ZnO Based SAW and FBAR devices for Lab-on-a chip Applications

W.I. Milne[1], X.Y. Du[1], Y.Q. Fu[3], J.Lu[4], X.Zhao[4] A.J. Flewitt[1],Luis Garcia-Gancedo[1], Greg Ashley[2],J.K. Luo[1,2]

[1] Electrical Division, Engineering Dept, Univ of Cambridge, UK
[2] University of Bolton, UK
[3] Herriot Watt University
[4] Dept of physics, Univ of Manchester

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Outline

1. Introduction to Lab-on-chip
2. ZnO film deposition and characterization
3. ZnO thin film surface acoustic wave devices
   - Micromixer
   - Surface treatment
   - Micropump
   - SAW sensors
4. FBAR devices
5. Lab-on-a-chip
6. Conclusions

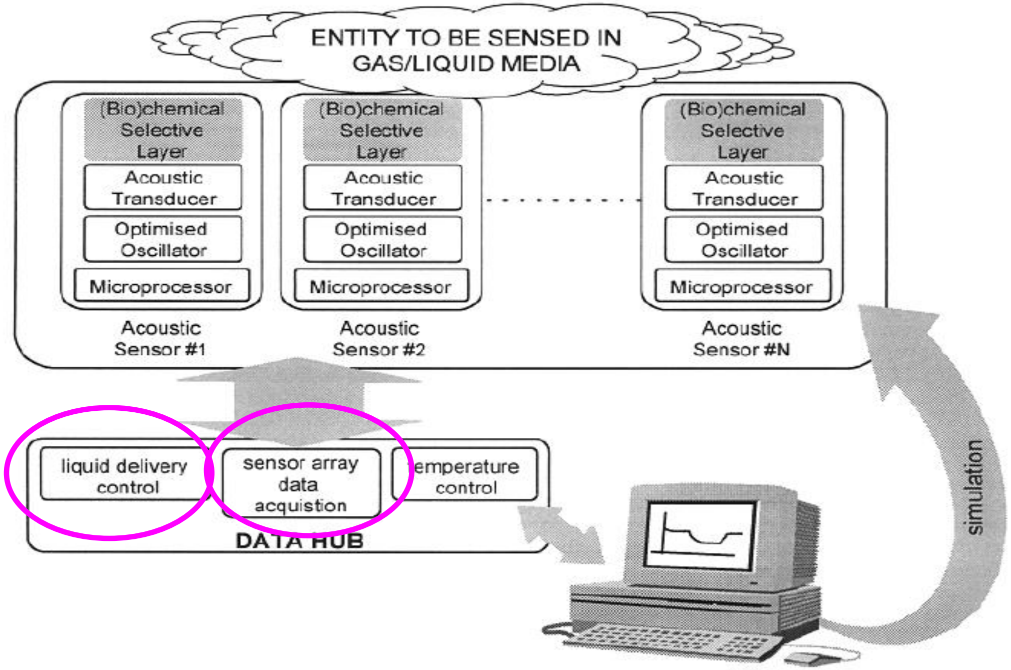
A Typical Lab-on-a-Chip

- Lab-on-a-chip typically consists of microfluidics, and electronics to perform filtering, sorting, reaction, detection…
- **Microfluidic systems**
  - Microchannels, microvalves, microfilter
  - Micropumps, Micromixers
  - Microreactor….
- **Biosensors:**
- Optical detection biosensor
- Thermal detection biosensor
- Electrochemical biosensors
  - Conductimetric
  - Amperometric
  - potentiometric
- Ion senstive FET biosensor
- Resonant biosensor
  - Quartz crystal mass sensor (QCM)
  - Surface acoustic wave (SAW)- high sensitivity and low detection limit (pg/ml)
  - However although this works the SAW device is big and difficult to scale down.
  - Our solution is to use FBAR sensors

- **Electronics**

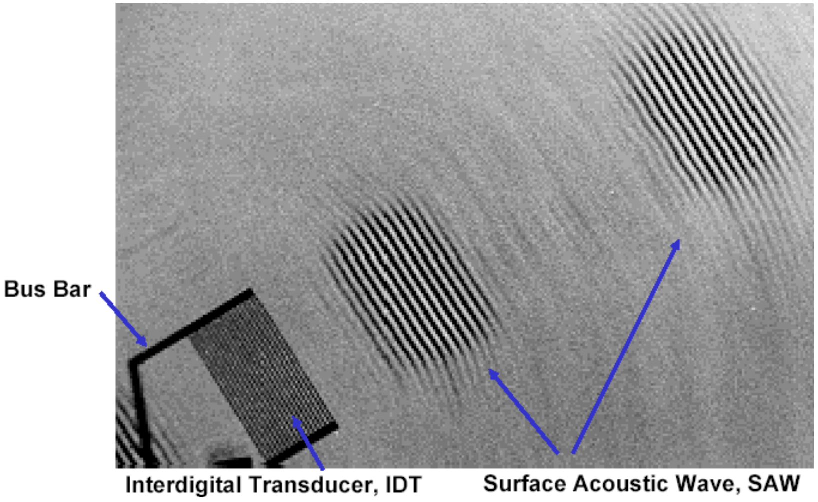
Objective

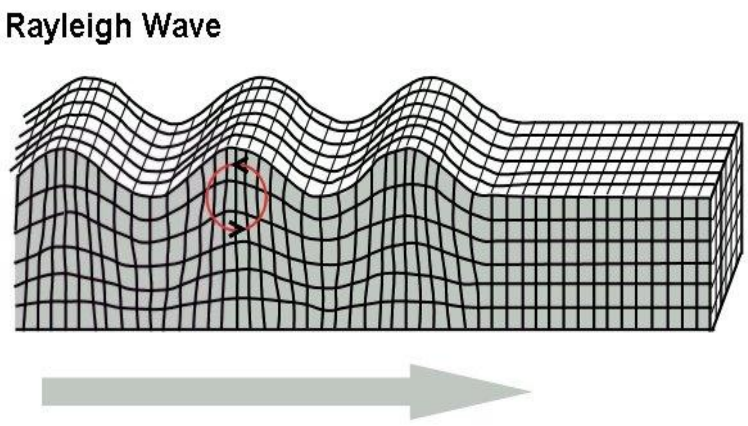
Integrated ZnO SAW based microfluidics (micropump and mixer) and sensing (microsensor) system

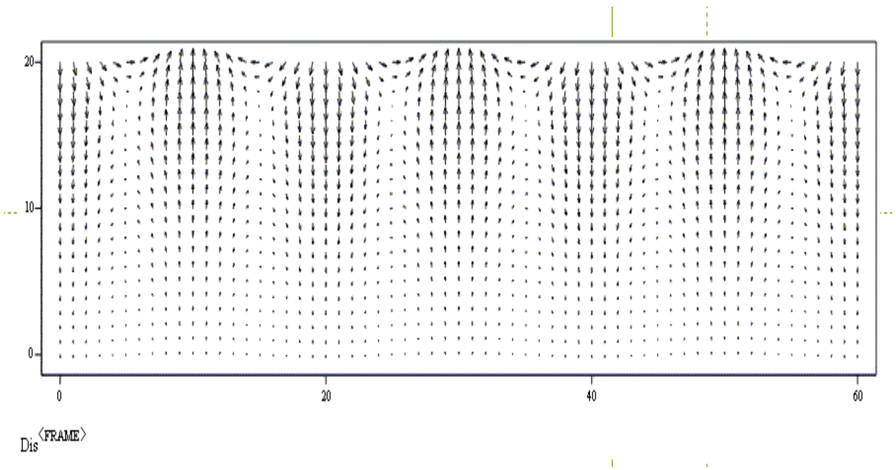


Surface acoustic wave (SAW)

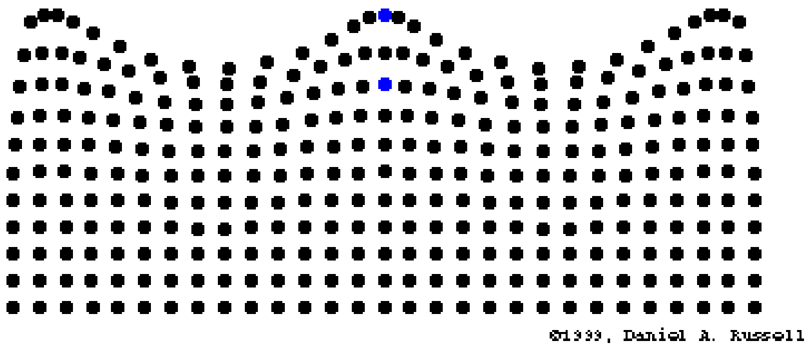
- Piezoelectric material
- RF signal
- Interdigital transducer
- Acoustic wave
- Surface propagation

