PRIFYSGOL ABERYSTWYTH UNIVERSITY

Strain propagation in Nano-crystalline ceramics

The laser pump-probe technique

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diamond

Topics covered

- Powder X-ray Diffraction (PXRD)
- Types of Strain
- The Experiment
- Reitveld Refinement
  - Topas
- Results, so far…

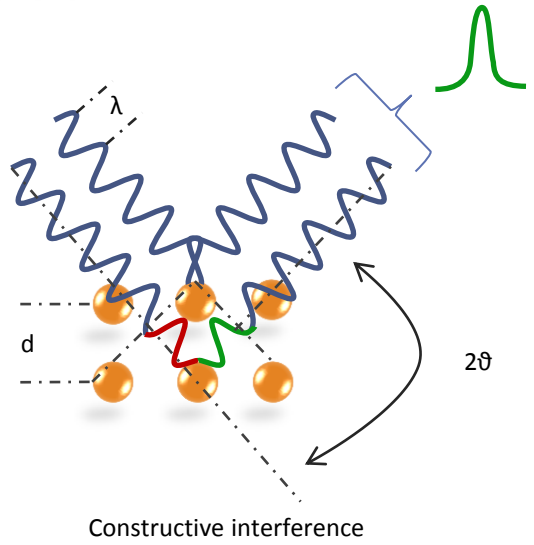
What's it all about?

- 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

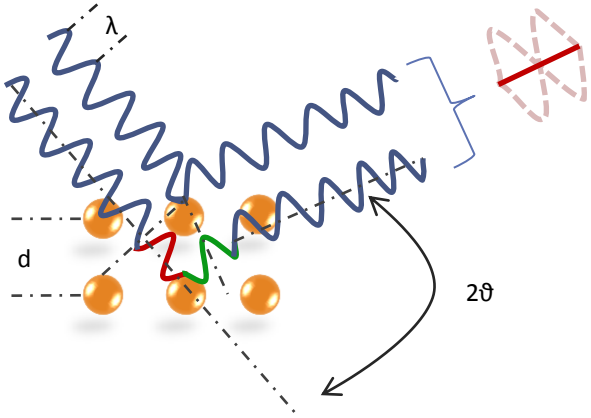
X-ray Diffraction

- Bragg Geometry
  - $n\lambda = 2dsin(\vartheta)$
- Not just the D-spacing
  - Sample contribution
  - Instrumental contribution
  - Source contribution

