Texture by Rietveld Refinement

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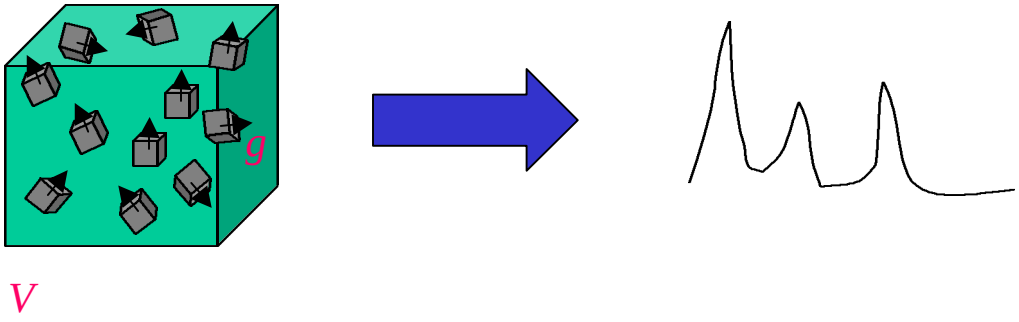
- H. Ledbetter (MSEL, NIST)

Outline

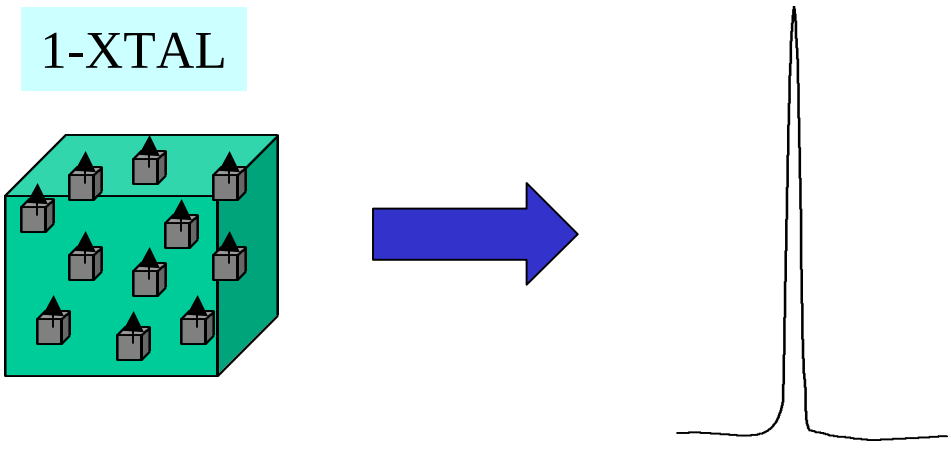
- Texture?
- Texture in RR:
  - Correction for
  - Determination of
- Why RR?
- Examples
  - Is the determination of texture important?
    - Residual stress
    - Phase analysis

Texture

RANDOM

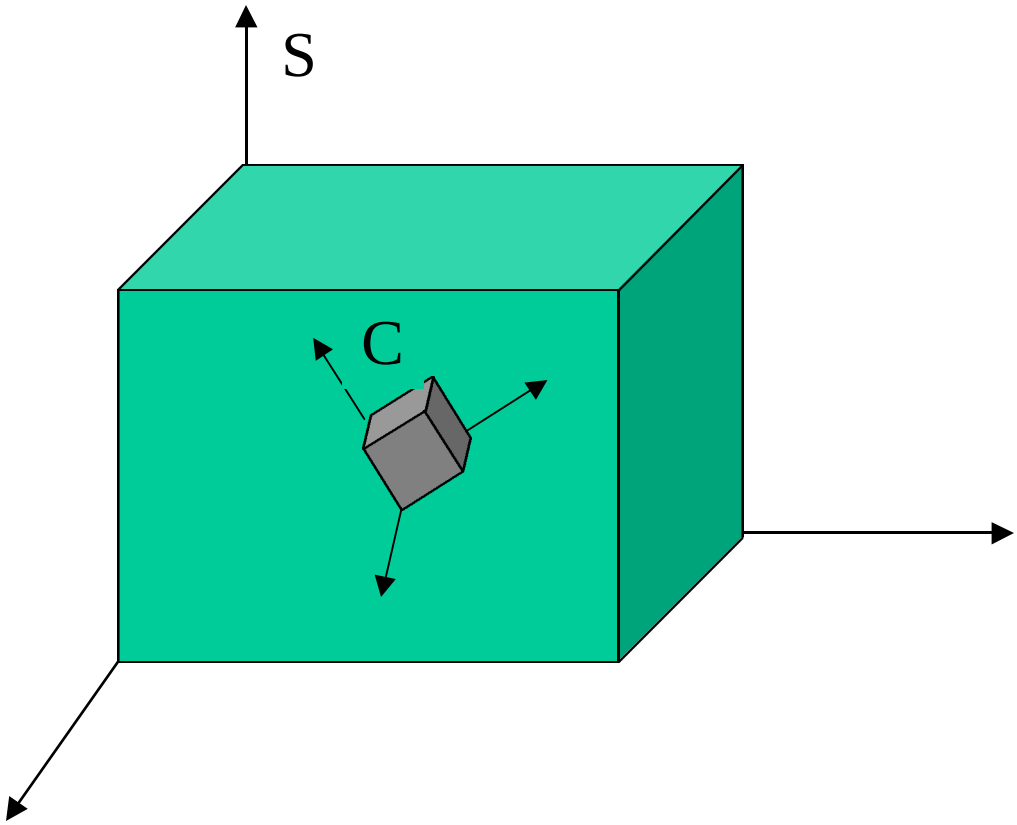


1-XTAL



$f(g) = dV/V/dg$

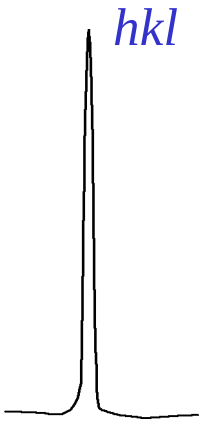
Texture Description



$$f(\varphi_1,\varphi_2,\varphi_3) = \sum_{l=0}^{\lambda}\sum_{m=-l}^{l}\sum_{n=-l}^{l} W_{lmn} Z_{lmn}(\cos\varphi_2) e^{-im\varphi_1} e^{-in\varphi_3}$$

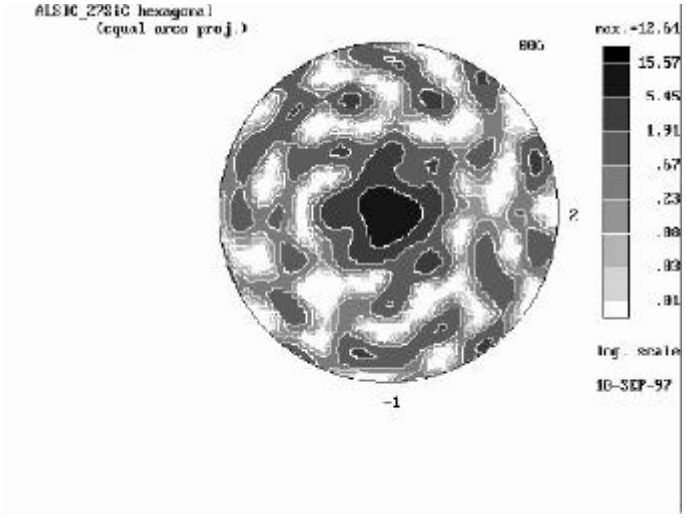
Euler angles φ_1, φ_2, φ_3

Intensity Measurements



- I (orientation):

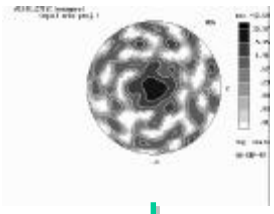
$$I(\psi,\phi) = \sum_{l=0}^{\lambda} \sum_{m=-l}^{l} Q_{lm} P_l^m(\psi) e^{-im\phi}$$



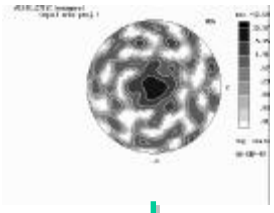
- Pole figure

Preferred orientation

Texture Calculation



+

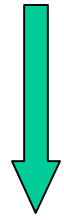


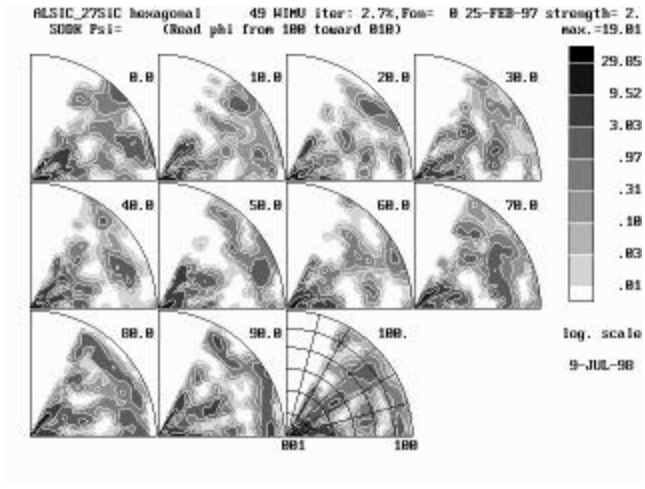
+

.....