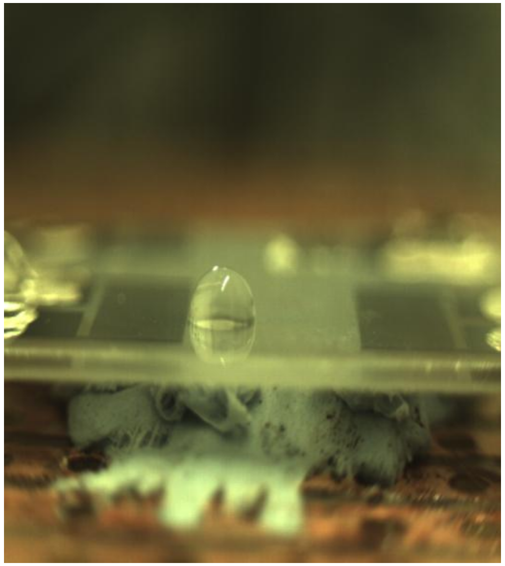


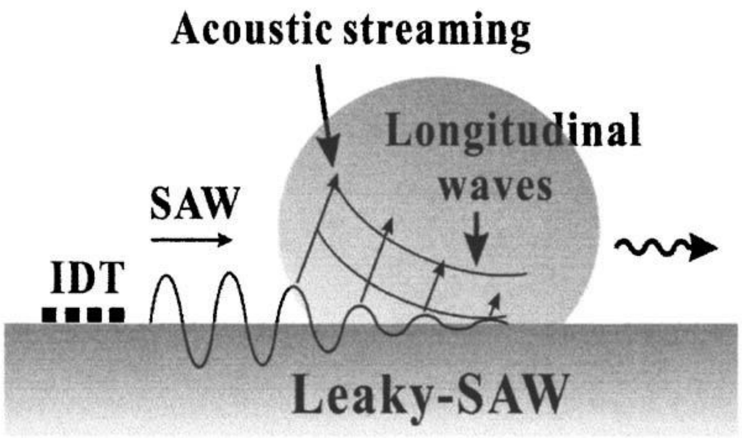




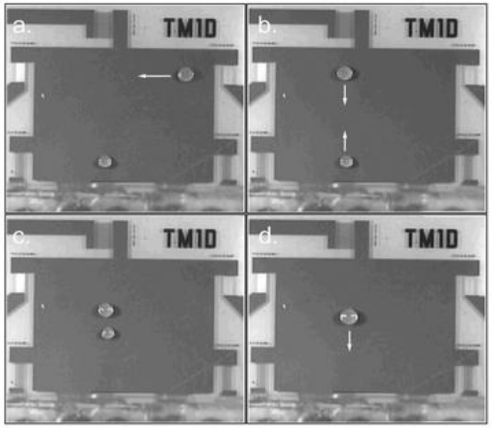
Principle for SAW pumping







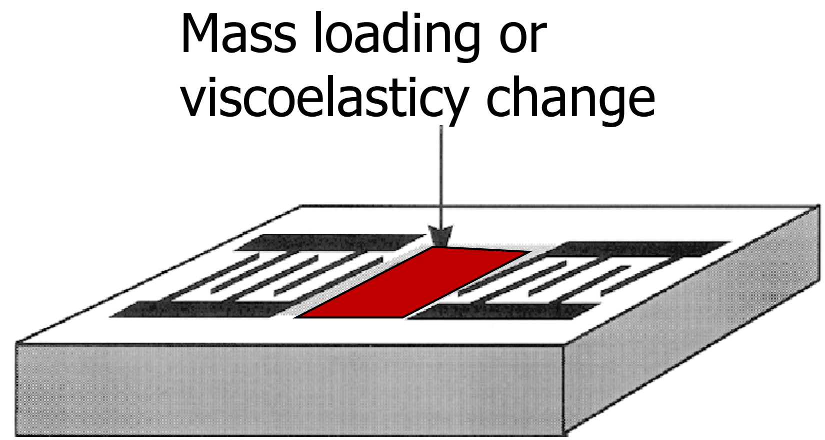
All on $LiNbO_3$

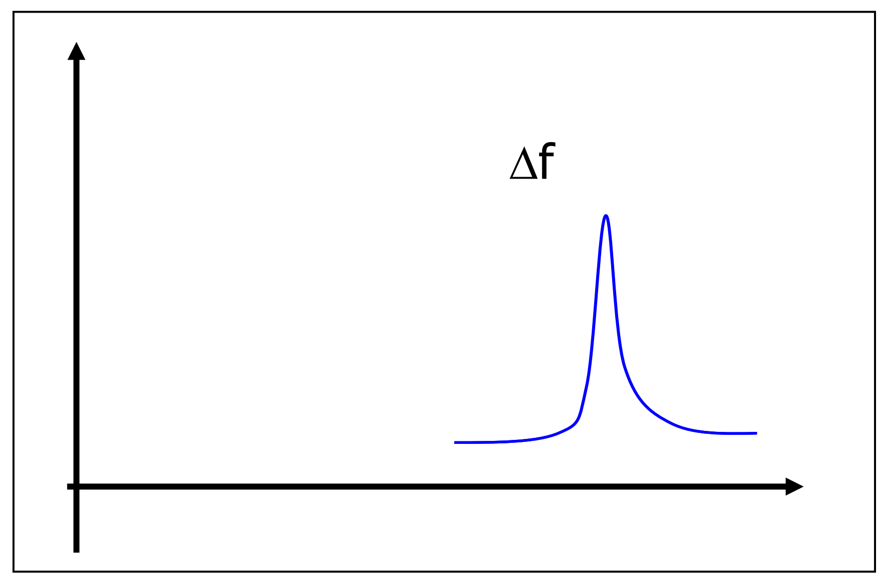


A. Wixforthm Superlattics Microstruct., 33 (2004) 389

Principle of SAW Sensor

- SAW is sensitive to
  - Mass loading
  - Viscosity change
  - Elasticity change
- SAW signals
  - Resonant frequency
  - Amplitude
  - Phase
  - Impedance
  - ……





- Sensitivity $\propto$ fr
- $\Delta f \propto \Delta m/m$, or $\Delta m \propto \Delta f * m$

- From an applications viewpoint SAWs operating at high frequency are preferred→ higher power density
- 2 approaches- either use reduced spacing for IDTs as resonant frequency is determined by spacing (but need sophisticated litho)
- OR use material with higher acoustic velocities (and perhaps utilise higher order modes- e.g.the Sezawa mode)

Piezoelectric materials

Material	SAW velocity v (m/s)	Coupling K^2(%)	ε (pF/m)	α_T (ppm/°C)
Quartz ST-X	3158	0.16	55	≈0
$LiNbO_3$ (YZ)	3485	4.5	460	91
$LiNbO_3$ (128°)	3921	5.7	-	57
ZnO	2715	1	-	40
$Bi_{12}GeO_{20}$ (100) (011)	1681	1.5	400	130

- However Lithium Niobate is expensive to use for such an application and cannot be integrated with Si – based ICs
- →Use SAW devices based on thin piezoelectric films grown onto other (lower cost)substrates→ lower cost, low power consumption and possible integration with Si
- We have chosen to investigate ZnO thin films
- For our initial results RF magnetron sputteringfilms from a zinc target in an Ar/Oxygen atmosphere- was used
- Now High Target Utilisation Sputtering(HiTUS) is used

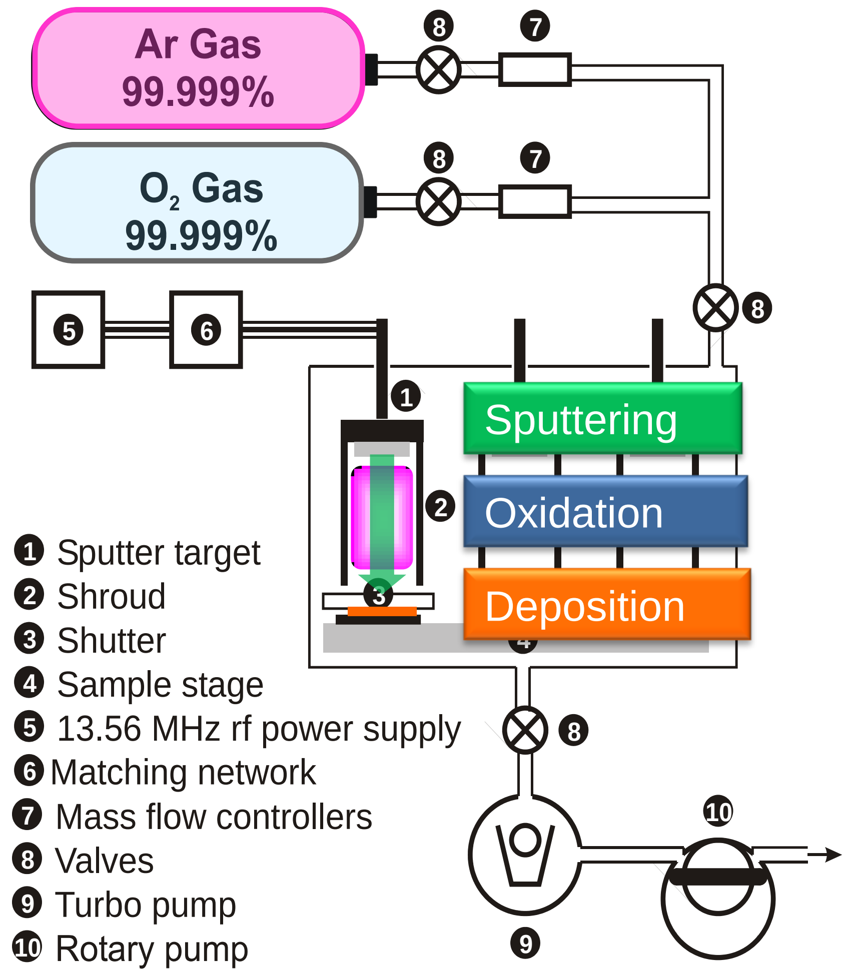
ZnO

- For application in acoustic wave devices, ZnO films must have the following properties:

- Ordered crystalline structure – good piezoelectric properties
- Smooth surface – surface roughness decreases *Q*
- High deposition rate – films thicker than 2 μm are needed
- Low stress and RT deposition – *possibility of using plastic substrates*
- High resistivity as mobile charges reduce piezoelectric transduction
- Cost effective, repeatable results

RF Magnetron Sputtering

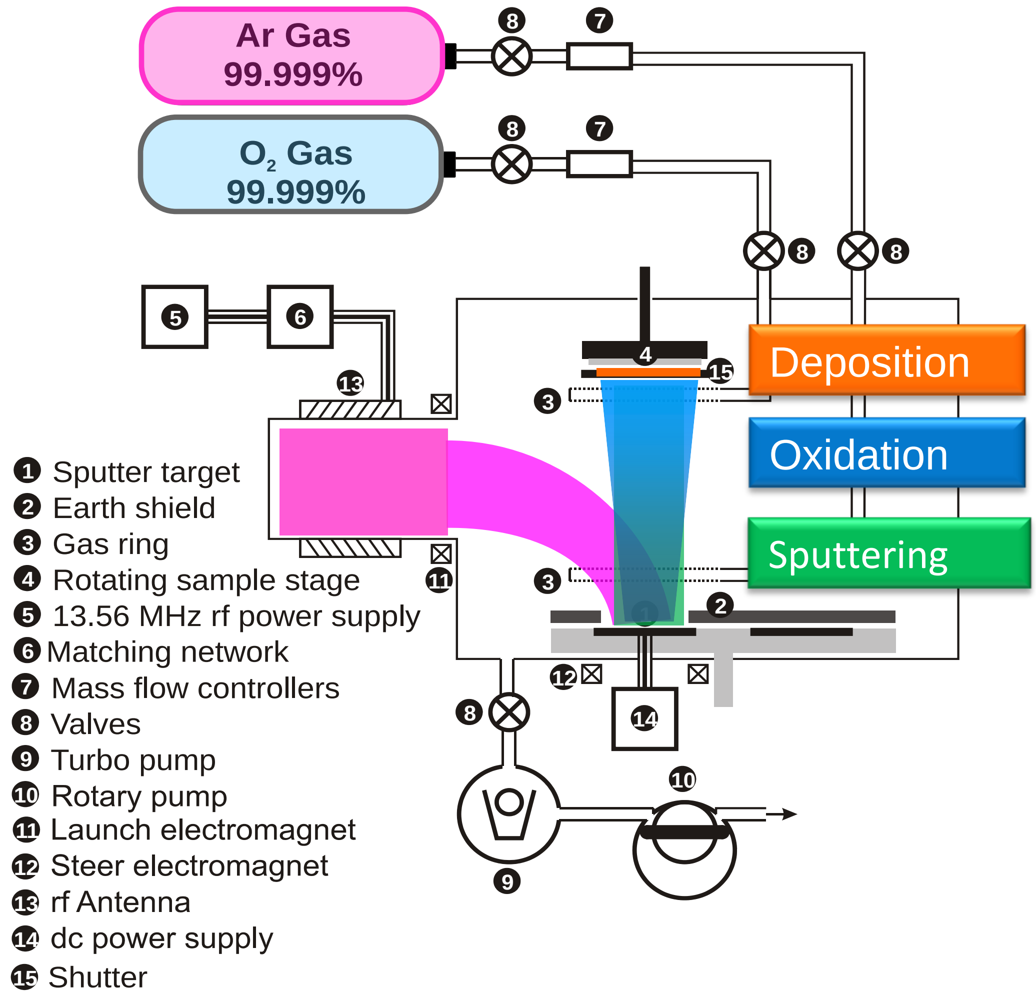
- ✓ Very easy growth technique
- ✓ Easy to optimise
- ✓ Established technology
- ✓ Low cost
- ✗ Ion energy is dependent on RF power
- ✗ Sample in direct line-of-sight of plasma → exposed to ion bombardment
- ✗ Higher growth rate → higher RF power → more ion bombardment → film damage/stress



Our sputtering choice:

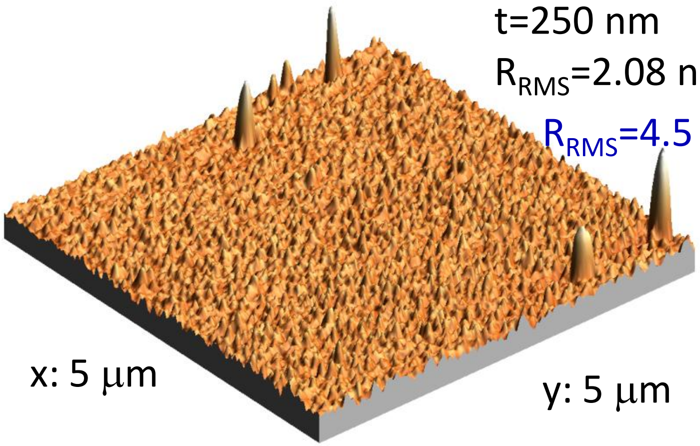
HiTUS System

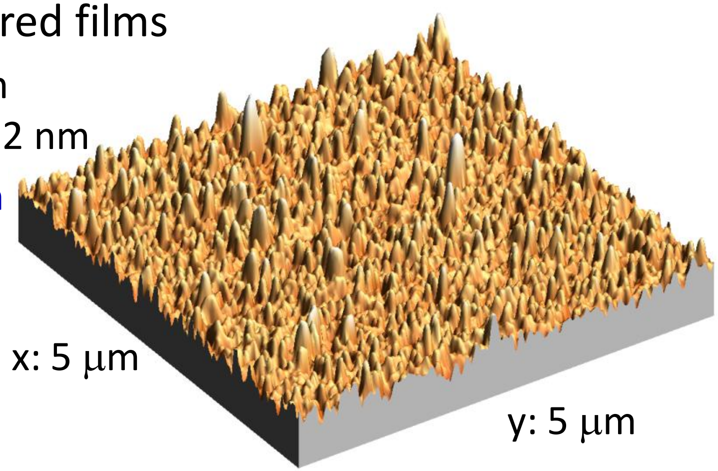
- ✓ Independent control of plasma density and sputtering ion energy
  - ✓ Excellent control of material properties including stress
- ✓ Sample away from sputtering plasma
  - ✓ Reduced ion bombardment on sample
- ✓ High target utilisation
  - ✓ High deposition rate

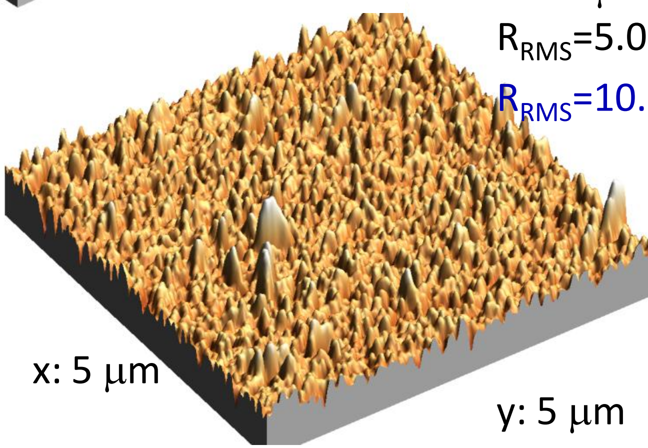


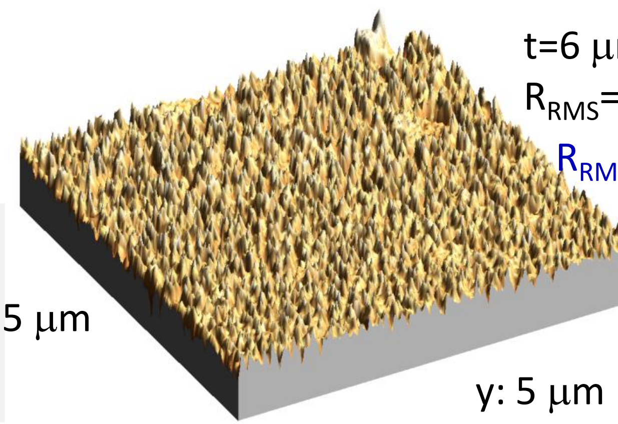
ZnO films resistivity surface roughness

- Film with low surface roughness are obtained
- Films are much smoother than magnetron sputtered films





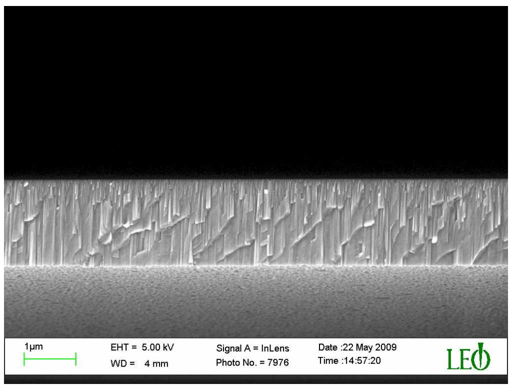


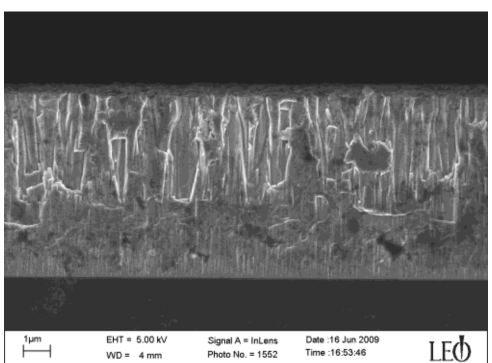


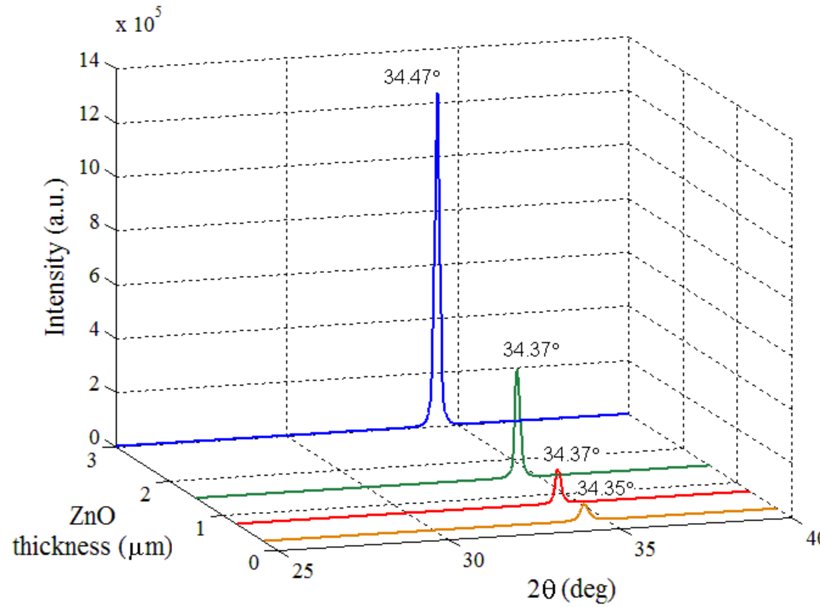
Crystallographic orientation

- ***c*-axis orientation** is normal to the substrate

SEM images







- Single peak at 2θ=34.37° with FWHM~0.2°
- The theoretical (002)-peak is positioned at 2θ=34.42°, meaning that our films have excellent *c*-axis orientation and possess very low stress

Film stress

Stress was calculated using Stoney's equation after measuring the change in curvature of the substrates after deposition

It was found to be less than an order of magnitude smaller than ZnO films obtained from magnetron sputtering

ZnO film thickness	Magnetron sputtering	HiTUS system
0.8 μm	~1.18 GPa	~60 MPa
1.8 μm	~1.34 Gpa	~82 MPa
2.2 μm	~1.31 Gpa	~92 MPa
4 μm	~1.53 GPa	~109 MPa

The room temperature HiTUS technique eliminated the stress induced by the mismatch in the thermal expansion coefficients of film and substrate, present in all other epitaxial and deposition techniques at high temperature

HiTUS benefits

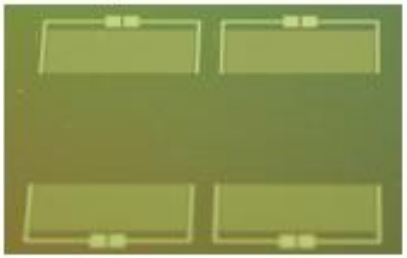
Novel Technology: HiTUS

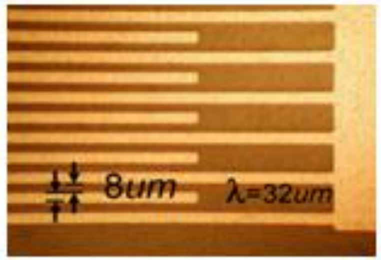
- ✓ Remote plasma, low ion-induced damage
- ✓ Large area compatible
- ✓ Higher deposition rate: *scalable for high-volume manufacturing*
- ✓ Low temperature process & low film stress: *plastic substrate compatible*

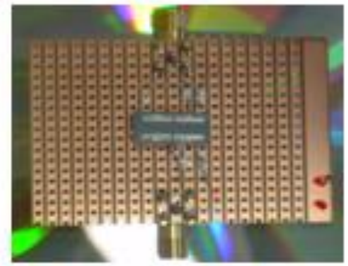
Improved ZnO material properties

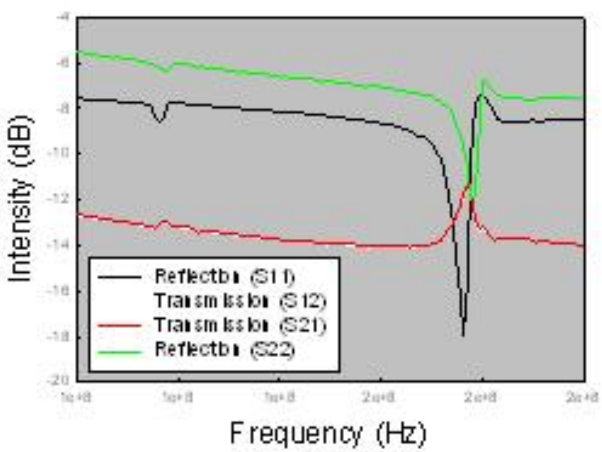
- ✓ Tuneable conductivity
- ✓ Very high deposition rate
- ✓ Smooth surface
- ✓ Low stress
- ✓ Excellent crystallographic orientation
- ✓ Repeatable, cost effective