$$Y(2\vartheta) = (Source \otimes Instrument) \otimes Sample$$



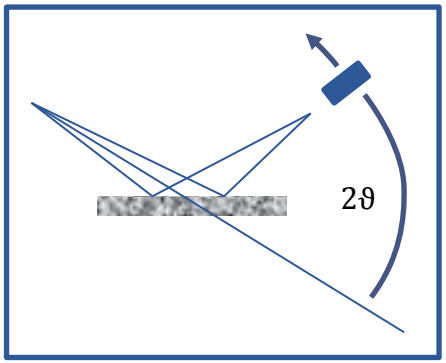
Constructive interference



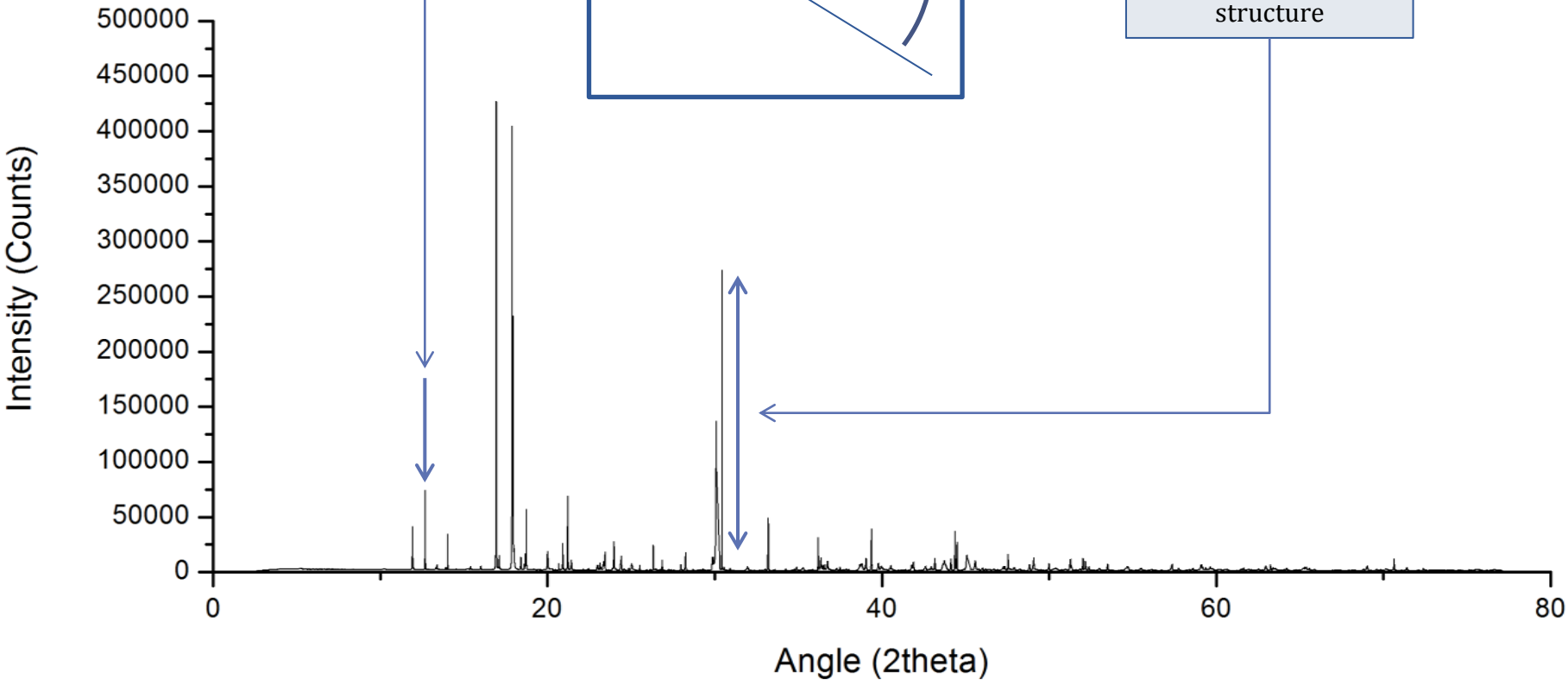
Destructive interference

Sample Contribution

1. Peak positions determined by size and shape of unit cell – internal structure

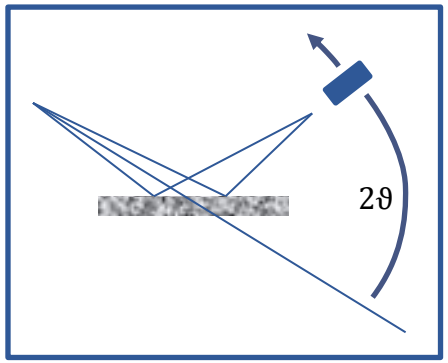


2. Peak Intensities determined by where atoms sit in the unit cell – internal structure

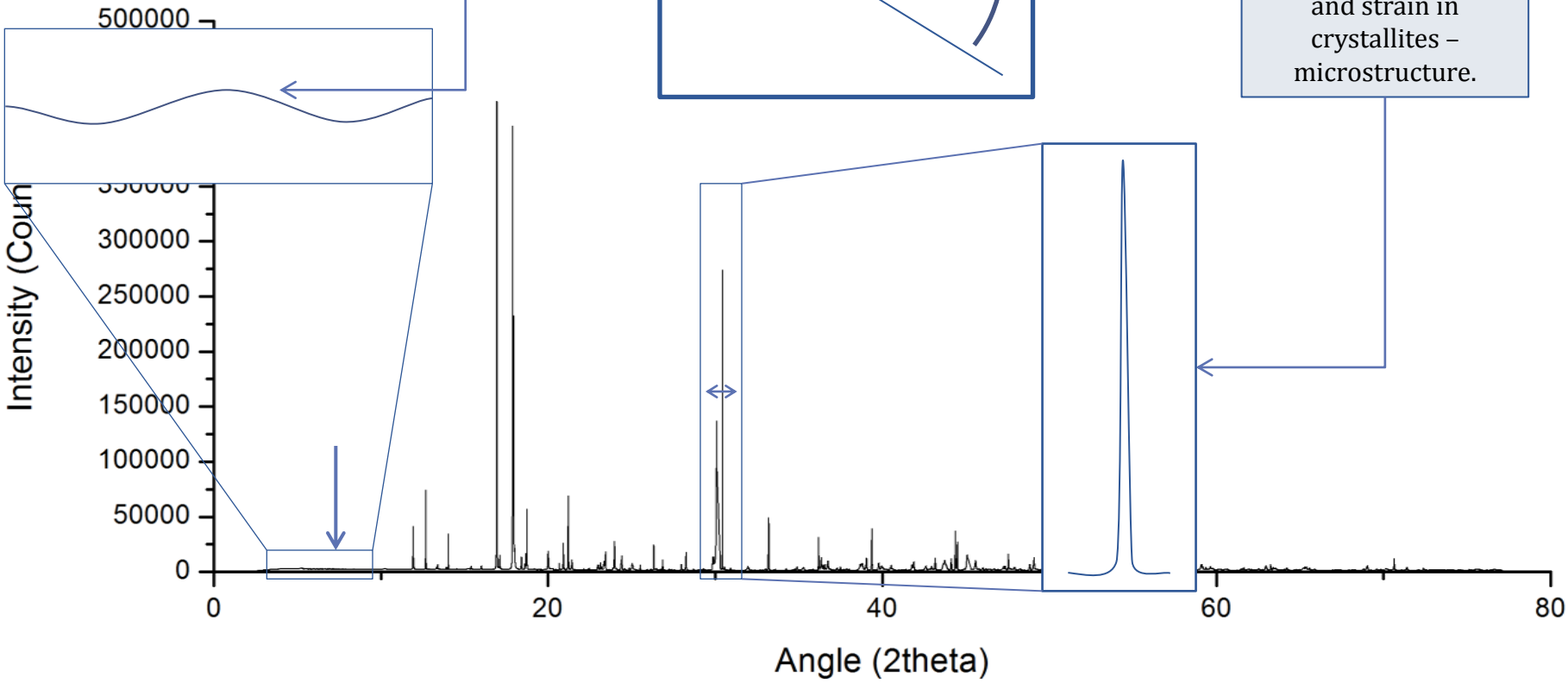


Sample Contribution, continued…

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

