Acoustic Wave Sensors

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Overview

1. The Lab
2. Basics of Acoustic Waves
    - Sensing principles
    - Acoustic wave modes and devices
3. Layer-Guided Acoustic Waves
    - Love waves & acoustic plate modes
    - Layer-guided devices: Theory of operating points and sensitivity
    - Layer-guided devices: Experimental confirmation
4. Sensor Research Examples
    - Atmospheric science: Particulate detection
    - Biological sciences: Peptide binding, steroids, sperm motility
    - Chemistry/Chemical engineering: Green solvents
    - Basic devices: Sectional guiding layers
5. Overview of Wetting

The Laboratory

The Laboratory

Themes & Expertise

Acoustic waves sensors

- Quartz crystals and surface acoustic waves

Wetting of surfaces

- Topography+chemistry, superhydrophobicity

Materials scientists

Physicists

Science

Acoustic wave sensors

- Theory of layer guided devices
- Applications of QCM and SAWs

Wetting & topography

- New superhydrophobic surfaces, superspreading
- Lquid marbles, electrowetting, hydrophobic soil
- Slip and slip boundary conditions

Multidisciplinary People

2 x Academics (Physicists, >2 incl. others)

3 x PhD Students (+ others joint)

- Physicists by first degree

5 x Research fellows

- Electrochemist/Physical chemist
- Applied physicist/acoustic waves
- Inorganic/protein chemist
- Materials synthesis (sol-gel)
- Engineer/Microfluidics

Facilities

Device and surface fabrication

- Lithography (<2 μm), metal deposition
- Inorganic/materials lab

Surface characterisation

- SEM/TEM/Confocal microscopy
- Contact/non-contact profilometry

Instrumentation & measurement

- RF Network analyzers, QCMs, laser cutting
- Krüss DSA, high speed camera, kV supplies,

Basics of Acoustic Waves

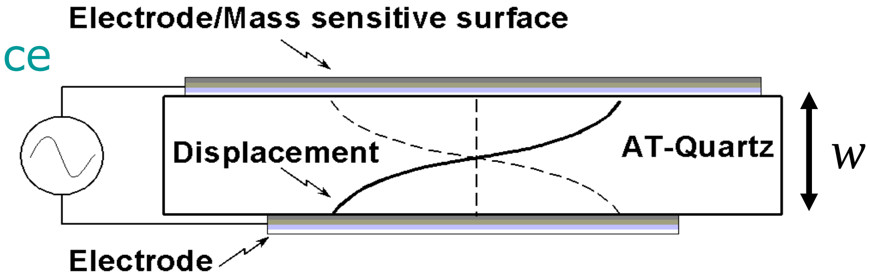
QCM Sensing Principles

Thickness Shear Mode Vibration

Quartz crystal microbalance - sharp resonance

Frequency given by quartz thickness, w

$v_s = f\lambda \quad \Rightarrow \quad f = 2v_s/w$



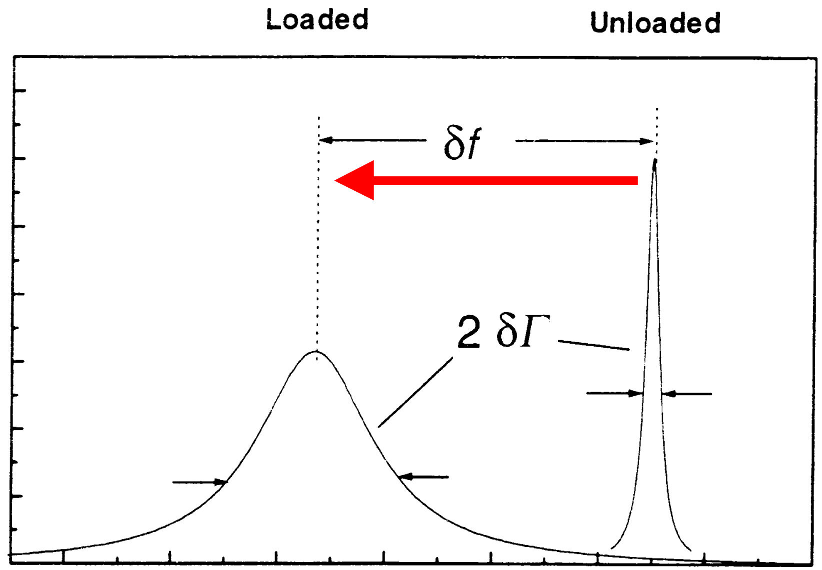
Mass Loading or Immersion

Frequency reduces due to mass

Resonance broadens due to polymer/liquid

Sauerbrey equation $\Rightarrow \Delta f \propto -f^2 \Delta m/A$

Kanazawa & Gordon $\Rightarrow \Delta f \propto -\sqrt{(\eta\rho)}\, f^{3/2}$



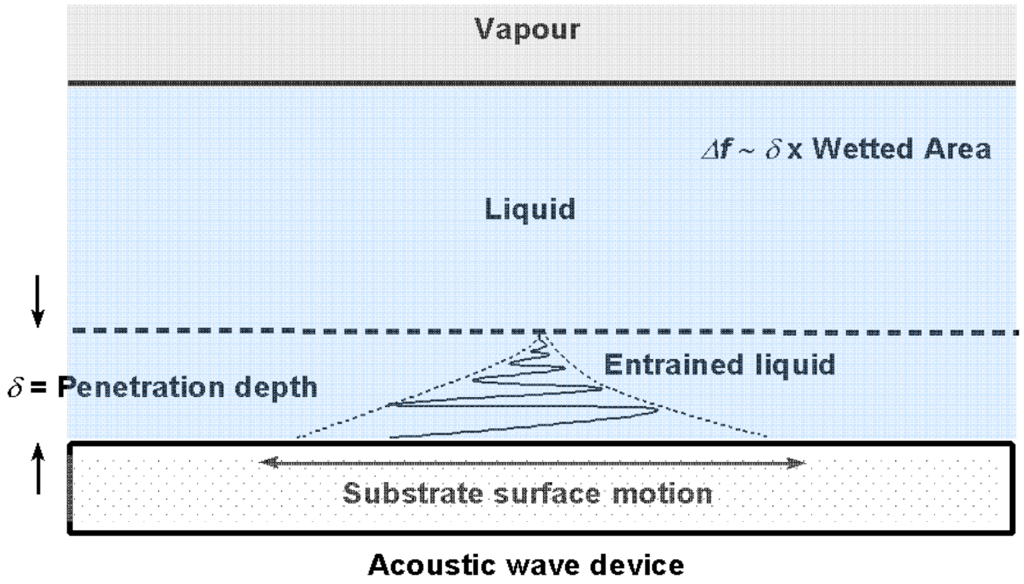
Sensitivity to mass or viscosity-density product increases with frequency

Liquids and Penetration Depth

Shear Mode Vibration

- Entrains liquid
- Liquid oscillation decays
- Penetration depth

$$\delta = (\eta / \pi f \rho)^{1/2}$$



Liquid Sensing

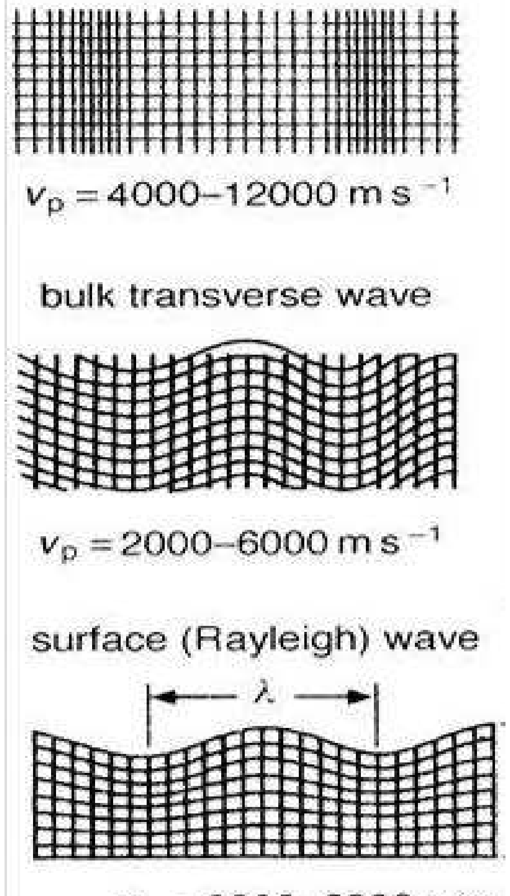
Sense liquid mass (via viscosity-density product) within penetration depth

	QCM		SAW	
For water	5 MHz	$\delta \sim$ 250 nm	500 MHz	$\delta \sim$ 25 nm

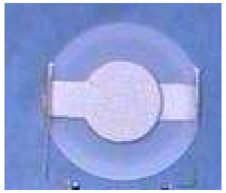
Penetration depth/sensing zone decreases with frequency

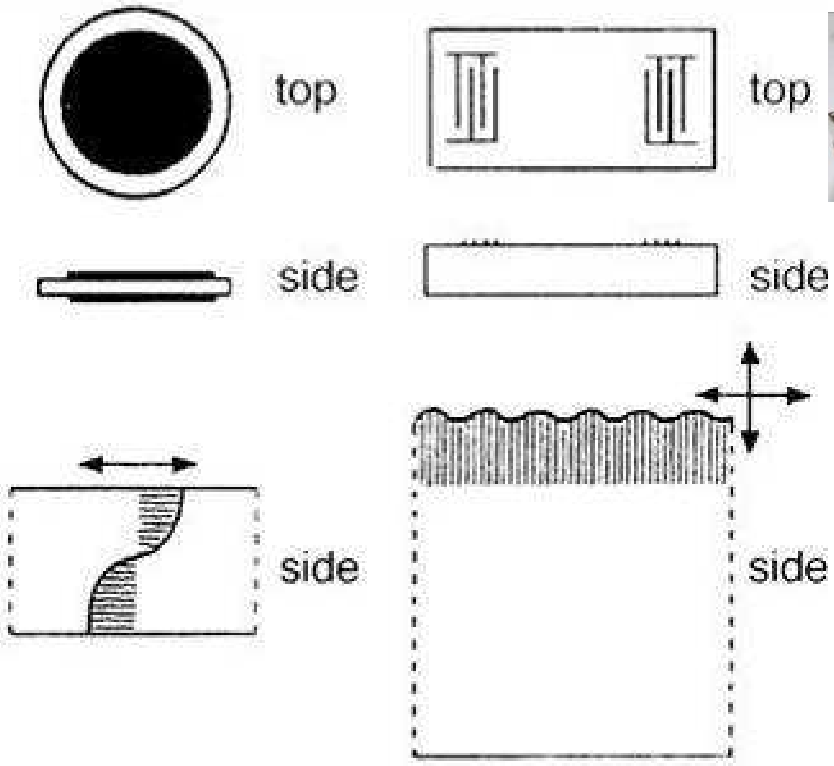
Acoustic Waves

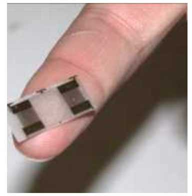
Acoustic Waves



QCM versus SAW





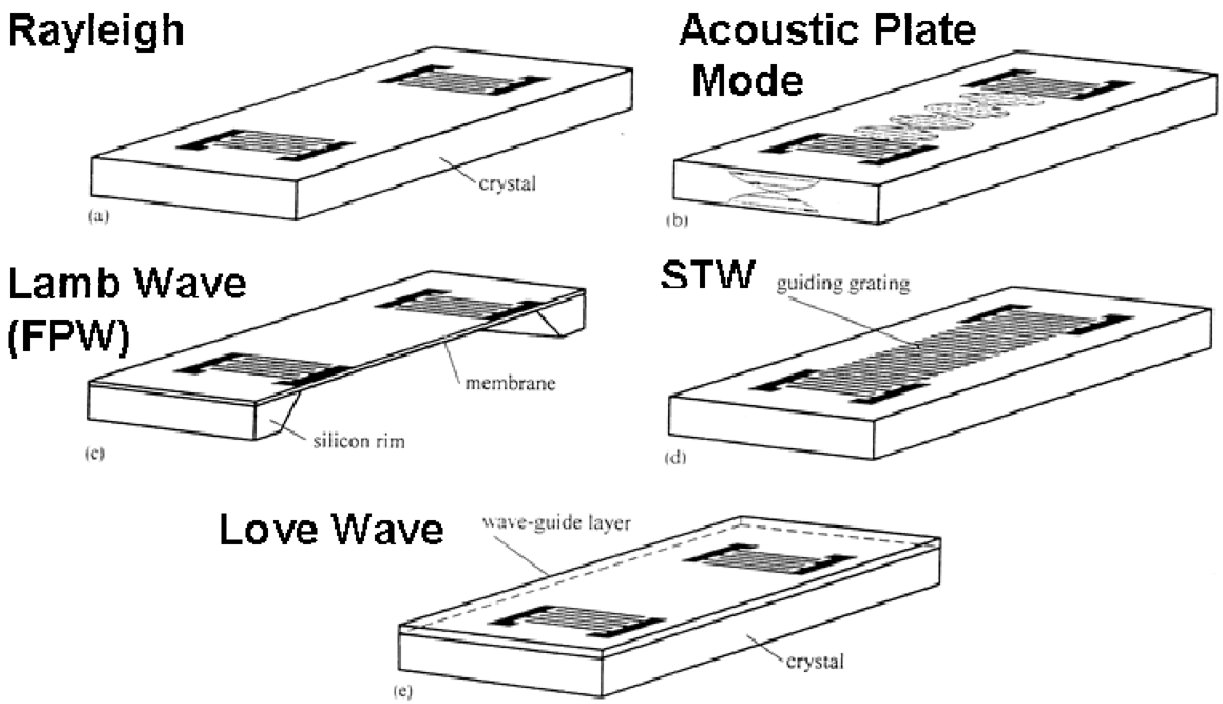


QCM – frequency determined by thickness

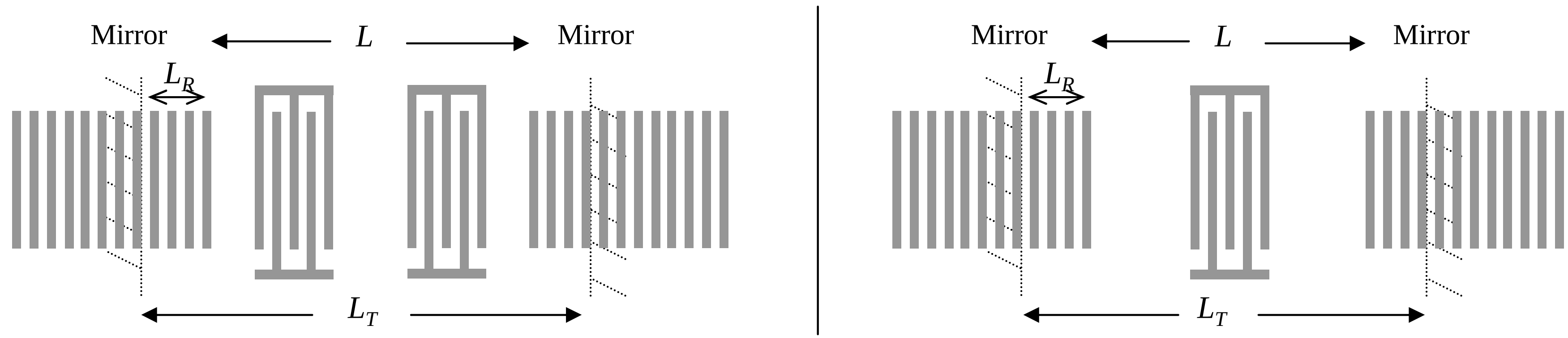
SAW – frequency determined by fingers

Acoustic Wave Modes

Delay Lines

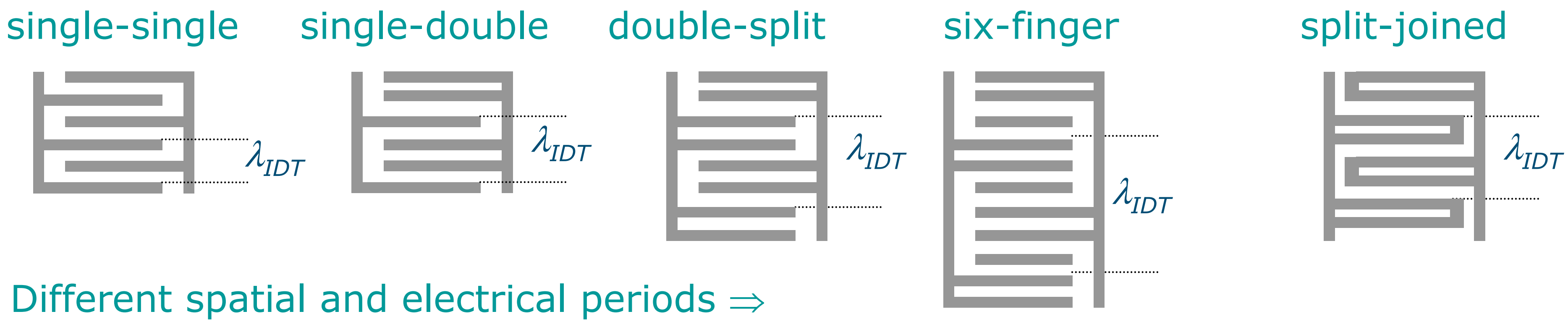


Resonators



SAW Transducer Design

IDT Configurations



Different spatial and electrical periods $\Rightarrow$

Harmonic operation: double-double gives higher frequency for same fabrication limits

possible multi-frequency operation along same path

Reflectivity: double-split and split-joined avoids reflections

Possible Design Considerations

Aperture: matching and beam/beam spreading

Number of fingers: bandwidth

End effects and guard electrodes

Reflecting structures: distributed reflection

Bidirectional and unidirectional transducers

Wireless operation

Acoustic Waves - Comparisons

Thickness Shear Mode

Quartz crystal microbalance (QCM)

