Photoacoustic determination of mechanical properties at microstructure level

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Studying microscopic elasticity in poroelastic materials

Motivation:
- Macroscopic elasticity matters in vibration decoupling/damping applications
- Macroscopic elasticity is different from microscopic elasticity: depends on microscopic elasticity and on frame morphology
- Microscopic elasticity is needed for understanding and tuning macroscopic elasticity

Challenges:
- The elastic moduli are frequency dependent and complex
- Classical methods are limited in frequency range
- The acoustic wavelength at the audio frequencies of interest is much longer than the pore size
- Guided wave propagation is intrinsically frequency dependent

Approach:
- Time-temperature superposition principle
- Guided acoustic wave dispersion analysis

Hooke’s law: 1678: “ut tensio sic vis”
Studying microscopic elasticity in poroelastic materials

Motivation:

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Foam slab: \( m \): mass, \( S \): surface, \( d \): thickness, \( k \): spring constant, \( M \): modulus, \( \eta \): damping constant

\[
\begin{align*}
\omega_1 - \omega_2 &= \frac{\eta}{m} \\
\omega_{\text{max}}^2 &= \frac{k}{m} - \frac{\eta^2}{4m^2}
\end{align*}
\]
Studying microscopic elasticity in poroelastic materials

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Elasticity of porous materials: static case

Hexagonal lattice:

\[
E_{\text{grid}} = \frac{2}{\sqrt{3}(3M + N)}
\]

Random lattice:

\[
E_{\text{grid}} = \frac{M + N}{M(3N + M)}
\]

With the compliances \( M \) and \( N \) given by:

\[
M = \frac{L}{Et_0}, \quad N = \frac{4L^3}{Et_0^3}, \quad \phi = \frac{t_0}{\sqrt{3L}} \left(1 - \frac{t_0}{4\sqrt{3L}}\right)
\]

When the filled volume fraction \( \phi \rightarrow 0 \), then

\[
M \rightarrow \frac{1}{\sqrt{3E\phi}}, \quad N \rightarrow \frac{1}{3\sqrt{3E\phi^3}} \quad \Rightarrow \quad E_{\text{random grid}} \rightarrow E \frac{\phi}{\sqrt{3}}
\]
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Chemical reagents (polyurethane)

Physical circumstances during production
• temperature
• gas pressure
• surfactant
• surface tension
• viscoelasticity
• vitrification

Morphology and composition of finally formed foam depend on
• timing of cease of chemical reaction
• timing of cease of reaction gas pressure driven foam cell expansion

For validation of tuning attempts and insight: characterization of final foam properties needed
Studying microscopic elasticity in poroelastic materials

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**Frequency dependent moduli: relaxation behavior**

**Temperature dependent relaxation frequency**
Studying microscopic elasticity in poroelastic materials

Challenges:

• The elastic moduli are frequency dependent and complex
• **Classical methods are limited in frequency range**
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Mass-spring resonance experiment

\[ k \approx \omega_0^2 m = \frac{MS}{d} \]

Dynamic mechanical analysis

\[ k = \frac{F}{\Delta x} = \frac{pSd}{\Delta xd} = \frac{pS}{\varepsilon d} = \frac{MS}{d} \]

Only one frequency at a time and per sample

Limited to low frequencies
Studying microscopic elasticity in poroelastic materials

Approach:

Photoacoustic and photothermal phenomena: extracting thermal and elastic information from spatial and temporal dependence of temperature and displacement.

2D, non-uniform excitation pattern

⇒ information on transport properties:
⇒ thermal diffusivity/diffusion length & acoustic velocity and damping/wavelength
Studying microscopic elasticity in poroelastic materials

Challenges:
- Classical methods are limited in frequency range

Approach:
⇒ elastic wave propagation analysis

\[ c = \sqrt{\frac{M}{\rho}} \]
Studying microscopic elasticity in poroelastic materials

Approach:
⇒ elastic wave propagation dispersion analysis

$c(\omega) = \sqrt{\frac{M(\omega)}{\rho}}$

Fit real and imaginary part of elastic moduli

2D Fourier transform

$\omega$

$k$

$t$

$x$

Probe-pump distance

Frequency (Hz)

