Thermoelastic and Mechanical Properties of SSP Materials: Simultaneous Ultrasonic and Synchrotron X-ray Studies at High P and T

Baosheng Li
(Stony Brook University)

Feb 19-20, 2019 DOE/SSAP Symposium
2018-2019 Participants  (DE-NA0002907, DE-NA0003886, )

Baosheng Li (SBU, PI)
Robert C. Liebermann (SBU, co-PI)
Nao Cai (SBU Postdoc)
Xington Qi (SBU, Graduate Student)
Siheng Wang (SBU, Graduate Student)
Sibo Chen (SBU, Graduate student)

Former participants
Matthew Whitaker (Beamline Scientist SBU/BNL)
Matthew Jacobsen (Staff Scientist, Institute for Defense Analyses (IDA))

Collaborators
Jianzhong Zhang (LANL)
Hongwu Xu (LANL)
Tony Yu (13-ID-D, GSECARSAPS/U of Chicago)
Yanbin Wang (13-ID-D, GSECARS, APS/U of Chicago)
David Welch (BNL/SBU), Qiang Li (BNL)
Haiyan Chen (6-BM/APS, COMPRES/SBU)
Project/Research Objectives

- **Understanding thermoelastic and mechanical properties of SSP materials under extreme conditions**
  - Sound velocity and density measurements at high pressure and temperature conditions (metallic, covalent, ionic) for precise equation of state determination

- **Education, Training, Technology Transfer**
  - Provide training for next generation scientists, collaborate with and transfer knowledge to national labs

- **Development of Experimental Techniques**
  - New techniques for materials characterization and data analysis
  - Extending ultrasonic measurements to $P > 30$ GPa to bridge the gap between static and dynamic/shock compressions
Our Tools at SBU

Single Stage, Boron Epoxy Pressure Medium, mm sized Sample, P <20 GPa T<1800K (6-BM, APS/COMPRES)

Double Stage MA-8 MgO Pressure Medium P <30 GPa, T<2500K Sample 5-100 mm^3 (also T25 at 13-ID-D, APS) (Test of P> 30 GPa in this press is in progress)

DAC, Liquid or solid Pressure Medium P to Mbar pressures sub-millimeter sample

(ab initio calculations, pseudopotential, FP-LAPW)
Integrated Ultrasonics+X-ray Technique in Multi-anvil, High-pressure Apparatus

- Accurate equation of state and sound velocities (10^{-3} precision)
- Characterization of stress (macroscopic and microscopic), yield strength, and rheology studies

Pressure can be directly calculated using measured V and K of sample (a.k.a “Absolute Pressure”)

**P-V-T Eos**

- CCD/SSD Detector
- Hi Pressure Apparatus
- Incident slits
- X-ray
- Ultrasonic Interferometer
- YAG and CCD Camera
- X-ray Imaging

**Data:**

- Al2O3
- Au
- NaCl

**Graph:**

- Energy, keV
- Count
- Intensity
Publications from 2/2018 to 2/2019


7. Wang, S., et al., Pressure induced softening in orthopyroxene (Am Min, under review)

Ph.D Thesis:

Thermally Induced Anomaly in the Shear Behavior of Magnetite at High Pressure

Yongtao Zou, 1,2,* Wei Zhang, 3 Ting Chen, 2 Xing-ao Li, 4 Chun-Hai Wang, 1 Xintong Qi, 2 Shanmin Wang, 1 Tony Yu, 5 Bingbing Liu, 6 Yanbin Wang, 5 Robert C. Liebermann, 2 Yusheng Zhao, 1 and Baosheng Li 2

1 Academy for Advanced Interdisciplinary Studies, and Department of Physics, Southern University of Science and Technology, Shenzhen 518055, China

2 Mineral Physics Institute, Australian National University, Canberra, Australia

3 School of Science, Southern University of Science and Technology, Shenzhen 518055, China

4 School of Science, Southern University of Science and Technology, Shenzhen 518055, China

5 Center for Advanced Study in Energy Generation, Department of Chemical Engineering, Seoul National University, Seoul 08826, South Korea

6 State Key Laboratory of Coal Resources and Safe Mining, China University of Mining and Technology, Xuzhou 221116, China

(Received 23 September 2017; published 10 May 2018)

Thermoelasticity and thermodynamic effects at high pressures and temperatures are important for understanding the phase transitions of iron oxide minerals. Here, we present new in situ x-ray techniques and high-pressure temperature measurements up to \( \sim 450 \) K, to investigate the pressure and temperature dependences of the magnetic susceptibility for magnetite. The pressure derivative of the Curie temperature is \( \partial B_S / \partial T = -0.0209(10) \) and the effective Gruneisen parameter is \( (\partial^2 G / \partial T^2)_p = -2.5(1) \). These results are consistent with previous studies on the magnetic properties of magnetite. We note that the high-pressure magnetic anomaly in magnetite is a function of the transition from high-pressure inverse spinel to normal spinel with increasing temperature. This finding provides new opportunities to gain a deeper understanding of the magnetic behavior of iron oxide minerals in magnetite-based geophysical systems and under Earth's mantle conditions.

