Elastic Strain and Stress by Rietveld Method

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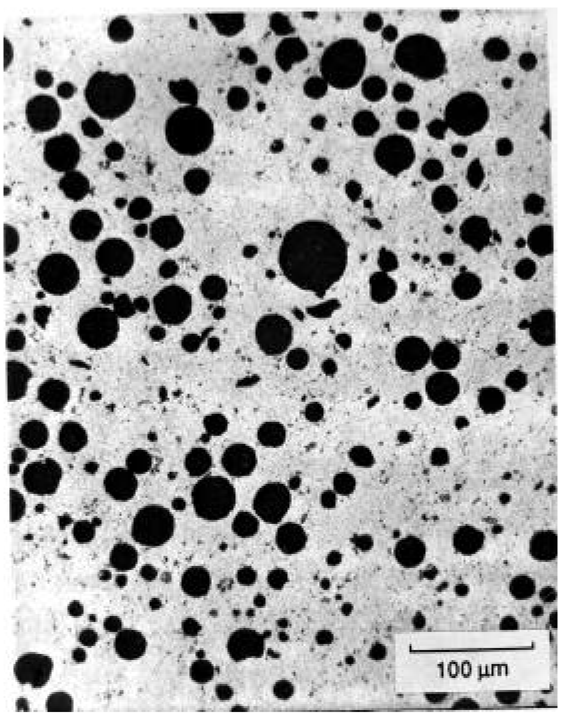
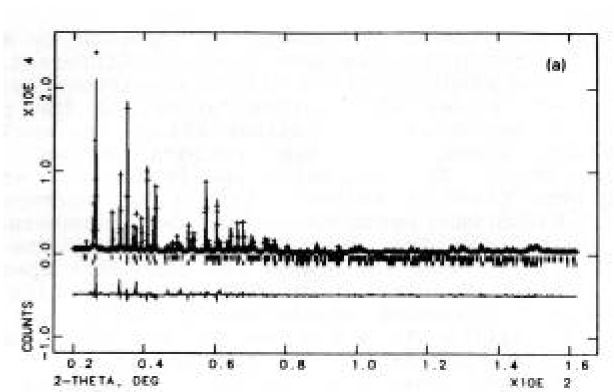
Why Rietveld refinement ?