Frequency measurement

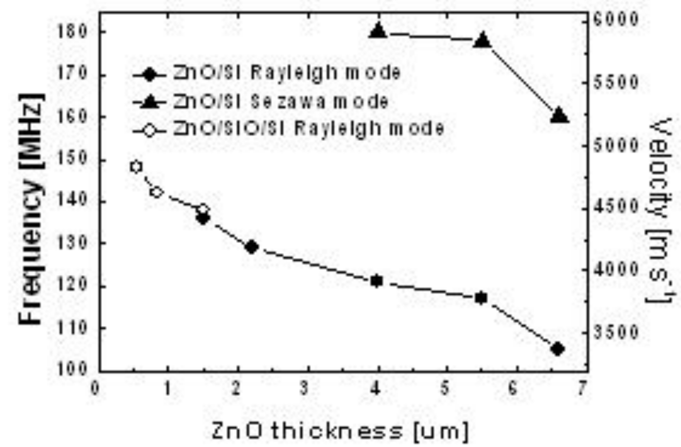




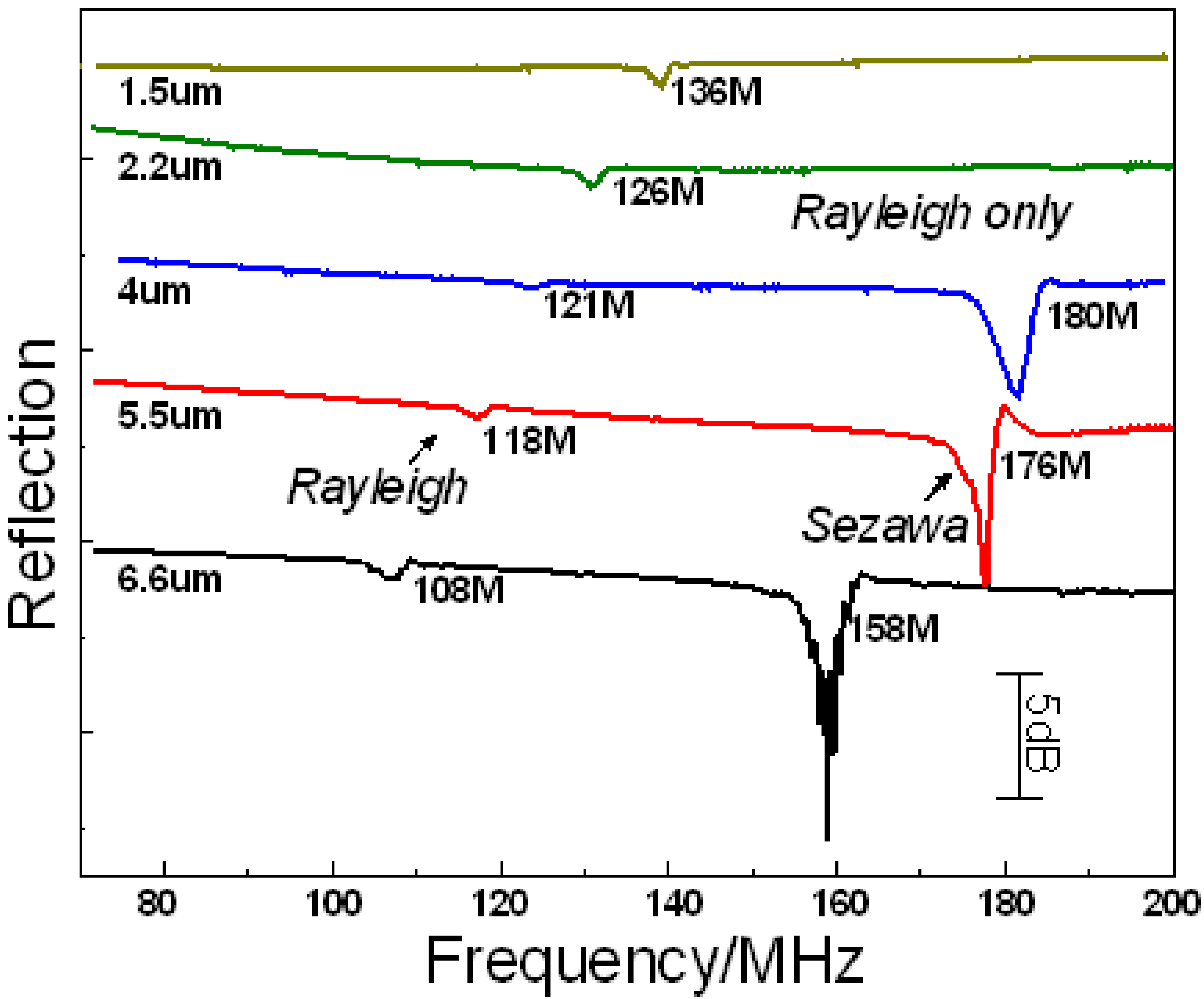




ZnO SAW frequency



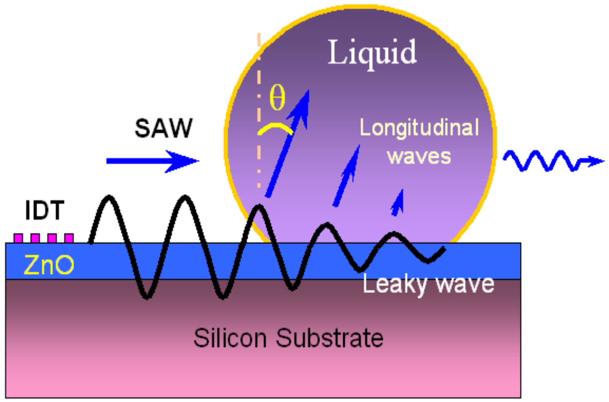
SAW frequency vs. film thickness

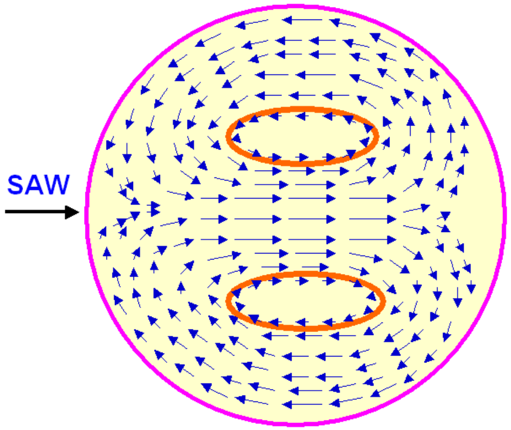


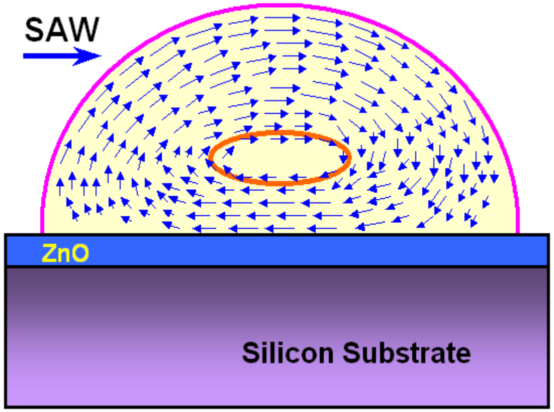
1.5um
136M
2.2um
126M
Rayleigh only
4um
121M
180M
5.5um
118M
Rayleigh
176M
Sezawa
6.6um
108M
158M
5dB
80
100
120
140
160
180
200
Frequency/MHz
Reflection

- As can be seen the Sezawa mode exists in addition to the Rayleigh mode- the most important parameter that governs its emergence is the ratio of the thickness of the ZnO layer to the SAW wavelength.
- The Sezawa mode does not exist for thickness of less than about 4 microns
- It is optimum to use the Sezawa Mode as it exhibits a higher electromechanical coupling coefficient – better for fluid motion
- BUT we also need Smooth Films because surface roughness will hinder wave transmission
- Initially (as shown in previous 2 slides) we used r.f. sputtered thick films
- For HiTUS a 1 micron thick film has a rms roughness 3.32 nm and for the 6 micron thick film has a rms roughness of only 13.1 nm so great improvement over RF sputtered films

SAW mixing inside water droplet on untreated r.f sputterd film



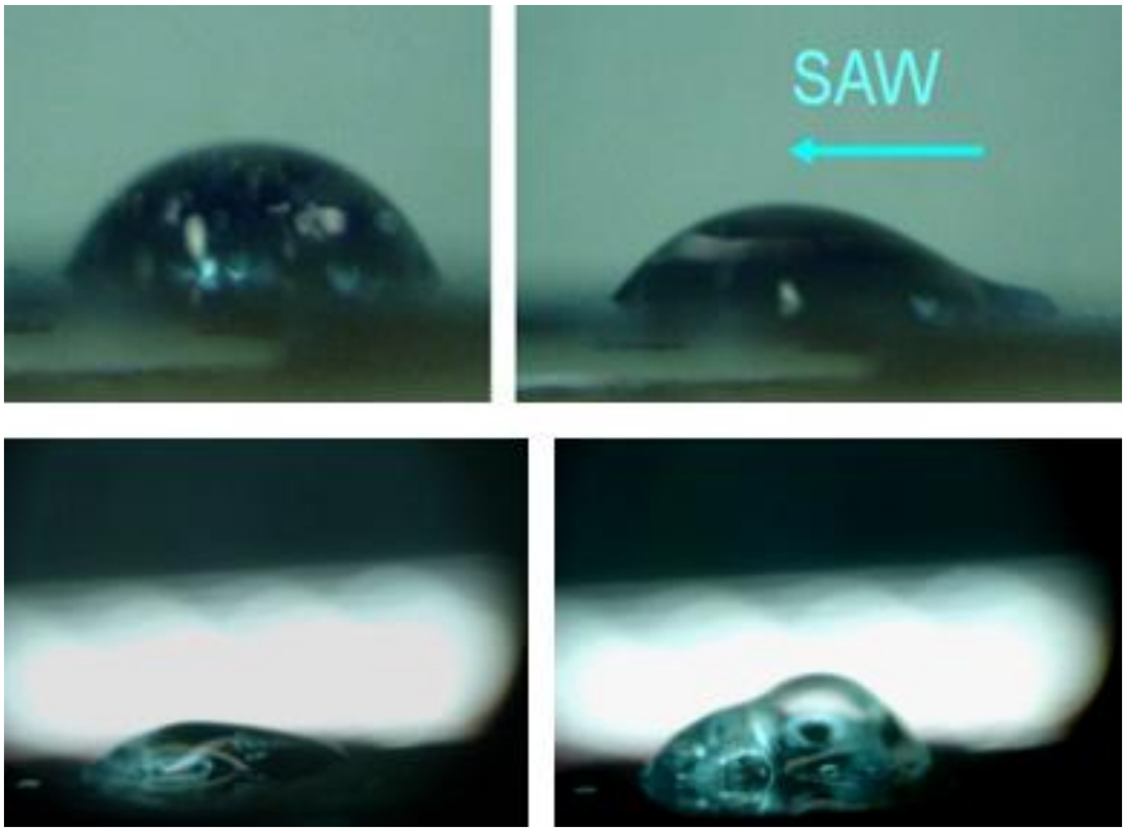




Schematic drawing of mixing patterns

- Here the liquid tries to move but is constrained by the hydrophillic surface .
- So we can see the ink particles are pushed along in the wave propagation direction until they hit the surface and are reflected-> reverse flow as shown
- Need to provide a hydrophobic surface