Surface Acoustic Waves (SAWs)

Rayleigh waves, Love waves, Surface transverse waves (STWs), Lamb waves/Flexural plate waves (FPWs)

Acoustic Plate Modes (APMs)

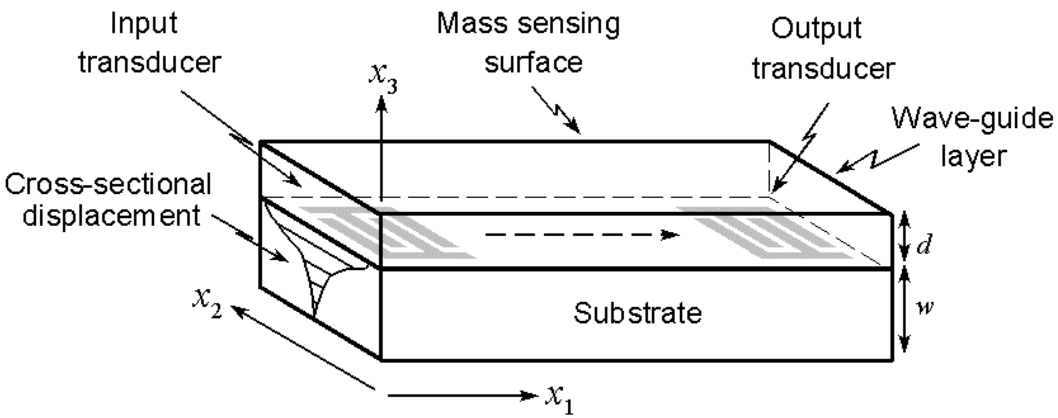
Shear horizontally polarised SAWs (SH-SAWs)
Surface skimming bulk waves (SSBWs)

Mode	Rel. Sens.	Complexity	Robustness	Gas/Liquid
QCM	Low	Low/Xtal	Med	g+l
SAW	High	Med/metal on Xtal	High	g
Love	High	Med/film+metal+Xtal	High	g+l
STW	High	Med/metal on Xtal	High	g+l
Lamb	High	High/membrane	Low	g+l
APM	Med	Med/metal on Xtal	Med	g+l

Layer-Guided Acoustic Waves

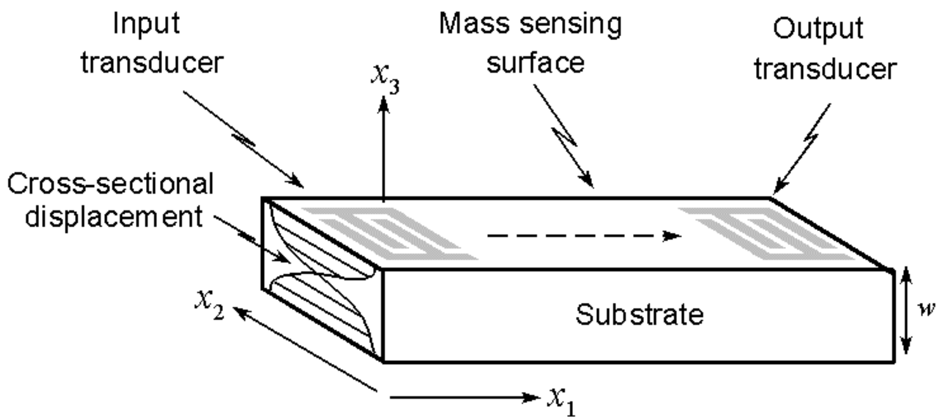
Love Waves versus SH-APMs

Love Wave



Layer guided SH-SAW with $v_l < v_s$
Surface localised wave
Increased sensitivity

SH-APM



"QCM with propagation"
Substrate resonance
Sensing via both faces

Increased sensitivity versus isolation between sensing face and transduction

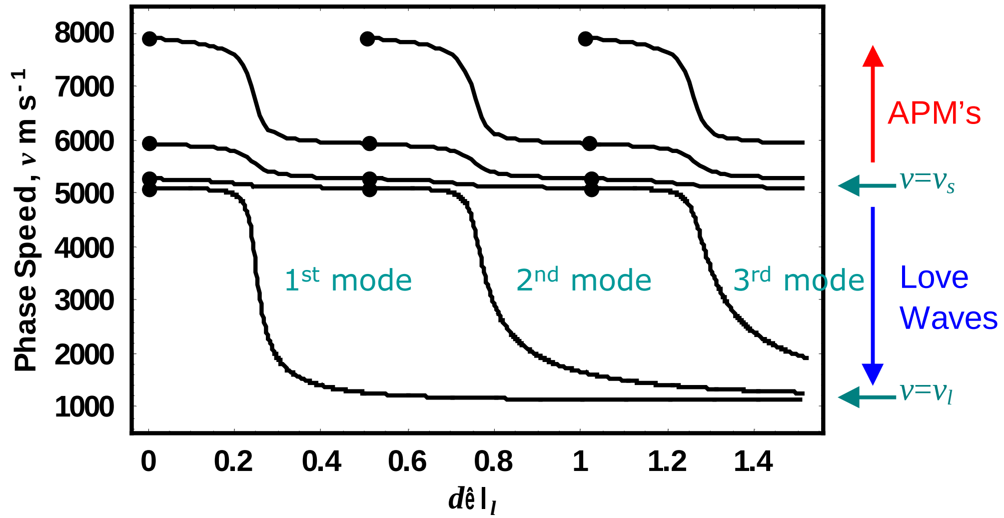
McHale, *et al, J. Appl. Phys.*, 2003, **93**, 675-690; *MST*, 2003, **14**, 1847-1853.

Layer-Guided SH-APMs

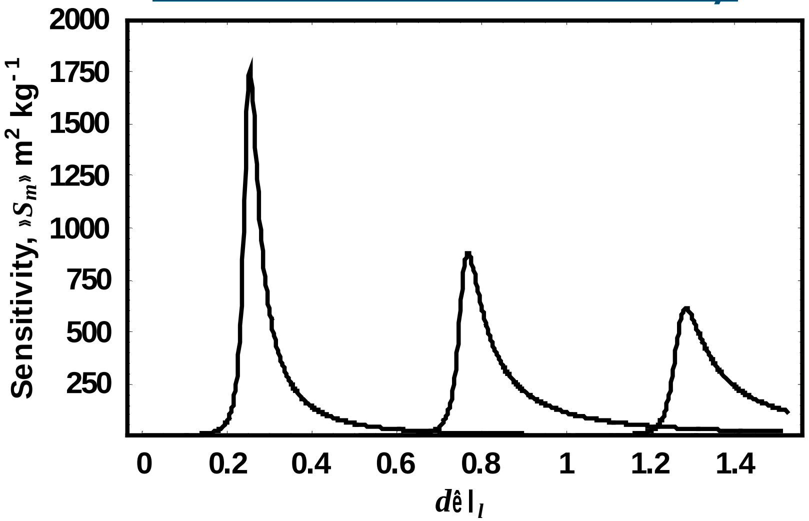
Theoretical Dispersion Curve

- Love device with thinned substrate
- Love waves for $v<v_s$
- Shear mode in substrate-to-shear mode in layer transition
- SH-APMs for $v>v_s$

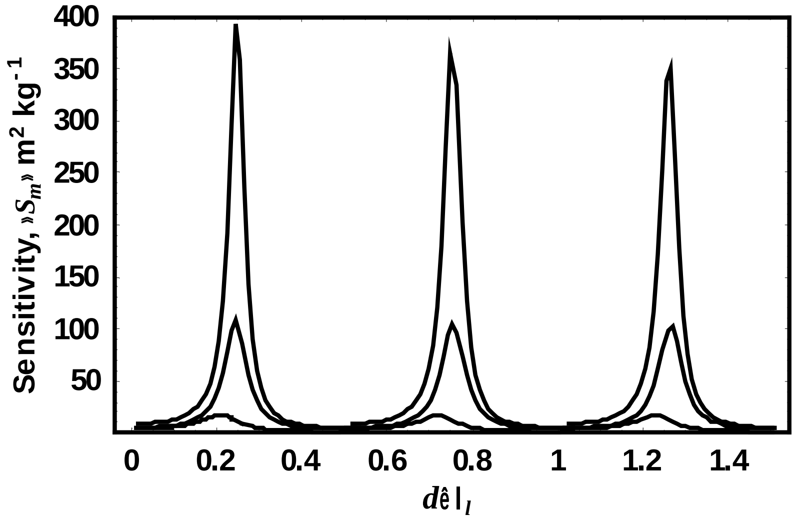
Mass/liquid sensitivity ∝ slope



Love Wave Sensitivity

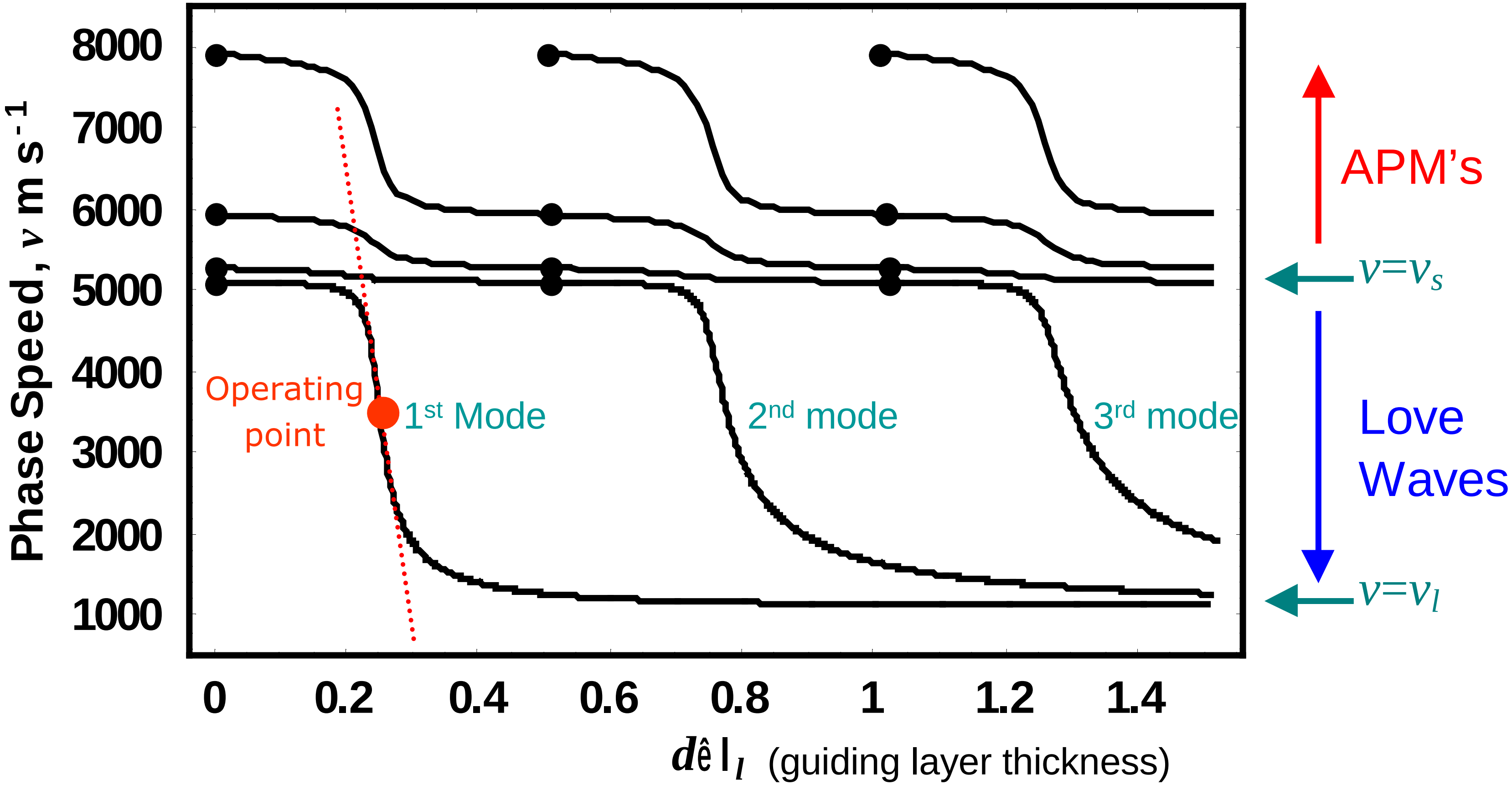


SH-APM Sensitivity



McHale, *et al, J. Appl. Phys.*., 2002, **91**, 5735-5744; 9701-9710.

