Dilatometry

PHY 590B S14

Sergey L. Bud’ko

Materials from the 2007 presentation of George Schmiedeshoff, Occidental College were heavily used.
Dilation: $\Delta V$ (or $\Delta L$)

Intensive Parameters:

- $T$: thermal expansion: $\beta = \frac{d\ln(V)}{dT}$
- $H$: magnetostriction: $\lambda = \frac{\Delta L(H)}{L}$
- $P$: compressibility: $\kappa = \frac{d\ln(V)}{dP}$
- $E$: electrostriction: $\xi = \frac{\Delta L(E)}{L}$
- *etc.*
A (very) Brief History

- **Heron** of Alexandria (0±100): Fire heats air, air expands, opening temple doors (first practical application...).

- **Galileo** (1600±7): Gas thermometer.

- **Fahrenheit** (1714): Mercury-in-glass thermometer.

- **Mie** (1903): First microscopic model.

- **Grüneisen** (1908): $\beta(T)/C(T) \sim \text{constant}$. 
Phase Transition: 

2\textsuperscript{nd} Order Phase Transition, Ehrenfest Relation(s):

$$\frac{dT_{N2}}{dp_c} = V_M T_{N2} \frac{\Delta \alpha_c}{\Delta C_p}$$

1\textsuperscript{st} Order Phase Transition, Clausius-Clapyeron Eq(s):

$$\frac{dT_{N1}}{dp_c} = \frac{\Delta V}{\Delta S} \approx V_M \frac{\Delta (\frac{\Delta L}{L})}{\Delta S}$$
Paul Ehrenfest (1880-1933)

PhD from Vienna Technical University
1904 – married Tatyana Alexeyevna Afanasieva – Russian mathematician
1907-1912 – St. Petersburg, Russia (happiest days of his life)
1912-1933 - professor in Leiden

Ehrenfest theorem
Ehrenfest paradox
Ehrenfest-Tolman effect
Classification of phase transitions
Ehrenfest time
Spinor

He was not merely the best teacher in our profession whom I have ever known; he was also passionately preoccupied with the development and destiny of men, especially his students. A. Einstein
**Grüneisen Theory** (one energy scale: $U_o$)

\[
\Gamma \equiv -\frac{\partial \ln U_o}{\partial \ln V} = V_M \kappa(T) \frac{\beta(T)}{C_p(T)} = \text{const.}
\]

\[U_o \propto V^{-\Gamma}\]

*e.g.:* If $U_o = E_F$ (ideal) then:  \[
\Gamma_{IFG} = \frac{2}{3}
\]
Grüneisen Theory (multiple energy scales: $U_i$ each with $C_i$ and $\Gamma_i$)

$$\Gamma \equiv \frac{\sum_i C_i(T) \Gamma_i}{\sum_i C_i(T)} = \Gamma(T)$$

Examples: Simple metals: $\Gamma \sim 2$

$$\Gamma_e = \frac{2}{3} + \frac{d \ln(m^*)}{d \ln(V)}$$

e.g.: phonon, electron, magnon, CEF, Kondo, RKKY, etc.
Example (Noble Metals):

After White & Collins, JLTP (1972).
(Γ_lattice shown.)
Example (Heavy Fermions):

\[ \Gamma_{HF}(0) \]

After deVisser et al. (1990)
Dilatometers

- Mechanical (pushrod *etc.*)
- Optical (interferometer *etc.*).
- Electrical (Inductive, Capacitive, Strain Gauges).
- Diffraction (X-ray, neutron).
- Others (absolute & differential).
push-rod with inductive readout
(Ames Laboratory thermal analysis facility in Wilhelm Hall)
Optical dilatometer - interferometer

nanometer resolution, commercially available
Strain gauges

Gauge factor, $GF = \frac{\Delta R/R}{\Delta L/L}$ is usually about 2 (for metal film gauges)
Strain gauges

**GOOD:**
- small, simple, cheap
- can be used with a rotator
- can measure $\Delta x$, $\Delta y$, $\Delta z$ in one experiment
- can be used in pressure cells

**NOT SO GOOD:**
- glue?
- calibration?
- low T sensitivity?
- re-usability?
- dissipation at low temperatures?
- “large” samples
- effects of magnetic field?
High resolution miniature dilatometer based on an atomic force microscope piezocantilever