- Multiple pole figures for different *hkl*

$$Q_{lm}$$





- ODF

$$W_{lmn}$$

Texture in RR

- Correction for a nuisance
  - Rietveld-Toraya:

$$P = G_2 + (1 - G_2)\exp(-G_1 \alpha^2)$$

  - March-Dollase:

$$P = (G_1^2 \cos^2\alpha + (1/G_1)\sin^2\alpha)^{-3/2}$$

Preferred-orientation correction

- Multiple preferred orientations!

- Correction and determination
  - GSAS (Von Dreele)

Harmonic method

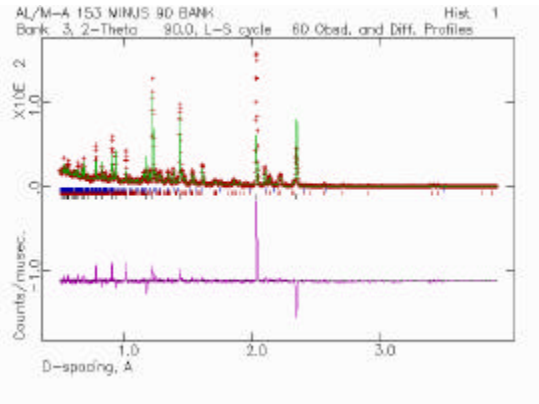
(W_{lmn} refinement)

  - RTA (Wenk)

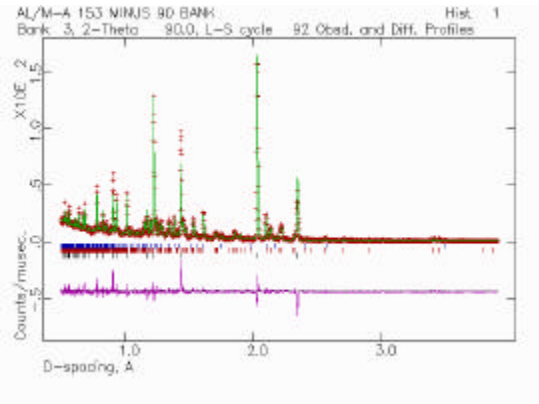
WIMV method
(Williams-Imhof-Matthies-Vinel)

(2-step)

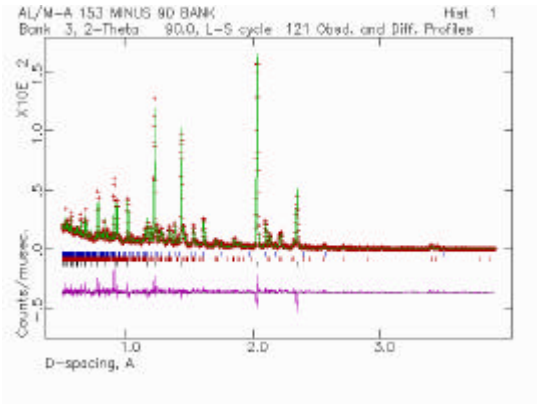
Comparison of M-D and S-F Texture Corrections



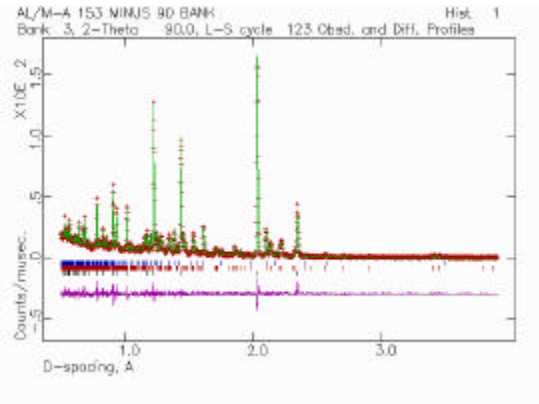
NO TEXTURE CORRECTION
R_{wp} = 31.9 %



[100] M-D CORRECTION
D = 8, 30 C, R_{wp} = 19.3 %



0.9 [100]+ 0.1 [110] M-D CORRECTION
D = 8, 30 C, R_{wp} = 17.7 %



S-F CORRECTION (CYL,L=14)
7 TERMS, D = 0, 1 C, R_{wp} = 16.4 %

Why Rietveld Refinement ?

- Harmonic method
  - Stable refinement (exact solution!)
  - Correction for texture even for 1 pattern
- When is recommended for the texture determination?
  - Low symmetries
  - Multiphase mixtures
  - Residual stress
- Complete diffraction pattern
  - Monochromatic sources
    - Position-sensitive detectors
    - Section of the pattern
  - Polychromatic sources
    - Neutron TOF
    - EDXRD

$BaTiO_3$ Surface Layer

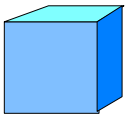
- Good ferroelectric

MELT

1618°C

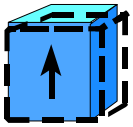
Hexagonal?

Cubic (Pm3m)



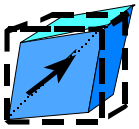
T_c 120°C

Tetragonal (P4mm)



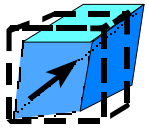
5°C

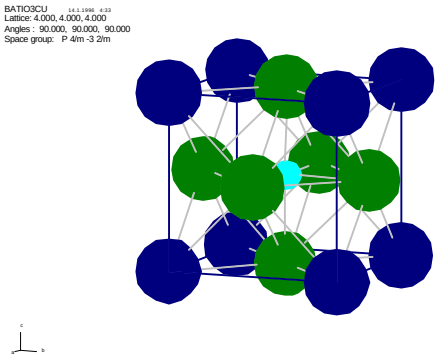
Orthorhombic (C2mm)