Generalized Love Waves – Operating Point



Love wave = Shear mode in substrate-to-shear mode in layer transition

Plate modes = Switch in order of resonance induced by layer

Increased mass/liquid sensitivity related to slope of dispersion curve

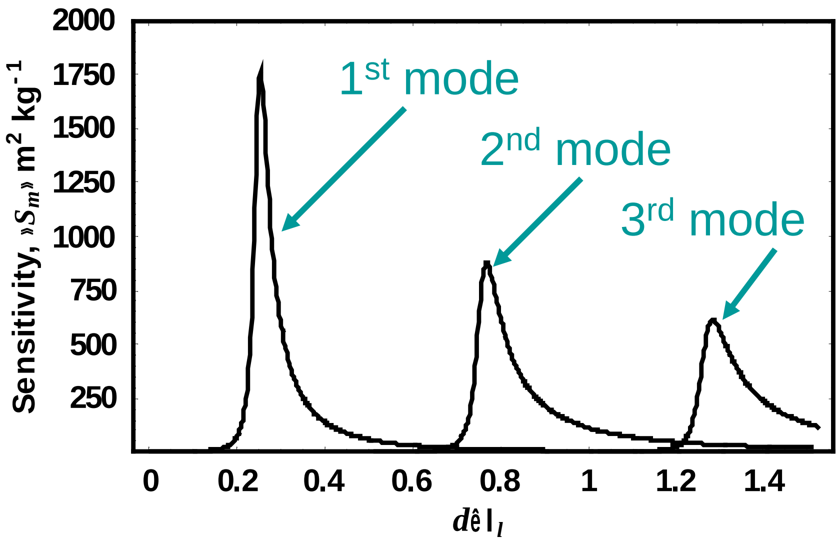
Phase Speed Mass Sensitivity

$$S_m = \lim_{\Delta m \to 0} \frac{1}{\Delta m}\left(\frac{\Delta v}{v_o}\right) \approx \frac{f_o}{\rho_l v_l}\left(\frac{d \log_e v}{dz}\right)_{z_0}$$

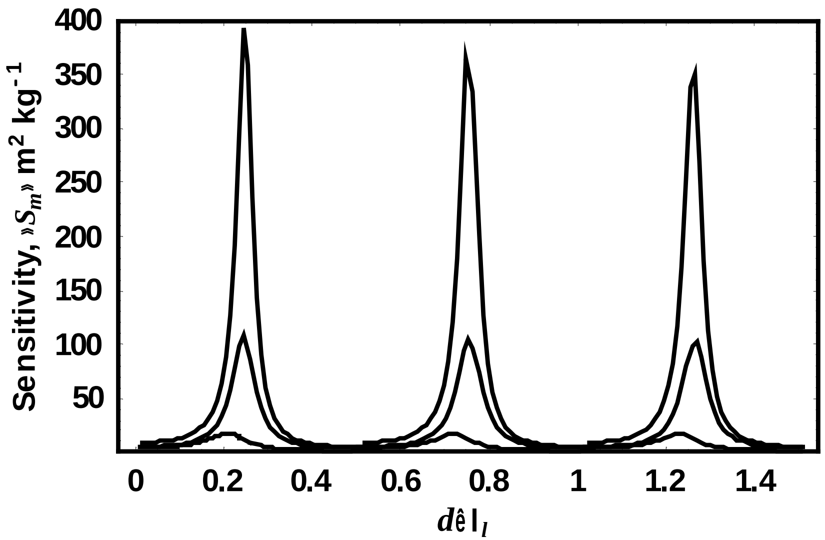
Δm is mass per unit area being sensed, $z = df/v_l$ is the normalized thickness

"Rigid" mass ⇒ Mass sensitivity is slope of dispersion curve

Love Waves



Layer-Guided SH-APMs



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Generalized Sauerbrey/Kanazawa & Gordon

Polymer Waveguide on Polymer Substrate

Complex velocity shift

$$\frac{\Delta v}{v_o} \approx \left(\frac{1-v_f^2 / v_o^2}{1-v_l^2 / v_o^2}\right)\left(\frac{d\log_e v}{dz}\right)_{z=z_o}\left(\frac{\tan\left(T_f^o h\right)}{T_f^o h}\right)\frac{\omega\rho_f h}{2\pi v_l^\infty \rho_l}$$

$\omega\Delta m/A$

$(\rho\eta\omega)^{1/2}$

Complex slope factor
from polymer waveguide

tan$x$/x factor gives mass/liquid loading limits

$$\left(\frac{\tan\left(T_f^o h\right)}{T_f^o h}\right) \to \begin{cases} 1 & h \to 0 \\ \dfrac{-\sqrt{-2j}}{2h\left(1-v_f^2 / v_o^2\right)}\sqrt{\dfrac{2\eta_f}{\omega\rho_f}} & h \to \infty \quad \text{and} \quad \omega\tau \to 0 \end{cases}$$

Sauerbrey/
solid limit

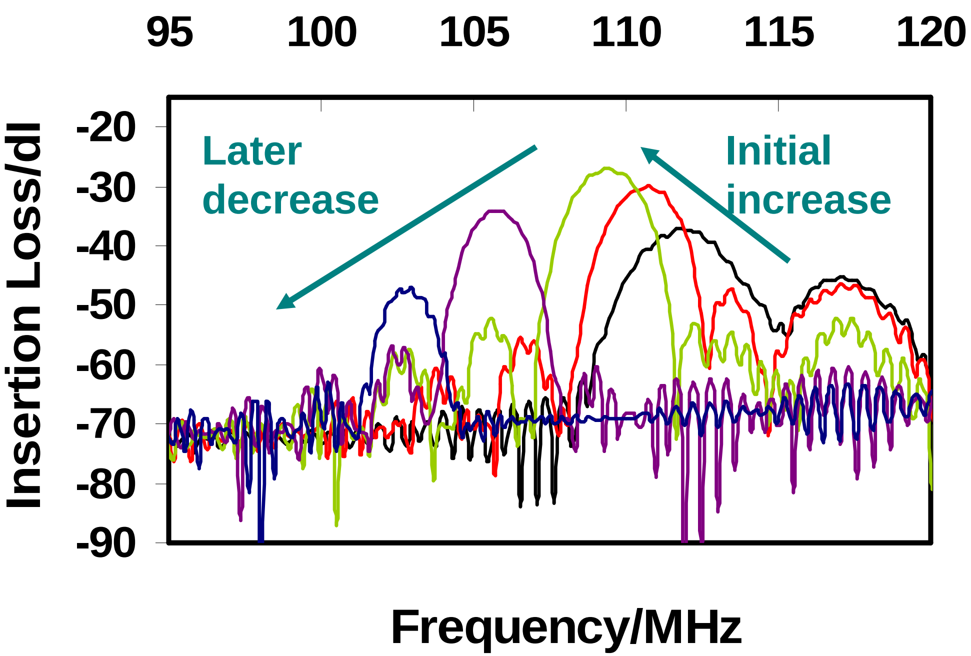
Kanazawa &
Gordon/liquid limit

McHale, *et al*, *J. Appl. Phys.*, 2003, **93**, 675-690; *MST*, 2003, **14**, 1847-1853.

Multiple Love Wave Modes

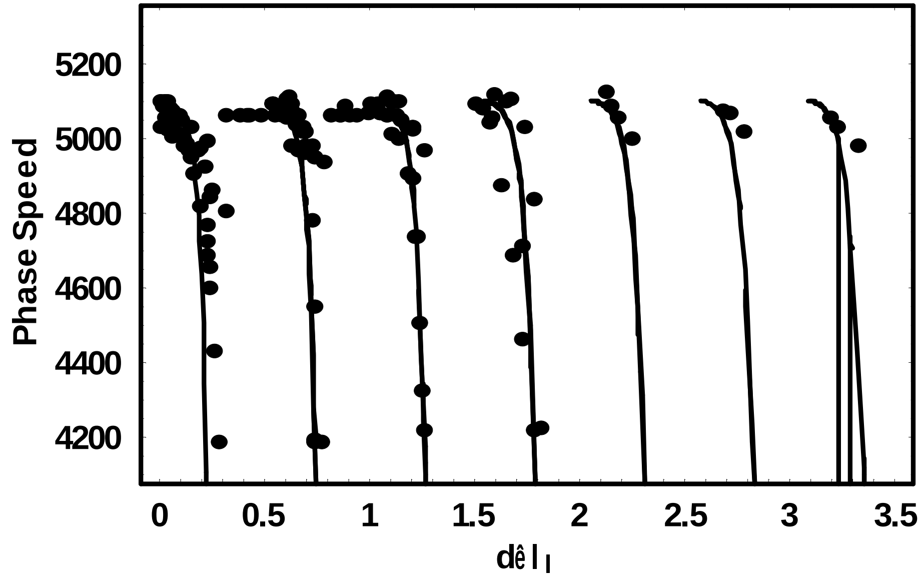
Spectra

Thick guiding layers
Photoresist layers
Quartz substrate (SSBW)



Experimental Results

Points = results for devices
110/330 and 309 MHz
Lines = theory



Newton, *et al, Euro. Phys. Lett.*, 2002, **58**, 818-822.

Experimental Data for Layer-Guided SH-APMs

25 MHz surface skimming bulk wave (SSBW)

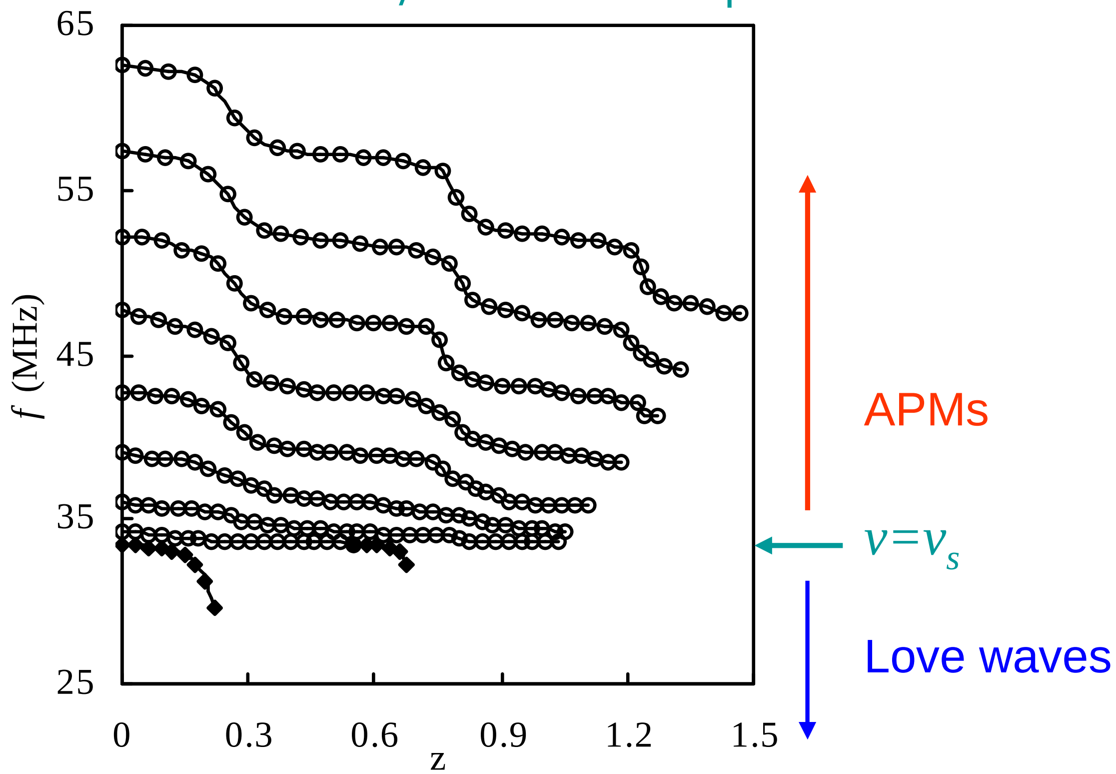
Propagation orthogonal to x-axis of thinned (200 mm) ST-Q substrate

IDT Face Coated

Love wave and SH-APM

are both sensitive

x-axis is d/λ with λ=IDT period

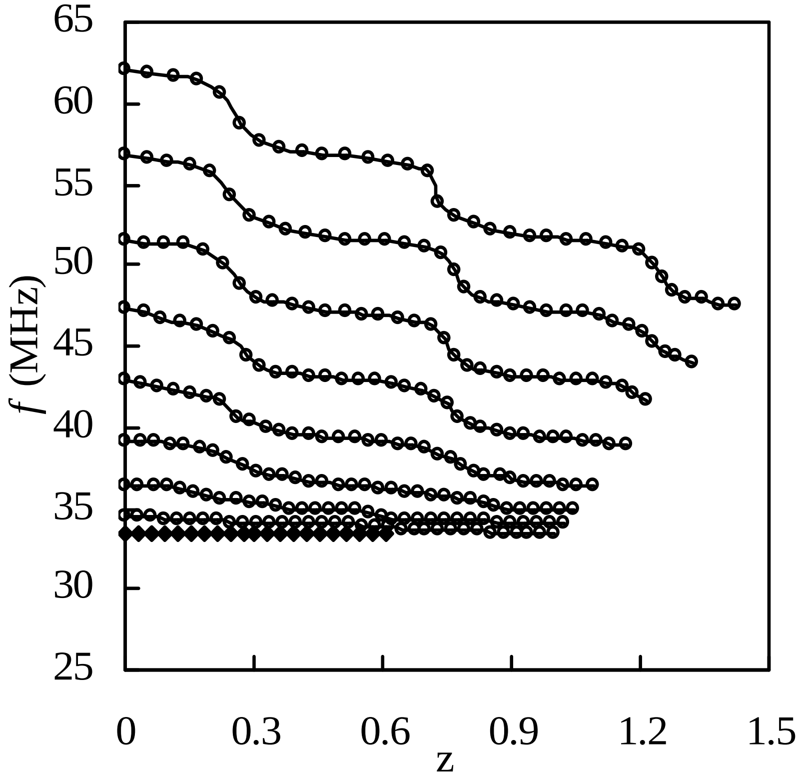


Opposing Face to IDTs Coated

SH-APM changes

Love wave insensitive

x-axis is d/λ with λ=IDT period



McHale, *et al, Appl. Phys. Lett.*, 2003, **82**, 2181-2183.

Newton, *et al*, *Sens. Act.*, 2004, **A109**, 180-185.

F. Martin, PhD Thesis, Nottingham Trent University (2002).

Love Waves and Higher Frequency

Established QCM Sensor Principle

Mass sensitivity $\propto$ Fundamental frequency

Higher frequency $\Rightarrow$ Higher mass sensitivity

Love Waves on a (Semi-) Infinite Substrate

Controlling factor is guiding layer thickness x frequency $z = d/\lambda_l = df/v_l$

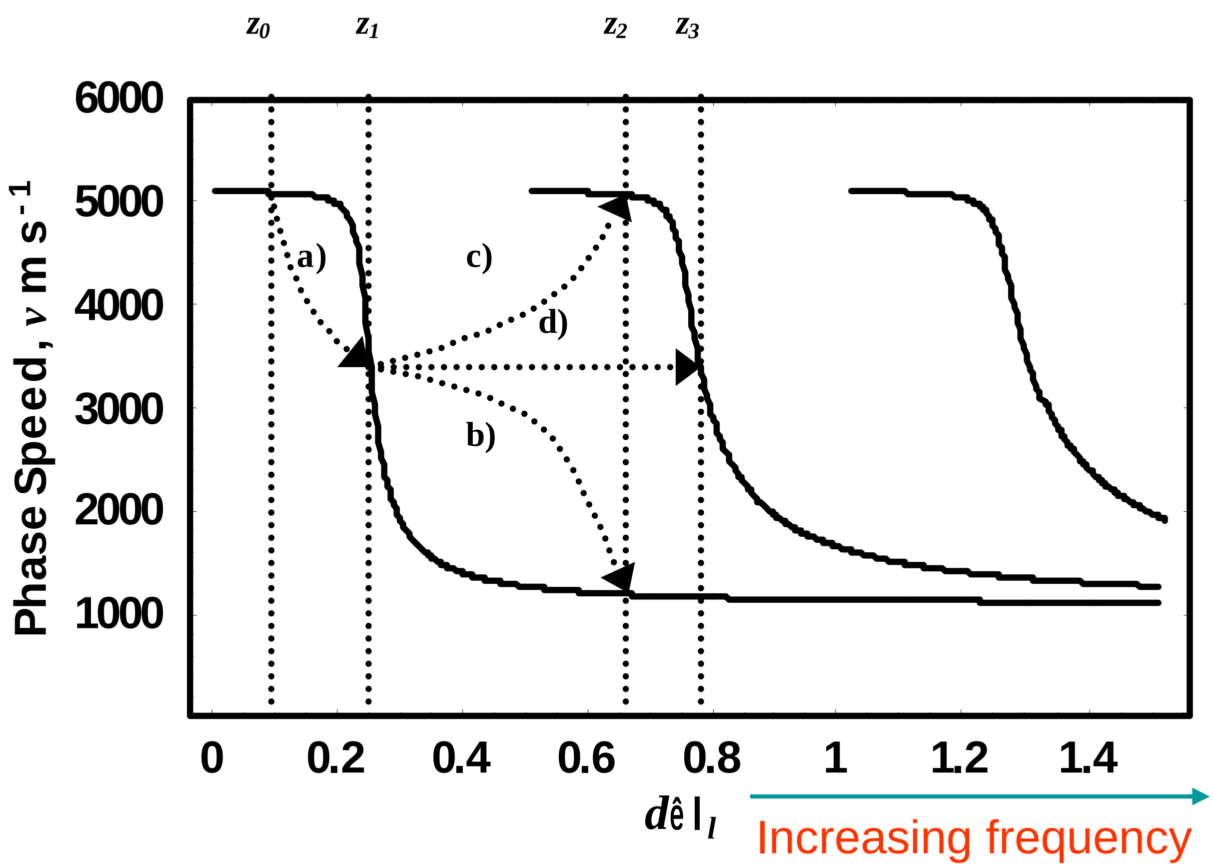
$$S_m = \lim_{\Delta m \to 0} \frac{1}{\Delta m}\left(\frac{\Delta v}{v_o}\right) \approx \frac{f_o}{\rho_l v_l}\left(\frac{d \log_e v}{dz}\right)_{z_0}$$

