AE development at Western University:
Microseismic monitoring of the olivine → spinel transition in fayalite under non-hydrostatic stress

Acoustic Emission Workshop
Jan. 29-31, 2016
GSECARS
The advanced Photon Source

Tim Officer, PhD candidate
Supervisor: Dr. Richard A. Secco

University of Western Ontario
The Problem:

In 1927 Kiyoo Wadati published the first paper clearly demonstrating the existence of deep earthquakes.
Dehydration

Embrittlement (e.g. serpentinite, antigorite)

\[ \alpha \rightarrow \gamma \] transition in Olivine

Shallow Earthquakes

Bimodal Depth Distribution

Green, 1994

Green, 2005
Natural Olivine

$$\text{(Mg}_{1.8}, \text{Fe}_{0.2})\text{SiO}_4$$

Fayalite

$$\text{Fe}_2\text{SiO}_4$$

[Diagram showing depth, pressure, and temperature relationships with phases: 
- Spinel
- Modified Spinel
- Olivine

Akaogi et al., 1989]

[Graph showing pressure vs. temperature with phases: 
Ahrensite (spinel structure)
Fayalite (olivine structure)

Ono et al., 2013]
The Goal

Measure acoustic emissions associated with transformational faulting in mantle silicates

• Conclusive evidence would include:
  ➢ The presence of the high pressure phase in the quenched sample via CT/SEM/XRD
  ➢ The occurrence of micro-faulting in the recovered sample via CT/SEM/TEM
  ➢ Acoustic emissions (AE) accompanying the transformation
  ➢ Locating the acoustic events within the sample
  ➢ First motion “analysis” of waveforms indicate shear faulting (only a crude analysis)

• Requires performing in situ acoustic measurements under non-hydrostatic stress at elevated temperature and pressure
The fayalite was synthesized in a gas mixing furnace with the help of Dr. Tony Withers at the University of Minnesota.

Starting powder

XRD Pattern for sample of synthesized fayalite from Hematite (Fe$_2$O$_3$) and Quartz (SiO$_2$)

Fayalite
Quartz
Fayalite Sintering Experimental Assembly

1. Fayalite powder
2. Ag sample container
3. BN
4. Graphite furnace
5. ZrO₂
6. Pyrophyllite
Sintering Fayalite

Fayalite powder must be *sintered* to create a solid homogeneous sample.

- 1000 ton cubic Press
- Ag sample container
- Pressure: 3.5 - 4 Gpa
- Temperature: 850 °C
- Duration: >10 hrs
High Pressure, High Temperature, *In Situ* Acoustic Emission Experiments on Sintered Fayalite
Experimental Setup
Vallen system (AMSY-6)

• Capable of eight acoustic channels (only use six)
• 40 MHz sampling rate
• 16 bit
• 2 GB data buffers
• Adjustable gain preamps
PICO Sensors from MISTRAS

• 100-1000 KHz response
• -65 °C - 177 °C 5 mm dia.
• 5 mm Dia. X 4mm height
• Sensitive to P-waves
An acoustic “hit” occurs when a transducer receives multiple threshold crossings in rapid succession.
Background Noise During Pressurization

Typical background hit rate upon the first pressurization cycle for a AgCl sample that behaves aseismically.

Large acoustic activity is observed below 150 psi due to gasket formation and compression of the cell.
The pump for the press induces electrical noise in the transducers.
Inserted Diamonite between the cubes and 3 of 6 Transducers
Pressurization to 1.5 - 5 GPa

- Spurious signals were removed as a result of the Diamonite™ discs
- The RMS noise was also substantially reduced
Finalized 18/11 mm Cell Design

Other possibilities used for thermocouple placement
Temperature Calibration Experiment:
Calibrating temperature at the thermocouple position to the center of the sample
Objective 2: Test Pressure Transducer using the Vallen system
Temperature gradient
(It’s several 0°C warmer in the center of the cell than the top/bottom)
Hot Pressed Fayalite Experiments - standardized procedure (but with varying P,T and deformation rate conditions)

- Pressurized slowly to desired confining pressure
- Let sit
- Resume slow pressurization and began heating
- Heated to desired temperature
- Resume pressurization to initiate deformation in the spinel stability field
- Depressurize slowly to avoid fracture upon decompression
Hits and Energy

![Graph showing hits and pressure over time.](image)

- **# of Hits/min.** vs. **Time (s)**
  - Orange line: Hits
  - Blue line: Pressure

![Graph showing energy and pressure over time.](image)

- **Energy (Arbitrary Units)** vs. **Time (s)**
  - Green line: Energy
  - Blue line: Pressure
Events $\geq 4$ hits; $<50\mu s$
Event Location
Velocity and Transducer Position estimation