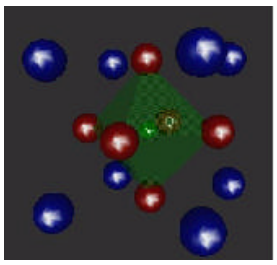


-90°C

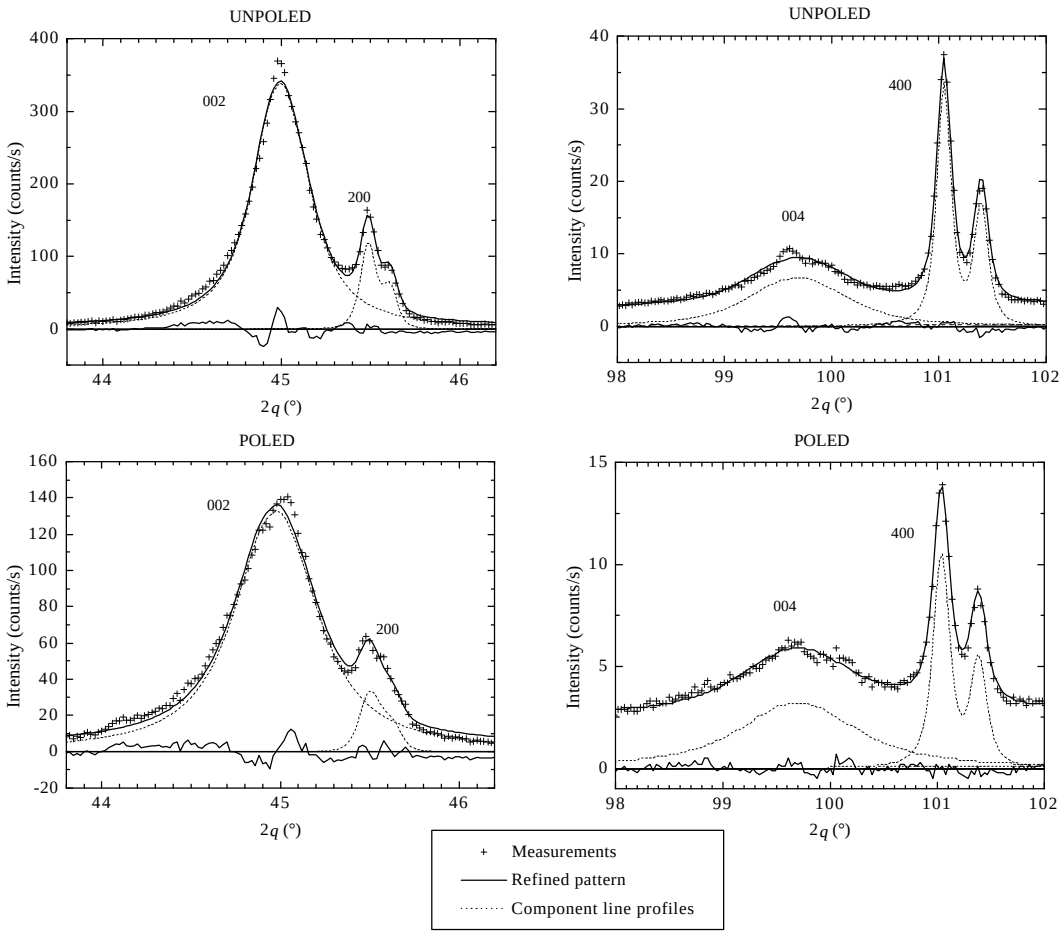
Rhombohedral (R3m)