J.-H. Park,¹ D. Graf,¹ T. P. Murphy,¹ G. M. Schmiedeshoff,² and S. W. Tozer¹

¹National High Magnetic Field Laboratory, Florida State University, Tallahassee, Florida 32310, USA
²Department of Physics, Occidental College, Los Angeles, California 90041, USA

(Received 18 September 2009; accepted 13 October 2009; published online 12 November 2009)
Piezoresistive cantilever dilatometer
Piezoresistive cantilever dilatometer

[a] [b] [c]

Capacitive Dilatometer (sample #2 thickness 0.4 mm)

Piezoresistive Dilatometer (sample #1 thickness 0.04 mm)

R (mΩ)

Cap (pF)

V_{AB} (μV)

Temperature (K)

H = 0 T
H = 10 T
H = 15 T

α - U

α_1
α_2
α_3
Piezoresistive cantilever dilatometer

**GOOD:**
- very small and compact
- can be used with a rotator
- atomic resolution
- can be used in pulsed fields
- environmentally insensitive

**NOT SO GOOD:**
- cell effects?
- calibration?
- device to device reproducibility?
- re-usability?
- dissipation at low temperatures?
fiber Bragg grating dilatometer

High resolution magnetostriction measurements in pulsed magnetic fields using fiber Bragg gratings

Ramzy Daou,1,4,a Franziska Weickert,1,4,a Michael Nicklas,1 Frank Steglich,1
Ariane Haase,2,4,a and Mathias Doerr2

1Max Planck Institute for the Chemical Physics of Solids, 01187 Dresden, Germany
2Institut für Festkörperphysik, TU Dresden, 01062 Dresden, Germany

(Received 1 February 2010; accepted 16 February 2010; published online 26 March 2010)
The Bragg wavelength, $\lambda_B$, of an FBG is related to the refractive index of the optical fiber, $n$, and the pitch of the grating, $\Lambda$,

$$\lambda_B = 2n\Lambda.$$  \hspace{1cm} (1)

The strain and temperature dependence of $\lambda_B$ are given by

$$\frac{\Delta \lambda_B}{\lambda_B} = \left(1 - \frac{n^2}{2} (P_{12} - \nu (P_{11} + P_{12}))\right) \frac{\Delta L}{L}$$

$$+ \left(\alpha(T) + \frac{1}{n} \frac{dn}{dT}\right) \Delta T.$$  \hspace{1cm} (2)

The strain response depends on the collection of terms $1 - (n^2/2) [P_{12} - \nu (P_{11} + P_{12})] \approx 0.76$, which has minimal temperature dependence from 2 to 300 K for FBGs in silica fiber. $P_{11}$ and $P_{12}$ are components of the strain-optic tensor and $\nu$ is the Poisson ratio. The strain response of an FBG is thus linear with the same constant of proportionality at all temperatures, and no calibration is required.
fiber Bragg grating dilatometer

GdSb 5.4K

GdSi 4.8K
fiber Bragg grating dilatometer

GOOD:
- intrinsically calibrated
- ~ 1A resolution
- can be used in pulsed fields
- environmentally insensitive
- little dissipation at low T

NOT SO GOOD:
- cell effects?
- only L || H
- re-usability?

suggested to work in piston – cylinder pressure cell ??
XRD dilatometry

Less accurate

Tedious/expensive

VERY useful for structural phase transitions (gives structural information)
Capacitive Dilatometer (Cartoon)

\[ C = \varepsilon_o \frac{A}{D} \]
- Cell body: OHFC Cu.
- BeCu spring (c).
- Stycast 2850FT (h) and Kapton (i) insulation.
- Sample (d).
Calibration

- Use sample platform to push against lower capacitor plate.
- Rotate sample platform (θ), measure C.
- $A_{\text{eff}}$ from slope (edge effects).
- $A_{\text{eff}} = A_0$ to about 1%
- "Ideal" capacitive geometry.
- Consistent with estimates.
- $C_{\text{MAX}} \gg C$: no tilt correction.