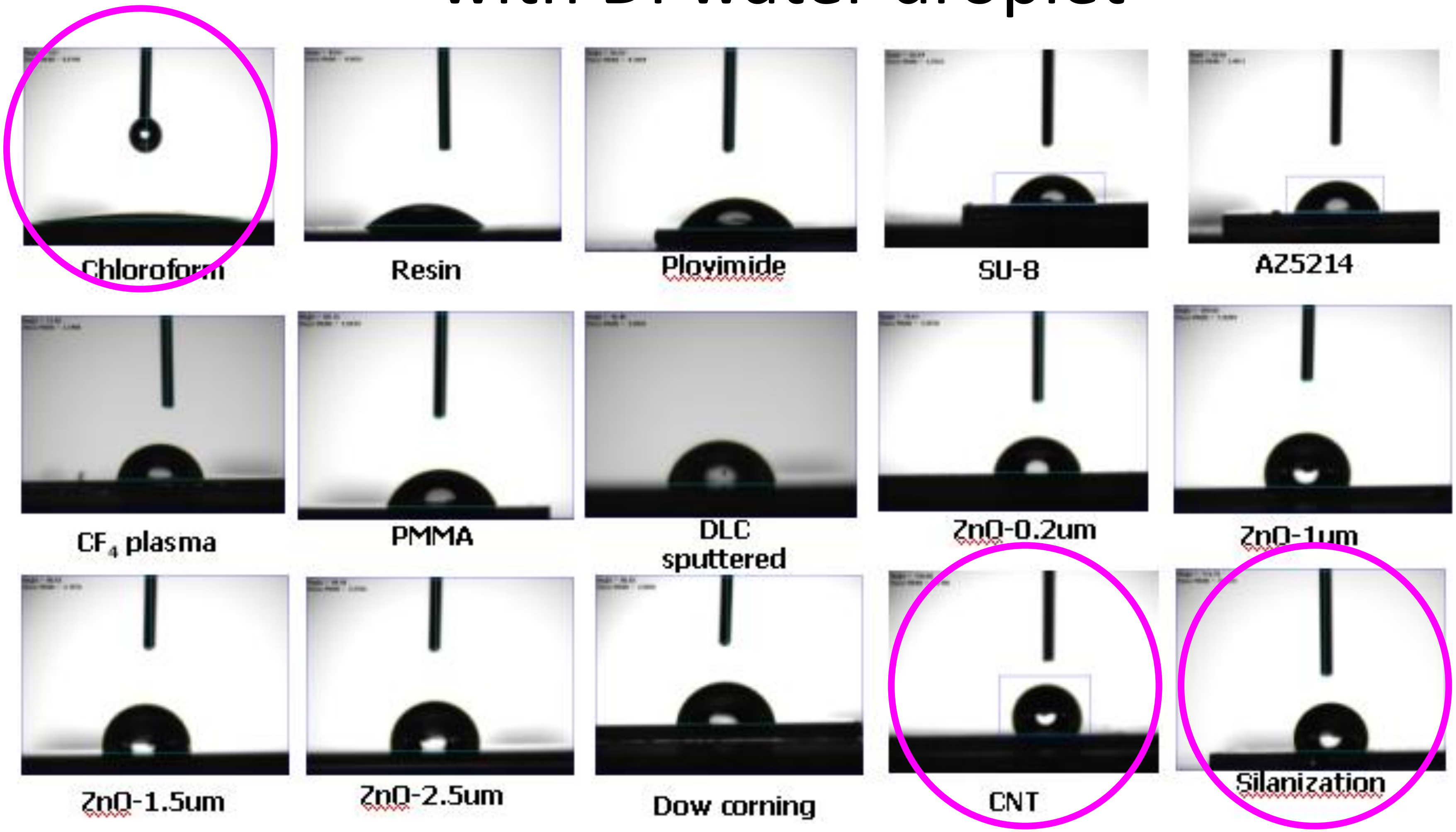
If ZnO film surface not treated..



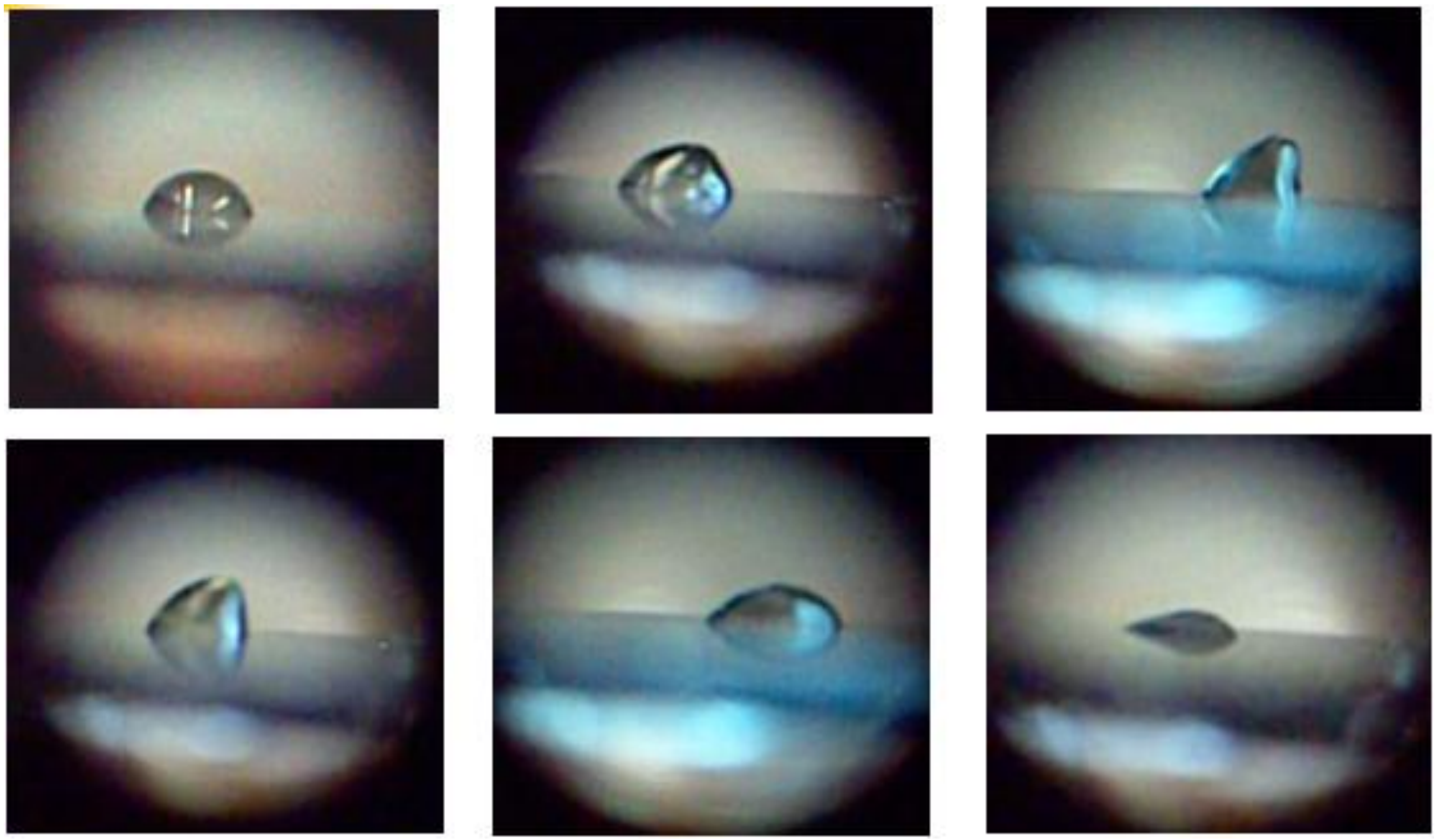
Droplet deformation

Droplet Evaporation

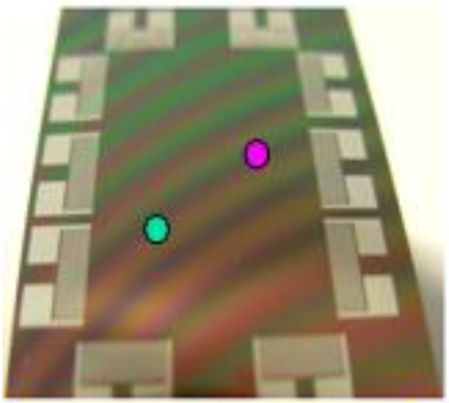
Contact angle of surface treated ZnO with DI water droplet

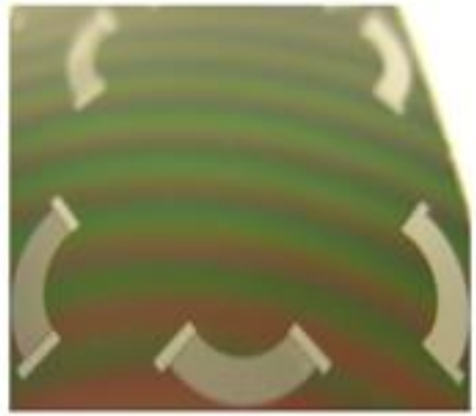


Cross-section observation of small water droplet moving with SAW

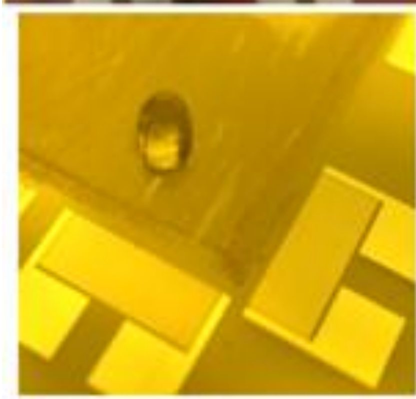


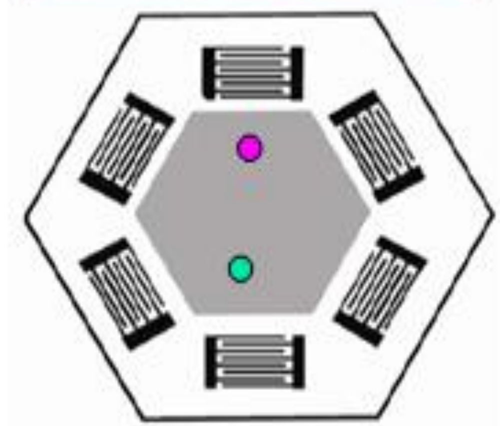
New ZnO based pump design

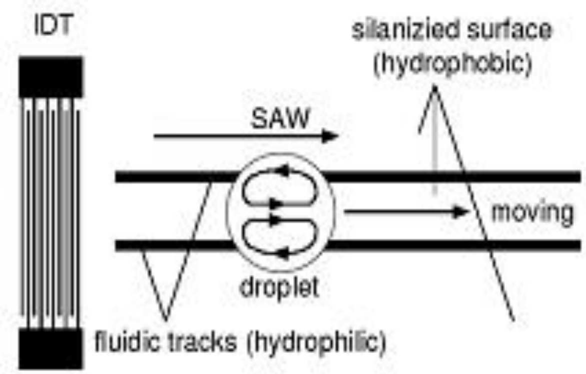




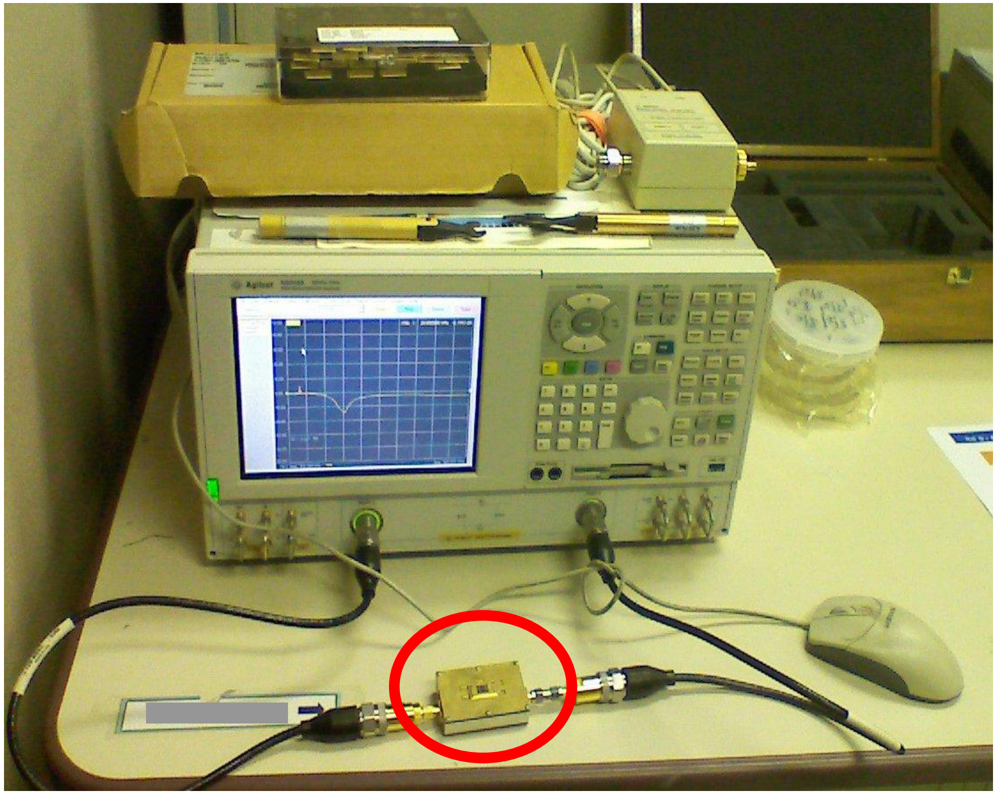
Hydrophilic borders constrain the liquid