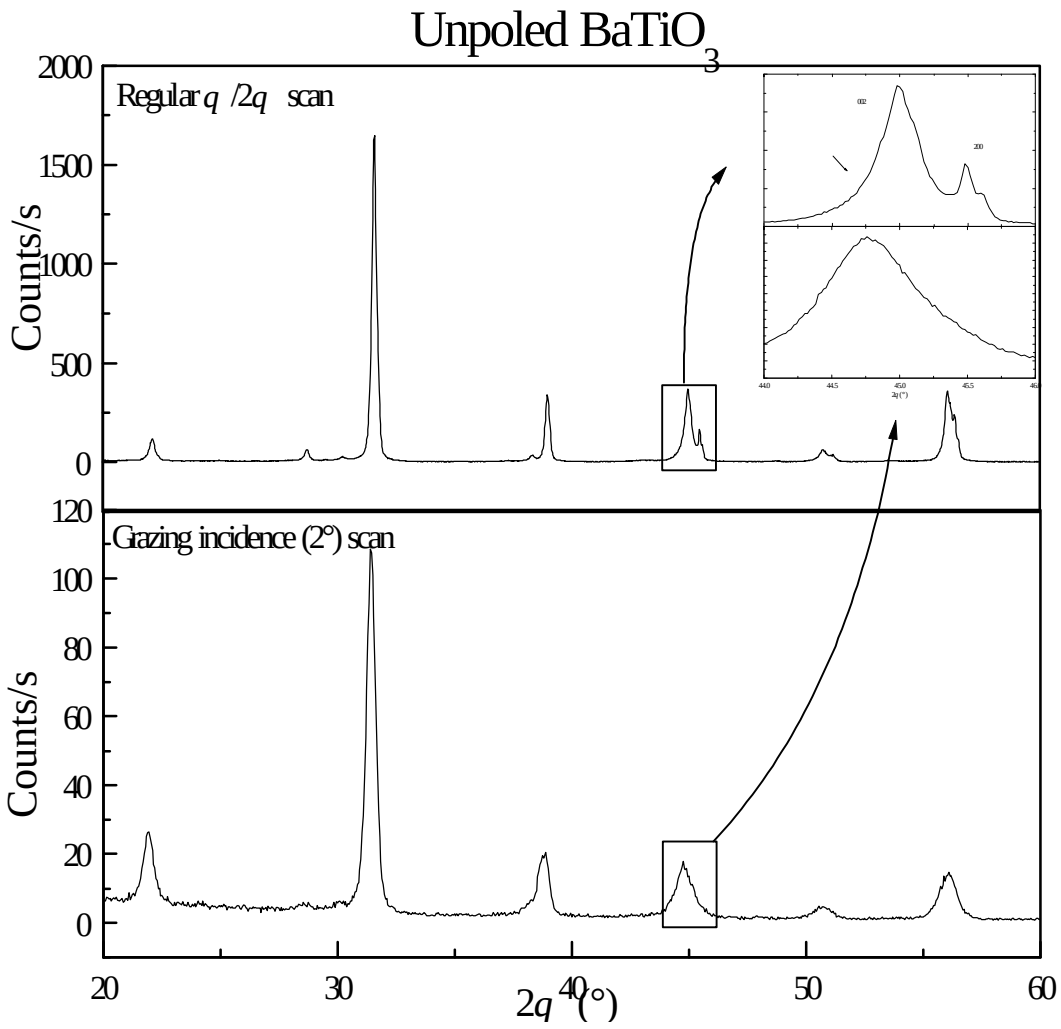




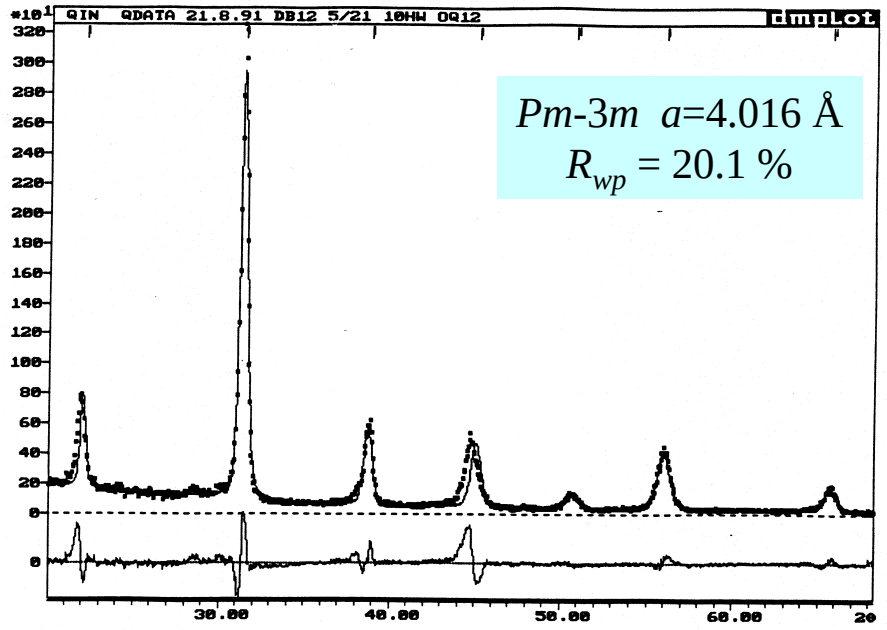
X-Ray Diffraction Patterns



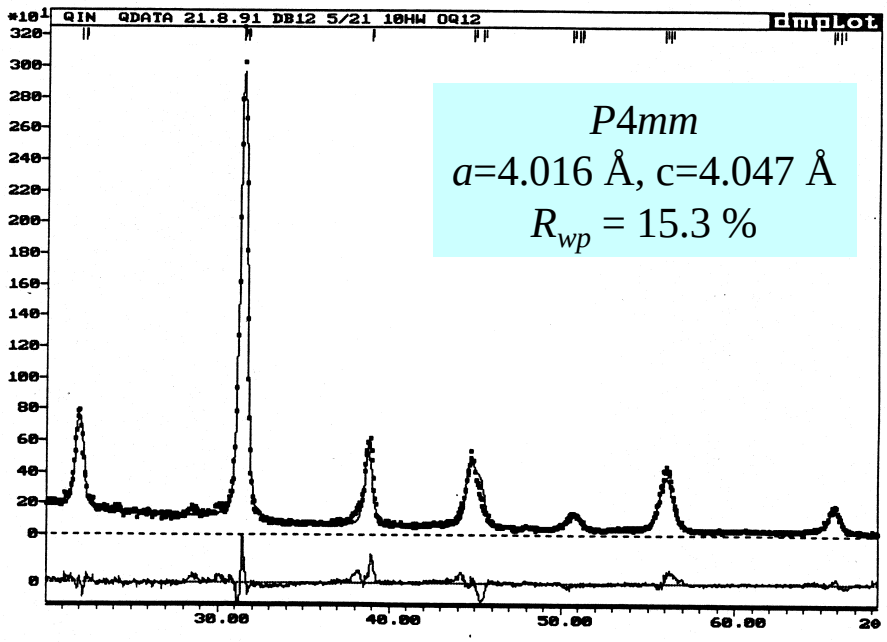
Grazing-Incidence Scan



Rietveld Refinement of the Grazing-Incidence Scan

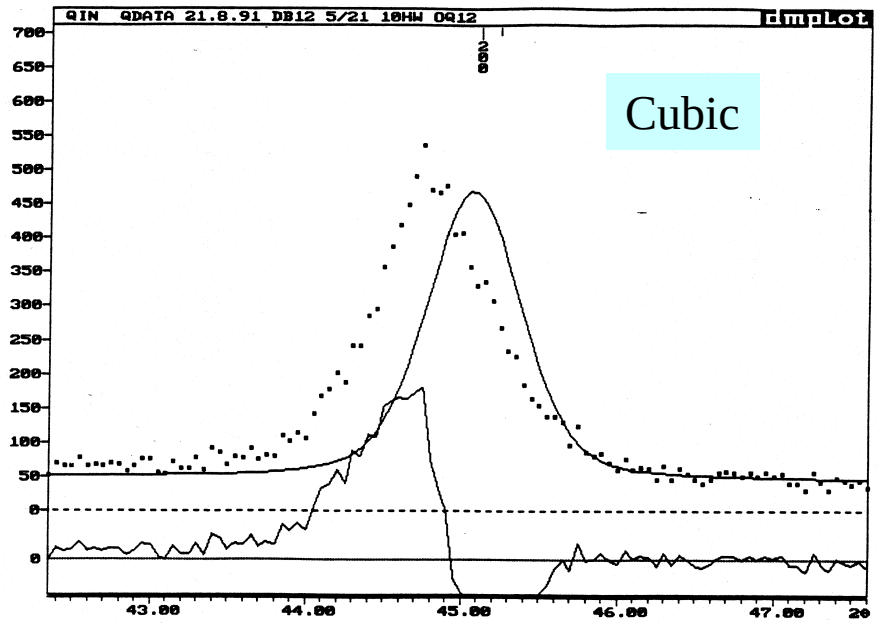


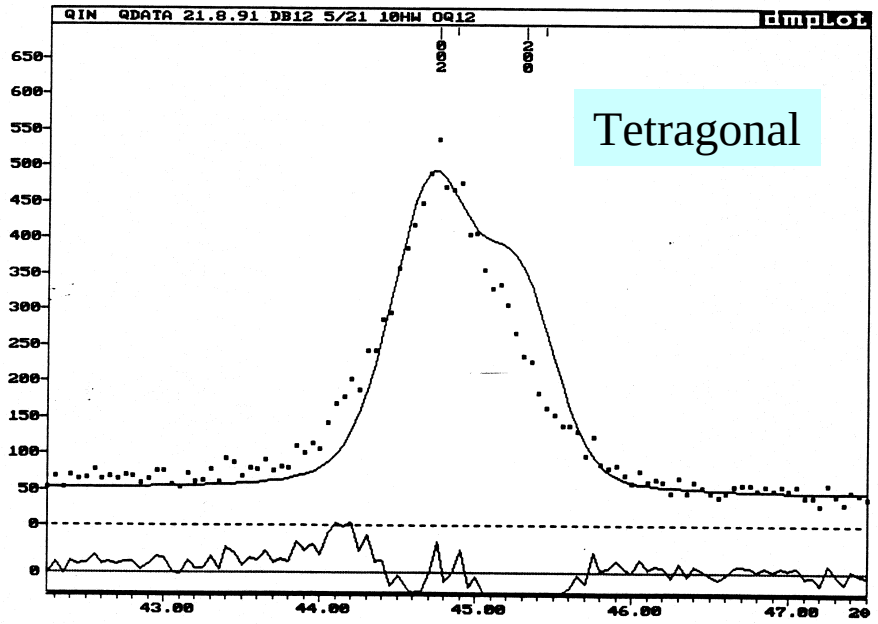
Pm-3*m* a=4.016 Å
R_{wp} = 20.1 %



*P*4*mm*
a=4.016 Å, c=4.047 Å
R_{wp} = 15.3 %

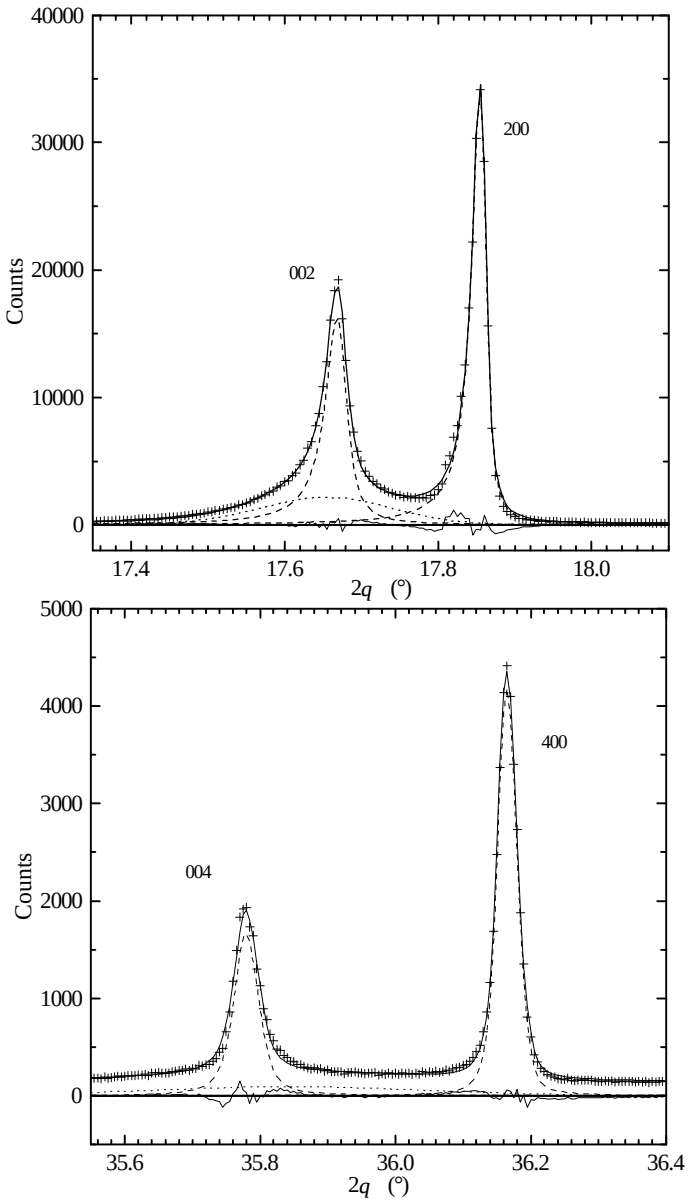
Enlarged View