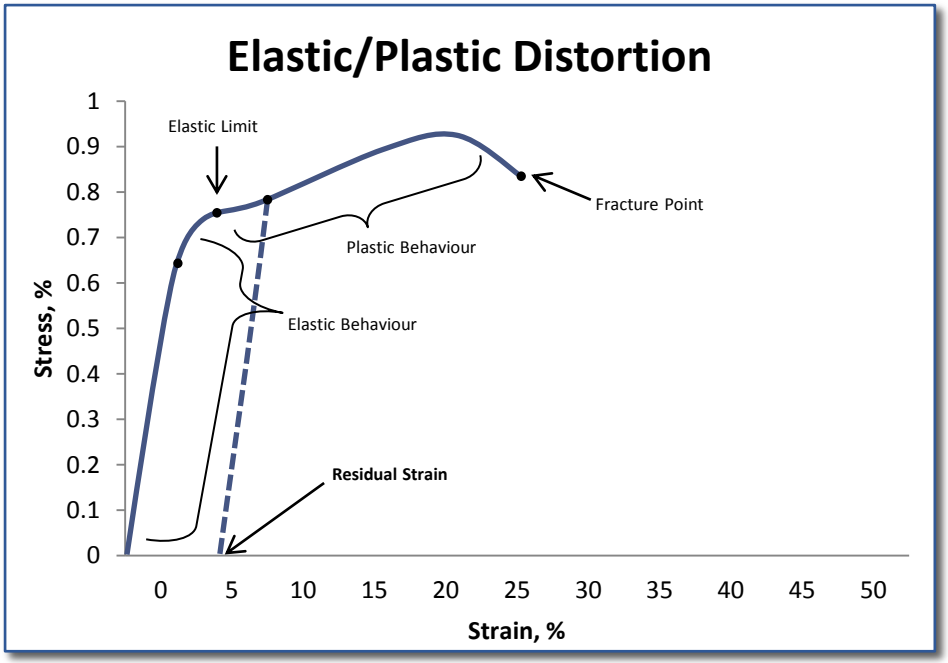


3. Peak widths determined by size and strain in crystallites – microstructure.



Types of Strain

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

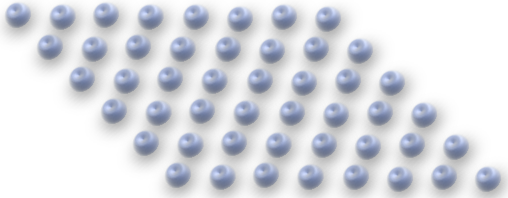


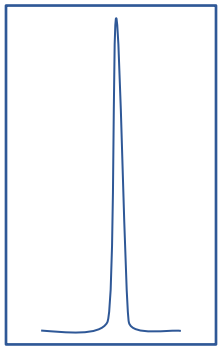
Strain…

- Macrostrain
  - Strain same over whole material
  - Peak shifts are a uniform
    - Magnitude is a $f(2\vartheta)$
    - Same direction
- Microstrain
  - Strain localised to a small region of sample
    - tens of unit cells
  - Peak broadening not uniform
    - Different magnitudes
    - Not as a $f(2\vartheta)$