Al/Mullite-Alumina Composites





- Al-6061 matrix

STRONG TEXTURE

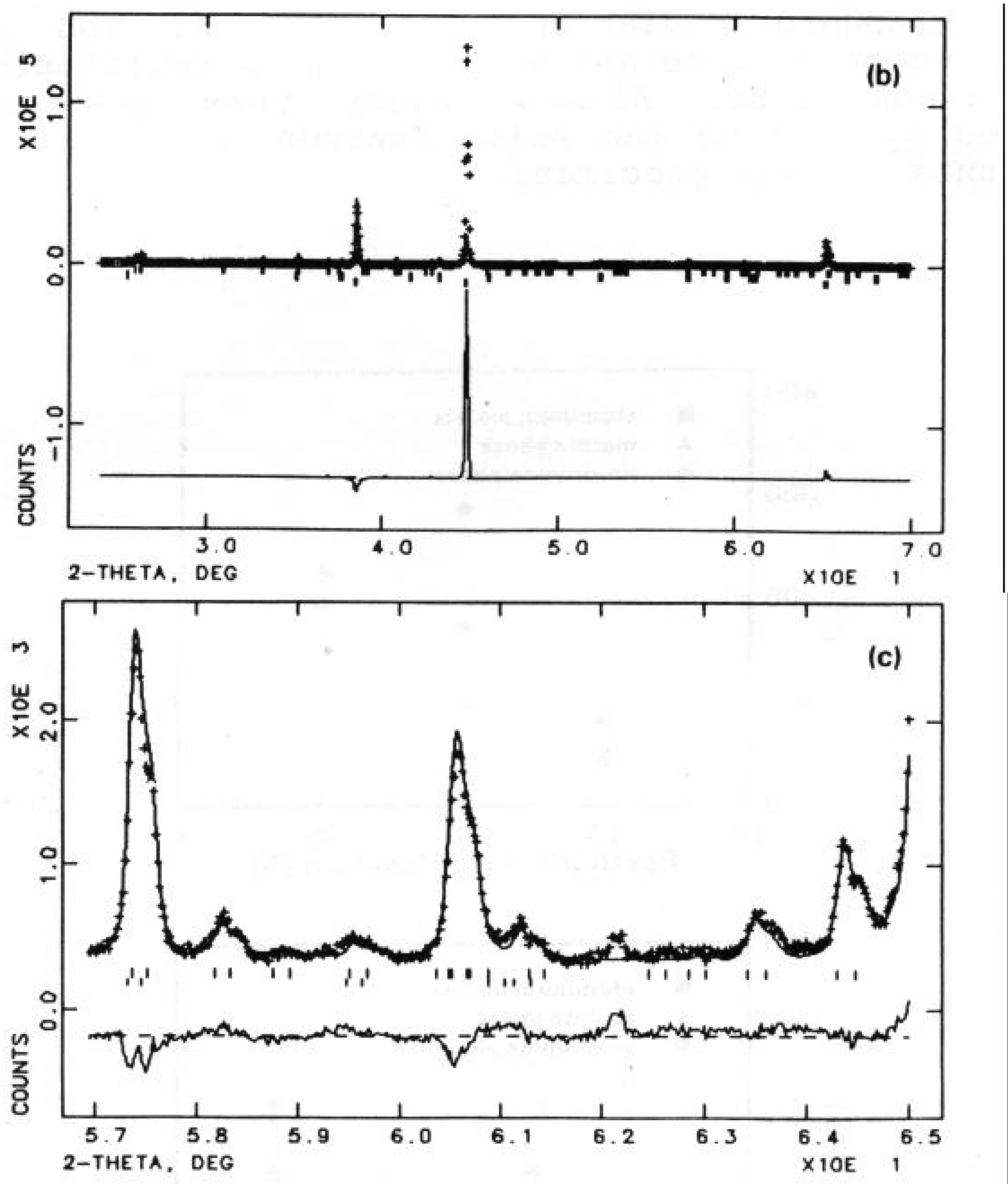
- α-alumina

STRESS ?

- 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

STRESS ?

X-Ray Diffraction



Consequences

- Complete diffraction pattern
  - All available *hkl*s   **TEXTURE !**
- Texture determination by Rietveld (GSAS)
  - Low symmetries
  - Multiphase mixtures
  - Residual stress
- Data collection
  - Monochromatic sources
    - Position-sensitive detectors
    - Section of the pattern
  - Polychromatic sources
    - Neutron TOF
    - EDXRD

Stress by diffraction

- Diffraction $\rightarrow$ STRAIN

$$\text{Strain} \xrightarrow{\text{TEXTURE}} \text{Stress}$$

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

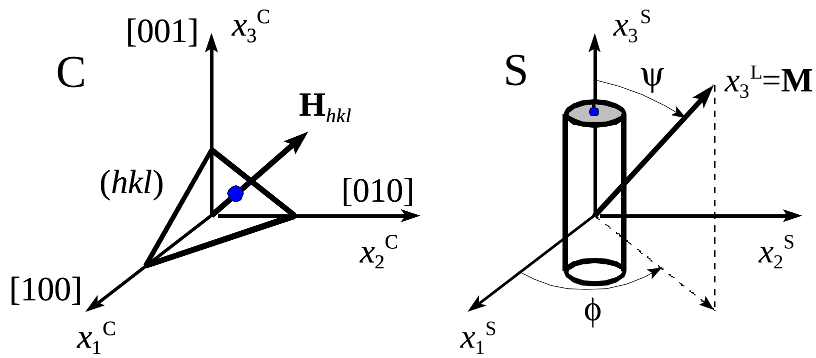
- Rietveld-refinement coding

$$\Delta T_h = (T - T_h) - \epsilon_{h,i} d - \epsilon_{h,a} d\, \Gamma$$

$$\Gamma_C = \beta_1^2\beta_2^2 + \beta_2^2\beta_3^2 + \beta_3^2\beta_1^2 = \frac{h^2k^2 + k^2l^2 + l^2h^2}{(h^2 + k^2 + l^2)^2}$$

$$\Gamma_H = \beta_3^2 = \frac{l^2}{\frac{4}{3}\frac{c^2}{a^2}(h^2 + hk + k^2) + l^2}$$