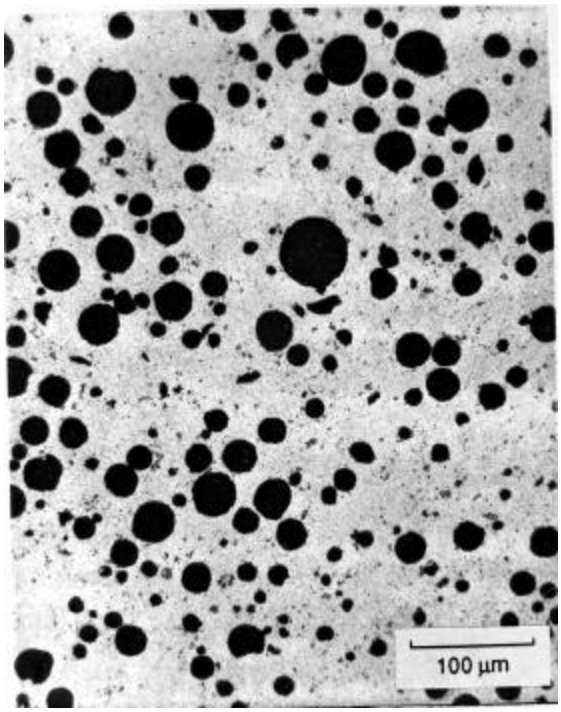


Synchrotron Data at 0.62 Å

NSLS X3B1 Data



Al/Mullite-Alumina Composites



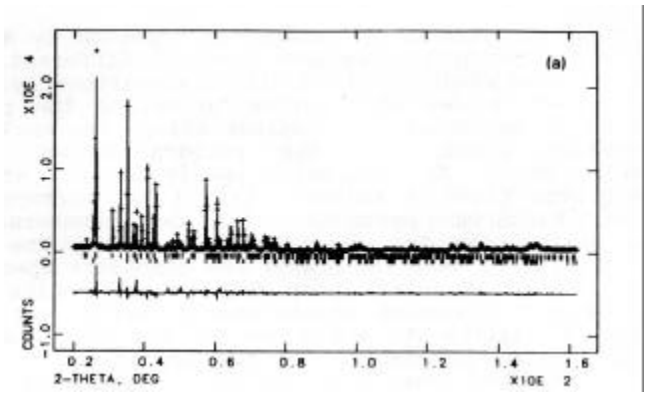
- Al-6061 matrix

STRONG TEXTURE

- α-alumina

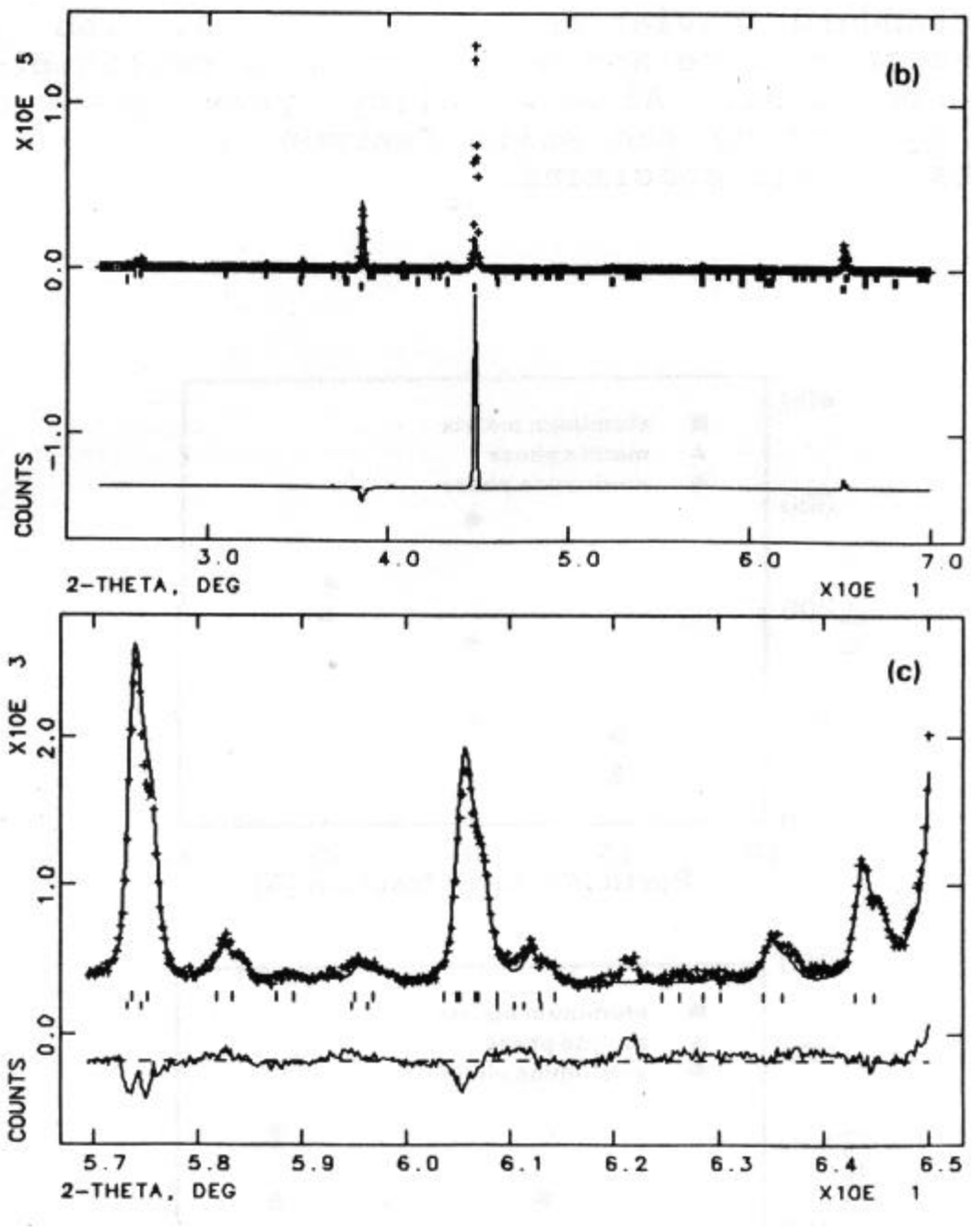
TEXTURE ?

- Mullite
  - $Al_2(Al_{2+2x}Si_{2-2x})O_{10-x}$
  - *Pbam* ($a \approx b \approx 7.6$ Å, $c \approx 2.9$ Å)
  - Incommensurate modulation



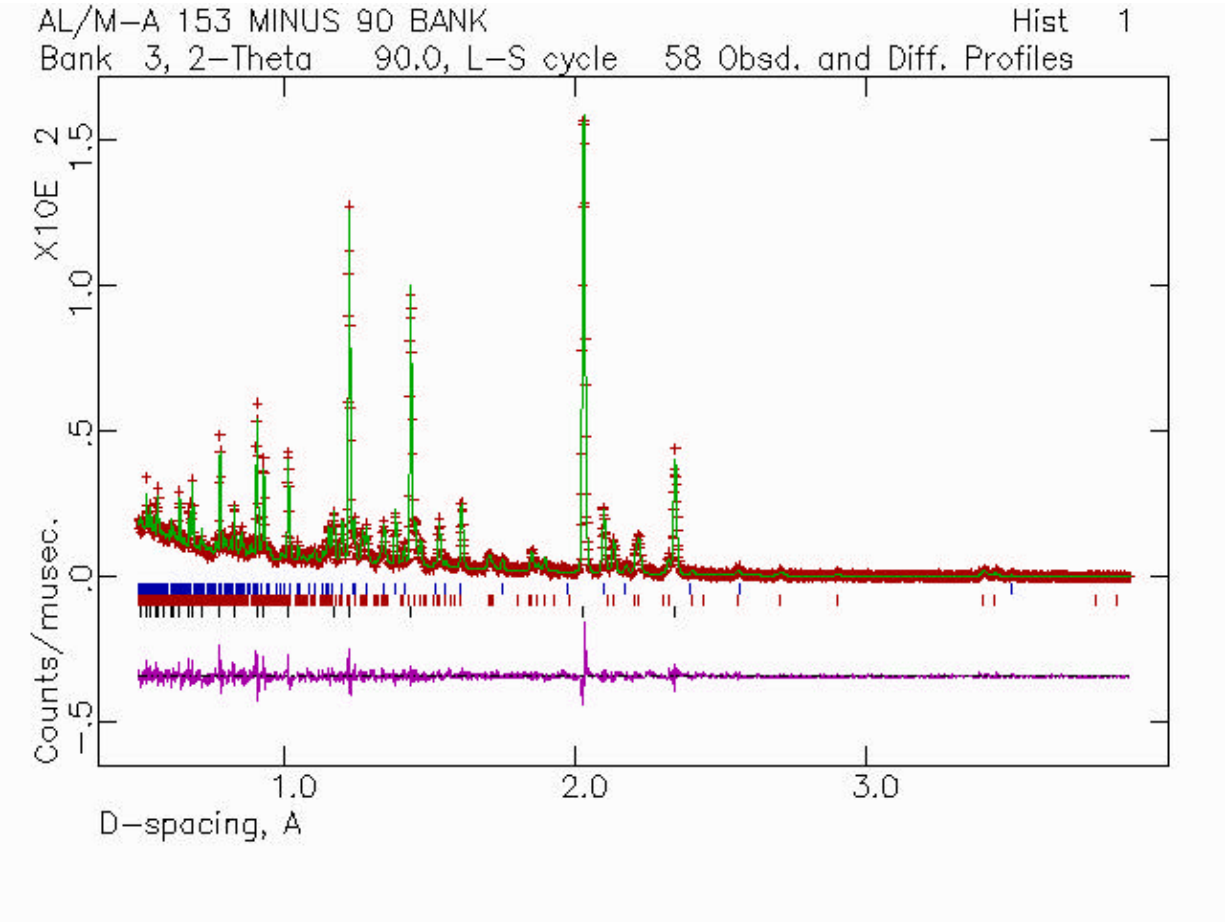
TEXTURE ?