Phase velocity (m/s)

2050
2100
2150
2200

$10^6$
$10^7$
$10^8$
$10^9$

$\omega$

$c=\omega/k$
Studying microscopic elasticity in poroelastic materials

Challenges:

- The elastic moduli are frequency dependent and complex
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Finite size effects, scattering due to mode conversion and reflection

Wavelength $\lambda \ll$ strut length: 😊

Wavelength $\lambda \sim$ strut length: 😉

Wavelength $\lambda \gg$ strut length: effective medium behavior or microscopic behavior?
Studying microscopic elasticity in poroelastic materials

Example of interaction of Lamb wave with vertical crack

→ incident mode: $A_0$

→ defect $= 0.4L$

Aluminium plate $L = 1$ mm, $\omega = 2.3 \times 10^6$ rad/s, 1198 basis modes, $A_0$ in
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Examples of scattering at irregularities

Scattering upon reflection on an irregular wall

Scattering of a periodic plane wave in a polycrystal
Studying microscopic elasticity in poroelastic materials

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Longitudinal, transverse and Rayleigh waves in homogeneous media: non-dispersive

Rayleigh waves: penetration depth < sample thickness ⇒ non-dispersive
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Waves in beams, plates, struts, membranes: wavelength > thickness ⇒ dispersive + multi-mode

How to untangle material dispersion from guided wave dispersion?

https://commons.wikimedia.org/wiki/Category:Beam_vibrationAnimations
Studying microscopic elasticity in poroelastic materials

Challenge: **Guided wave propagation is intrinsically frequency dependent**

Ideal world scenario of extraction of elastic modulus from wave propagation characteristics

2D Fourier transform

Fit real and imaginary part of elastic moduli

$c = \omega/k$
Studying microscopic elasticity in poroelastic materials

Challenge: **Guided wave propagation is intrinsically frequency dependent**

Real world scenario: combined dispersion due to guided wave character and material relaxation
Studying microscopic elasticity in poroelastic materials

**Challenge:** Guided wave propagation is intrinsically frequency dependent

Real world scenario: combined dispersion due to guided wave character and material relaxation

Dispersion due to Lamb wave character

Combined dispersion due to Lamb wave character and material relaxation

Challenging to unravel dispersion due to material relaxation from total dispersion (guided wave & relaxation)
Studying microscopic elasticity in poroelastic materials

Challenge: complex morphology of struts and membranes

Strategy:
- Investigate influence of fiber cross section by simulations

<table>
<thead>
<tr>
<th>cross section shape</th>
<th>Young's modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fitting result with infinite plate</td>
</tr>
<tr>
<td>square</td>
<td>3.8 ± 0.2</td>
</tr>
<tr>
<td>triangular</td>
<td>2.9 ± 0.4</td>
</tr>
<tr>
<td>circular</td>
<td>3.4 ± 0.2</td>
</tr>
</tbody>
</table>
Studying microscopic elasticity in poroelastic materials

Approach:

- **Time-temperature superposition principle**
- Guided acoustic wave dispersion analysis

\[ E(\omega_1, T_1) = E_\infty + \frac{\Delta E}{1 + i \frac{\omega_1}{\omega_{\text{rel}}(T_1)}} \]

is to be found in experimentally inaccessible \( \omega_1, T_1 \) range

\[ \left( \frac{E(\omega_2, T_2) - E_\infty}{\Delta E} \right)^{-1} - 1 = i \frac{\omega_2}{\omega_{\text{rel}}(T_2)} \]

is known from experiment in feasible \( \omega_2, T_2 \) range

\[ \omega_{\text{rel}}(T_1) = \omega_x \exp \left( -\frac{B}{T_1 - T_0} \right) \]

is known by extrapolating VFT law from fit of B and T_0 in experimentally accessed \( \omega_2, T_2 \) range

\( E_\infty \) and \( \Delta E \) are known from experimentally accessed \( \omega_2, T_2 \) range