Mass sensitivity $\propto$ Frequency $\times$ Slope Factor

Slope operating point $z_o \propto d \times f$

Increasing frequency <u>may</u> or <u>may not</u> increase sensitivity

Love Waves and Frequency Hopping



<u>No Mode Change</u>

Transition a) ⇒ Higher mass sensitivity

Transition b) ⇒ Lower mass sensitivity

<u>Mode Change</u>

Transition c) ⇒ Lower mass sensitivity

Transition d) ⇒ Higher mass sensitivity

Sensor Research Examples

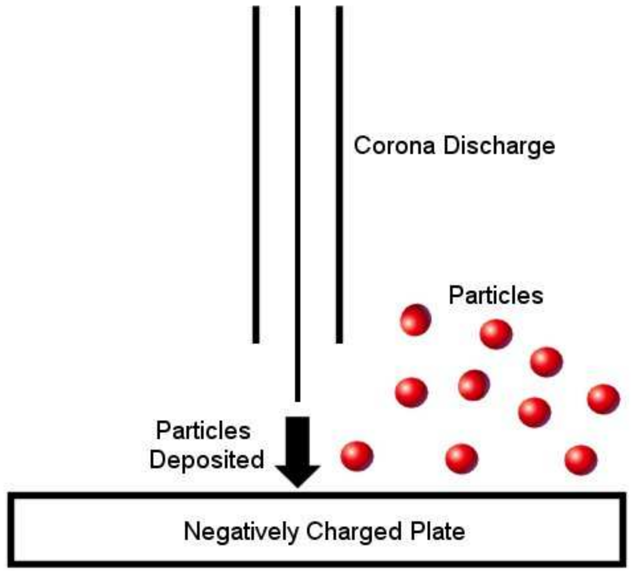
Examples 1 - Atmospheric Sciences

Particulates

EP-SAW for Atmospheric Particulates

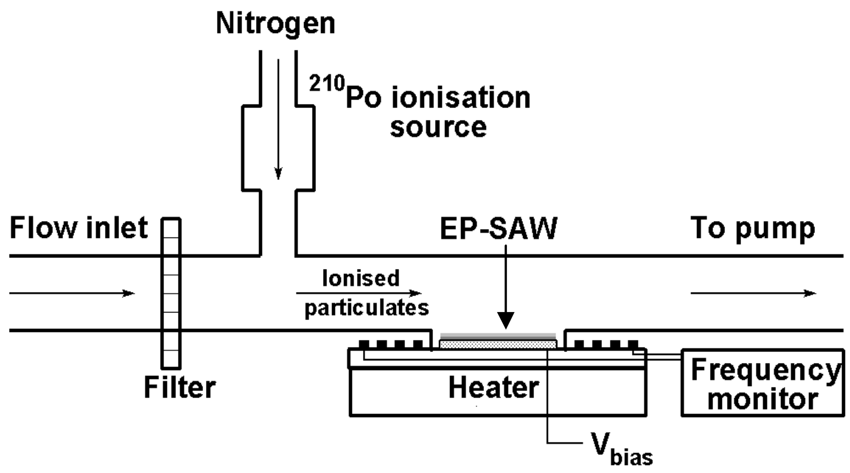
Electrostatic Precipitation (EP)

Charged particles deposited on collector
Established principle for atmospherically borne microorganisms
High (99-100%) efficiency



System Design

Air sampled via filter
Particles ionised by N_2^+
Particles collected onto path of biased metallised SAW
i.e. Electrostatic precipitation

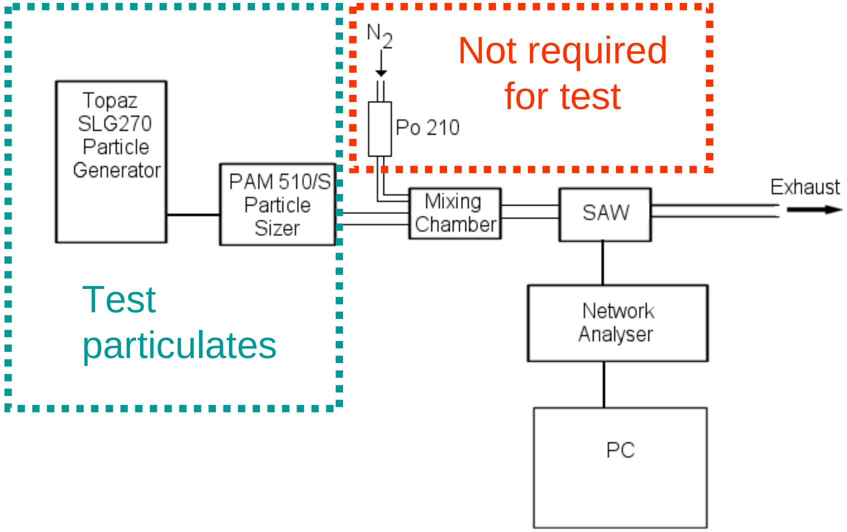


Stanley, *et al*, *NDT&E*, 2005, **20**, 3-7.

Particulate Response with Bias

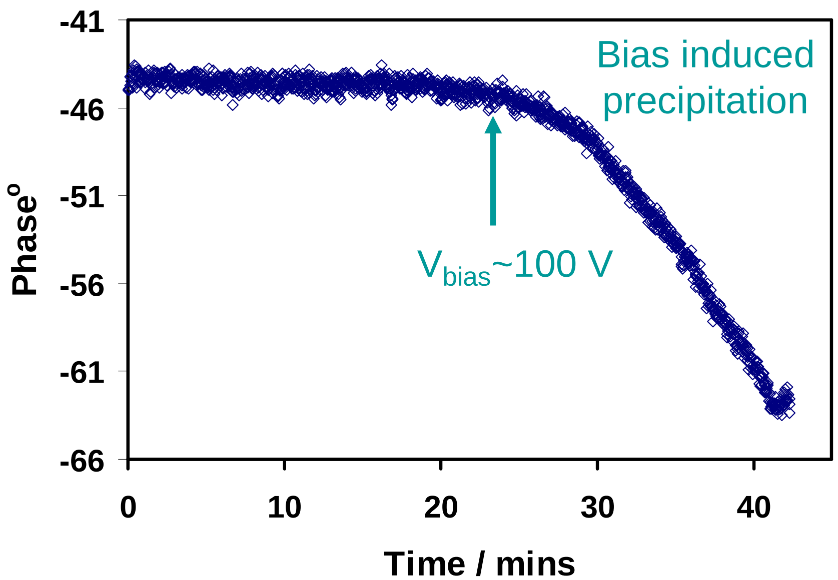
Test Device Configuration

$LiNbO_3$ - Rayleigh-SAW, $\lambda \sim 45\ \mu m$
86 MHz & 253 MHz
Test particulates via a NaCl aerosol:
mono-disperse with ~ $(2.0 \pm 0.1)\mu m$



Results

Voltage of plate increased
to > 120 V in 20 V steps
⇒ Phase changes

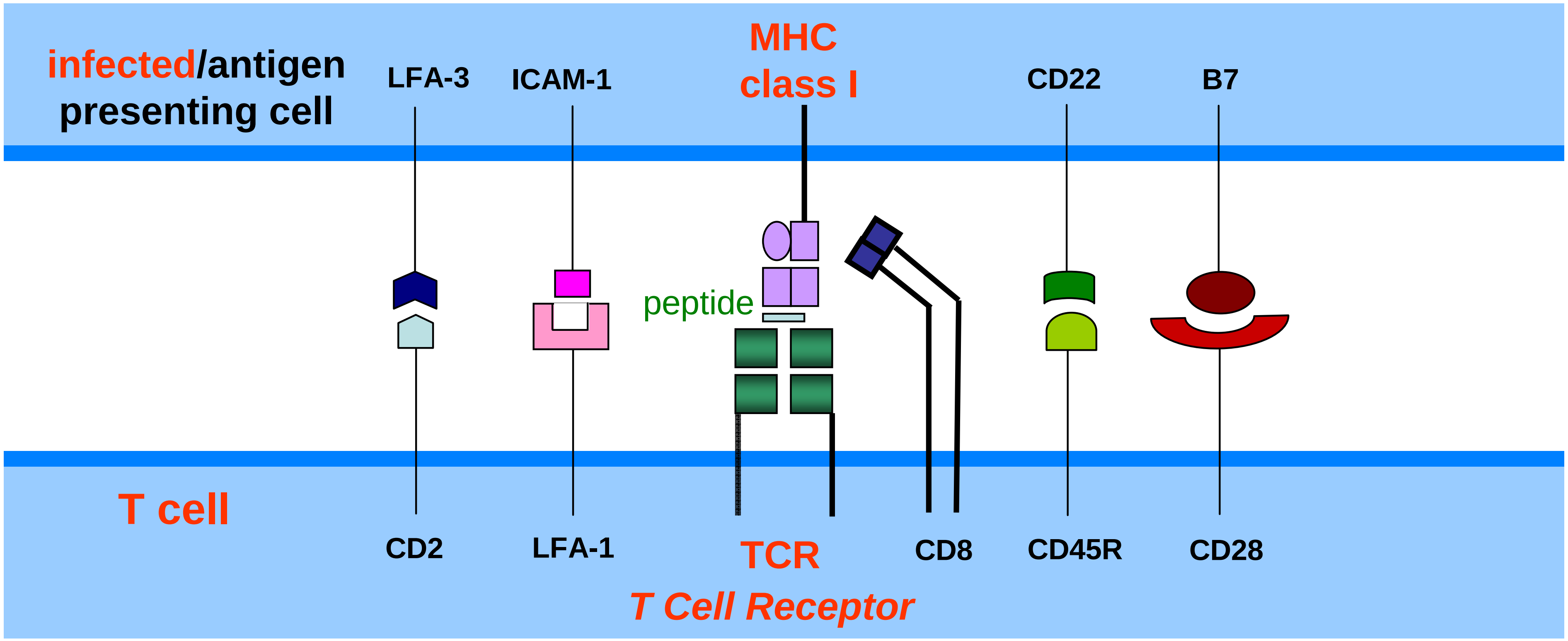


Examples 2 - Biological Sciences

Immune System and Vaccines

Peptides and T-Cells

1. Infection/virus broken into peptide fragments and presented on cell surface
2. Cytotoxic T-cells attach to peptides and "read" peptide sequence
3. If foreign, cell is killed by release of a cytotoxic chemical
4. Major histocompatability complex (MHC) antigens are responsible for the expression of peptides on the infected cell
5. Vaccine introduces peptide to the T-cell – Aim is to find suitable peptides



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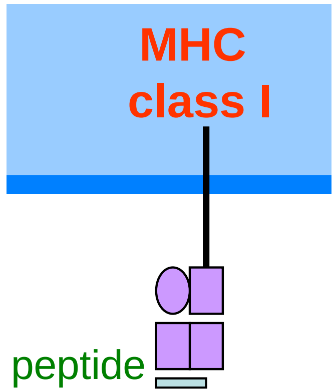
Sensor Strategy

Make this the acoustic wave sensor

Recognition layer is MHC protein

Detect peptide specific binding

Screen for suitable peptides (from the 1000's that exist) with specificity and strong affinity for the MHC



Current State-of-Art

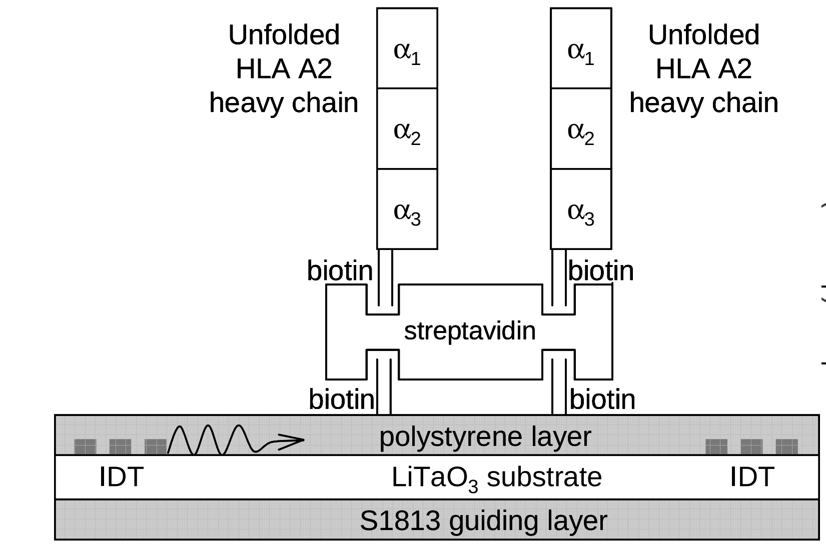
Cellular peptide-MHC assays

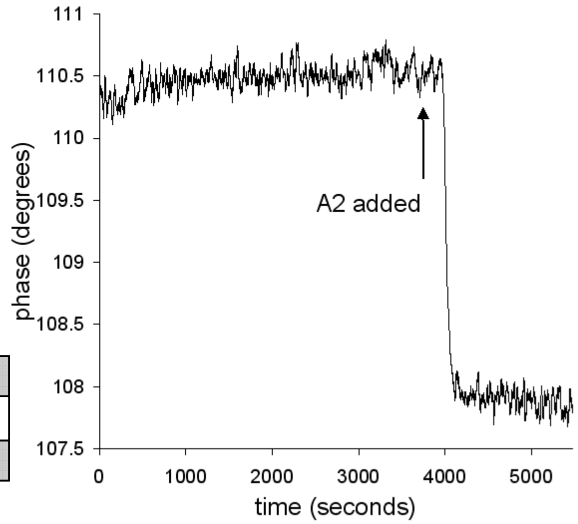
→ yes/no and not real-time

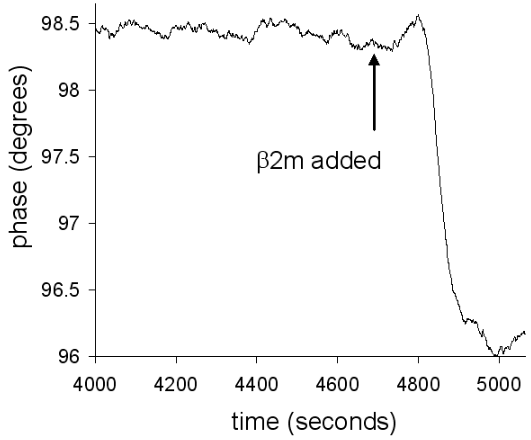
Sensitive, real-time and non-cellular based assay would assist vaccine development

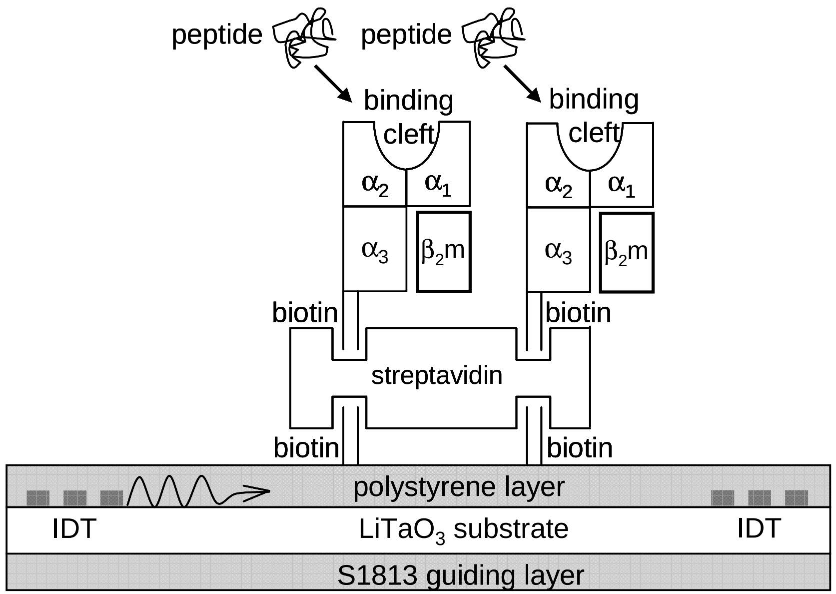
Flow Cell with Love Wave Screening Device

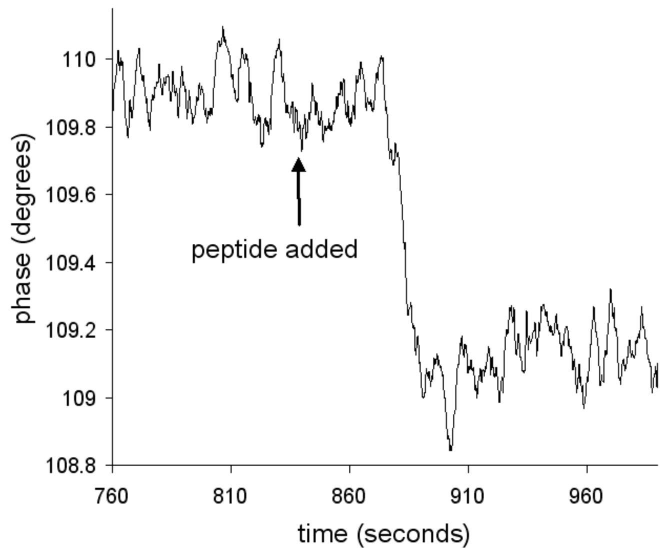
β_{2m} protein binds to A2 and folds to create peptide specific binding cleft











Stanley, *et al*, *Analyst*, 2006, **136**, 892-894.