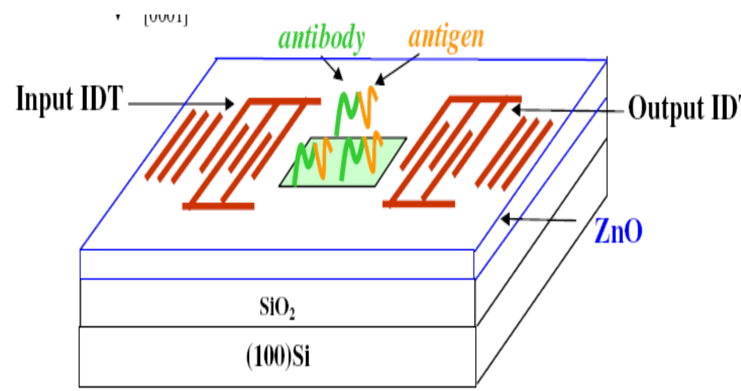


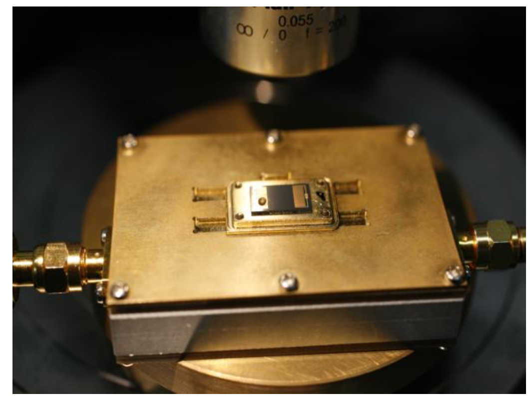




ZnO SAW sensing test set-up



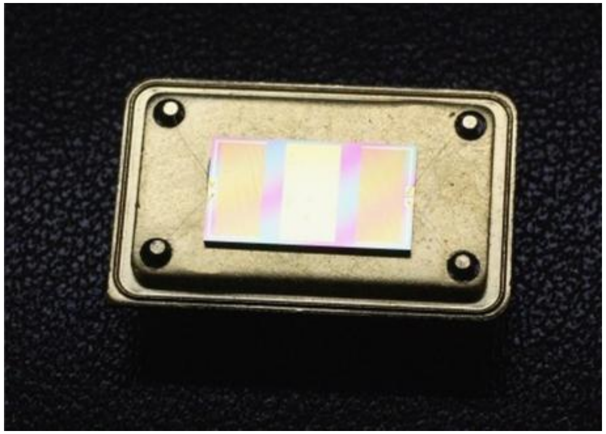


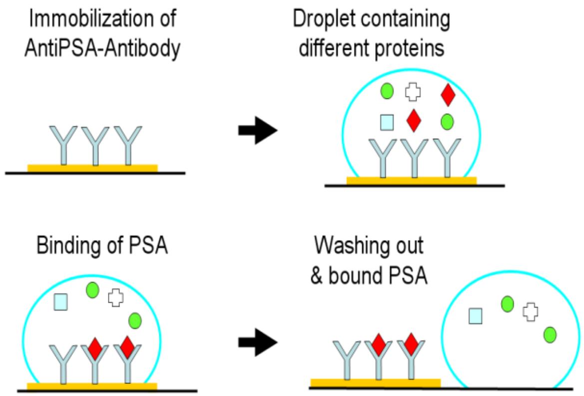


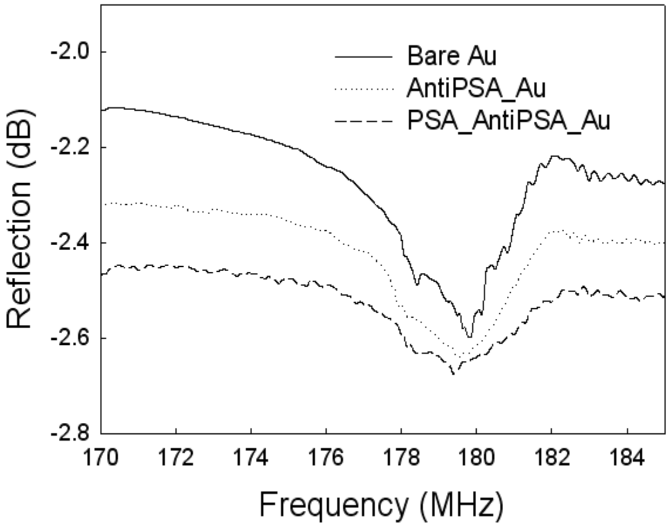
Prostate specific antigen (PSA) is the target mol.

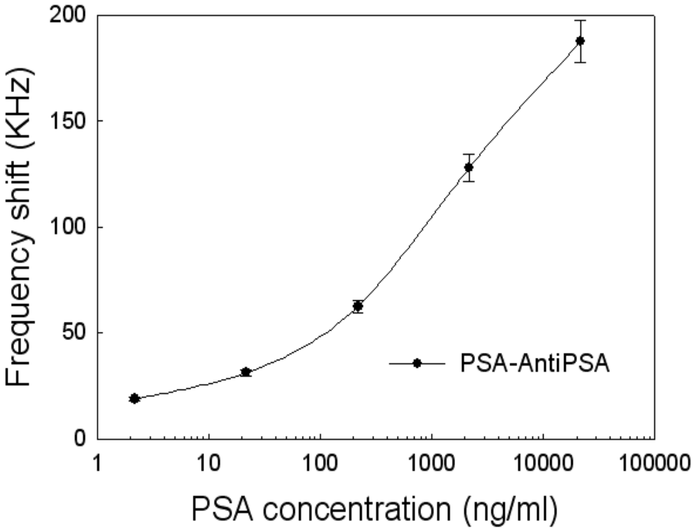
We begin by adding a layer of a monoclonal anti-PSA antibody in a phosphate solution- measure the res frequency and then add the PSA containing solution and measure again- the larger the shift in freq the higher the PSA amount

Biodetection Results by SAW Device









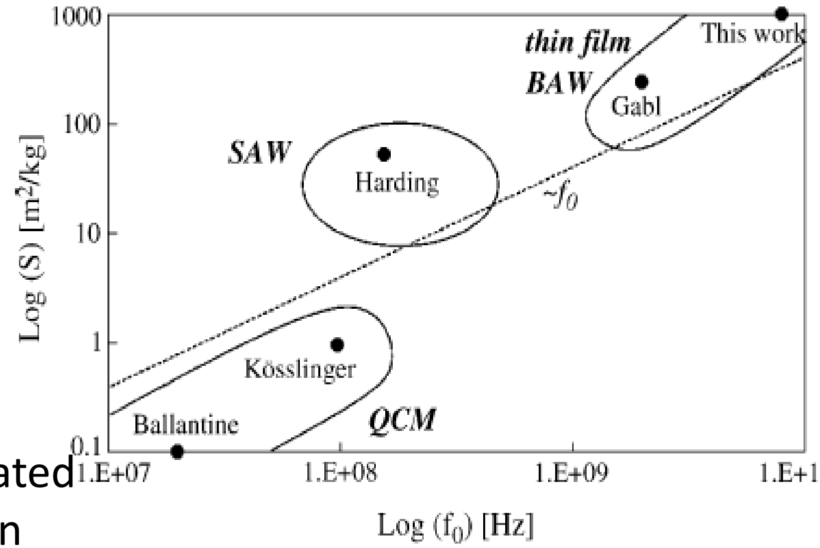
Lee et al. Int. Electron Device Meeting 2007, Washington, USA, 10, Dec. 2007.

Advantages of SAW-based Biosensor

- High sensitivity (high fr) QCM have a mass detection limit of a few nanograms, limited by low operation frequency (5 to 20 MHz) due to substrate thickness
- Low detection limitation (small base mass)
- Detection in liquid possible (Love-wave mode)
- Real time detection & monitoring
- Lab-on-chips (integration of microfluidic & electronics)
  - Micropump, micromixer & microchannel
- Multi-time usage
- Electronic signal, simple detection system
- However SAW device is big, not easy to scale down.
- The solution is to use a Thin Film Bulk Acoustic Resonator (FBAR) sensor

Advantages of FBAR Sensors

- Small dimensions
- Small base mass
- Very high sensitivity
- Low cost
- Electronics signal
- Parallel detection by array of FBARs
- Lab-on-a-chip detection system with integrated FBARs and microfluidics for multi-detection in parallel
- Possess all common advantages of label free biosensors



Comparison of sensitivity of acoustic wave devices

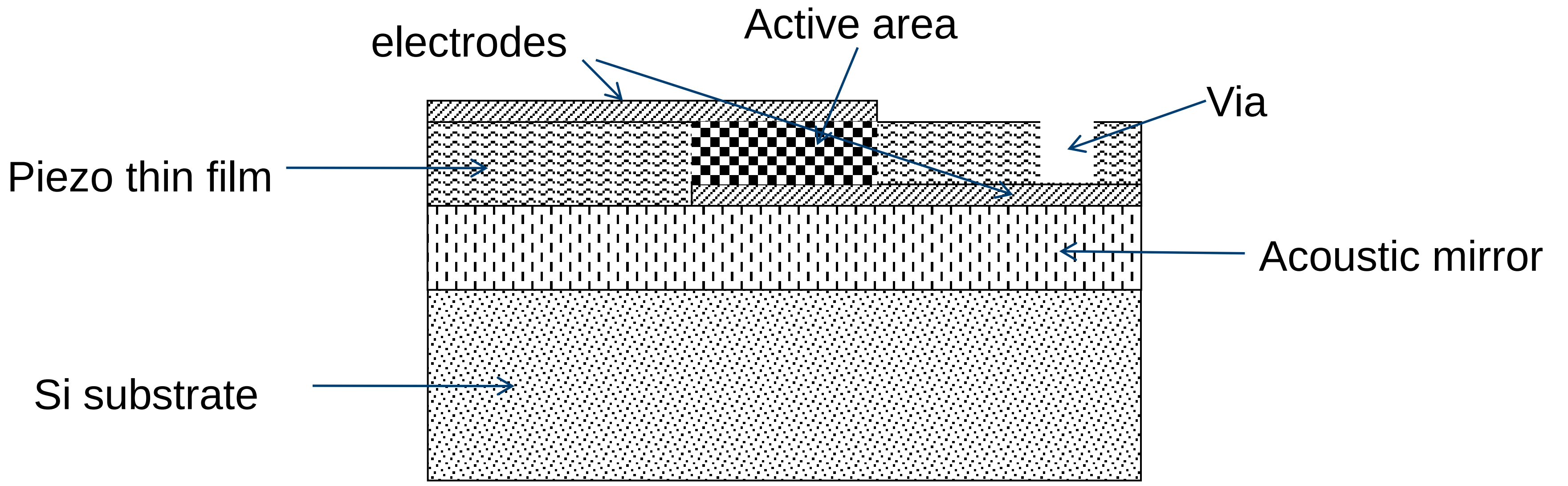
(Courtesy of S.R.Mermet)

Film Bulk Acoustic Resonators (FBARs)

- FBARs are a **nano-version of QCMs**
- **Thin films** of piezoelectric materials are utilised as the active material instead of Quartz
- FBARs resonate in the **GHz range** instead of MHz – so much **higher sensitivity** can be achieved
- Recent years have seen explosion of interest in the FBAR device as **potential replacement for the QCM**
- Allows for incorporation into **Lab-on-Chip, CMOS integration, microfluidics integration** and other associated technologies
- **Cost effective** photolithographic microfabrication