X-Ray Diffraction



TOF Neutron Diffraction

IPNS (ANL) Measurements



Influence of Texture on a Stress State in Material

- Diffraction → STRAIN

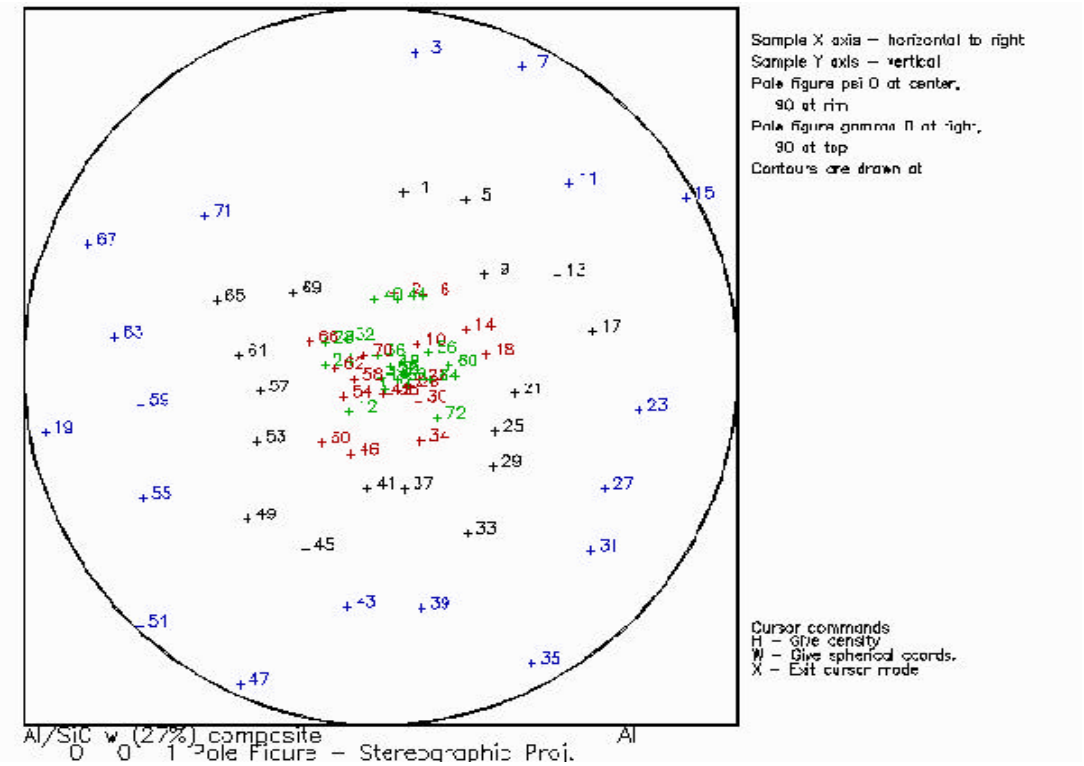
TEXTURE

Strain → Stress

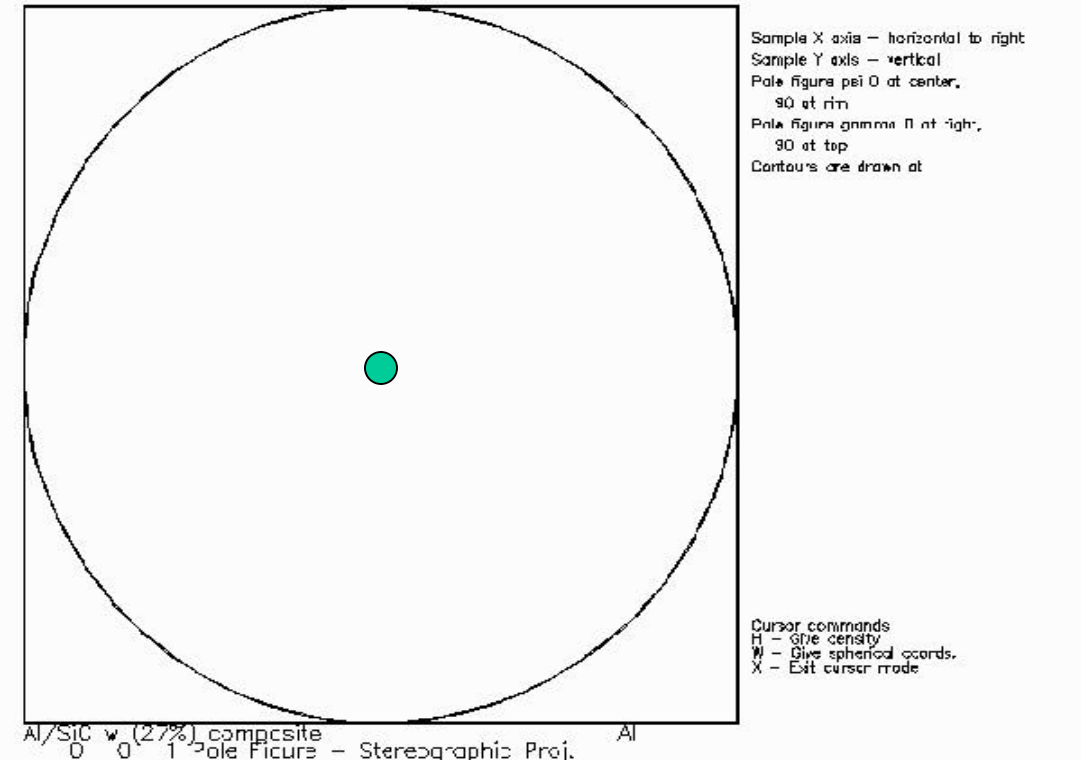
$$\sigma_{ij} = C_{ijkl}\, e_{kl}$$

- Texture and strain from the same data:
  - 13-18 specimen orientations
  - 4 patterns / orientation 52-72 patterns
  - Rietveld refinement:
    - > 250,000 data points !
    - > 1,800 refinable parameters !