Strain continued

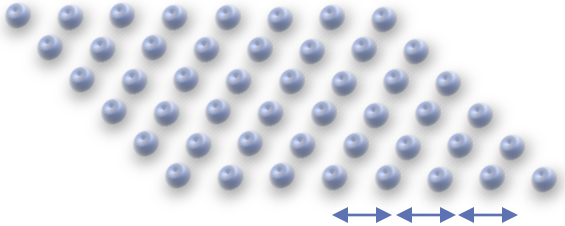
Unstrained

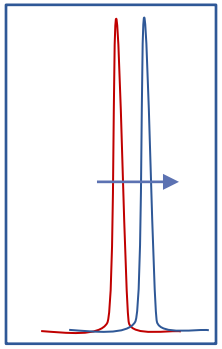




Macrostrain

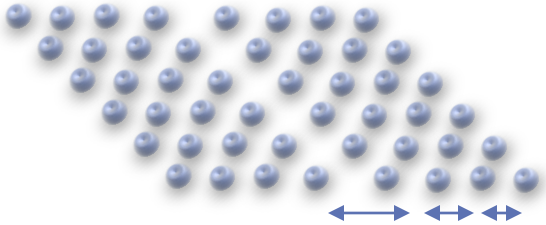
- *Peak shift*

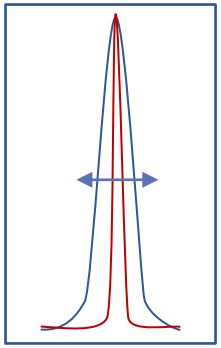




Microstrain

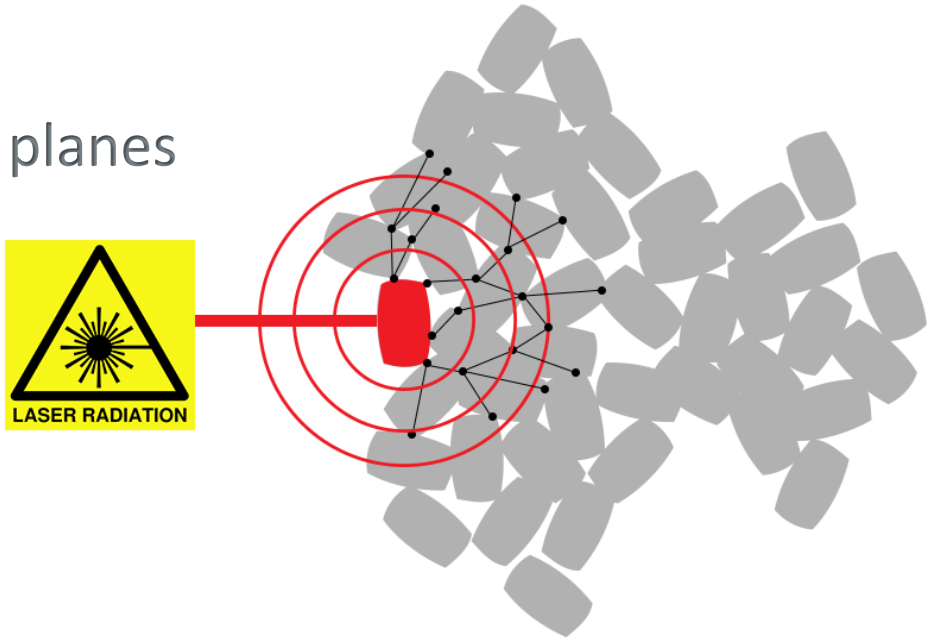
- *Peak broadening*



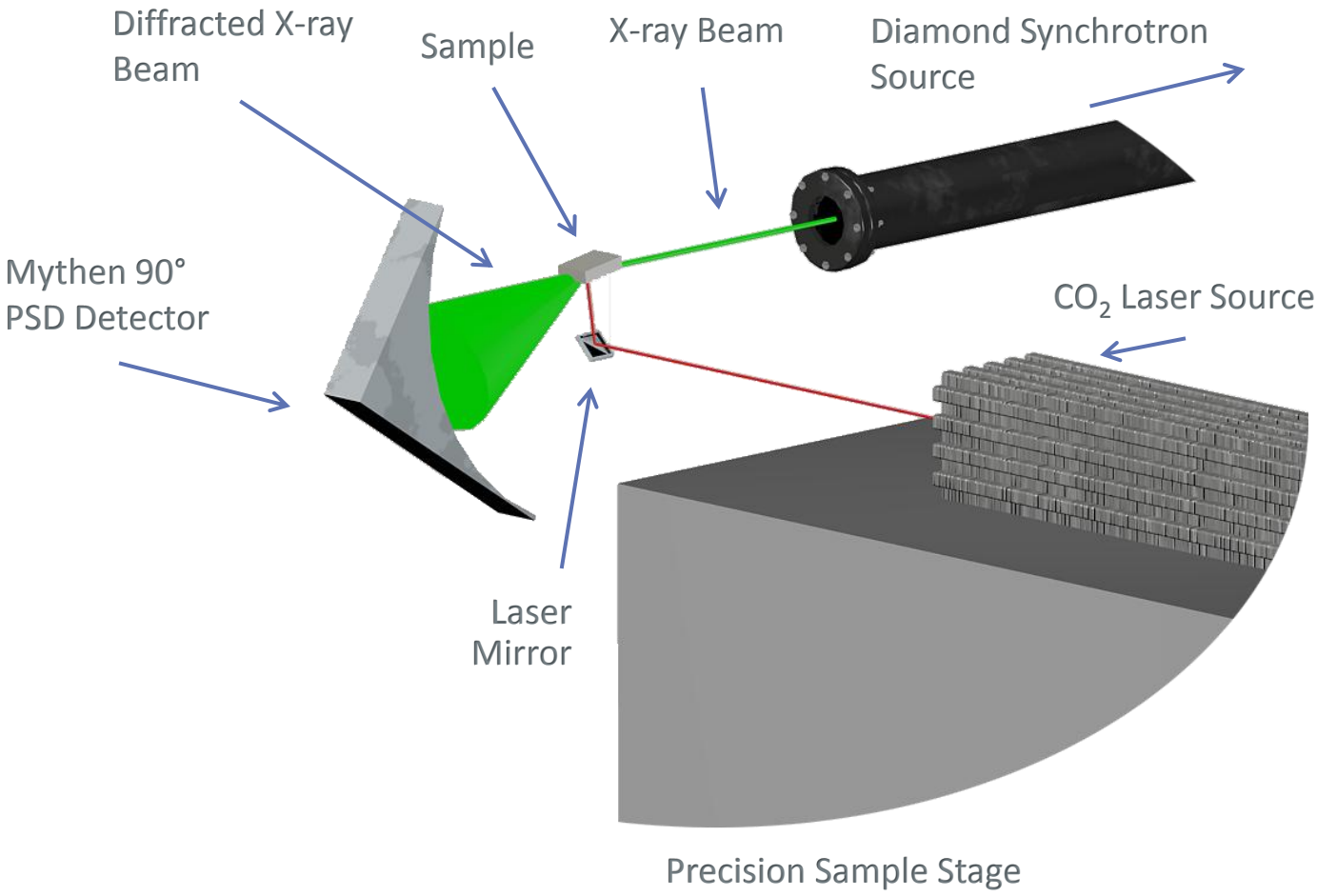


Dynamic Strain

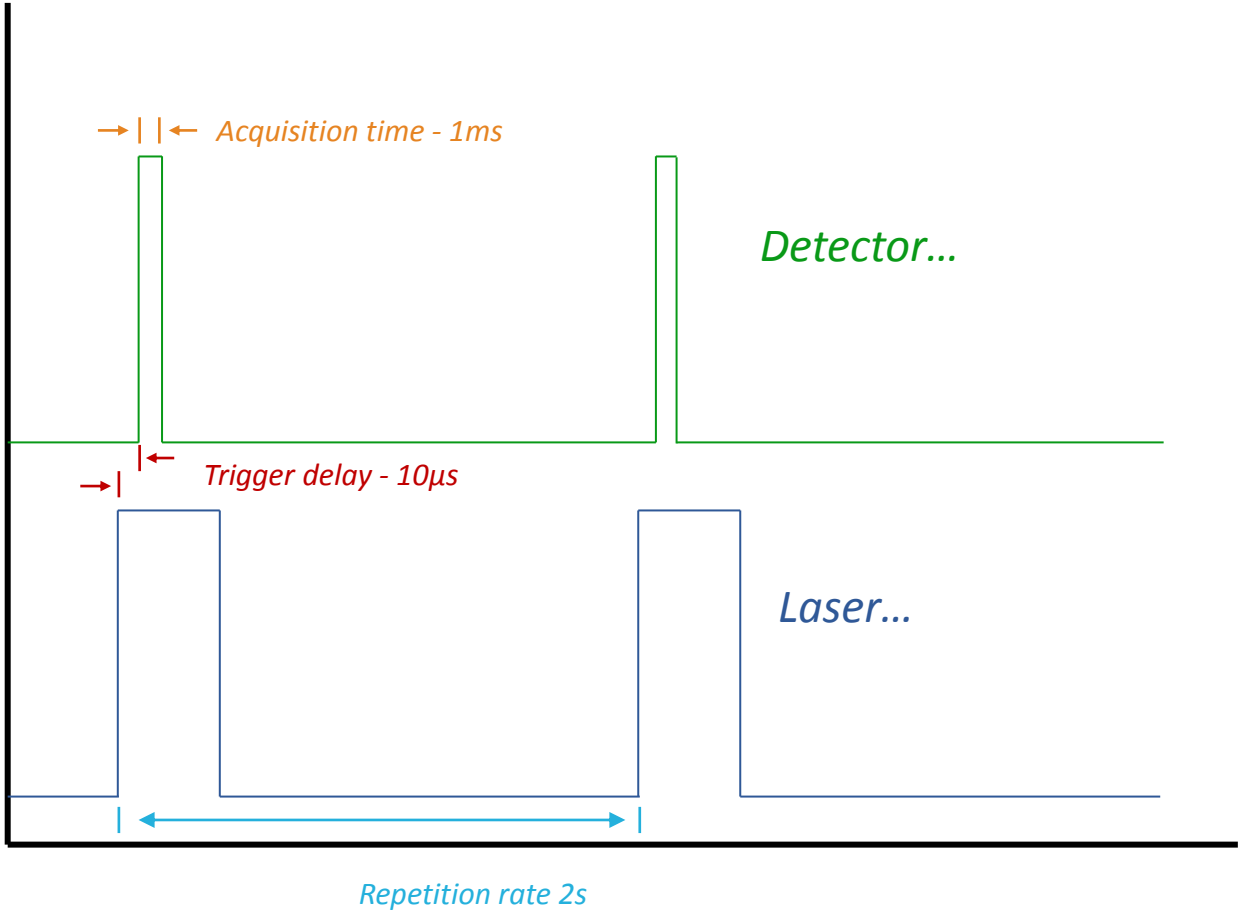
- Energy Propagation
  - Thermal effect - radial
  - Thermo-kinetic effect – planes
    - Grain boundary?
  - Very quick
  - $V \approx 3.7 \times 10^6$ m/s
- Why Nano-crystalline?
  - Grain boundary
  - Slower propagation



The Experiment



How does it work?