Pulsing Transducer

Anvils

Receiving Transducer

![Graph showing the relationship between sound velocity and pressure.](image)
The Vallen system locates events (but not to sub mm accuracy)

<table>
<thead>
<tr>
<th>Id</th>
<th>DSET</th>
<th>HHMMSS</th>
<th>MSEC</th>
<th>NSEC</th>
<th>CHAN</th>
<th>A</th>
<th>R</th>
<th>THR</th>
<th>E(TE)</th>
<th>PA0</th>
<th>TRAI</th>
<th>X</th>
<th>Y</th>
<th>Z</th>
<th>CNTS</th>
<th>SIGS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>La</td>
<td>Label 1: '16:15 Resume'</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DT</td>
<td>Wednesday, August 26, 2015,</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Host Time: 4:15 PM</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>LE</td>
<td>633700:19:00 112.9043</td>
<td>112.9043</td>
<td>2</td>
<td>68.2</td>
<td>8.4</td>
<td>40</td>
<td>7.39E+03</td>
<td>380.625</td>
<td>352</td>
<td>1.78</td>
<td>-15.96</td>
<td>-1.66</td>
<td>134</td>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ht</td>
<td>633800:19:00 112.9046</td>
<td>112.9046</td>
<td>3</td>
<td>75</td>
<td>20.2</td>
<td>40</td>
<td>2.61E+04</td>
<td>380.625</td>
<td>353</td>
<td>1.78</td>
<td>-15.96</td>
<td>-1.66</td>
<td>134</td>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ht</td>
<td>633900:19:00 112.9046</td>
<td>112.9046</td>
<td>5</td>
<td>79.5</td>
<td>27.8</td>
<td>40</td>
<td>6.32E+04</td>
<td>380.625</td>
<td>354</td>
<td>360</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ht</td>
<td>634000:19:00 112.9069</td>
<td>112.906875</td>
<td>1</td>
<td>61.4</td>
<td>24</td>
<td>40</td>
<td>1.62E+03</td>
<td>380.625</td>
<td>355</td>
<td>168</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ht</td>
<td>634100:19:00 112.9069</td>
<td>112.9069</td>
<td>7</td>
<td>54.7</td>
<td>7.8</td>
<td>40</td>
<td>5.58E+02</td>
<td>380.625</td>
<td>356</td>
<td>105</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ht</td>
<td>634200:19:00 112.9077</td>
<td>112.907725</td>
<td>6</td>
<td>54.7</td>
<td>24.6</td>
<td>40</td>
<td>5.07E+02</td>
<td>380.625</td>
<td>357</td>
<td>110</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Arrival Time Error (The Vallen system records the time of threshold crossing as the arrival time of the waveform.)

Threshold crossing time.

Amplitude (mV) vs Time (μs)

Δt = 4.5 μs
Akaike Information Criterion (AIC)

Automatic Arrival Time Picker

\[ AIC(k) = k \ln(\text{var}(y(1-k))) + (n_{\text{samp}} - k - 1) \ln(\text{var}(y(k+1:n_{\text{samp}}))) \]

- Separates the signal into two separate signals (background noise and waveform)
- Compares the natural log of the variances of the two signals from 1:n and from n+1:n_{\text{samp}} for each point in the data
- The arrival time is point corresponding to the minimum of the AIC function (i.e. when difference in variances is largest)
- To use this algorithm you need to select the right size window since it is an auto-recursive process
Akaike Information Criterion
Arrival Time Determination

\[ AIC(k) = k \ln(\text{var}(y(1-k))) + (nsamp - k - 1) \ln(\text{var}(y(k+1:nsamp))) \]

For the \( k^{th} \) sample of a waveform of \( n \) samples
Difference in visual and AIC picks for 135 picks

Difference in Manual and AIC picks (# of points)
**Inversion Algorithm**

- **Input data** \( d = [x_i, y_i, z_i, t_i]^T \) positions and arrival time of \( i^{th} \) transducer

- The arrival times are described by
  \[
  t_i = t + \frac{\sqrt{(x_i - x)^2 + (y_i - y)^2 + (z_i - z)^2}}{v} \quad \text{where} \quad v
  \]

- Model parameters \( m = [x, y, z, t]^T \) are the unknown event source time and hypocenter location

- Given the observed arrival times we try to obtain a model \( m \) that best fits the data

- The problem is solved by beginning with a starting model \( m^0 \) at the center of the assembly that predicts data \( d^0 \). We seek changes \( \Delta m \) that improve on our starting model

- The problem is linearized using the first term of a Taylor series expansion and solved iteratively by computing changes to the model using a least squares inversion such that
  \[
  \Delta m = (G^T G)^{-1} G^T \Delta d \quad \text{where} \quad G_{ij} = \frac{\partial d_i}{\partial m_j}
  \]

