred Orientation) at the Lujan Center at LANSCE
HIPPO: Stacks of diffraction spectra for deformed limestone
Relative intensity differences indicative of texture

Simultaneous analysis of 384 spectra (48 detectors x 8 rotations)
with the Rietveld method
2D Multiplot for 150° bank omega -61.7
measured data only

2D Multiplot for 90° bank omega -61.7
measured data only

2D Multiplot for 40° bank omega -61.7
measured data only

150 deg bank

90 deg bank

40 deg bank
HIPPO Automatic Sample Changer
Rietveld Analysis
with MAUD

(Materials Analysis Using Diffraction by Luca Lutterotti)
How to get ODF?

- from individual orientations
- from pole figures
- from diffraction spectra
Conventional method: from pole figures to ODF

New approach: from diffraction spectra to ODF
Limestone Standard: Refining 256 Spectra Simultaneously for Texture and Structure with the Rietveld Method (MAUD)

Lutterotti et al. 2002
What influences the spectrum?

- Instrumental features (wavelength etc.)
- Crystal structure (lattice, atomic positions)
- Microstructure (size, strain)
- Texture (ODF)
Cycle 1: scale factors and background
Cycle 2: previous + detector distance
Cycle 3: previous + texture
Pole figures for round robin limestone standard

HIPPO Limestone standard, e-WIMV 10 deg

HIPPO measured in 20 minutes, ILL in 4 hours

ILL D20 Limestone standard, e-WIMV 10 deg
Two approaches
1) Harmonic Method (Fourier approach)
   Termination errors, odd coefficients
1) Direct Methods (Tomography)
   WIMV, Entropy etc.
ECAP aluminum

L=12
L=14
L=16
WIMV
Data Quality

- Comparison with other techniques (neutron-electron)
- Internal consistency (observed-recalculated)
- Round Robin
BRC420 pole figures from electron/neutron diffraction

1335 max.

1 m.r.d.

1 min.

log. scale
equal area proj.
Round Robin limestone
Advantages of neutrons

Low absorption / high penetration:
- bulk samples (not surfaces)
- large samples (coarse grained)
- environmental stages

High spectral resolution:
- low symmetry materials (e.g. minerals, HTS, Pu)
- Composites (rocks, metal matrix etc.)

Scattering power:
- Be, D, D₂O, Al-Si
Neutron Diffraction for Texture and Strain Analysis

**Texture**
- Geesthacht (monochromatic)
- ILB Saclay (monochromatic)
- ILL D1B, D19 and D20 (monochromatic, banana)
- IPNS GPPD (TOF)
- LANSCE HIPPO (TOF)
- Dubna SKAT (TOF)
- ISIS SXD (TOF)

**Strain**
- Chalk River (monochromatic)
- Dubna EPSILON (TOF)
- IPNS GPPD (TOF)
- LANSCE SMARTS (TOF)
- ISIS ENGIN-X (TOF)
- Geesthacht (monochromatic)
Iron

bcc – fcc – bcc
Fe

Kurdjumov-Sachs

Burgers
Phase transformations in iron

Kurdjumov-Sachs 1934:

\{110\}<111> \text{bcc} \quad \{111\}<110> \text{fcc}

\{110\} is the densest packed plane in bcc, \{111\} is close-packed plane in fcc, \langle111\rangle (bcc) and \{110\} fcc are closest-packed directions

Burgers 1934:

\{110\}<111> \text{bcc} \quad \{0001\}<11-20> \text{hcp}

\{110\} is the densest packed plane in bcc, \{0001\} is close-packed plane in hcp, \langle111\rangle (bcc) and \{11-20\} hcp are closest-packed directions
ULC Iron, ESRF, June 2004
ULC steel, in situ neutron diffraction with HIPPO (LANSCE)
Wenk, Huensche, Kestens Trans. Mat. 2006
24 variants

KS

12 variants

NW

3 variants

Bain
ICE
NCD at Lujan

Bennett, Wenk, Durham and Stern (1997) Phil Mag. A76
Quartz

trigonal – hexagonal – trigonal

Texture Memory
Low quartz ($\alpha$), trigonal

High quartz ($\beta$), hexagonal
Young’s modulus for quartz
Sci 293 Quartzite mylonite: Texture memory
Mechanical Dauphiné twinning in quartz
Quartz in compression (Tullis, 1970)

Inverse Young’s modulus for quartz
Quartz (Novaculite): IPF before and after heating to 650°C, **no texture memory**

Tullis 200°C before phase transformation
Neutron diffraction with HIPPO

max=1.94
min=0.30

Tullis 200°C after phase transformation

max=1.02
min=0.99
Geological applications
Mechanical twinning in Quartz
In SITU Stressing

SMARTS, ENGIN-X
N1−2;qa;400C;s=−3
Bank no. = 1 Two-theta = 89.53

Mouse (keyboard): Left(H) – Height, Right(W) – Location Both(X) – exit
\[ I = N \left\{ (1+m) F_{h0l}^2 + (1-m) F_{0hl}^2 \right\} \]
$100\% = N \left( F_{h0l}^2 + F_{0hl}^2 \right)$
Neutron Diffraction

Main advantages:

• Low absorption (bulk samples, good statistics vs. EBSD, environmental cells: P, T, \( \sigma \))
• High spectral resolution for composites and low symmetry compounds, no defocusing (Rietveld method)

Main disadvantages:

• Weak scattering
• Complex data processing
• Limited access
Conclusions

• Neutron diffraction an increasingly used method for quantitative texture analysis.

• Neutron diffraction for in situ experiments $p$, $T$, $\sigma$.

• Time-resolved experiments to investigate kinetics.

• Neutron diffraction to determine residual strain.

• An exciting prospect for students in earth sciences.