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Examples 2 - Biological Sciences

Steroids and Sperm Motility

Molecularly Imprinted Polymers

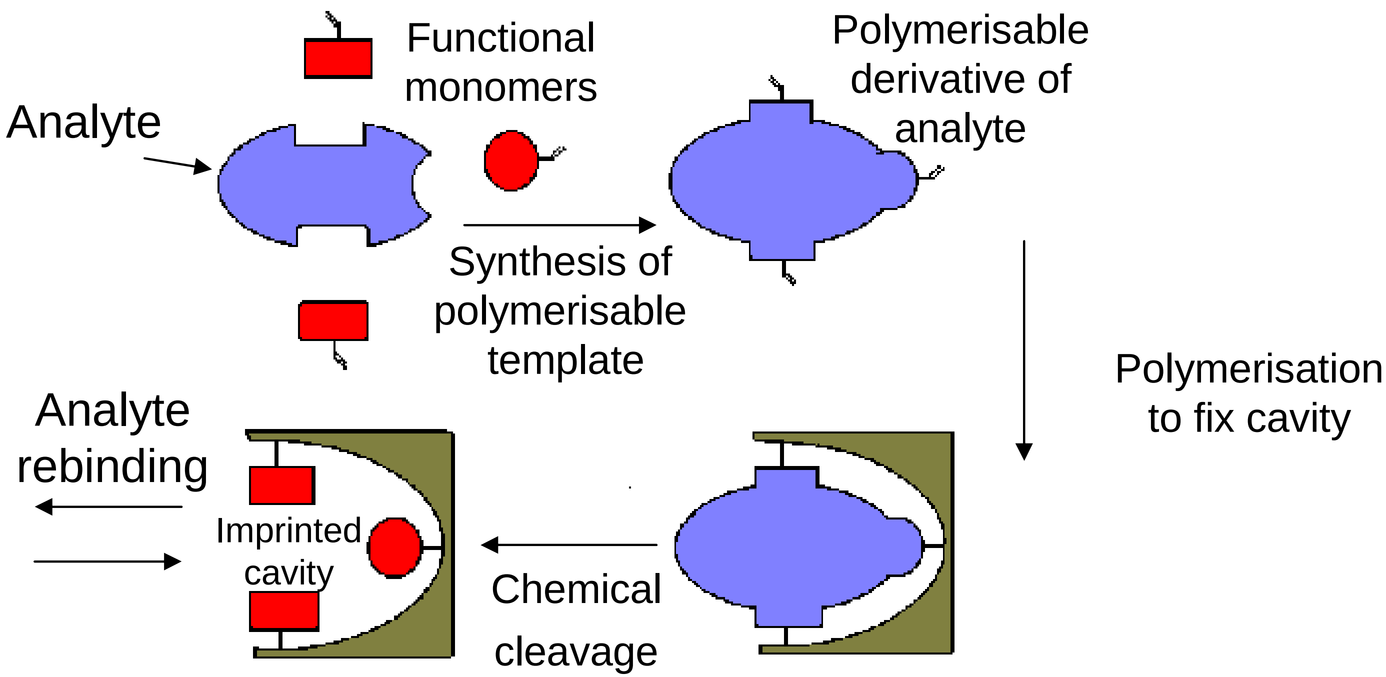
<u>Target Applications (*Liquid Phase*)</u>

Recognition/selectivity via molecularly imprinted polymers (MIPs)

Applications: monoterpenes, amino acids, *topical steroids*

Tailor made enantioseparation materials

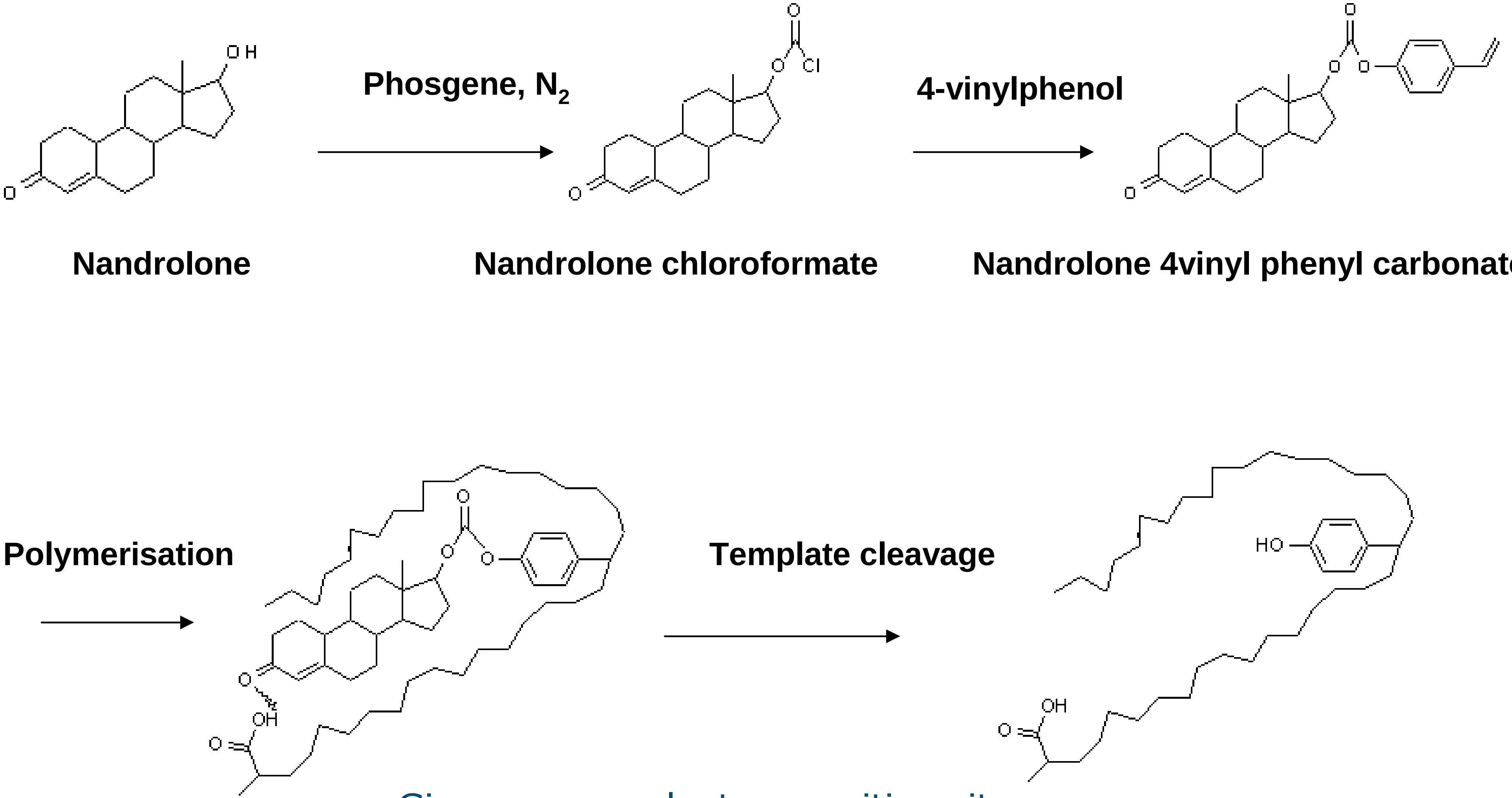
<u>MIP - Polymer Type Artificial Receptor</u>



Percival, *et al, Anal. Chem.*, 2001, **73**, 4225 –4228; 2003, **75**, 1573-1577.

Synthesis of Nandrolone MIP

Covalent Approach (Scheme 1)



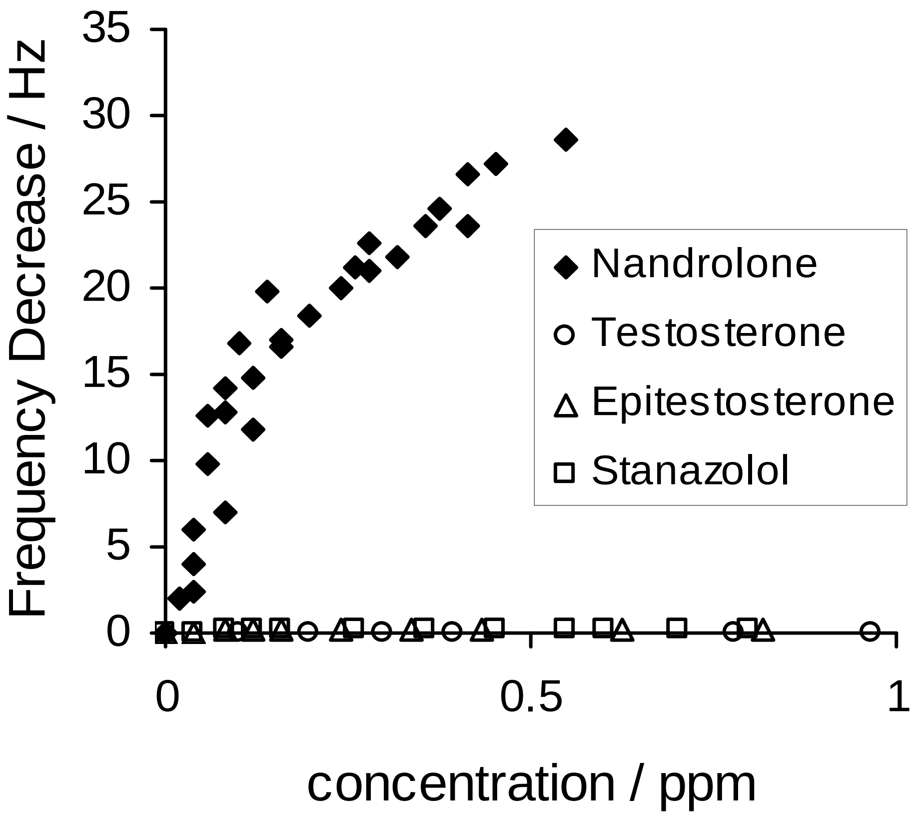
Gives non-covalent recognition sites

Selectivity to Nandrolone

QCM Coating

Spin coated/cast layer

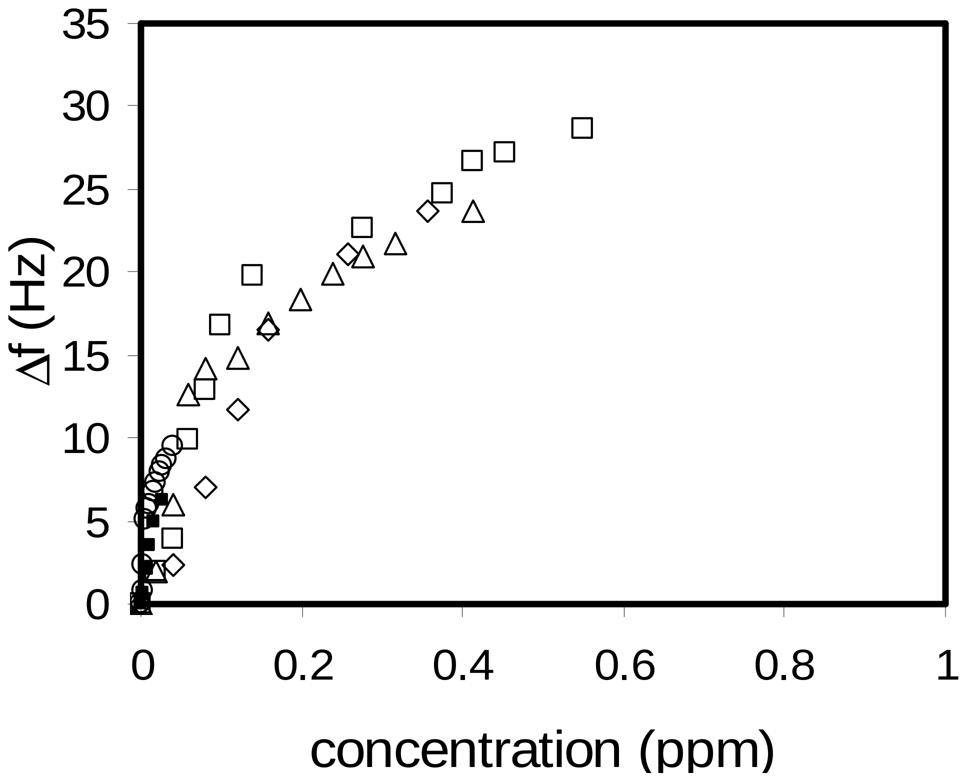
Covalent imprinting strategy



Response to Replicates

One-shot screening

Test data for 5 crystals

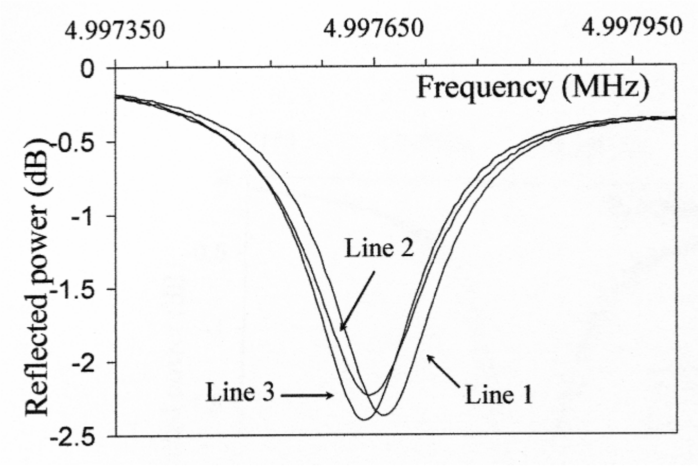


Percival, *et al*, *Analyst*, 2002, **127**, 1024-1026.

Sperm Motility

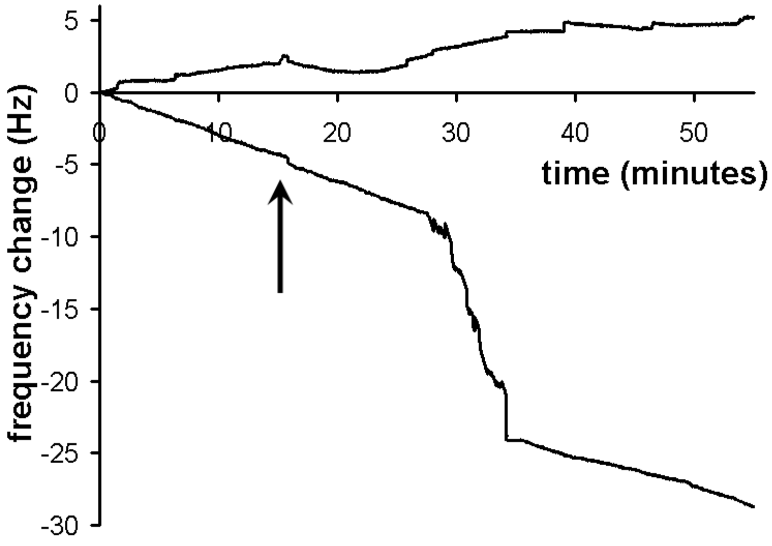
Veterinary Artificial Insemination (VetAI)

- Sperm Quality Assessment & Detection Device (SQuADD)
- Time of flight/swim
- 5 MHz QCM (or use other AWS device)
- Frequency drop relative to reference
- Crystal pre-coated with sperm ‘sticky’ material



Experimental Sequence

- Stabilisation of signal in PBS
- Addition of sperm (arrow)
- Time of arrival data – swim speed



Newton, *et al, Appl. Phys. Lett.*, 2007, **90**, art. 154103.