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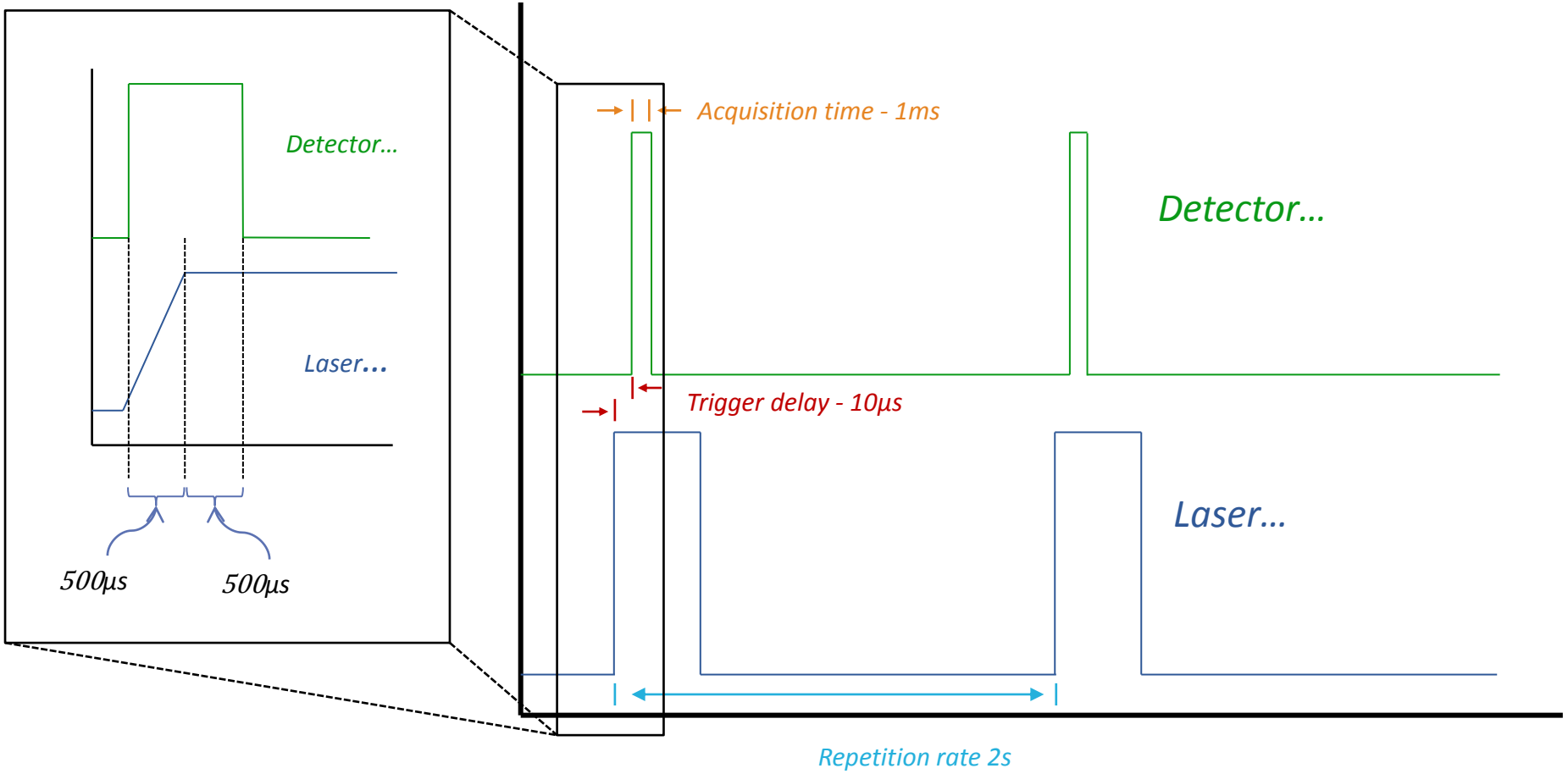
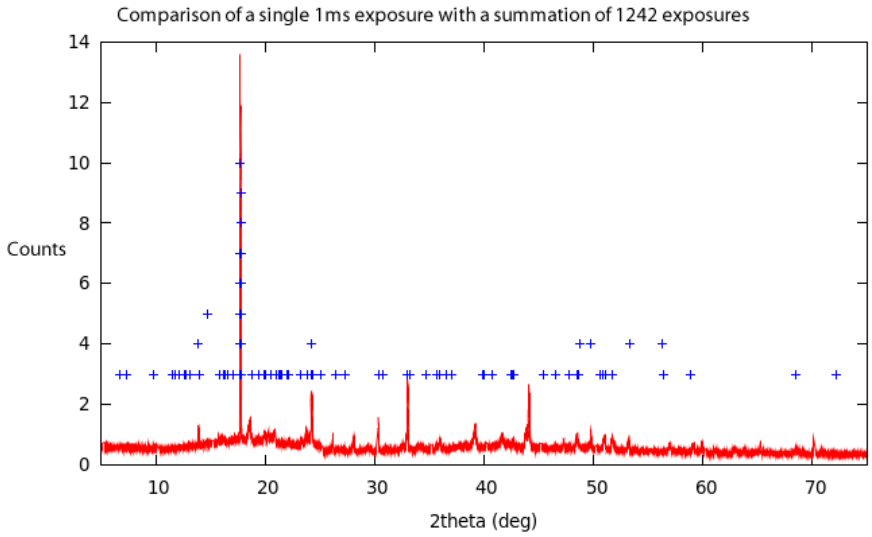
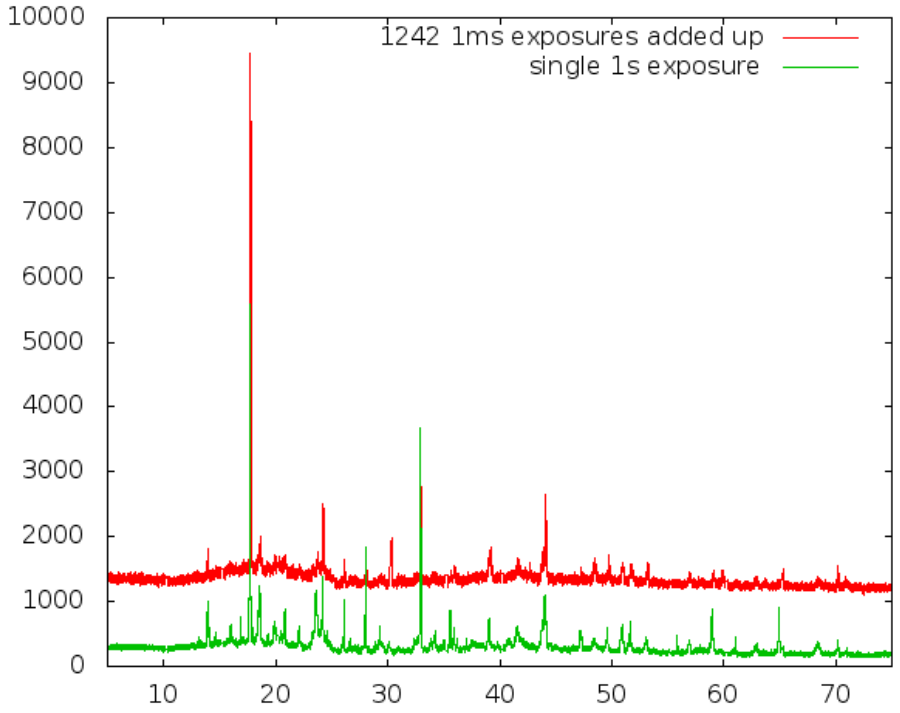