Strain measurements



$$\boxed{C} \xleftarrow{a(\varphi_1,\varphi_2,\varphi_3)} \boxed{S} \xrightarrow{\gamma(\psi,\phi)} \boxed{L}$$

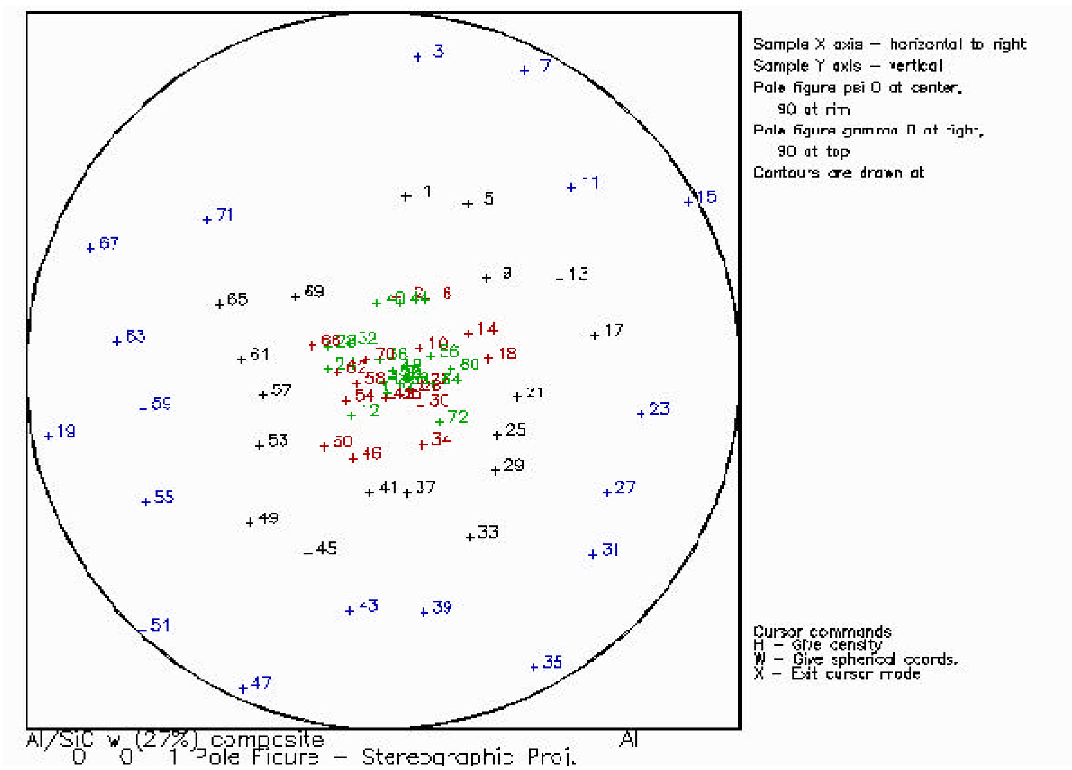
$$\mathbf{x}^{\mathbf{L}}_{i} = \gamma_{ij}\,\mathbf{x}^{\mathbf{S}}_{j}, \qquad \gamma_{ij} = \psi_{ik}\,\phi_{kj} = \begin{pmatrix} \cos\phi\cos\psi & \sin\phi\cos\psi & -\sin\psi \\ -\sin\phi & \cos\phi & 0 \\ \cos\phi\sin\psi & \sin\phi\sin\psi & \cos\psi \end{pmatrix}; \quad \mathbf{H}_{\mathrm{hkl}} \parallel x_3^L.$$

$$e_{33}^{\mathrm{L}} \equiv e_{\psi\phi} = d_{\psi\phi}/d_0 - 1$$

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

6 unknowns: over-determined system, least-squares

Neutron TOF measurements LANSCE (Los Alamos)



- 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 !