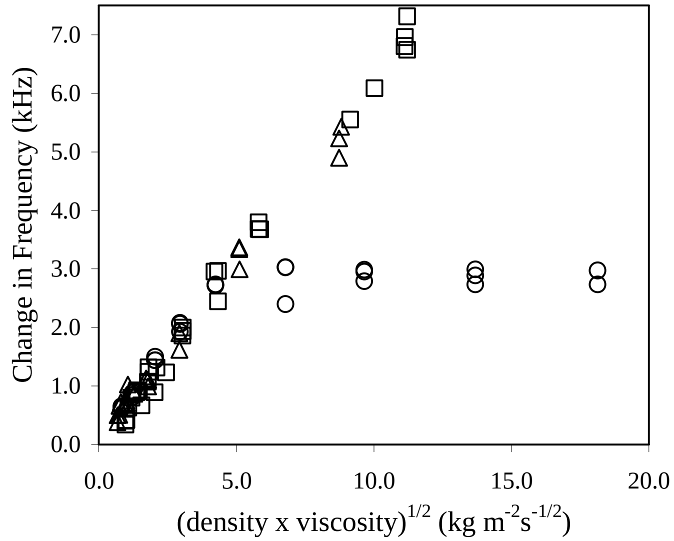
Examples 3 - Chemistry/Chemical Eng.

Green Solvents

Ionic Liquids

Determining Physical Properties

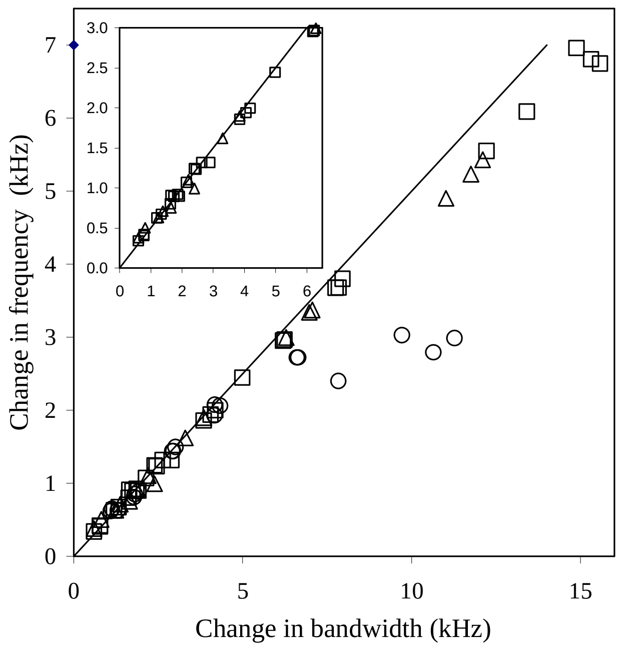
Room temperature ionic liquids (RTIL's)
Green because non-volatile
Millions of simple IL's, billions of binary ILs, …
Designer solvents
Poorly characterised



QCM

Can measure density-viscosity product, but can also determine whether Newtonian via coupled frequency shift-bandwidth increase

$$\Delta f = -\Delta B/2$$



Data

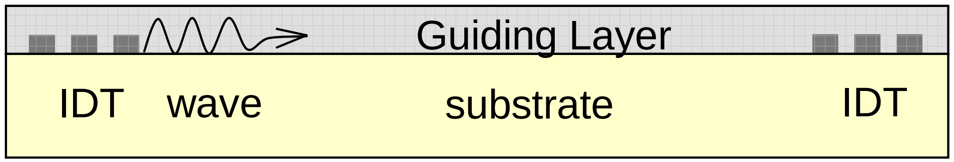
Polydimethylsiloxane oil - known non-Newtonian at higher molecular weights (ooo)
Two ionic liquids [C_4mim][OTf] (□□□) and [C4mim][NTf_2] (ΔΔΔ)

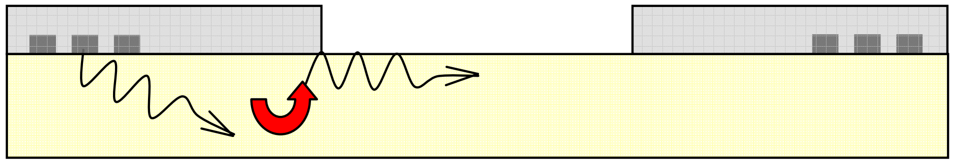
McHale, *et al*, *Anal. Chem.*, 2008, to be submitted.

Examples 4 – Novel Devices

Sectional guiding layers

Sectional Guiding Layers

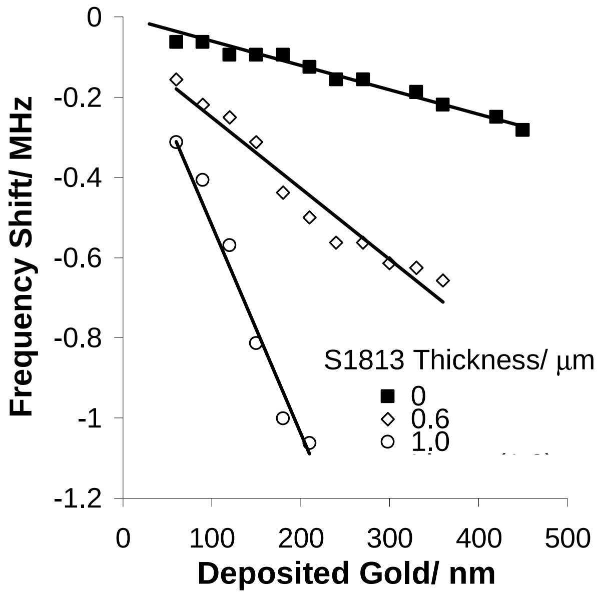


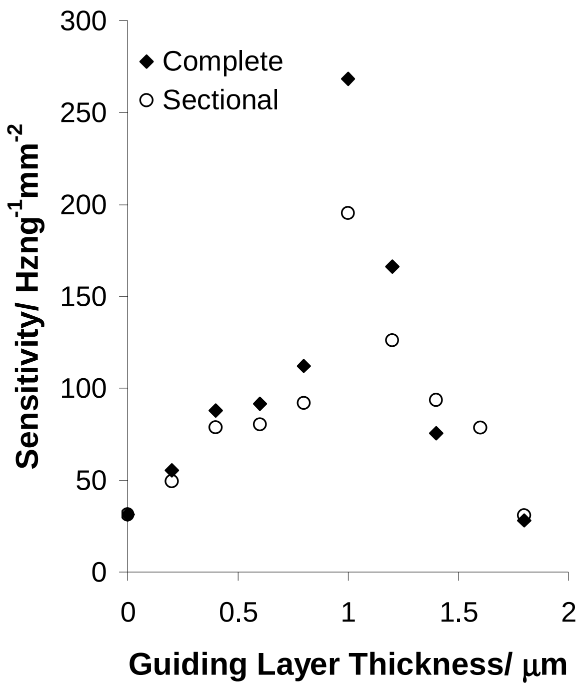


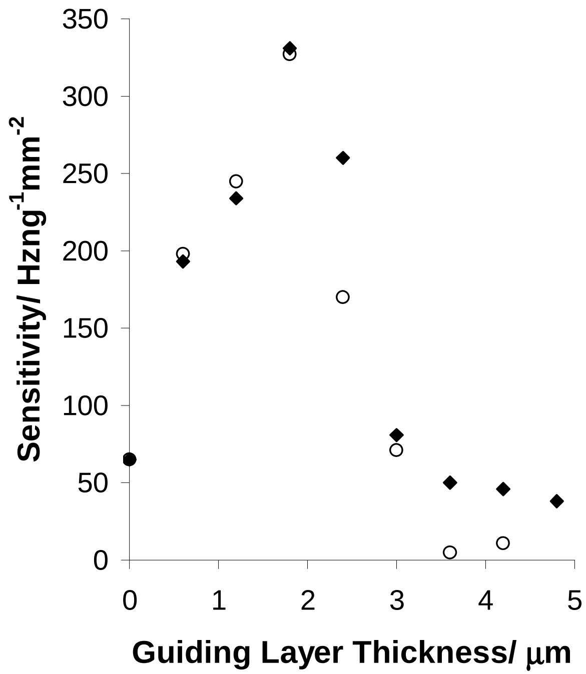
Mass Sensitivity

S1813 guiding layer on quartz and $LiTaO_3$ with 100 MHz device

AU deposited between IDTs to determine mass sensitivity





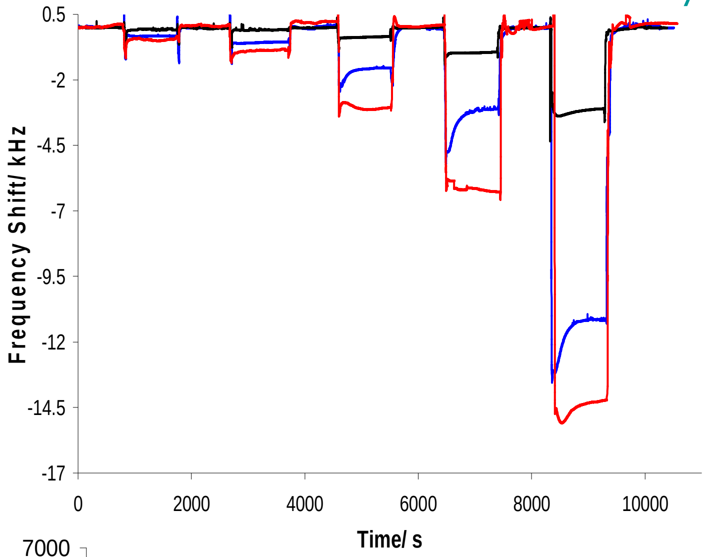


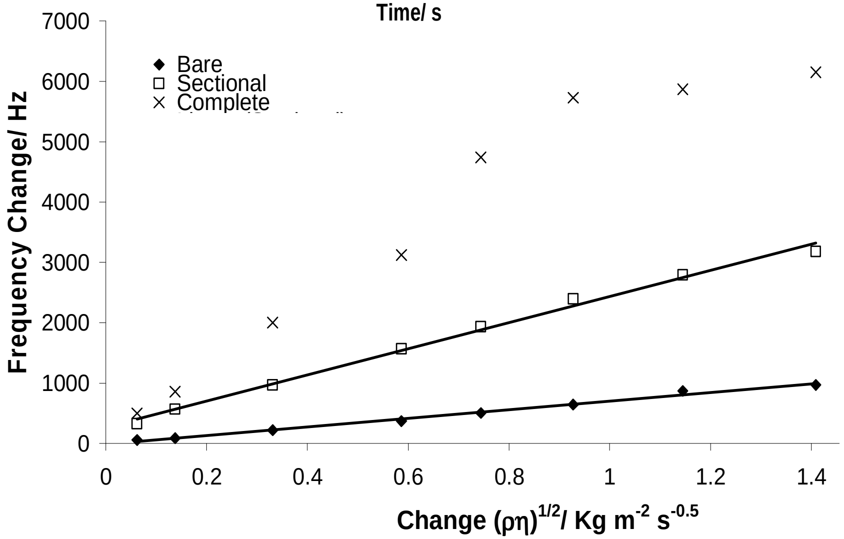
Roach, *et al*, *Sensors*, 2007, **7**, 2539-2547; Roach *et al*. to be submitted 2008.

Liquid Sensing

Water-glycerol mixtures

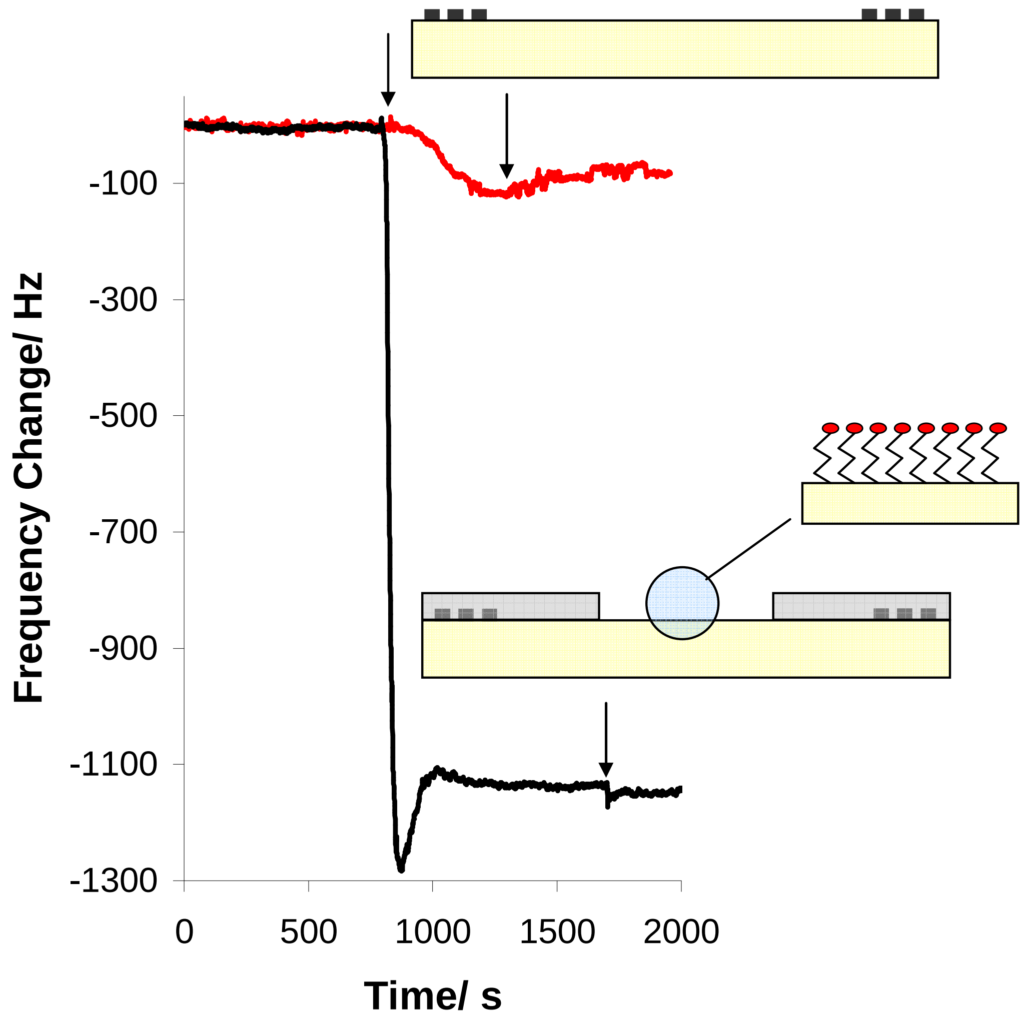
5% 0% 30% 50% 70% Glycerol





Albumin adsorption

0.5 mg ml^{-1} BSA in PBS



Other Research - Wetting

Topography and surface chemistry

Selected Examples

Slip and Slip Boundary Condition

- *Nano-scale superhydrophobicity: Suppression of protein adsorption and promotion of flow-induced detachment*, Lab on a chip (2008) accepted.
- *Decoupling of the liquid response of a superhydrophobic QCM*, Langmuir 23 (2007) 9823-9830.
- *Surface roughness and interfacial slip boundary condition for QCMs*, J. Appl. Phys. 95 (2004) 373-380.
- *Contact angle-based predictive model for slip at the solid-liquid interface of a transverse-shear mode acoustic wave device*, J. Appl. Phys. 94 (2003) 6201-6207.
- *Influence of viscoelasticity and interfacial slip on acoustic wave sensors*, J. Appl. Phys., 88 (2000) 7304-7312.

Novel Applications of Superhydrophobicity

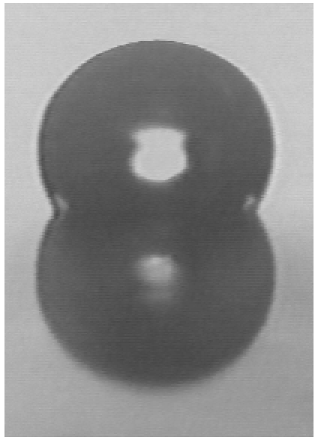
- *Electrowetting of non-wetting liquids and liquid marbles*, Langmuir 23 (2007) 918-924.
- *Implications of ideas on super-hydrophobicity for water repellent soil*, Hydrol. Proc. 21 (2007) 2229-2238.
- *A lichen protected by a superhydrophobic and breathable structure*, J. Plant Physiol. 163 (2006) 1193-1197.
- *Plastron properties of a super-hydrophobic surface*, Appl. Phys. Lett. 89 (2006) Art 104600.