FBARs

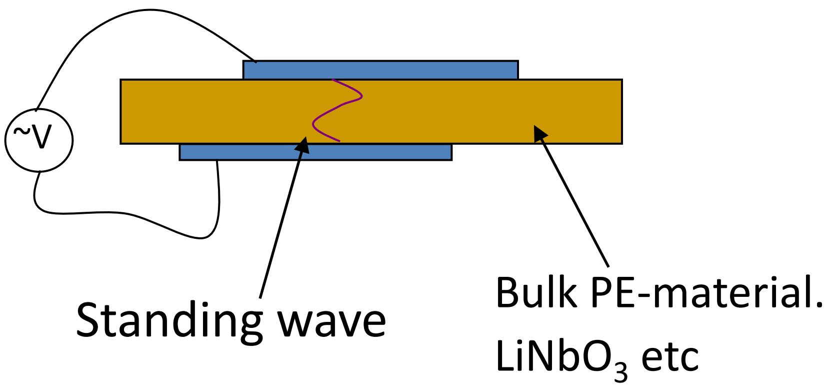
- Small size (in the order of μm^2). They can be used anywhere!
- Possibility of arrays for multi detection
- Possibility of wireless excitation



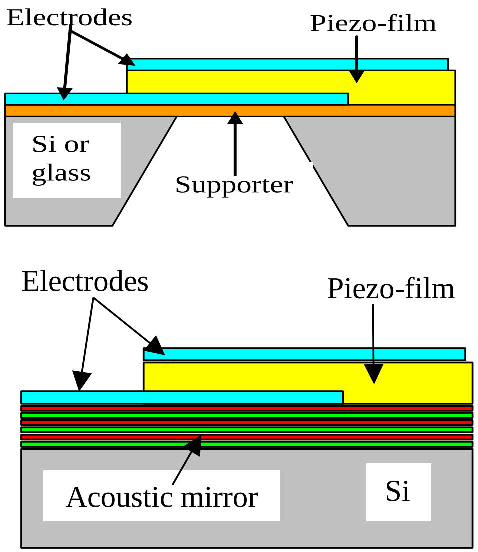
FBAR Biosensor Array

What is FBAR (Film bulk acoustic wave resonator)?

- Operating principle is same as the QCM, but the device dimensions are at least two orders of magnitude smaller
  - QCM: d~1cm, t~500 μm
  - FBAR: LxWxt ~ 100x100x2μm
- Extremely high sensitivity due to much reduced base mass and high resonant frequency → $10^{-(15\sim18)}$g