DOI: 10.1103/PhysRevApplied.10.024009
Anomalous shear modulus at P and T: Magnetite Fe₃O₄

$Fd\bar{3}m$, AB₂O₄, Tet A site: Fe³⁺, Oct B site: Fe²⁺ and Fe³⁺ (=> 2.5+)
Pr phase transitions at 300 K
Pr I-II: ~4 GPa; Pr II-III: ~9 GPa.
* e.g., Chesnut and Vohra, 2000; Baer et al., 2003; Dmtriev et al., PRB 2004
Experimental and theoretical studies on the elasticity of tungsten to 13 GPa

Xintong Qi,¹,a) Nao Cai, ² Ting Chen,¹ Siheng Wang,¹ and Baosheng Li¹,²
¹Department of Geosciences, Stony Brook University, Stony Brook, New York 11794, USA
²Mineral Physics Institute, Stony Brook University, Stony Brook, New York 11794, USA

(Received 13 June 2018; accepted 28 July 2018; published online 17 August 2018)

Compressional ($V_P$) and shear wave ($V_S$) velocities of polycrystalline tungsten have been measured up to $\sim$13 GPa at room temperature using ultrasonic interferometry in a multi-anvil apparatus. Using finite strain equation of state results, the elastic constants determined through inversion and theoretical calculations have been compared to previous studies.

![Graphs showing elastic moduli as a function of pressure](image-url)
Poster by Cai et al.: Elasticity of cerium to ~20 GPa

Crystal structure of Ce within 20 GPa
γ: FCC; α: FCC; α’: oC4; α’’: mC4; ε: tI2, BCT.

Characteristics of sound velocity across α’’-BCT phase transition.
Experimental Study on the elasticity of iron-nickel alloy at high pressure: Fe$_{0.90}$Ni$_{0.10}$

Sound velocities at BCC-HCP Transition
Summary of Shear Wave Velocity vs. Density

Vs (km/s)

Density (g/cm³)

X. Qi Ph. D Thesis, 2019
Summary of Compressional Velocity vs. Density

![Graph showing the relationship between compressional velocity (Vp) and density for various elements like Zr, Nb, Mo, Fe, Hf, Ta, and W. The graph highlights the trend across different densities.](image)

X. Qi Ph. D Thesis, 2019
# Thermoelastic properties of hafnium

<table>
<thead>
<tr>
<th></th>
<th>$K_{S0}$ (GPa)</th>
<th>$K_{T0}$ (GPa)</th>
<th>$K_0'$</th>
<th>$\partial K/\partial T$ (GPa/K)</th>
<th>$\partial^2 K/\partial T^2$ (GPa/K^2)</th>
<th>$G_0$ (GPa)</th>
<th>$G_0'$</th>
<th>$\partial G/\partial T$ (GPa/K)</th>
<th>$\partial^2 G/\partial T^2$ (GPa/K^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>111.0</td>
<td>110.9</td>
<td>3.4</td>
<td>-0.019</td>
<td>-3.6x10^{-4}</td>
<td>51.3</td>
<td>0.8</td>
<td>-0.028</td>
<td>0.00028</td>
</tr>
<tr>
<td>2</td>
<td>82.2</td>
<td>82.2</td>
<td>3.9</td>
<td>-0.0012</td>
<td>-4.87x10^{-5}</td>
<td>53.3</td>
<td>0.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>109.5</td>
<td>109.5</td>
<td>3.9</td>
<td></td>
<td></td>
<td>54.7</td>
<td>0.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>110.4</td>
<td>110.4</td>
<td>3.7</td>
<td></td>
<td></td>
<td>56.2</td>
<td>0.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>108.0</td>
<td>108.0</td>
<td>3.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>112.9</td>
<td></td>
<td>3.3</td>
<td></td>
<td></td>
<td>56.2</td>
<td>0.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>116.7</td>
<td></td>
<td>3.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>108</td>
<td></td>
<td>3.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1. This study, finite strain fitting with velocity data. $\alpha = a+bT$ with $a=5.93x10^{-6}$ K^{-1} and $b = 3.19x10^{-8}$ K^{-2}.
2. This study, high temperature Birch-Murnaghan equation of state, with $a=8.7x10^{-6}$ K^{-1} and $b = 6.23x10^{-8}$ K^{-2}.
3. Qi et al, this study
5. Pandey et al. 2014.
Birch’s Law \[ V_B = a \langle M \rangle + b \rho \]
Birch's Law: Why Is It So Good?
Author(s): Dae H. Chung

\[
\omega = V k \quad (2)
\]