Strain by Rietveld refinement

- Strain determined from:
  - Particular d_{hkl}
  - Lattice parameters from Rietveld refinement:

$$\langle d \rangle = \frac{1}{4\pi} \int_0^{\pi} \mathrm{d}\psi \sin\psi \int_0^{2\pi} \mathrm{d}\phi \; d_{\psi\phi}$$

$$d_{\psi\phi} = \frac{\int_0^{2\pi} \mathrm{d}\omega \; d_{33}^{\mathrm{L}} f(g)}{\int_0^{2\pi} \mathrm{d}\omega \; f(g)}; \quad f(g) = \frac{\mathrm{d}V / V}{\mathrm{d}g}$$

- Quasi-isotropy:

$$\langle e \rangle \equiv \langle d \rangle / d_0 - 1 = (e_{11} + e_{22} + e_{33}) / 3$$

Strain

- Hydrostatic and deviatoric components:

$$e_{ij} = e'_{ij} + \delta_{ij} e^{\mathrm{H}}$$

$$e_{\psi\phi} = \gamma_{3k}\gamma_{3l} e_{kl} = e^{\mathrm{H}} + \gamma_{3k}\gamma_{3l} e'_{kl}$$

- Two possibilities:
  - Lattice parameters refined (d_0 known):

$$\langle e \rangle \equiv e^{\mathrm{H}} = (e_{11} + e_{22} + e_{33})/3$$

$\epsilon_{h,i} \Rightarrow e'_{kl}$ **no need for d_0 (!)**

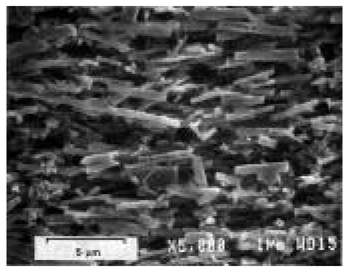
  - Lattice parameters held constant at a_0:

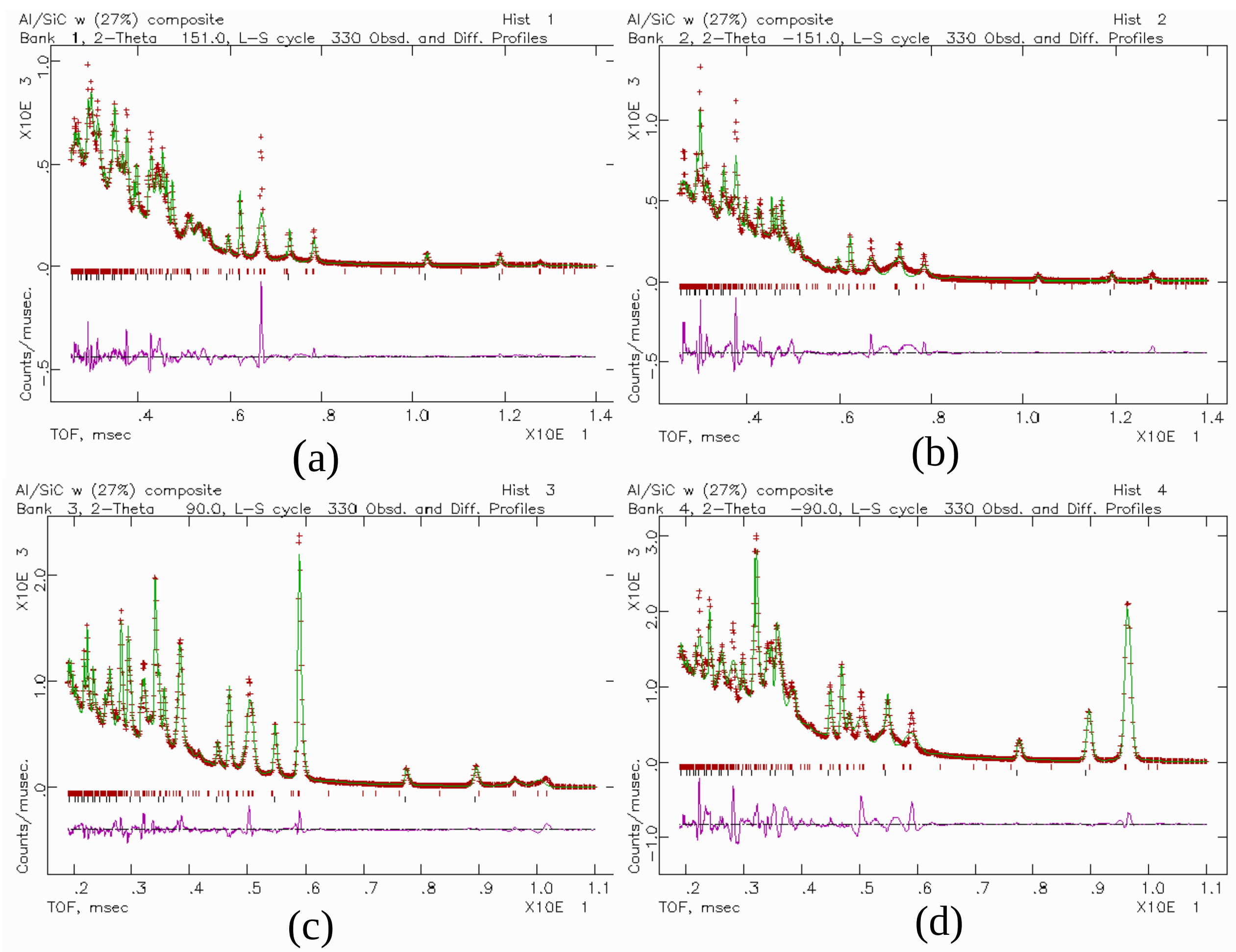
$$\langle e \rangle \equiv 0 = e'_{11} + e'_{22} + e'_{33}$$