Measurements at Different Specimen Orientations

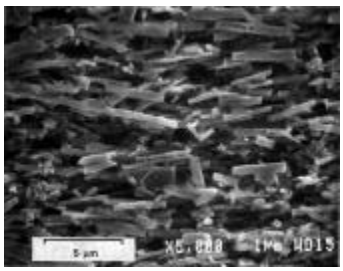


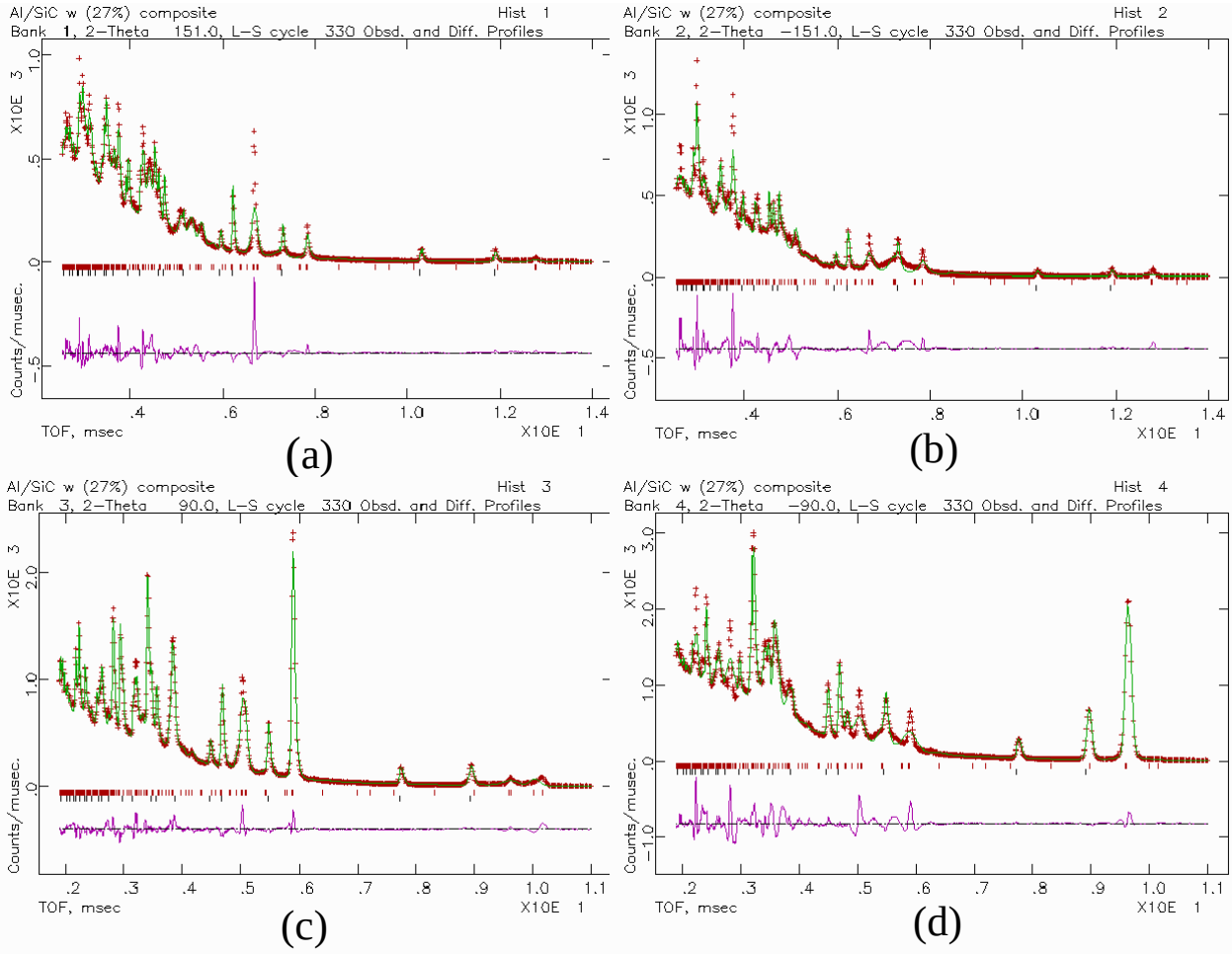
Coupled /2 Scan



LANSCE (LANL) Measurements

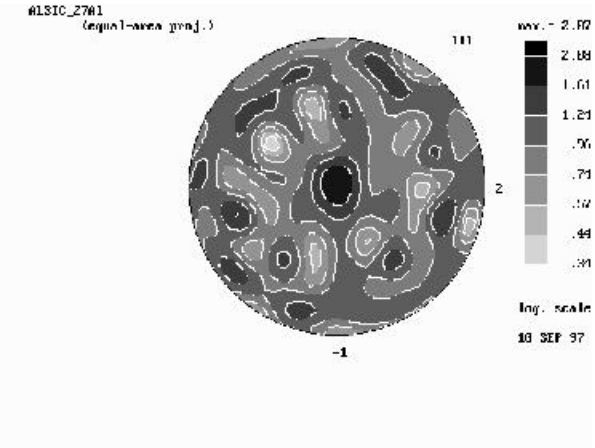
Al (6061)/SiC_w (hexagonal) — extrusions