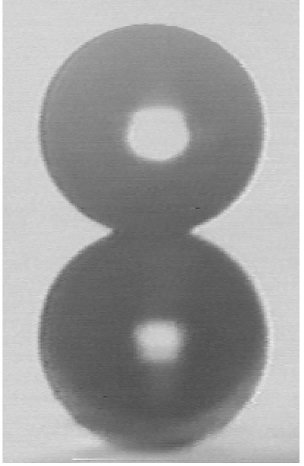
Theory of Wetting

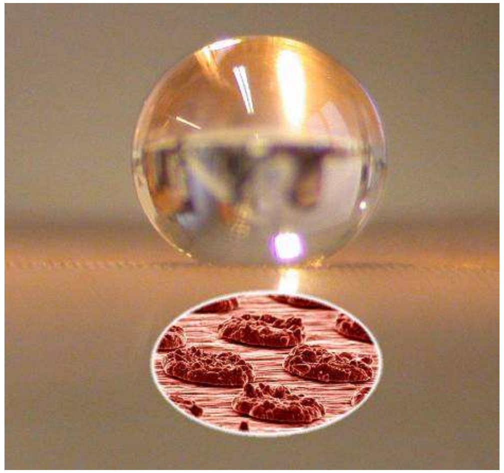
- *Liquids shape up nicely, Nature Materials* (Invited "New & Views" item) 6 (2007) 627-628.
- *Analysis of droplet evaporation on a super-hydrophobic surface*, Langmuir 21 (2005) 11053 - 11060.
- *Contact angle hysteresis on super-hydrophobic surfaces*, Langmuir 20 (2004) 10146-10149.

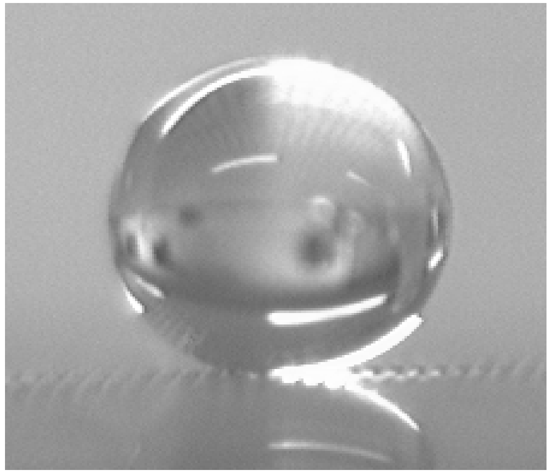
Materials

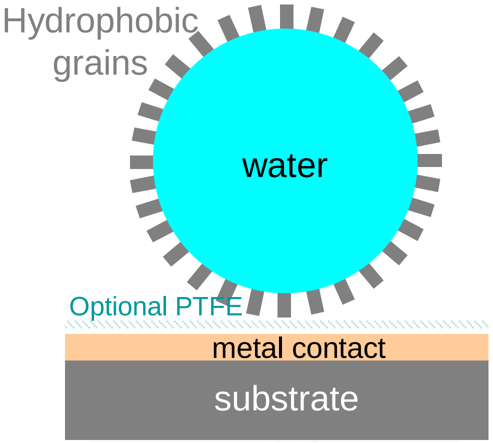
- *Porous materials show superhydrophobic to superhydrophilic switching*, Chem. Comm. (25) (2005) 3135-3137. (See also Nature Highlight/News "Quick change for super sponge" Published on-line 20/7/05).
- *Wetting and wetting transitions on copper-based super-hydrophobic surfaces*, Langmuir 21 (2005) 937-943.
- *Topography driven spreading*, Phys. Rev. Lett. 93 (2004) Art 036102.
- *Dual-scale roughness produces unusually water repellent surfaces*, Adv. Maters. 16 (2004) 1929-1932.
- *Intrinsically super hydrophobic organo-silica sol-gel foams*, Langmuir 19 (2003) 5626-5631.

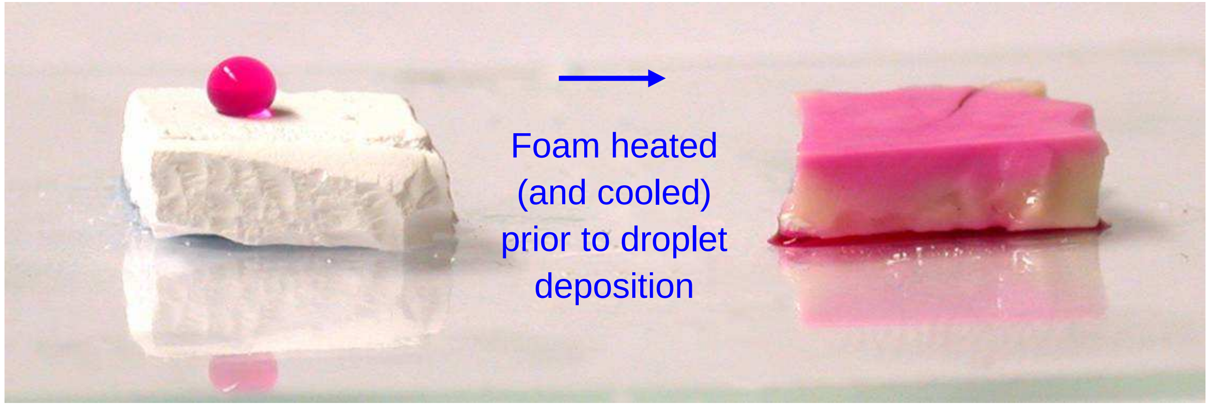


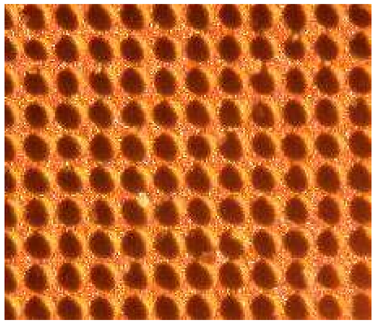


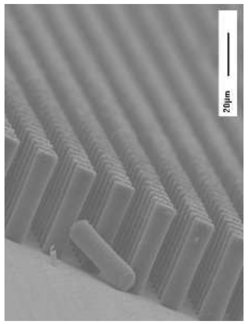


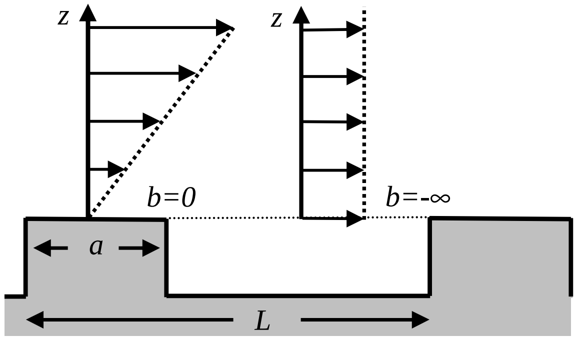



Hydrophobic grains
water
Optional PTFE
metal contact
substrate


Foam heated (and cooled) prior to droplet deposition






z
z
b=0
b=-∞
a
L

Acknowledgements

EPSRC (and BBSRC and EU and ….)	Funding of Research
Research Group	Doing the Work

Dr Mike Newton, Dr Neil Shirtcliffe, Dr Fabrice Martin, Dr Simon Stanley,

Dr Carl Evans, Dr Paul Roach, Ms Nicola Doy, Mr Shaun Atherton,

Mr Steve Elliott + Mr Jeremy Simons

Prof. Rees, Prof. Dodi, Dr Hughes	Biological Sciences
Dr Percival	Atmospheric Sciences
Dr Gizeli and Dr Melzak	Biotechnology/Love Waves
Prof. Thompson, Dr Lücklum Dr Hayward, Mr Ellis	QCM and Slip
Prof. Allen, Prof. Hardacre, Dr MacInnes, …	Ionic Liquids

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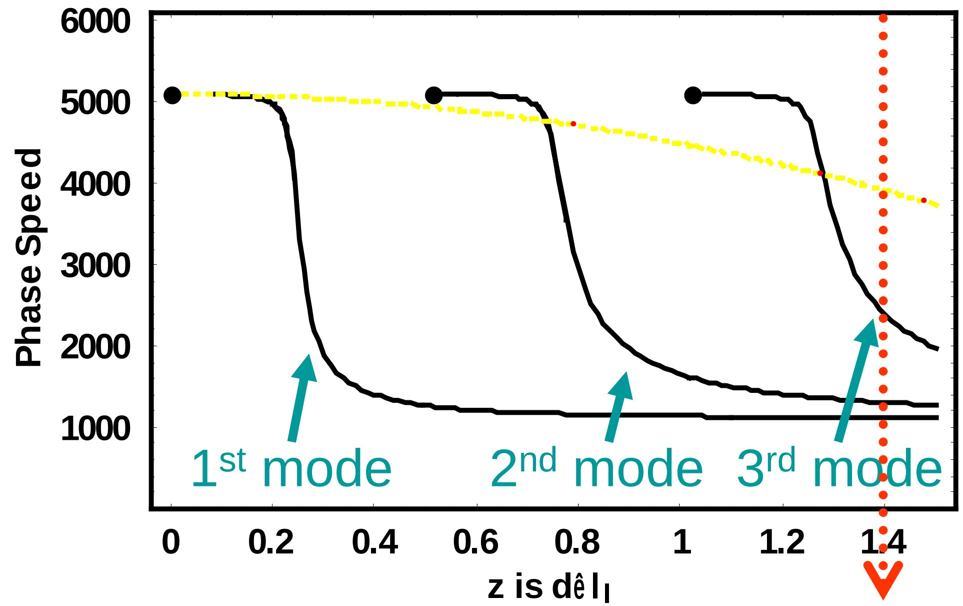
Thank you for your attention

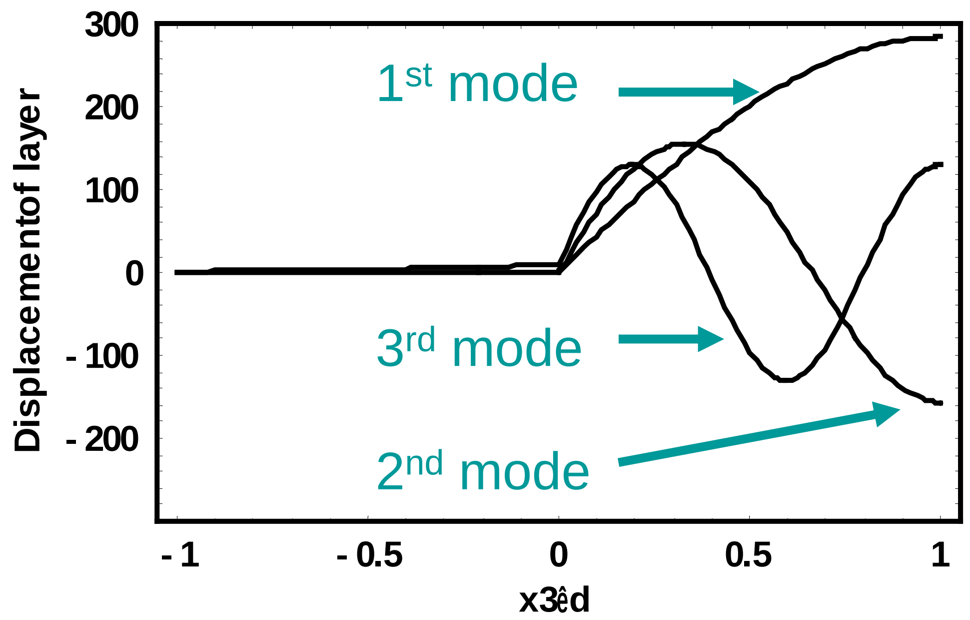
Love Waves

Theoretical dispersion curve

(Insertion loss is unchanged by an elastic guiding layer)

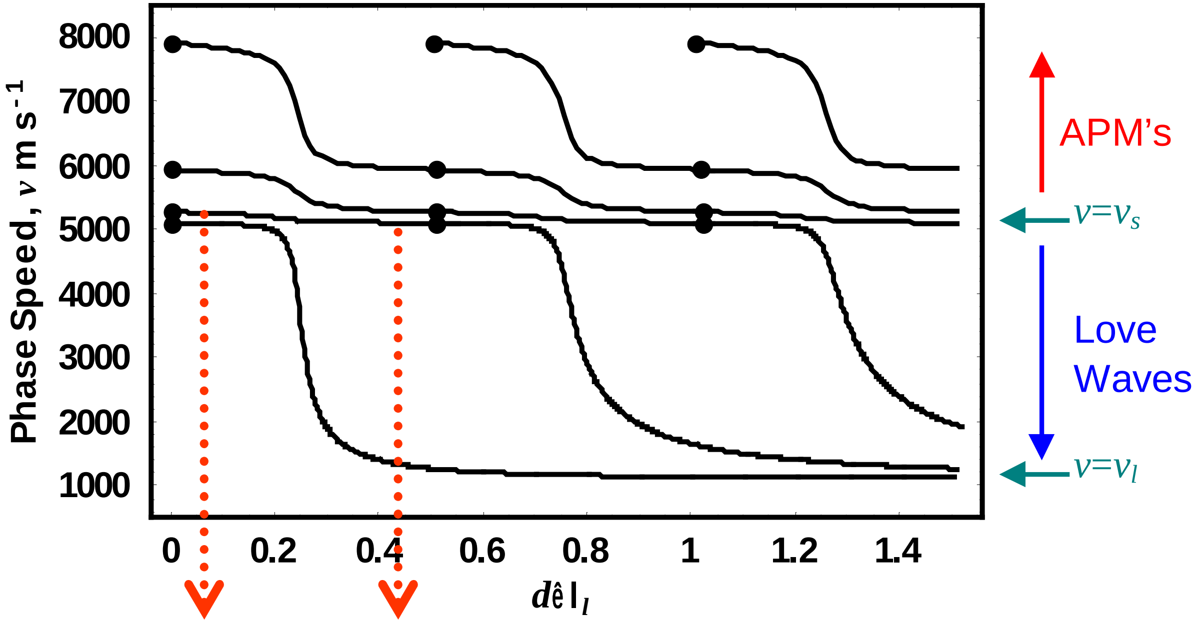


Displacements for first three modes (z=1.3)