$\epsilon_{h,i} \Rightarrow e^{\mathrm{H}} + e'_{kl}$

LANSCE (LANL) Measurements

Al (6061)/SiC_w (hexagonal) — extrusions



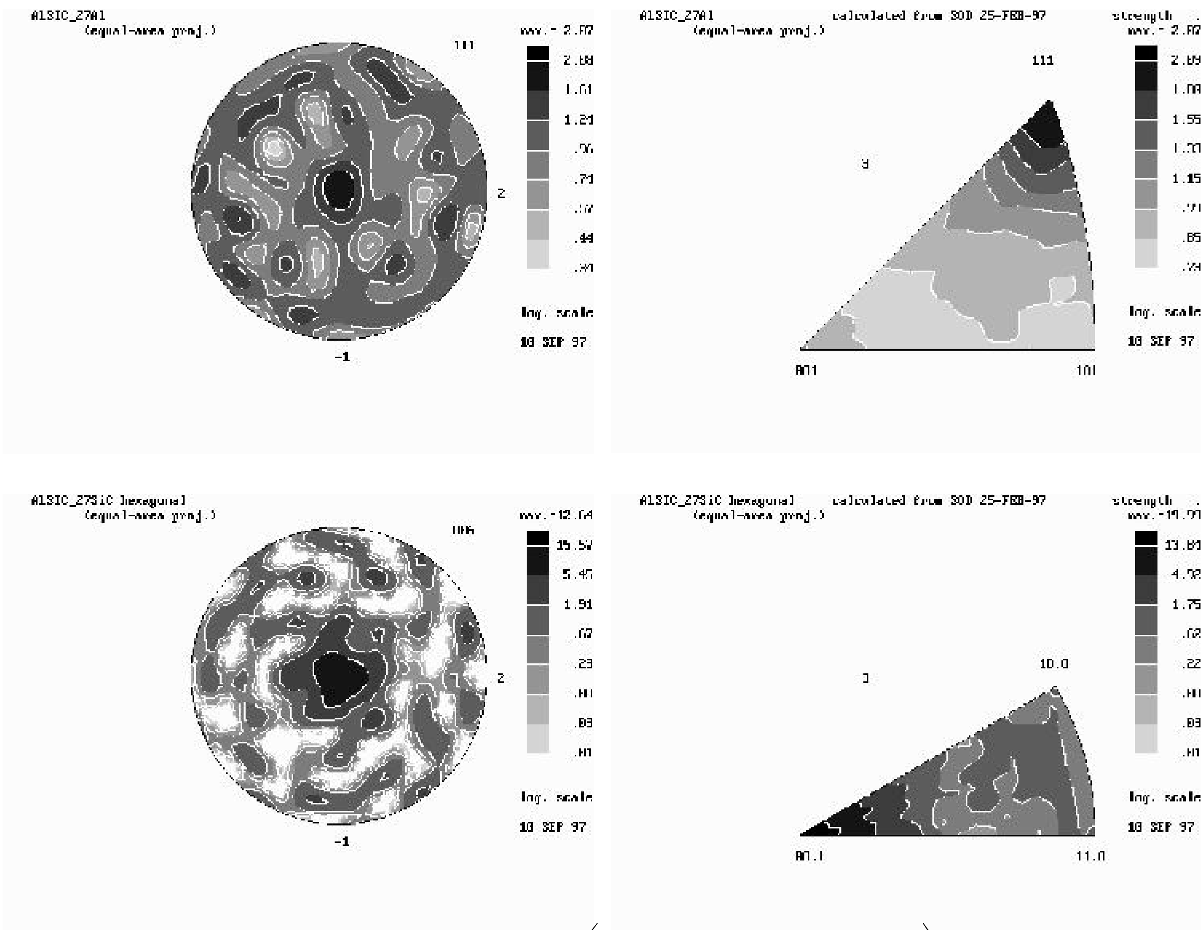


Complete Strain Tensor
Al/SiC$_w$ Composite

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)

