Strain propagation in Nano-crystalline ceramics

The laser pump-probe technique

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Topics covered

• Powder X-ray Diffraction (PXRD)
• Types of Strain
• The Experiment
• Reitveld Refinement
  – Topas
• Results, so far...
What’s it all about?

• Short intense influx of energy
  – High peak intensity
  – low integrated intensity

• Cause a shockwave
  – Wavefront of high strain
    • Propagates through grains
    • High propagation velocity
    • High peak strain intensity
X-ray Diffraction

- **Bragg Geometry**
  - \( n\lambda = 2d\sin(\theta) \)

- **Not just the D-spacing**
  - Sample contribution
  - Instrumental contribution
  - Source contribution

\[ Y(2\theta) = (Source \otimes Instrument) \otimes Sample \]
1. Peak positions determined by size and shape of unit cell – internal structure

2. Peak Intensities determined by where atoms sit in the unit cell – internal structure
Sample Contribution, continued...

3. Peak widths determined by size and strain in crystallites – microstructure.

4. Background oscillations may contain information about short range order in the material.
Types of Strain

- Intrinsic Strain
  - Current state
- Residual Strain
  - Modify Intrinsic Strain
- Dynamic Strain
  - Time resolved
Strain...

- **Macrostrain**
  - Strain same over whole material
  - Peak shifts are a uniform
    - Magnitude is a $f(2\theta)$
    - Same direction

- **Microstrain**
  - Strain localised to a small region of sample
    - tens of unit cells
  - Peak broadening not uniform
    - Different magnitudes
    - Not as a $f(2\theta)$
Strain continued

Unstrained

Macrostrain
  • Peak shift

Microstrain
  • Peak broadening
Dynamic Strain

- **Energy Propagation**
  - Thermal effect - radial
  - Thermo-kinetic effect – planes
    - Grain boundary?
  - Very quick
  - $V \approx 3.7 \times 10^6 \text{ m/s}$

- **Why Nano-crystalline?**
  - Grain boundary
  - Slower propagation
The Experiment

- Diamond Synchrotron Source
- Mythen 90° PSD Detector
- Sample
- X-ray Beam
- Diffracted X-ray Beam
- Laser Mirror
- CO₂ Laser Source
- Precision Sample Stage
How does it work?

Detector...

Laser...

Acquisition time - 1ms

Trigger delay - 10μs

Repetition rate 2s
How does it work?

Detector...

Laser...

Acquisition time - 1ms

Trigger delay - 10μs

Repetition rate 2s

500μs 500μs
Data Quality

Comparison of a single 1ms exposure with a summation of 1242 exposures

Counts

2theta (deg)

1242 1ms exposures added up
single 1s exposure
Data Analysis

- Rietveld Refinement
  - Least Squares fitting
    - Instrumental
    - Source
    - Sample
  - Structural refinement
- Pawley le Bail
  - Least Squares fitting
  - Not structural
  - Not intensity dependent
The Sample

• Blend
  • 60% Alumina
  • 40% Zirconia
  • Potassium Binder

• Results...
  • Two phase refractory
    » Alumina Zirconia (tetragonal)
    » Zirconia (monoclinic)
  • Potassium (minor phase)
Results

Strain vs Displacement

Macroscopic Strain vs Displacement from shock site (mm)

- a (Al60Zr40)
- c (Al60Zr40)
- beta (Zirconia)
- a (Zirconia)
- b (Zirconia)
- c (Zirconia)
Results

Strain vs Displacement

Macroscopic Strain

Displacement from shock site (mm)
Results

Strain vs Displacement

- Macroscopic Strain
- Displacement from shock site (mm)

- a (Al60Zr40)
- c (Al60Zr40)
- beta (Zirconia)
- a (Zirconia)
- b (Zirconia)
- c (Zirconia)
Results

Strain vs Displacement

- Macroscopic Strain vs Displacement from shock site (mm)

- Different materials represented with different markers:
  - Diamond (Al60Zr40)
  - Beta (Zirconia)
  - Alpha (Zirconia)

- Data points indicate strain values at various displacement points.
Conclusion so far...

• Results
  • Strain clearly visible
  • $Strain \propto \frac{1}{r}$
    » $r = \text{distance from shock site}$
  • Not due to thermal expansion
    » Coeff. Thermal Expansion $10^{-6}/^\circ\text{C}$
    » Strain too large to be thermal expansion

– Frame Analysis...
  • No residual thermal expansion
Next Step

• Rolling Average
  – Small frames ~250ms
  – Refine strain on small frames
    • More noisy
    • More strain in each frame
Thanks for Listening

This work was funded by EU FP7, grant PIAP-GA-2011-286110-Intercer2