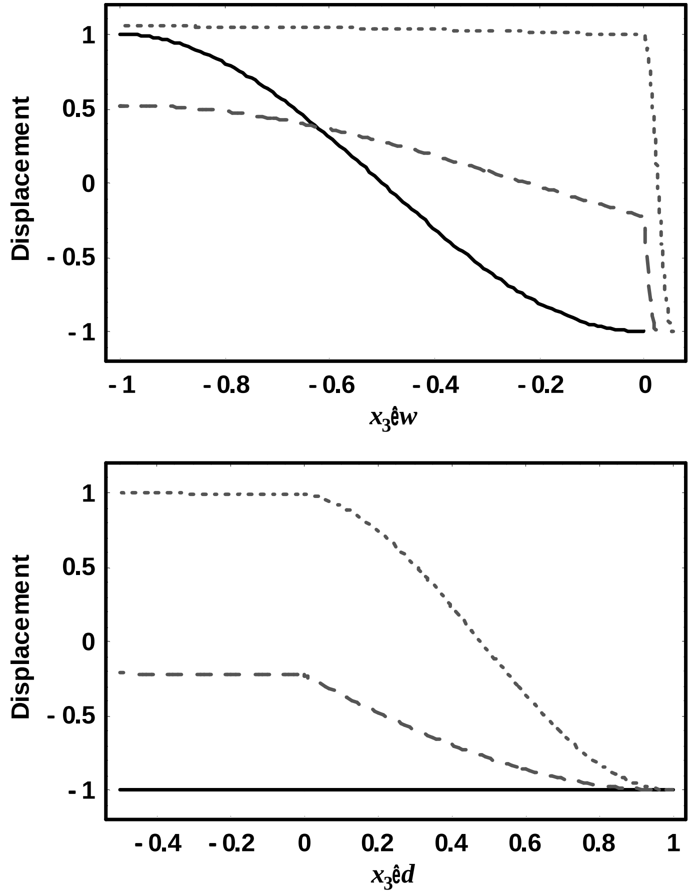
Substrate + Mass Guiding Layer

Dispersion Curve



Points = Anti-node moving from substrate to layer

Evolution of 1st SH-APM



Solid → dashed with increasing guiding

Dispersion and Group Velocity

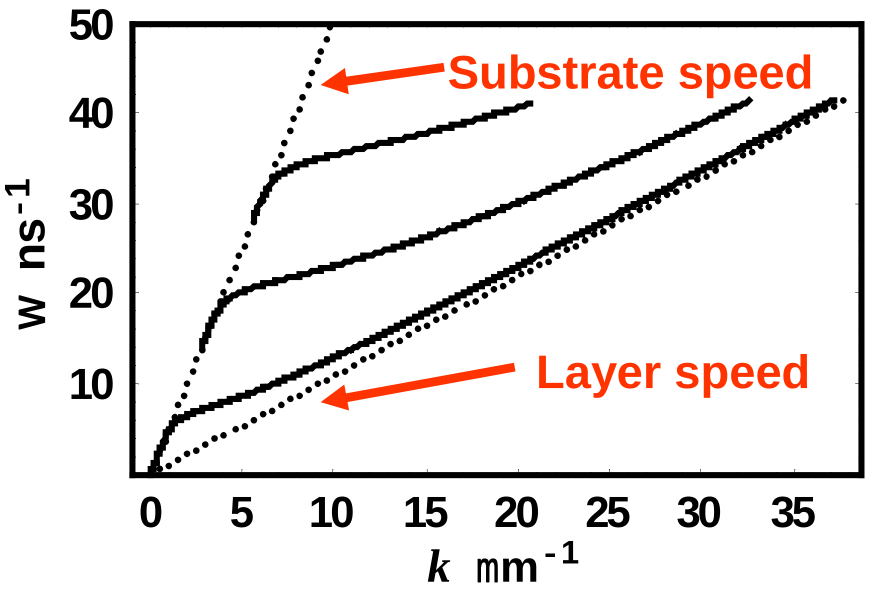
Guiding Layer Induces Dispersion

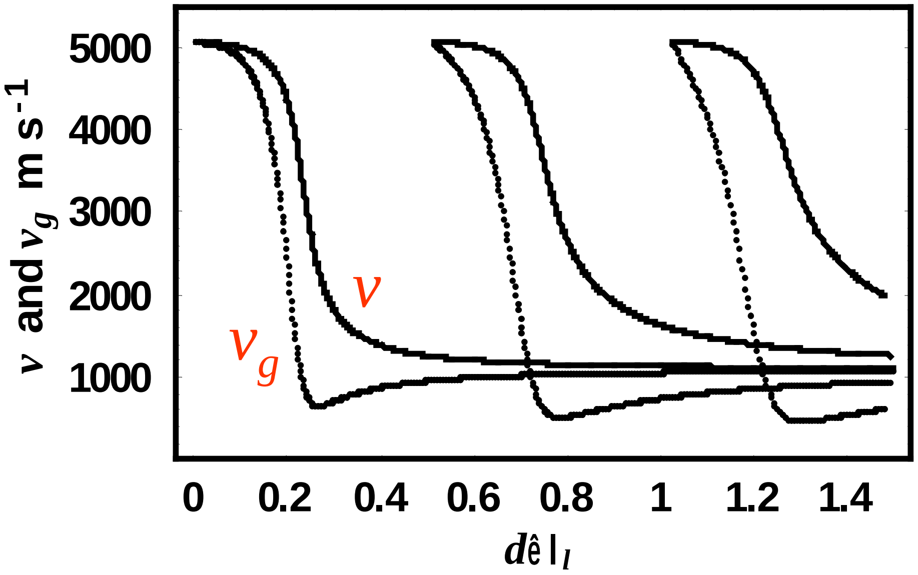
Phase velocity $v = f\lambda$ or $v = \omega/k$

Group velocity $v_g = d\omega/dk$

Group velocity is slope of the (ω, k) dispersion curve

Example 0.25 μm polymer guiding layer on Quartz with $w \rightarrow \infty$





Group Velocity Mass Sensitivity

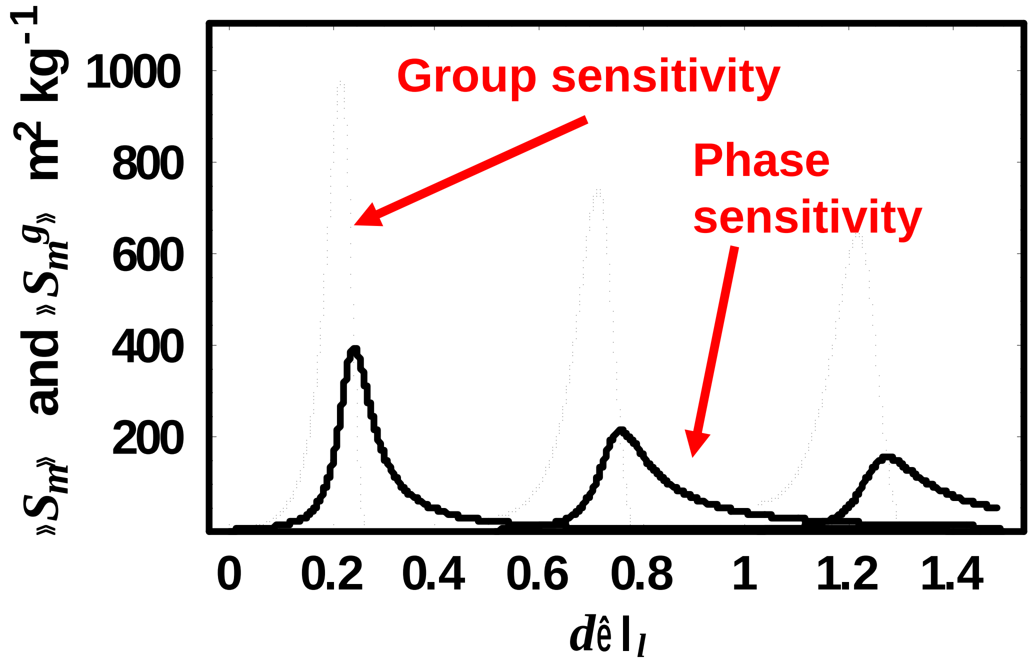
$$S_m \approx \frac{1}{\rho_l d}\left(1-\frac{v}{v_g}\right)=\frac{1}{\rho_l d}\frac{\left(v_g-v\right)}{v_g}$$

"Rigid" mass

Mass sensitivity is fractional deviation of the phase velocity from the group velocity divided by mass per unit area due to the guiding layer

Define a Group Velocity Sensitivity

$$S_m^g=\frac{f_o}{\rho_l v_l}\left(\frac{d\log_e v_g}{dz}\right)_{z=z_o}$$

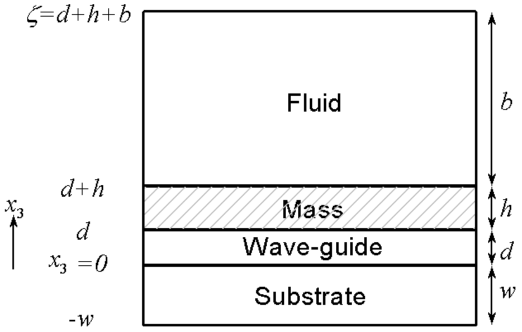


NTU

Insertion Loss for Polymer Waveguide

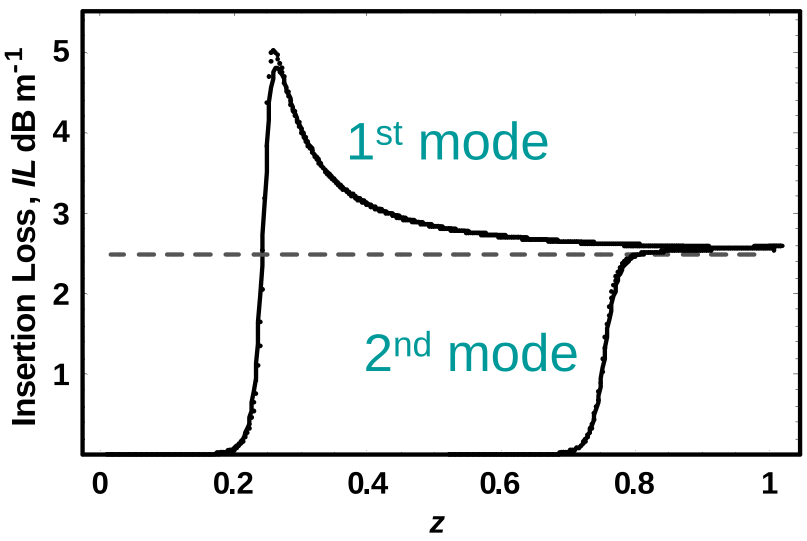
Cases considered

1. Wave-guide layer is viscoelastic
2. Mass layer deposited from liquid or from vacuum
3. Mass may be omitted (i.e. liquid phase sensitivity)

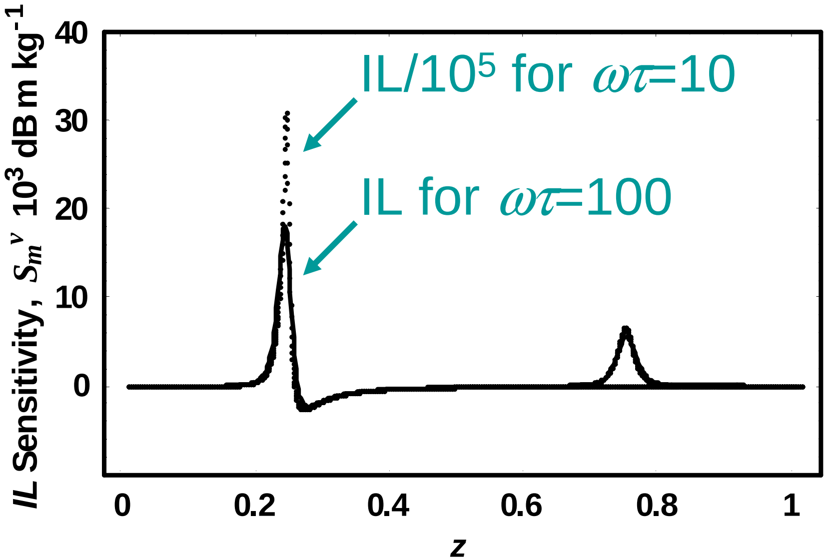


Mass/liquid sensitivity can be derived for phase velocity & insertion loss

Love Wave Insertion Loss



Love Wave IL Sensitivity



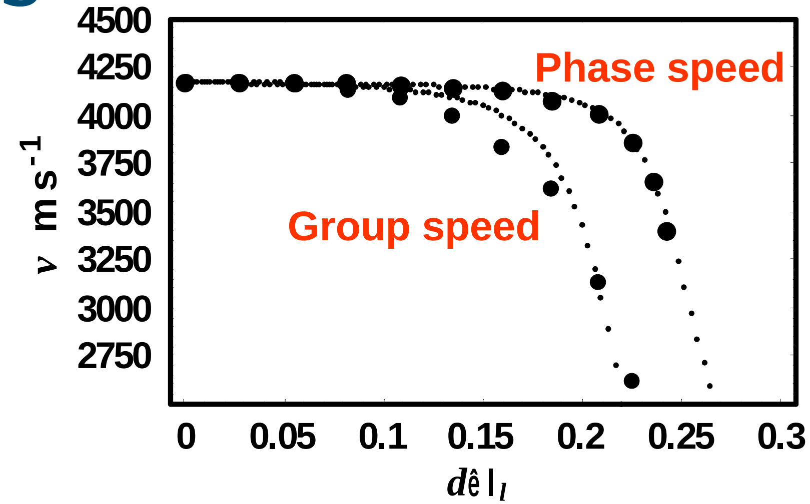
Group Velocity Data - Solids

Phase and Group Velocity

36° XY $LiTaO_3$

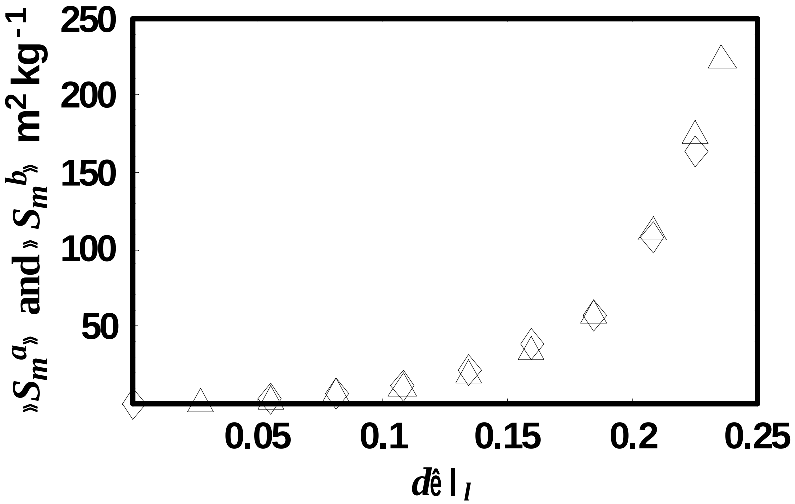
Hardbaked photoresist

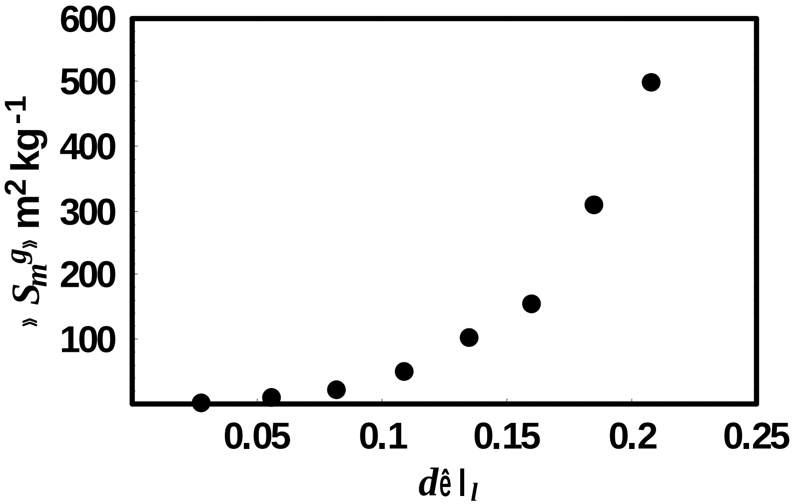
Dotted curves are fits



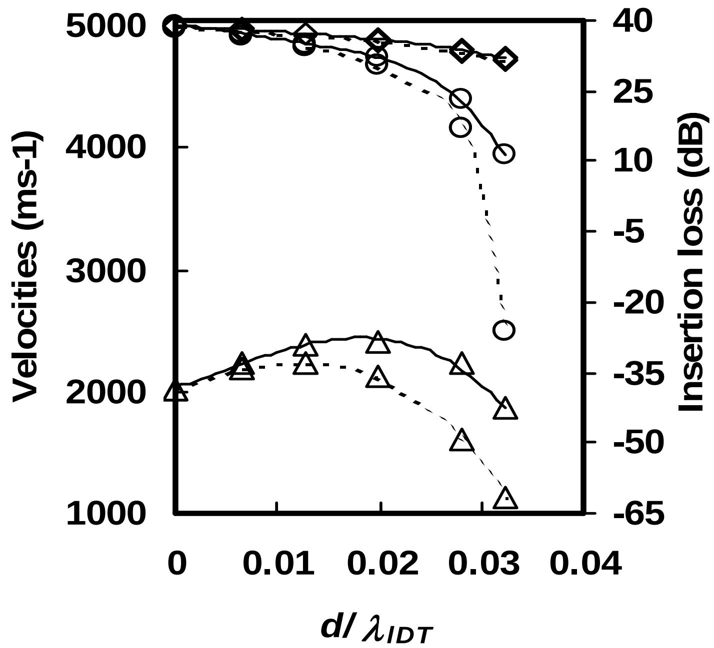
Mass Sensitivity

$$S_m^b = \frac{1}{\rho_l d}\left(1 - \frac{v}{v_g}\right) \qquad S_m^a = \frac{1}{\rho_l}\left(\frac{d \log_e v}{dx}\right)_{x=d} \qquad S_m^g = \frac{1}{\rho_l}\left(\frac{d \log_e v_g}{dx}\right)_{x=d}$$





Group Velocity Data - Liquids



Deposition of Guiding Layer

ST-Quartz + photoresist

Poly (ethylene glycol) solutions

= Phase velocity = Grp Velocity

Δ = Insertion loss Dotted = water

Sensitivity at Operating Points

Phase and group velocities