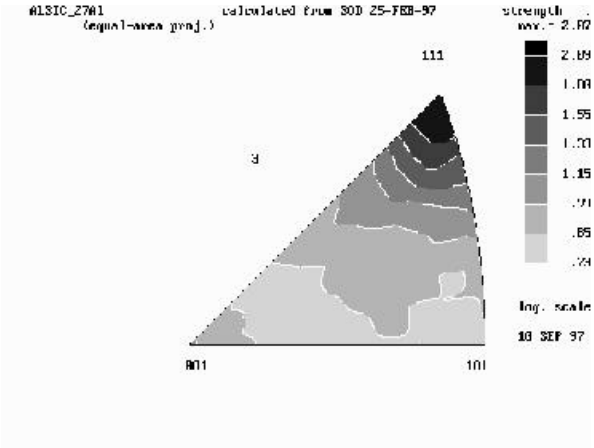


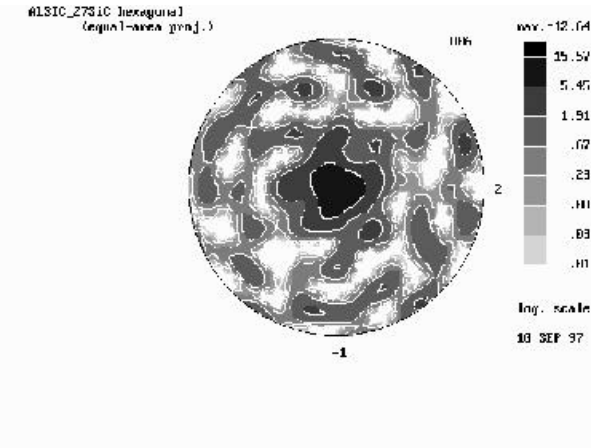


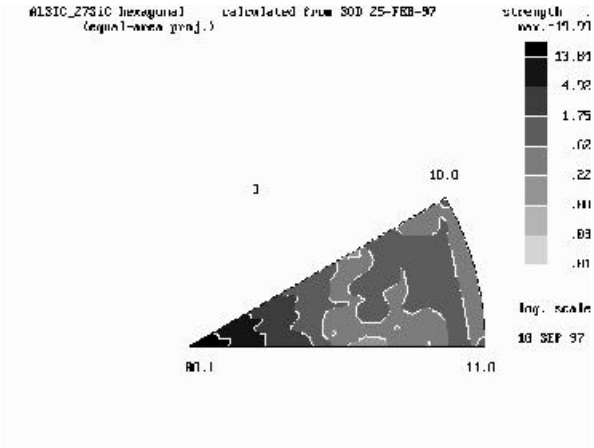
Pole Figures

W_{lmn} → Q_{lm} → Pole Figures

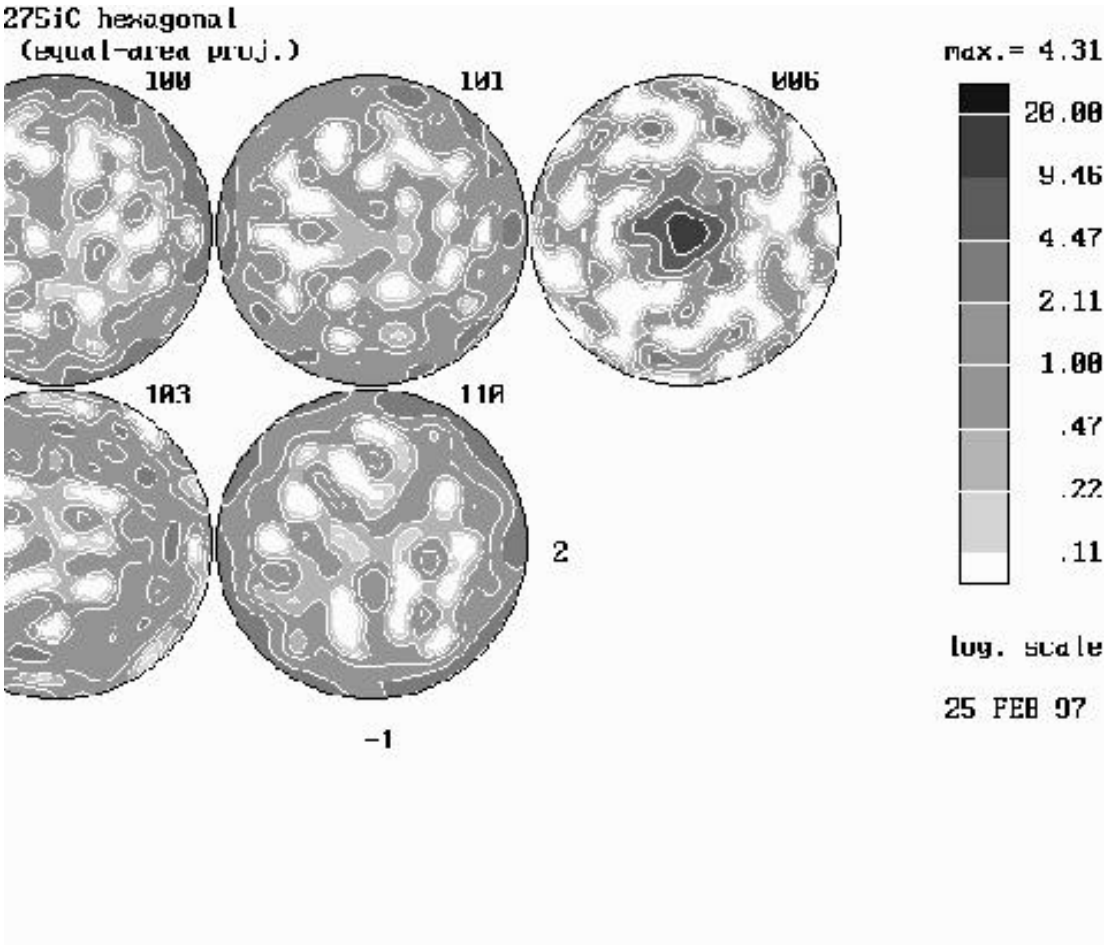




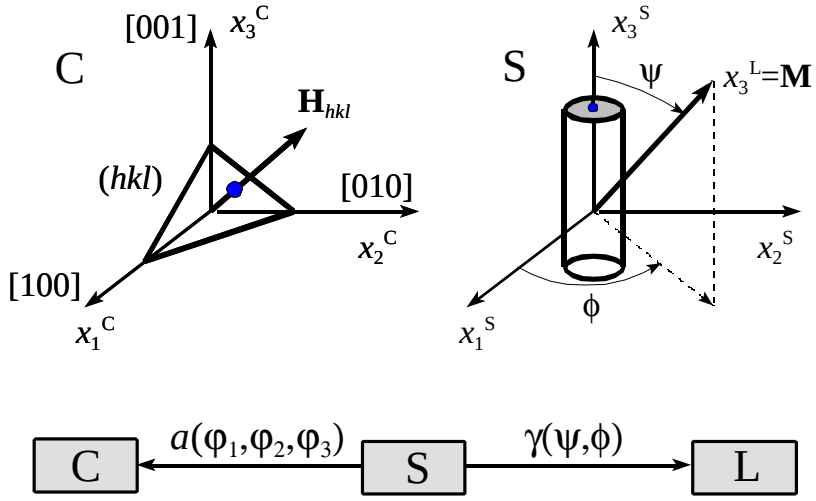




Any Pole Figure is Constructed



Strain Determination



$$e_{\psi\phi} = e_{33} + [e_{11}\cos^2\phi + e_{12}\sin 2\phi + e_{22}\sin^2\phi - e_{33}]\sin^2\psi + [e_{13}\cos\phi + e_{23}\sin\phi]\sin 2\psi$$

6 unknowns: over-determined system, least-squares

(Balzar, Von Dreele, Bennett & Ledbetter,
Journal of Applied Physics, in press)

Complete Strain Tensor Al/SiC_w Composite

Strain (10^{-3})	**Al**	**SiC**
e_{11}	0.10(5)	-0.43(5)
e_{22}	0.13(5)	-0.33(4)
e_{33}	2.03(5)	0.64(7)
e_{12}	-0.13(4)	-0.10(4)
e_{13}	0.01(5)	0.17(6)
e_{23}	0.01(5)	0.02(6)

Calculation of Stresses

- Isotropic:

$$\sigma_{ij} = \frac{1}{S_2/2}\left(e_{ij} - {}_{ij}\frac{S_1}{S_2/2 + 3S_1} e_{kk} \right)$$

- Anisotropic:

$$\langle s_{ijkl} \rangle = T_{ijklmnop}\, s_{mnop}$$

$T_{ijklmnop} = \mathrm{f}(W_{lmn})$! crystal & specimen symmetry

Stress

ISOTROPIC

ANISOTROPIC

Al (total)

$$\begin{bmatrix} 135(6) & -5(2) & 7(3) \\ \cdot & 133(7) & -9(3) \\ \cdot & \cdot & 231(6) \end{bmatrix}$$

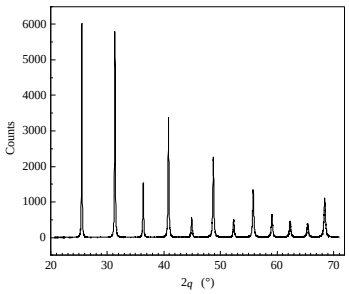
$$\begin{bmatrix} 136(?) & -5(?) & 18(?) \\ \cdot & 135(?) & -26(?) \\ \cdot & \cdot & 244(?) \end{bmatrix}$$

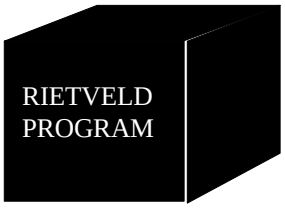
SiC (deviatoric)

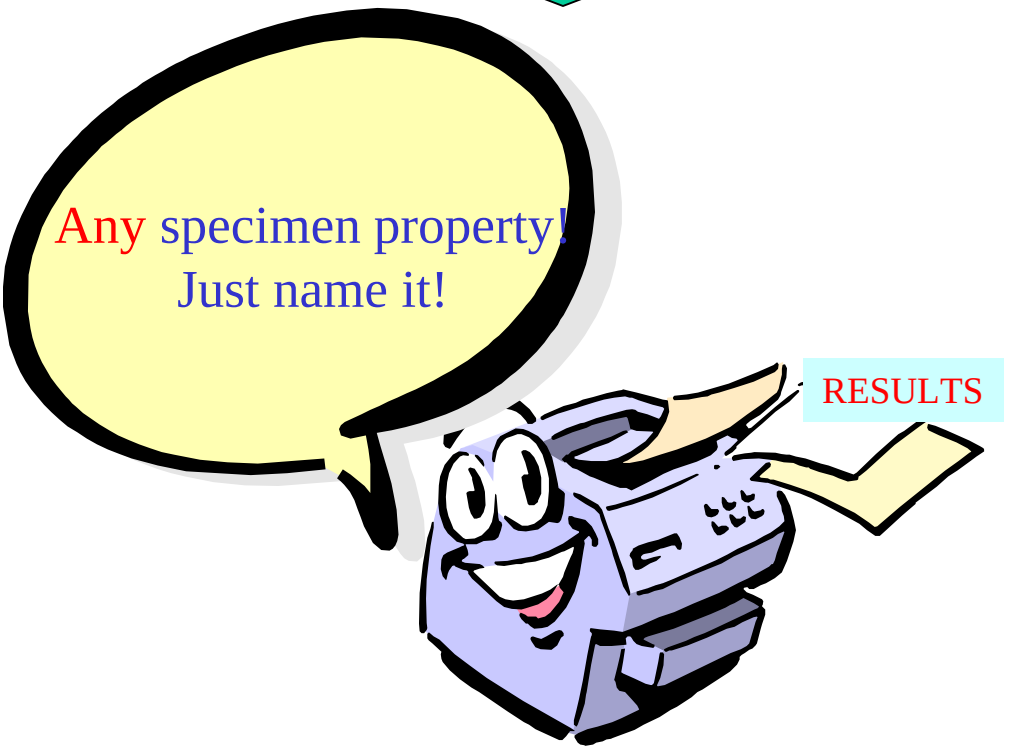
$$\begin{bmatrix} -113(17) & -21(12) & 64(18) \\ \cdot & -117(20) & -118(21) \\ \cdot & \cdot & 195(33) \end{bmatrix}$$

$$\begin{bmatrix} -290(?) & -14(?) & 57(?) \\ \cdot & -293(?) & -106(?) \\ \cdot & \cdot & 240(?) \end{bmatrix}$$

Future ?


6000
5000
4000
3000
2000
1000
0
Counts
20
30
40
50
60
70
2q (°)


RIETVELD
PROGRAM


Any specimen property!
Just name it!
RESULTS