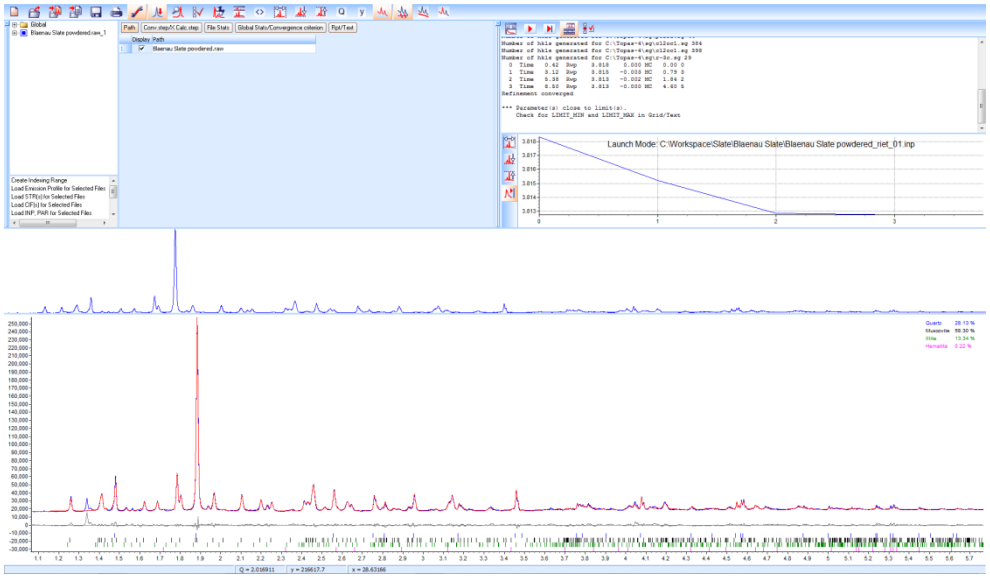
Data Quality





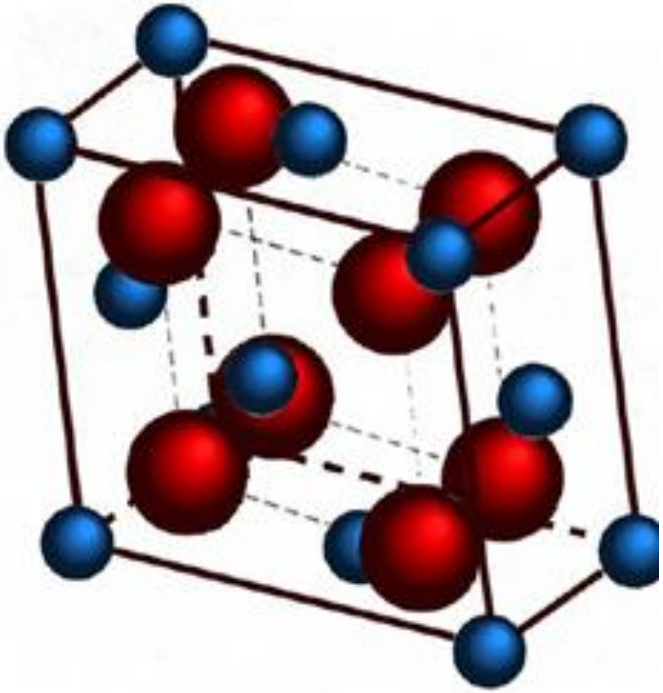
Data Analysis

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



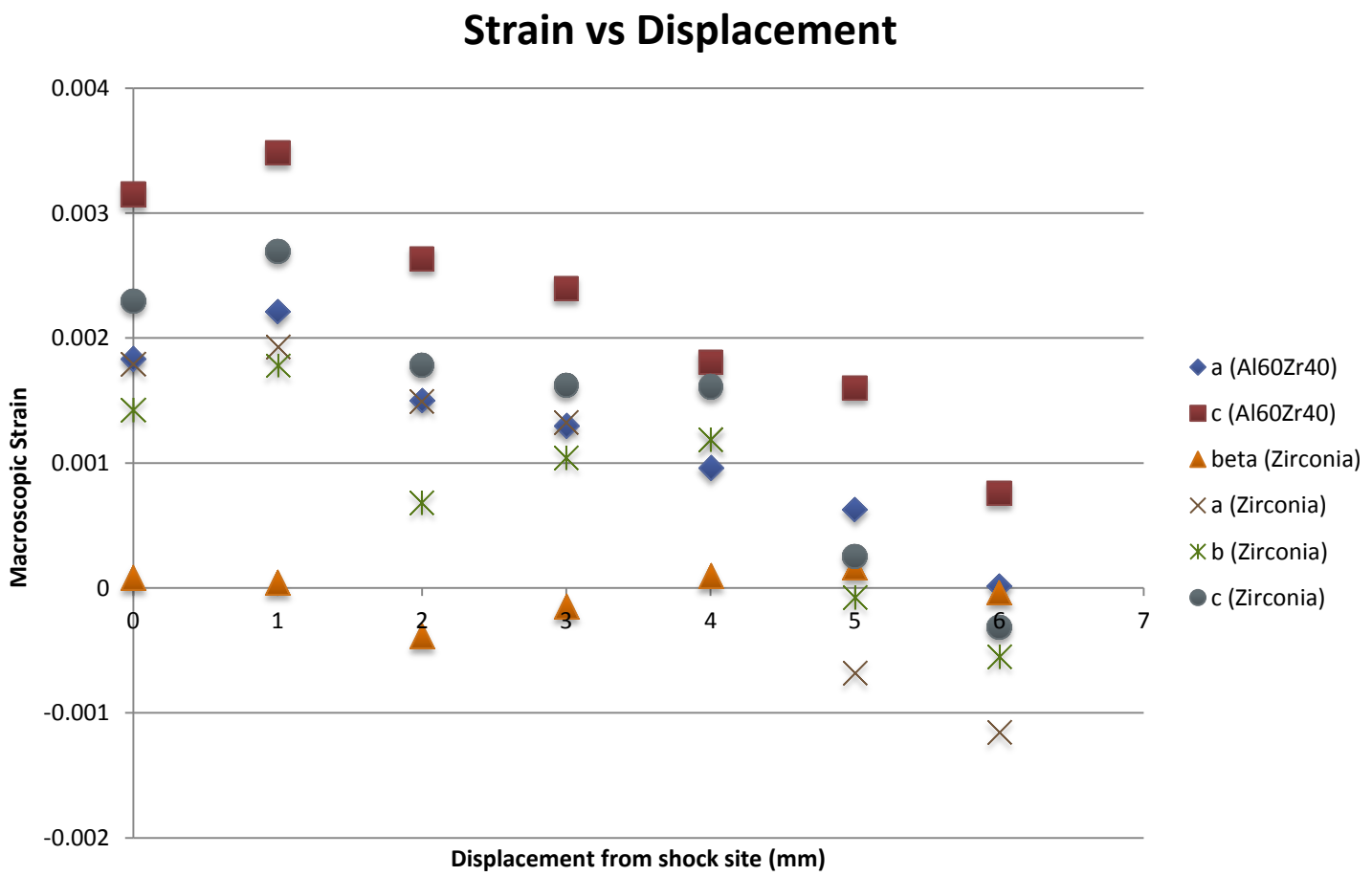
The Sample

- Blend
  - 60% Alumina
  - 40% Zirconia
  - Potassium Binder
- Results…
  - Two phase refractory
    - » Alumina Zirconia (tetragonal)
    - » Zirconia (monoclinic)
      - Potassium (minor phase)