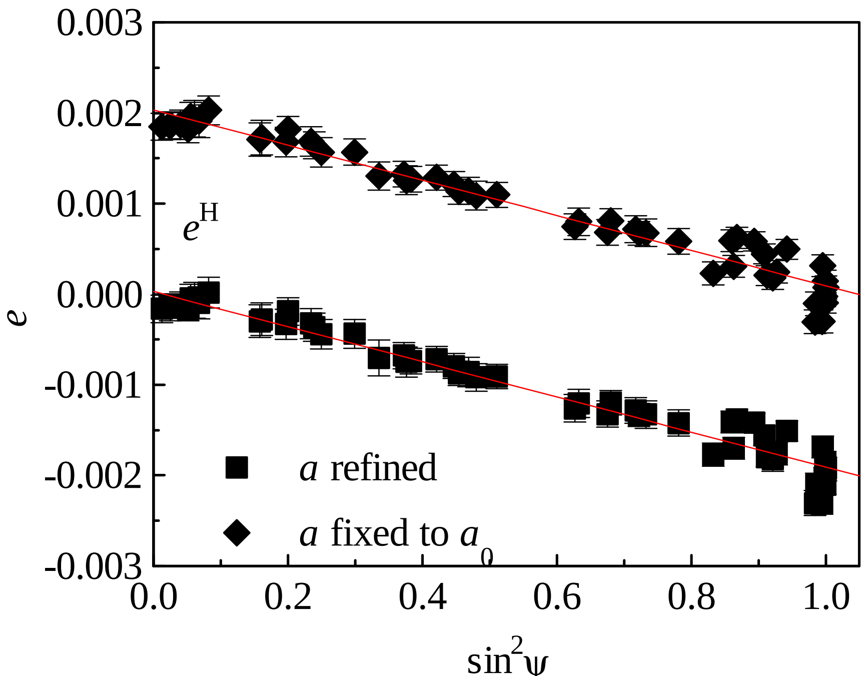
Pole Figures

W_{lmn} → Q_{lm} → Pole Figures

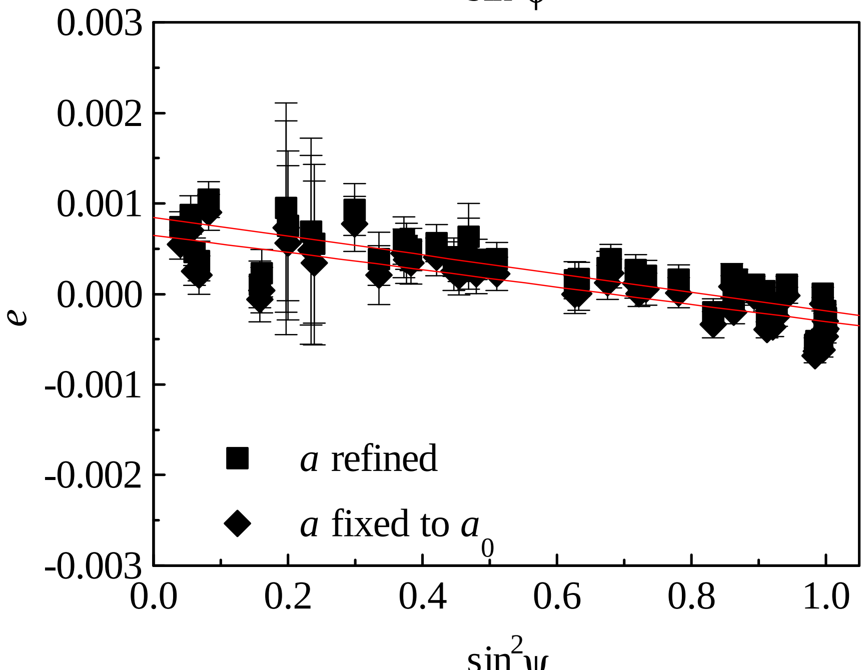


$$e(\psi) = e^{\mathrm{H}} + e'_{33} + \left(\frac{e'_{11} + e'_{22}}{2} - e'_{33} \right) \sin^2\psi$$

$\sin^2\psi$ plots



Al



SiC

Comparison of results

Al

Strain (10^{-3})	Linear Fit	Complete Tensor
e^H	2.00(8)	2.01(10)
e_{11}'	-1.91(10)	-1.91(6)
e_{22}'		-1.86(5)
e_{33}'	0.03(4)	0.02(4)

SiC

Strain (10^{-3})	Linear Fit	Complete Tensor
e^H	-0.19(14)	-0.14(11)
e_{11}'	-0.19(8)	-0.31(5)
e_{22}'		-0.22(4)
e_{33}'	0.84(7)	0.83(7)

Calculation of Stresses

- Isotropic:

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

- Anisotropic:

$$< s_{ijkl} > \; = \; T_{ijklmnop} \, s_{mnop}$$

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

Stress

ISOTROPIC

ANISOTROPIC

Al (total)

$$\begin{bmatrix} 135(6) & -5(2) & 7(3) \\ \cdot & 133(7) & -9(3) \\ \cdot & \cdot & 231(6) \end{bmatrix} \qquad \begin{bmatrix} 136(?) & -5(?) & 18(?) \\ \cdot & 135(?) & -26(?) \\ \cdot & \cdot & 244(?) \end{bmatrix}$$