Taking the partial derivative of Eq. 2 and evaluating it term by term, we find that

\[
\left( \frac{\partial \ln V}{\partial \ln \rho} \right) = \gamma - 1/3 \quad (3)
\]

\[V = A(\overline{m}) \rho^\lambda\]

\[V: \text{Sound wave velocity}\]

See also
Shankland (1977, Geophysical Surveys, 3, 69-100)
Liebermann and Ringwood (1973);
Anderson, O.L (1973, JGR)

Shaner, et al. (LA-UR--87-2233, Report,

\[\frac{d \ln v_b}{d \ln \rho} = \frac{1}{2} \left( \frac{d \ln K}{d \ln \rho} - 1 \right)\]

\[= \frac{1}{2} (dK/dP -1)\]
Sound velocity and density measurements of liquid iron up to 800 GPa: A universal relation between Birch’s law coefficients for solid and liquid metals

Tatsuhiro Sakaiya\textsuperscript{a,*}, Hideki Takahashi\textsuperscript{a}, Tadashi Kondo\textsuperscript{a}, Toshihiko Kadono\textsuperscript{b,1}, Yoichiro Hironaka\textsuperscript{b}, Tetsuo Irifune\textsuperscript{c,d}, Keisuke Shigemori\textsuperscript{b}

<table>
<thead>
<tr>
<th>Element</th>
<th>Phase</th>
<th>Formula</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ta</td>
<td>Liquid</td>
<td>$0.30\rho - 1.98$</td>
<td>Shock (Shaner et al., 1984)</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>$0.19\rho + 1.65$</td>
<td></td>
</tr>
<tr>
<td>Mo</td>
<td>Liquid</td>
<td>$0.65\rho - 1.53$</td>
<td>Shock (Hixson et al., 1989)</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>$0.40\rho + 3.25$</td>
<td></td>
</tr>
<tr>
<td>V</td>
<td>Liquid</td>
<td>$1.25\rho - 3.91$</td>
<td>Shock (Dai et al., 2001)</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>$0.65\rho + 3.53$</td>
<td></td>
</tr>
<tr>
<td>NaCl</td>
<td>Liquid</td>
<td>$2.28\rho - 0.85$</td>
<td>Shock (Boness and Brown, 1990)</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>$2.62\rho - 1.13$</td>
<td>BS (Campbell and Heinz, 1992)</td>
</tr>
<tr>
<td>CsBr</td>
<td>Liquid</td>
<td>$0.57\rho + 0.23$</td>
<td>Shock (Boness and Brown, 1993)</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>$0.85\rho - 1.23$</td>
<td></td>
</tr>
<tr>
<td>W</td>
<td>Liquid</td>
<td>$0.42\rho - 3.70$</td>
<td>IEX (Hixson and Winkler, 1990)</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>$0.62\rho - 6.70$</td>
<td></td>
</tr>
<tr>
<td>Pb</td>
<td>Liquid</td>
<td>$0.11\rho - 1.82$</td>
<td>IEX (Hixson et al., 1985)</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>$0.45\rho - 1.82$</td>
<td></td>
</tr>
</tbody>
</table>
Test of Birch’s Law: W

\[
\frac{V_\phi}{V_{\phi_0}} = (A - B + C) + (B - 2C) \left( \frac{\rho}{\rho_0} \right) + C \left( \frac{\rho}{\rho_0} \right)^2
\]
Birch’s Law with Ultrasonic and DAC Data
Summary of Activities and Outlook

• **Sound velocities** of transition metals (and alloys) and lanthanides have been studied at high pressure and high temperatures using ultrasonic and X-ray studies, the derived elasticity, $K$, $G$, $\partial K/\partial P$, $\partial G/\partial P$, $dK/dT$, $dG/dT$ contribute to those databases maintained at national labs for SSP studies.

• **Towards a higher pressure range (P > 30 GPa).** Progress have been made to apply these measurements to lanthanides (Ce, Pr) to further bridge the gap between static [multi-anvil and DAC] and dynamic [shock] high-pressure experiments. The current experimental data are used for exploration of Birch’s law.

• **Education and training,** Provided Education and training for the next generation scientists for the stewardship science program as well as technology transfer for conducting advanced experiments using X-ray/neutron source at APS/LANL.