Scaling of relaxation frequency:

\[ \omega_{\text{rel}}(T_1) = \omega_{\text{rel}}(T_2) \exp \left( +\frac{B}{T_2 - T_0} - \frac{B}{T_1 - T_0} \right) \]

Scaling of frequency:

\[ \frac{i \omega_1}{\omega_{\text{rel}}(T_1)} = \frac{\omega_1}{\omega_2} \frac{i \omega_2}{\omega_{\text{rel}}(T_2)} \exp \left( -\frac{B}{T_2 - T_0} + \frac{B}{T_1 - T_0} \right) = \frac{\omega_1}{\omega_2} \left( \frac{E(\omega_2, T_2) - E_\infty}{\Delta E} \right)^{-1} \exp \left( -\frac{B}{T_2 - T_0} + \frac{B}{T_1 - T_0} \right) \]

Substitution of relaxation behavior into scaled frequency term:

\[ E(\omega_1, T_1) = E_\infty + \frac{\Delta E}{1 + i \frac{\omega_1}{\omega_{\text{rel}}(T_1)}} = E_\infty + \left( \frac{E(\omega_2, T_2) - E_\infty}{\Delta E} \right)^{-1} \exp \left( -\frac{B}{T_2 - T_0} + \frac{B}{T_1 - T_0} \right) \]
Studying microscopic elasticity in poroelastic materials

Approach:

- **Time-temperature superposition principle**
- Guided acoustic wave dispersion analysis

\[ M(t, T_1) \]

shift factor \( \alpha_t(T_2/T_1) \)

(e.g. Williams-Landel-Ferry (WLF) relation)

\[ M(\alpha_t t, T_2) \]
Studying microscopic elasticity in poroelastic materials

Approach:
- Time-temperature superposition principle
- Guided acoustic wave dispersion analysis

Validation step: piezoelectrically and photoacoustically excited guided waves along a nylon wire

Approach:
- Local excitation: small source, short wavelength $\lambda$, high frequency $f_{\text{exc}}$
- Local detection:
  - vibrometer probe spot size $<<\lambda$
- Local guided wave velocity and damping
  $\rightarrow$ local real and imaginary part of elastic modulus
Studying microscopic elasticity in poroelastic materials

**Approach:**

- Time-temperature superposition principle
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**Approach:**

- Local excitation: small source, short wavelength $\lambda$, high frequency $f_{\text{exc}}$
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Young’s modulus (+ Poisson’s ratio) by curve fitting
Studying microscopic elasticity in poroelastic materials

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Validation step: piezoelectrically and photoacoustically excited guided waves along a nylon wire
Studying microscopic elasticity in poroelastic materials

**Approach:**
- Time-temperature superposition principle
- **Guided acoustic wave dispersion analysis**

**Validation step:** piezoelectrically and photoacoustically excited guided waves along a nylon wire

\[
v_{A0,f \rightarrow 0} = \sqrt{c_T \left(1 - \frac{c_T^2}{c_L^2}\right)^{1/4}} \sqrt{\frac{2\pi fd}{\sqrt{3}}}
\]

\[
v_{50,f \rightarrow 0} = 2c_T \left(1 - \frac{c_T^2}{c_L^2}\right)^{1/2}
\]

$\gamma_{\text{wire, ps}} = (4.5 \pm 0.1)$ GPa

$\gamma_{\text{wire, ns}} = (2.92 \pm 0.07)$ GPa

$\gamma_{\text{wire, ps}} = (3.61 \pm 0.07)$ GPa

Poisson ratio: 0.4 fixed or fitted with large uncertainty

\[
\rho_{\text{nylon}} = 1200\text{kg/m}^3 \quad d_{\text{nylon}} = 150\text{micron}
\]
Studying microscopic elasticity in poroelastic materials

Approach:
• Time-temperature superposition principle
• **Guided acoustic wave dispersion analysis**

Validation step: piezoelectrically and photoacoustically excited guided waves along a nylon wire

\[ Y_{wire, piezo-needle} = (4.5 \pm 0.1) \text{ GPa} \]
\[ Y_{wire, ps} = (2.92 \pm 0.07) \text{ GPa} \]
\[ Y_{wire, ns} = (3.61 \pm 0.07) \text{ GPa} \]
\[ Y_{wire, DMA, TT} = (5 \pm 1) \text{ GPa} \]
Studying microscopic elasticity in poroelastic materials