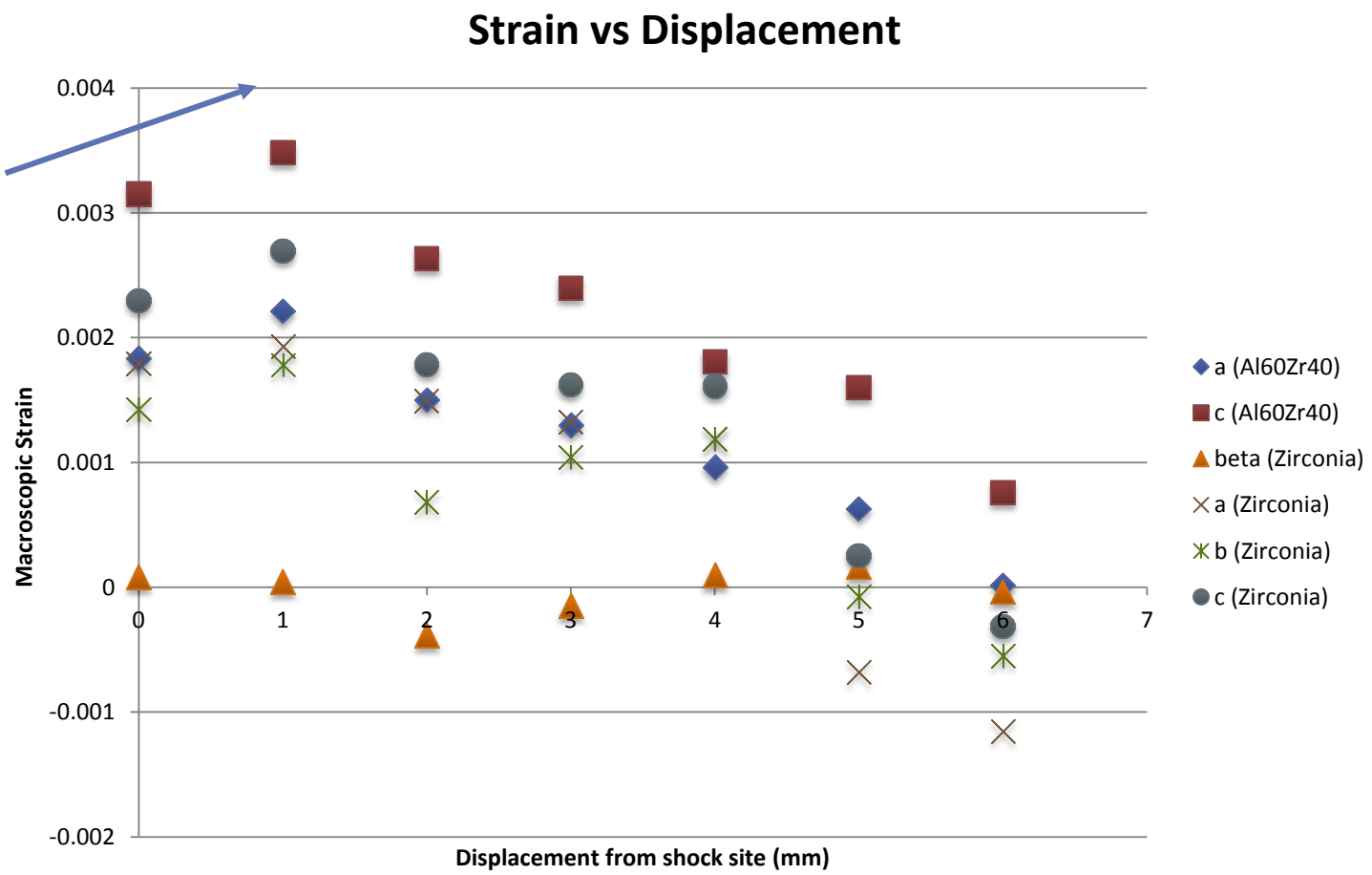


Monoclinic Zirconia

Results


Strain vs Displacement
Macroscopic Strain
0.004
0.003
0.002
0.001
0
-0.001
-0.002
0
1
2
3
4
5
6
7
Displacement from shock site (mm)
a (Al60Zr40)
c (Al60Zr40)
beta (Zirconia)
a (Zirconia)
b (Zirconia)
c (Zirconia)