- The model is improved with each successive iteration until model changes are sufficiently small.
Iterations of the model to find location

Vallen: $x = 1.78$
Corrected: $x = -0.77$

Vallen: $y = -15.96$
Corrected: $y = 1.17$

Vallen: $z = -1.66$
Corrected: $z = 15.12$

The Vallen software used a velocity of 6 mm/µs
The Location algorithm used a velocity of 6.6 mm/µs based on pulsing runs made before and after the experiment
The event locates within the gasket and is primarily explosive
Comparison of location results between the Vallen software and my inversion program for an experiment with an AgCl sample.
HPF_AE_5
Five and Six hit events
HPF_AE_5
Four, Five and Six hit events

Vallen Locations
HPF_AE_5: Strain

Strain \approx 41\%

Strain Rate \approx 1.36 \times 10^{-3}

D3: 3.40 millimeter
P,T space explored

<table>
<thead>
<tr>
<th>Pressure (Gpa)</th>
<th>Temp. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.5 – 7.5</td>
<td>540</td>
</tr>
<tr>
<td>6 – 8</td>
<td>550</td>
</tr>
<tr>
<td>5 – 8</td>
<td>775 - 950</td>
</tr>
<tr>
<td>4.5 – 8.7</td>
<td>775 - 913</td>
</tr>
<tr>
<td>6 – 8</td>
<td>850</td>
</tr>
<tr>
<td>5.8 – 8.7</td>
<td>872</td>
</tr>
<tr>
<td>6 – 8.5</td>
<td>1000</td>
</tr>
</tbody>
</table>
Future Work

• All of the processes involved in designing and constructing my experiments has been accomplished and the system is in full working order.

• Run ~5-10 more experiments on hot pressed fayalite samples with in situ acoustic monitoring under varying conditions of pressure, temperature and strain rate to induce HPHT faulting

• CT scan recovered samples to look for microstructures associated with faulting

• Grind and polish the samples to look for traces of microstructures associated with faulting.
Hg Experiment: Measuring the transition from Liquid to Solid Hg using discontinuity in travel time

Cell Design and Geometry

Anvils

Pulsing Transducer

Receiving Transducer

“Double Capsule” Sample Container
Hg Phase Diagram

Phase Transformation
1.2 Gpa , 25 °C

Hg Liquid

Hg I
(Solid)
Measuring Travel Time Along Three Axis

A total of six transducers provides three sets of acoustic paths through the sample: one parallel to the sample axis and two oblique at an angle of 70.5°.
The phase transition in Hg is indicated by the abrupt change in travel time vs. pressure. Large triangles represent the onset and conclusion of the phase change. The arrows indicate the 2nd pressurization cycle (up) and the 1st and 2nd depressurization cycles (down).
Travel Time Along Each Independent Path

- Comparison of travel time data from all three independent ray paths.
- Data acquired during the first depressurization cycle.
- The travel times for rays traveling parallel to the sample axis are longer since this path passes through the largest amount of Hg (the slowest component in the assembly).
- Travel times for oblique ray paths are similar but not identical.
Sample pressure vs. oil pressure calibration curves for the 3000 multi-anvil apparatus.

- Blue calibration curve is based on the known transition pressures for Bi I-II, Bi III-V and Sn I-II.
- The orange calibration curve includes the two Hg L-\(\alpha\) transition pressures measured in this study.
Detection of a P-induced liquid ⇔ solid-phase transformation using multiple acoustic transducers in a multi-anvil apparatus

Timothy Officer and Richard A. Secco*

Department of Earth Sciences, Western University, London, Canada ON N6A 5B7

(Received 24 December 2014; final version received 25 March 2015)

A technique for detecting and measuring phase transitions in a multi-anvil apparatus by measuring the change in travel time for a longitudinal sound wave as a function of pressure is reported. The system measures the time for pulsed ultrasonic signals to travel through a high pressure assembly with a sample in the center. Upon phase change from liquid to solid, the travel time shows an abrupt decrease due to the intrinsic increase in velocity in the sample and a reduced delay between the triggering of an amplitude threshold and the arrival of the waveform. As a proof of concept, results are shown for mercury as it undergoes pressure-induced liquid ⇔ solid transitions at room temperature. We propose that this non-destructive technique may be valuable in situations where other in situ probing techniques cannot be readily used to provide information about changes of state and potentially to study transition kinetics at high pressures as well.

Keywords: phase transitions; ultrasonics; high pressure; multi-anvil apparatus; Hg
Questions/Advice ?/!

References:

Ono S. et al., 2013. In situ observation of a phase transition in Fe2SiO4 at high pressure and high temperature, 40, 811-816


References (cont.):

Green, H.W., 1994. Solving the paradox of deep earthquakes, Scientific American, 64-71