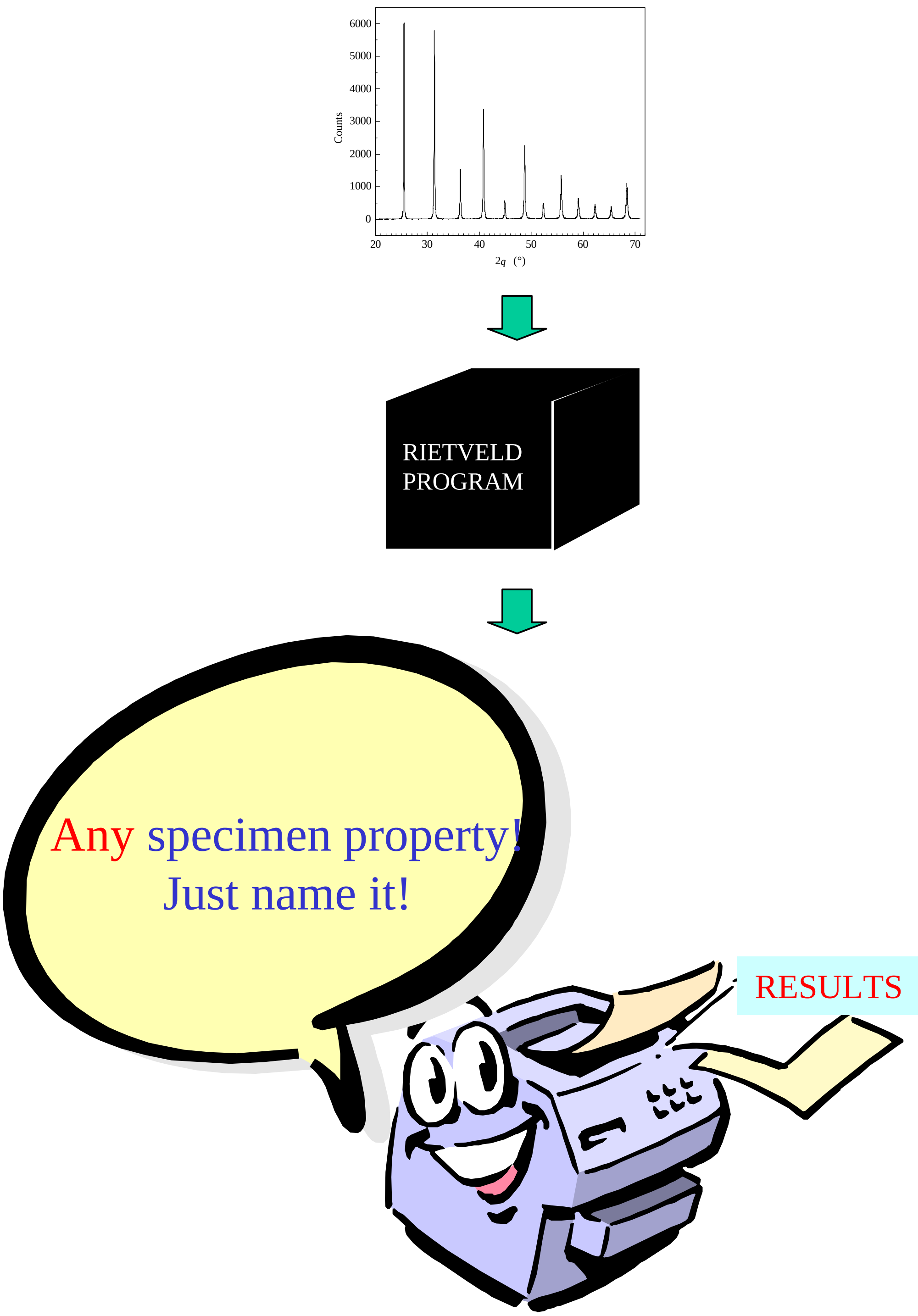
SiC (deviatoric)

$$\begin{bmatrix} -113(17) & -21(12) & 64(18) \\ \cdot & -117(20) & -118(21) \\ \cdot & \cdot & 195(33) \end{bmatrix} \qquad \begin{bmatrix} -290(?) & -14(?) & 57(?) \\ \cdot & -293(?) & -106(?) \\ \cdot & \cdot & 240(?) \end{bmatrix}$$

Acknowledgements

- R. B. Von Dreele (LANSCE, Los Alamos National Laboratory)

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