Approach:
- Time-temperature superposition principle
- **Guided acoustic wave dispersion analysis**

Photoacoustically excited guided waves along a 2D foam-like polyamide grid
Studying microscopic elasticity in poroelastic materials

Approach:

- Time-temperature superposition principle
- Guided acoustic wave dispersion analysis

Photoacoustically excited guided waves along a regular polyamide 2D grid

Cubic/orthorhombic grids/lattices with Slenderness/unit cell Aspect Ratio (AR) 1:1 and 2:1 and different unit cell dimensions (15mm and 7.5mm)
Studying microscopic elasticity in poroelastic materials

Approach:
- Time-temperature superposition principle
- **Guided acoustic wave dispersion analysis**

Photoacoustically excited guided waves along a regular polyamide 2D grid

**Cubic grid 7.5mm**

For comparison/calibration: single strut
Studying microscopic elasticity in poroelastic materials

Approach:
- Time-temperature superposition principle
- Guided acoustic wave dispersion analysis

Photoacoustically excited guided waves along a regular polyamide 2D grid

Cubic grid 7.5mm

Comparison between
- experimental
- FEM-simulated dispersion curve

Effects of mode conversion at strut crossings ~ phononic bandgaps
Studying microscopic elasticity in poroelastic materials

Approach:

- Time-temperature superposition principle
- Guided acoustic wave dispersion analysis

Photoacoustically excited guided waves along a regular polyamide 2D grid
Studying microscopic elasticity in poroelastic materials

**Approach:**
- Time-temperature superposition principle
- **Guided acoustic wave dispersion analysis**

Photoacoustically excited guided waves along a regular polyamide 2D grid
Studying microscopic elasticity in poroelastic materials

Approach:
- Time-temperature superposition principle
- Guided acoustic wave dispersion analysis

Photoacoustically excited guided waves along a random polyamide 2D grid

<table>
<thead>
<tr>
<th>Sample</th>
<th>Young’s modulus (GPa)</th>
<th>Poisson’s ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Random</td>
<td>2.00 ± 0.05</td>
<td>0.33 ± 0.02</td>
</tr>
<tr>
<td>3D 1:1</td>
<td>2.01 ± 0.02</td>
<td>&lt; 0.35</td>
</tr>
<tr>
<td>3D 1:2</td>
<td>1.50 ± 0.02</td>
<td>&lt; 0.35</td>
</tr>
</tbody>
</table>

For comparison: single strut:
Studying microscopic elasticity in poroelastic materials

Approach:
- Time-temperature superposition principle
- **Guided acoustic wave dispersion analysis**

Example of fitting analysis
Studying microscopic elasticity in poroelastic materials

Intermediate summary:
• With photoacoustic excitation, a wide frequency range is accessible
• Preliminary results on simplified model system:
  • cm size struts
  • periodical and random network structure
  • non-relaxing temperature range: frequency independent material behavior
• Single strut-like dispersion can be obtained from strut embedded in porous frame
• Elastic properties of single struts can be extracted but large fitting covariance, mainly on Poisson’s ratio

Future work:
• Experiments in temperature range where material’s behavior is frequency dependent
• Exploit time-temperature superposition to extrapolate high frequency results to audio frequency results
• Experiments on real foams with (sub-)mm size struts
• Investigation of in-plane grid elasticity
• Full-field vibrometry for simultaneous assessment of macro- and microscopic dynamics
Nanoporous silicon
Nanoporous silicon

Fast and sensitive displacement detection

Heterodyne diffraction method
Characterization of porous silicon
Characterization of porous silicon

\[ c' = \frac{(c_{11} - c_{12})}{2} = 27.5 \pm 0.25 \text{ GPa} \]

\[ c_{44} = 40.1 \pm 0.1 \text{ GPa} \]
Characterization of porous silicon
THANK YOU FOR YOUR ATTENTION

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