Operating Region

$C_{\text{MAX}} \geq 65 \text{ pF}$
Cell Effect

\[ \alpha_{Sample} = \frac{1}{L} \frac{dL}{dT} \left|_{Sample+Cell} - \frac{1}{L} \frac{dL}{dT} \left|_{Cell+Cu} + \alpha_{Cu} \right. \]

Much smaller cell effect, above \( \sim 10K \), using quartz (or sapphire?) instead of Cu for cell body. See, for example, Neumeier et al. (2005) and references therein.
Tilt Correction

- If the capacitor plates are truly parallel then $C \to \infty$ as $D \to 0$.
- More realistically, if there is an angular misalignment, one can show that $C \to C_{\text{MAX}}$ as $D \to D_{\text{SHORT}}$ (plates touch) and that

$$D = \frac{\varepsilon_0 A}{C} \left[ 1 + \left( \frac{C}{C_{\text{MAX}}} \right)^2 \right]$$


- For our design, $C_{\text{MAX}} = 100 \text{ pF}$ corresponds to an angular misalignment of about $0.1^\circ$.
- Tilt is not always bad: enhanced sensitivity is exploited in the design of Rotter et al. (1998).
Kapton Bad (thanks to A. deVisser and Cy Opeil)

- Replace Kapton washers with alumina.
- New cell effect scale.
- Investigating sapphire washers.
The dilatometer is sensitive to magnetic torque on the sample (induced moments, permanent moments, shape effects...).

Manifests as irreproducible magnetostriction (for example).

Best solution (so far...): glue sample to platform.

Duco cement, GE varnish, N-grease...

Low temperature only. Glue contributes above about 20 K.
Hysteresis ~Bad

- Cell is very sensitive to thermal gradients: thermal hysteresis. But slope is unaffected if T changes slowly.

- Magnetic torque on induced eddy-currents: magnetic hysteresis. But symmetric hysteresis averages to “zero”:
Quantum Oscillations

Thermal expansion and magnetostriction of pure and doped RAgSb$_2$ (R = Y, Sm, La) single crystals

S L Bud’ko$^1$, S A Lam$^{+1,2}$, P C Canfield$^1$, G D Samolyuk$^1$, M S Torikachvili$^3$ and G M Schmiedeshoff$^3$

$$\epsilon_i = -MH \frac{\partial \ln S_m}{\partial \sigma_i}$$

Can estimate uniaxial pressure derivatives of the extremal FS cross-sections
Tricritical Phenomena at the $\gamma \to \alpha$ Transition in Ce$_{0.9-x}$La$_x$Th$_{0.1}$ Alloys

J. C. Lashley,$^1$ A. C. Lawson,$^1$ J. C. Cooley,$^1$ B. Mihaila,$^1$ C. P. Opeil,$^1$ L. Pham,$^1$ W. L. Hults,$^1$ J. L. Smith,$^1$ G. M. Schmiedeshoff,$^2$ F. R. Drymiotis,$^3$ G. Chapline,$^4$ S. Basu,$^5$ and P. S. Riseborough$^5$
Anisotropic thermal expansion and magnetostriction of YNi$_2$B$_2$C single crystals

S I. Bud'ko$^1$, G M Schmiedeshoff$^2$, G Lapertot$^{1,3}$ and P C Canfield$^1$

![Graph showing anisotropic thermal expansion and magnetostriction of YNi$_2$B$_2$C single crystals.](image)
Magnetic-Field-Induced Lattice Anomaly inside the Superconducting State of CeCoIn$_5$:
Anisotropic Evidence of the Possible Fulde-Ferrell-Larkin-Ovchinnikov State

V.F. Correa,$^1$ T.P. Murphy,$^1$ C. Martin,$^1$ K.M. Purcell,$^{1,2}$ E.C. Palm,$^1$ G.M. Schmiedeshoff,$^3$
J.C. Cooley,$^3$ and S.W. Tozer$^1$

---

---
The Good & The Bad

- Small (scale up or down).
- Open architecture.
- Rotate *in-situ* (NHMFL/TLH).
- Vacuum, gas, or liquid (magnetostriction only?).
- Sub-angstrom precision.
- Cell effect (T ≥ 2K).
- Magnetic torque effects.
- Thermal and magnetic hysteresis.
- Thermal contact to sample in vacuum (T ≤ 100mK?).
Recommended:

- Book (broad range of data on technical materials *etc.*): *Experimental Techniques for Low Temperature Measurements*, Ekin, 2006.
- ...and references therein.