Effect of internal stress on peak position: problems and opportunities

HCP materials

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APD IV
Outline

- Interphase and intergranular stresses
- Effect on Rietveld refinement
- Opportunities to determine crystal properties
  - Impact of high energy neutrons on structure
  - Use of internal stresses to determine operating deformation modes
- Conclusions
Constraints on phases and grains

What is impact of interactions between phases and different grain orientations on stress / deformation?
Thermal anisotropy

- e.g. Zirconium-Niobium alloy; two phase HCP and BCC

- $10.1 \times 10^{-6} / ^\circ C$
- $7.4 \times 10^{-6} / ^\circ C$
- $5.3 \times 10^{-6} / ^\circ C$
Diffraction measurement of elastic strain

\[ \lambda = 2d \sin \theta \]

\[ \varepsilon = \frac{\Delta d}{d} \]

Diffraction peaks measured in specific direction in sample

(cubic example shown)
Relationship $d-hk.l$ and $a$ & $c$

\[ d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad \text{cubic} \]

\[ \frac{1}{d_{hkl}^2} = \frac{4}{3} \left( \frac{h^2 +hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad \text{hexagonal} \]

Will hold for single crystal, a single-crystal powder, and a “stress-free” polycrystal
Deviation from ideal relation due to: thermal stresses
Deviation from ideal relation due to: thermal stresses – effect of texture

![Graph showing strain vs. angle from c-axis for different {hk.l} values.]
How to handle in Rietveld refinement?

e.g. GSAS:
“RSTR” – isotropic strain parameter,
“RSTA” – assumes cosine variation with angle from c-axis
Elastic anisotropy

Elastic anisotropy; different stiffnesses in different crystallographic directions
Plastic anisotropy

Plastic anisotropy; introduces non-linearity

fcc structure
\{111\}<110> slip
Examples of intergranular strains in Zr

Elastic strain (E-6)

Macro applied stress (MPa)

b) RD-RD
Deviation from ideal relation due to: plasticity induced stresses

![Graph showing strain vs angle with various strain levels and angles plotted on the x and y axes.](image)
Deviation from ideal relation due to: plasticity induced stresses – response depends on texture

![Graph showing strain vs. angle]

- Strain values for different angles and percentages.
- The graph illustrates the deviation from ideal relation due to plasticity induced stresses, with the response depending on texture.
Deviation from ideal relation due to: plasticity induced stresses – response depends on texture.
After 3% strain

strain [$10^{-6}$]

angle from c-axis

RD

ND

TD

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After 3% strain

angle from c-axis

strain \times 10^{-6}
How to handle in a Rietveld refinement?

Most extreme peaks, typically strain of ~0.001

Response differs too far from relation to allow accurate peak width and/or peak intensity fit while maintaining peak positions

  e.g. pseudo-anisotropic broadening

Solution

  1) Allow peak positions to vary freely?
  2) Incorporate models of plasticity?
Deformation modes observed in single crystal hcp Zr

(1010) <1120> prismatic slip

(0001) <1120> basal slip

(1011) <1123> pyramidal slip

(1012) <1011> tensile twin
Understanding strains – models of polycrystalline plasticity

Single crystal properties

+ Crystallographic texture (ODF)

+ Crystal interactions with matrix

↓ Macroscopic & polycrystal properties
e.g. self-consistent elasto-plastic

Single crystallite
• elastic and plastic anisotropy
• relate crystallite strain / stress state to macro applied strain / stress.

Matrix
• homogenous average of all the crystallites
• iterate to solution
Model: self-consistent elasto-plastic

Keep track of each grain, its stress state and its plastic strain.

Fixed parameters:
• Single crystal elastic constants
• Texture (⇒ grain population)
• Plastic slip directions / planes

Fitted parameters, to get best agreement with experiment
• Critical resolved shear stress, \( \tau \)
• Hardening gradient \( \phi \)
  i.e. only 2 fitting parameters for a slip system
(Perhaps more complex plastic law)
Use of zirconium reactors worldwide

- Zircaloy Fuel Cladding:
  - Pressurized or unpressurized \( \text{H}_2\text{O} \) coolant
  - Temperatures range from ambient to \( >300^\circ \text{C} \)

PHWR:
- Zr-2.5Nb* pressure tube contains coolant
- Zircaloy-2 ‘calandria’ tube, separates hot pressure tube from moderator

Research reactors:
- Reflector vessel walls

Metallurgy somewhat analogous to titanium
* 2 phase alloy, c.f. Ti-6Al-4V
Neutron Economy

Zr alloy components
Effect of neutron irradiation

On average, each atom is displaced from its position about once a year.
Residual structure after decay of thermal spike

- Vacancy clusters
- Interstitial clusters
- Individual vacancies (freely migrating)
- Individual SIAs (freely migrating)

Subsequent Evolution

- Vacancies and interstitials migrate through the lattice and the microstructure evolves
- Usually a dense dislocation “loop” structure generated over first few dpa
Dislocation loops

Differences in migration rates / anisotropy lead to different densities of \(<a>\) and \(<c>\) loop dislocations
Hardening of Steel

- UE decreases with dose
- YS increases with dose

Similar effect in zirconium alloys

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Unirradiated Experiment, Load Parallel to Hoop

Unirradiated Model, Load Parallel to Hoop

Very low hardening – plot vs macroscopic strain

Experiment

Model
Irradiated Experiment, Load Parallel to Hoop

Irradiated Model 1, Load Parallel to Hoop

True strain [%]

Elastic Strain [$10^{-6}$]

Irrad Model

Irrad experiment
# Impact of irradiation on model (fit) CRSS

<table>
<thead>
<tr>
<th>Mode</th>
<th>Unirradiated [MPa]</th>
<th>Irradiated [MPa]</th>
<th>Increase [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prism &lt;a&gt;</td>
<td>160</td>
<td>370</td>
<td>210 (x2.3)</td>
</tr>
<tr>
<td>Basal &lt;a&gt;</td>
<td>170</td>
<td>370</td>
<td>200 (x2.2)</td>
</tr>
<tr>
<td>Tensile twin</td>
<td>450</td>
<td>n/a</td>
<td>n/a</td>
</tr>
<tr>
<td>&lt;c+a&gt;</td>
<td>460</td>
<td>500</td>
<td>0 (x1.1)</td>
</tr>
<tr>
<td>Macroscopic (experimental 0.2% yield)</td>
<td>530 / 740</td>
<td>820 / 950</td>
<td>(x1.5 / x1.3)</td>
</tr>
</tbody>
</table>
Wilkens type full pattern LPA
Dislocation density ($10^{14} \text{ m}^{-2}$)

- \(<a>\) - Red circle
- \(<c+a>\) - Black triangle

Unirradiated

Unirradiated

Deformation

Deformation

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Conclusion

- Internal stresses change location of peaks enough that we must account for them in Rietveld refinement
- Can study these changes to determine crystal
  - Irradiation changed response (load sharing) of differently (crystographically) oriented grains.
  - Effect of irradiation on critical resolved shear stress in extruded Zr2.5Nb was determined
- Can be correlated with dislocation populations measured by peak width analysis (typically ex situ)
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