Results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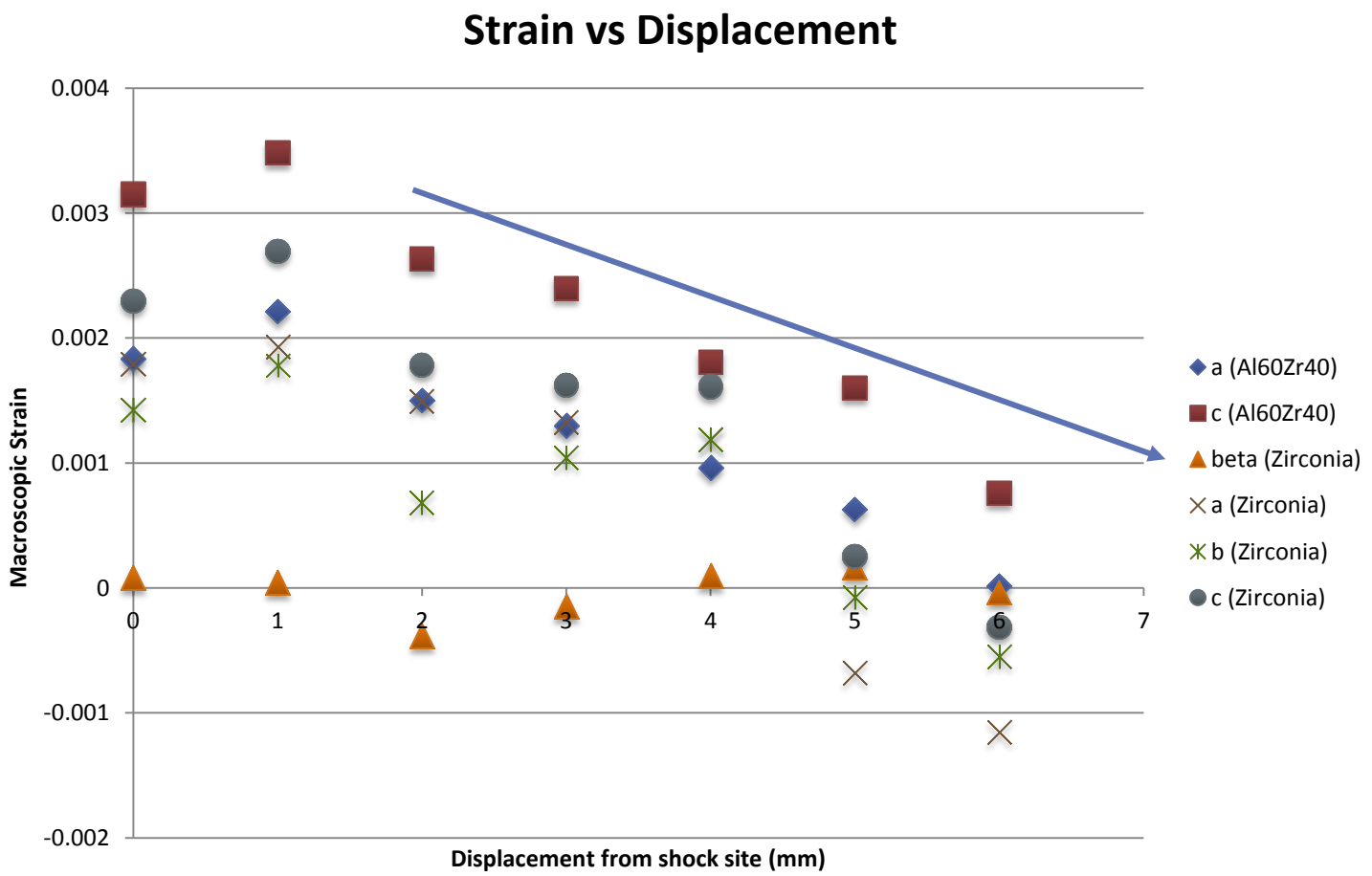
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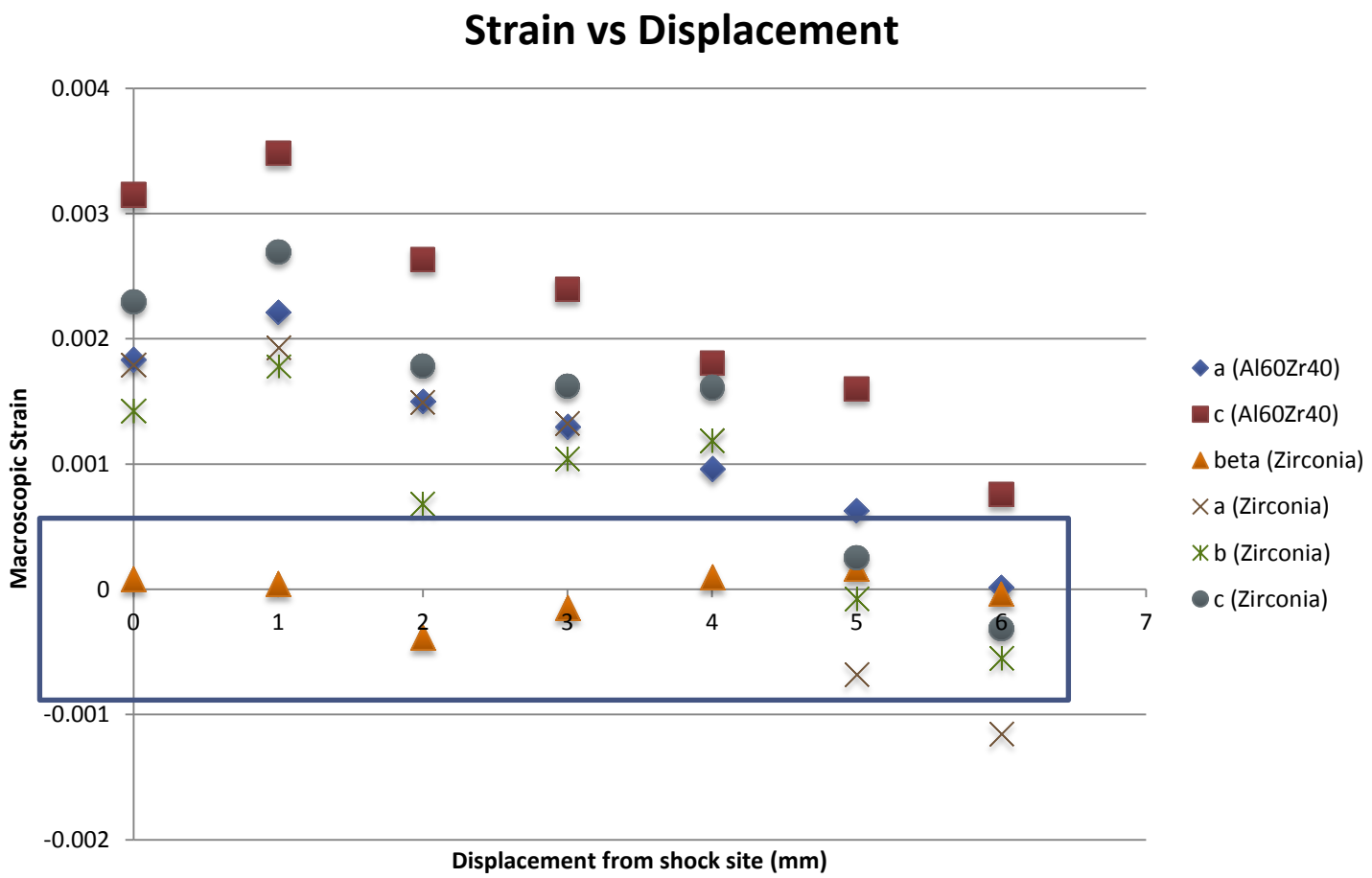
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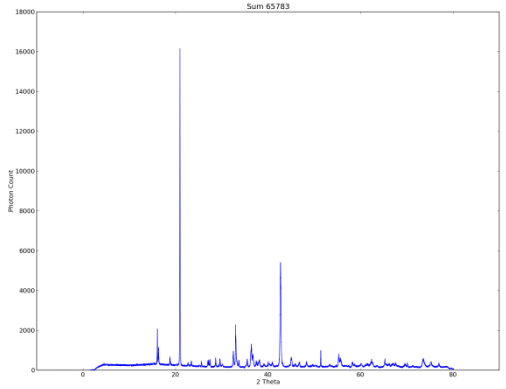
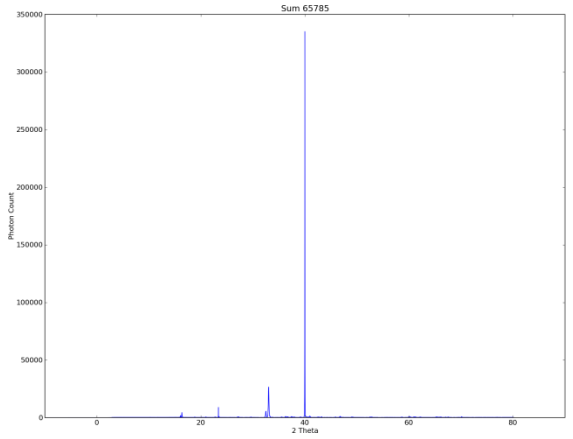
Conclusion so far…

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





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Thanks for Listening

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diamond