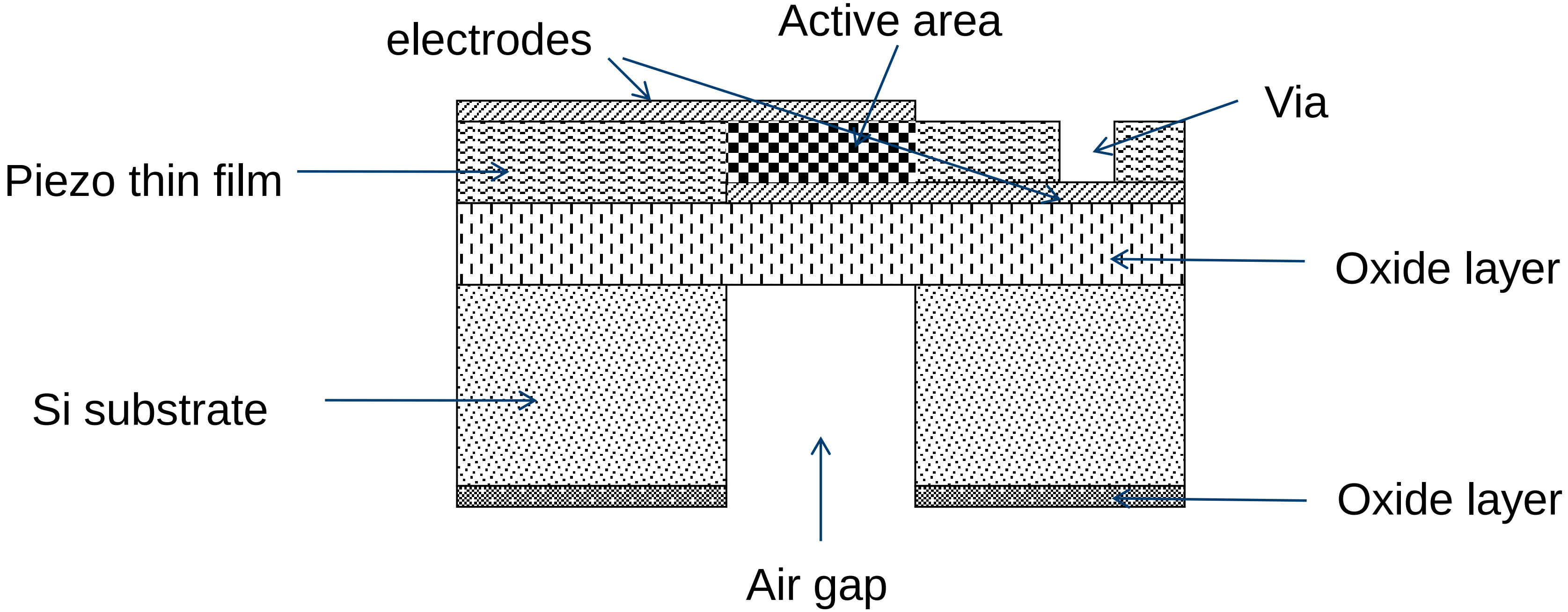


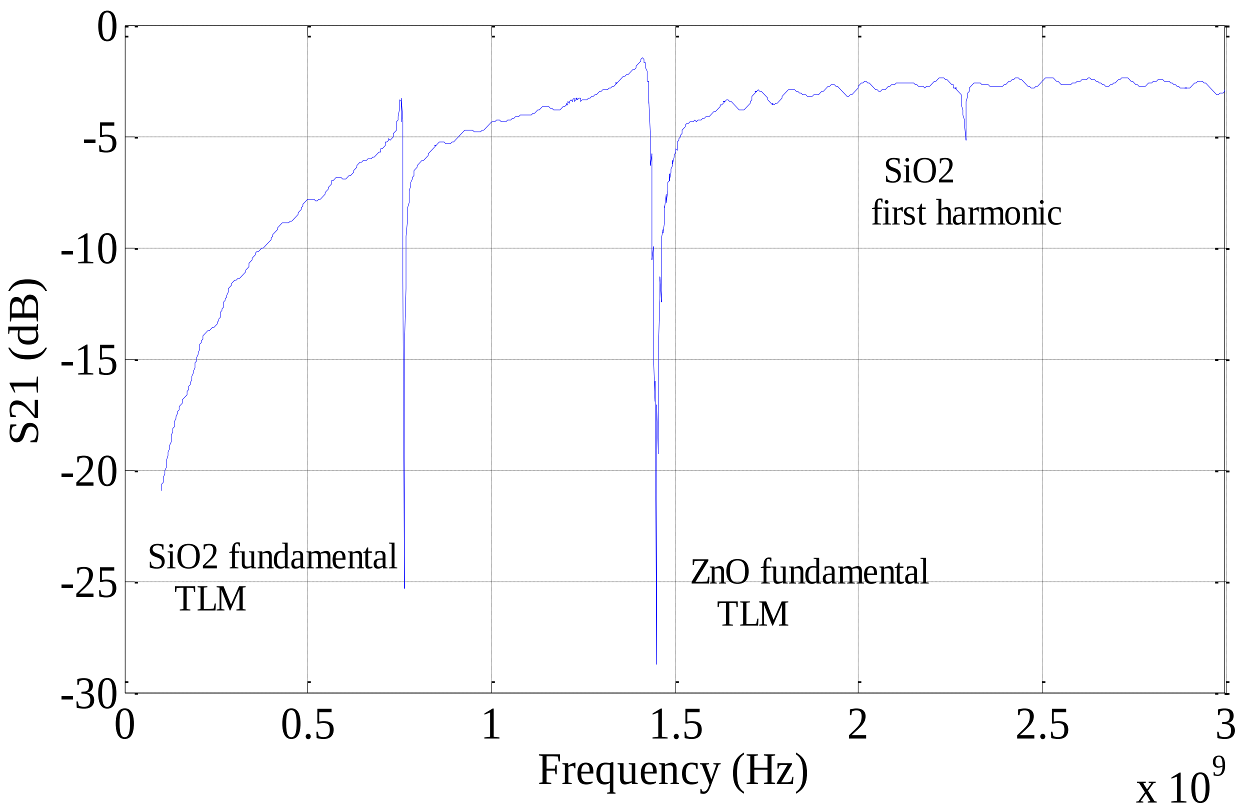
QCM



Two types of FBARs

- FBARs with free-standing membrane

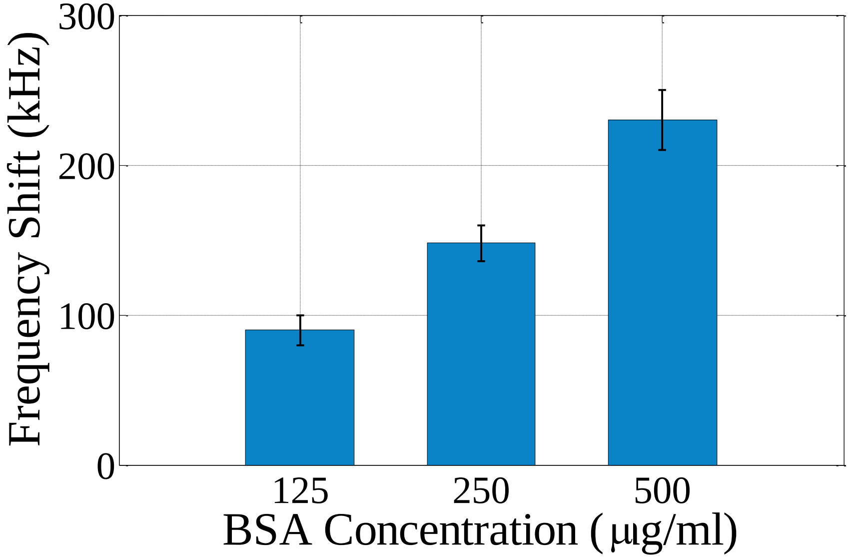




Quality factors higher than 1000 were achieved

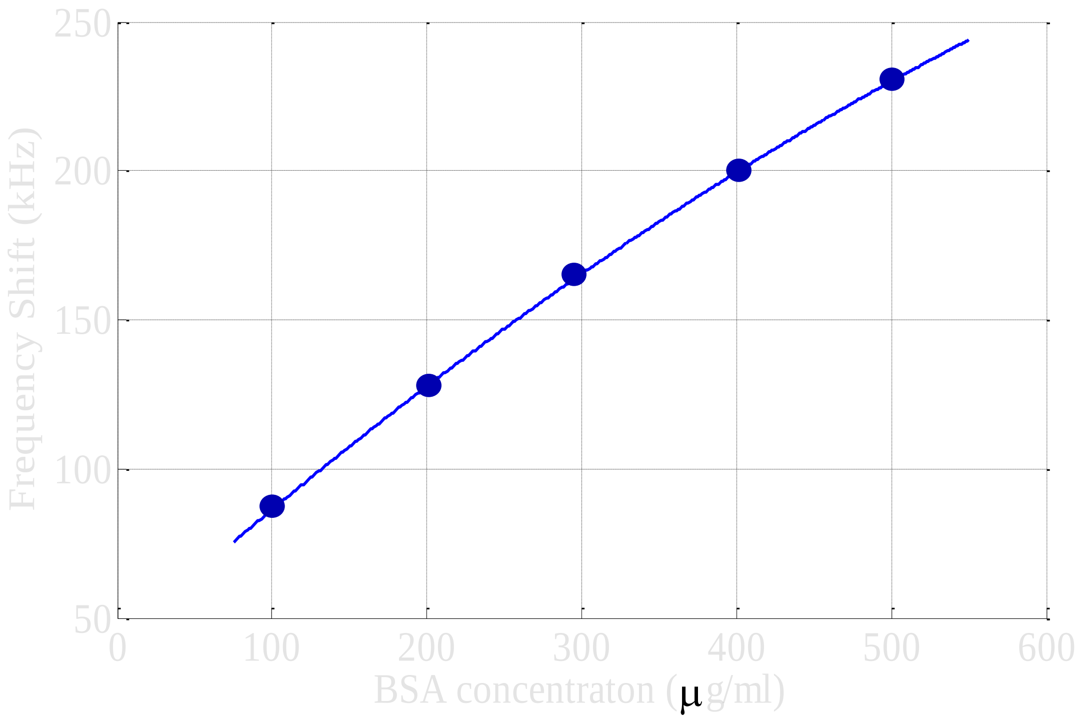
Protein detection through mass loading

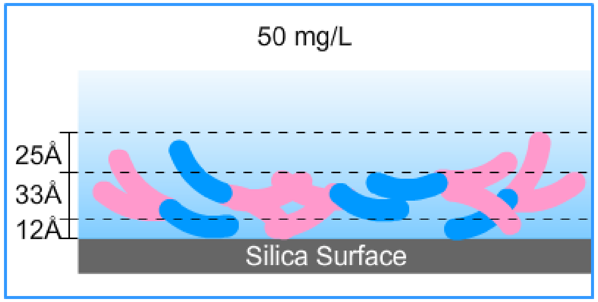
Bovine Serum Albumin (BSA) solutions with different concentrations were then placed on the top of different sets of identical FBARs and their responses to mass-load from physically adsorbed protein coatings were investigated.



Protein mass loading

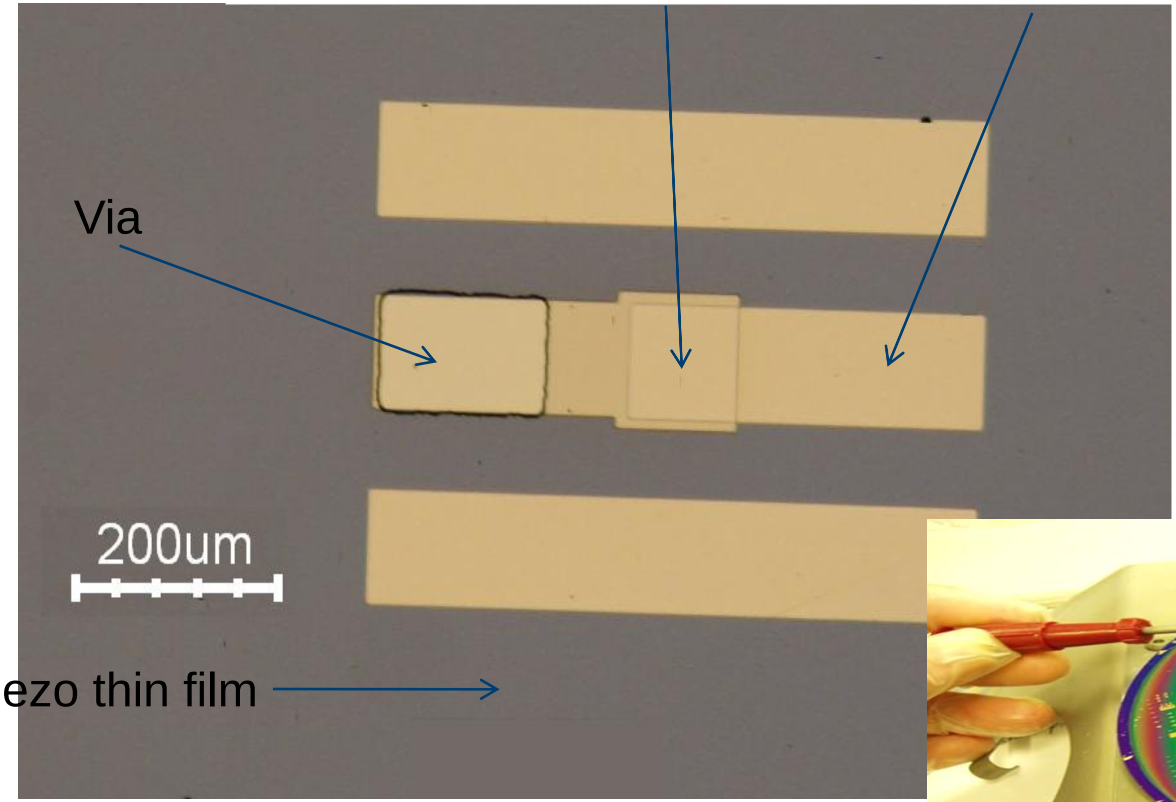
FBAR resonating at 1.5 GHz

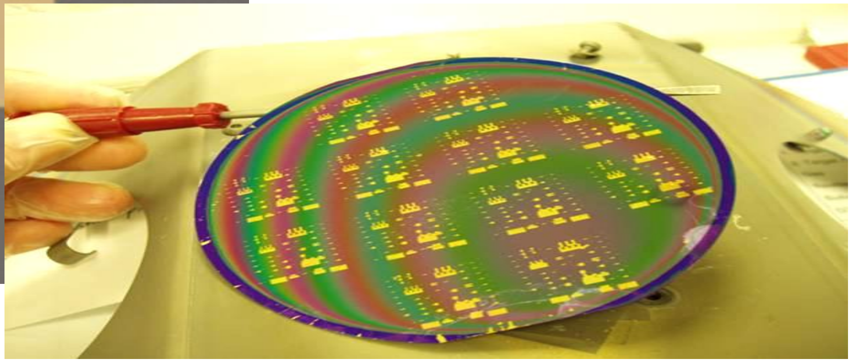




- Frequency changes were some three orders of magnitude greater than that of a QCM for a given BSA load
- FBAR has significantly better mass loading sensitivity!

FBARs fabricated

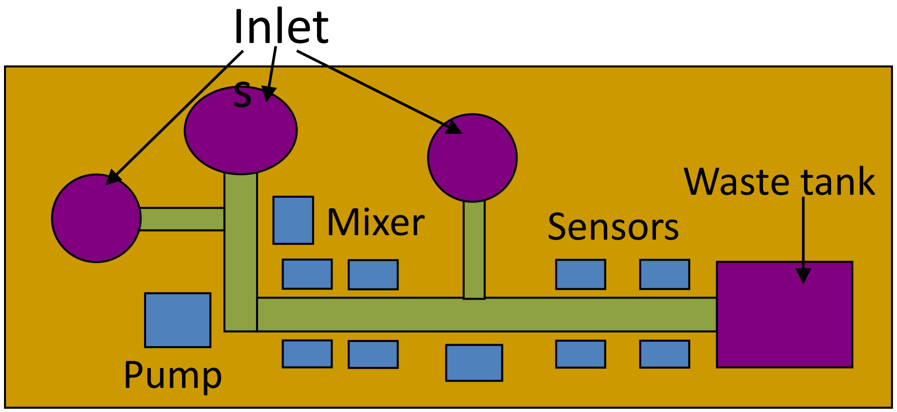


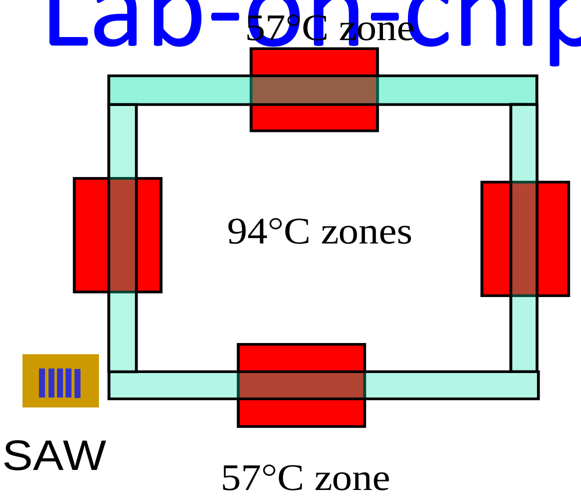


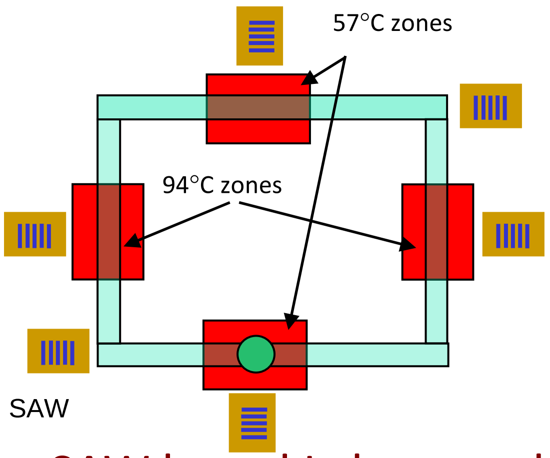
Lab-on-chip Bio-detection System

- SAW devices and FBAR array can all be made on a thin film piezoelectric substrate to form lab-on-chip biodetection system driven by SINGLE acoustic-wave-mechanism
- Surface acoustic wave can be used for pumping liquids and mixing reagents to maximise binding of target/probe molecules and minimize non-specific binding
- Surface acoustic wave can be used for remote-heating to speed up biochemical reactions
- SAW-sensors and FBAR array will be used for multi-detection in parallel for accurate diagnosis, detection, identification and monitoring of infectious agents
- SAW and FBARs will also be used for monitoring other physical properties such as temperature, humidity, UV-light exposure etc

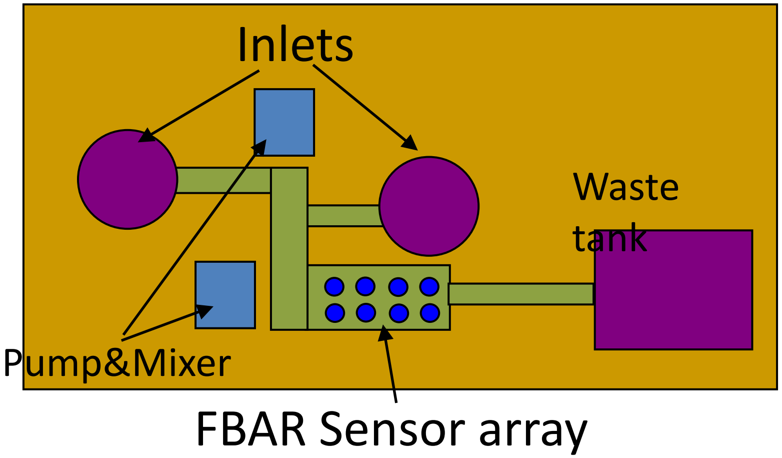
Acoustic Wave-based Lab-on-chips







SAW based Lab-on-a-chip



Acoustic wave-based lab-on-a-chip

Conclusions

- High quality, c-axis oriented ZnO thin films obtained on Si-substrates using an RF magnetron sputtering system.
- SAW devices with ZnO thin films fabricated
- Both Rayleigh & Sezawa mode waves obtained.
- Streaming/pump effects investigated as a function of signal amplitude, frequency, droplet size and surface chemical condition of the substrate
- Droplet streaming, mixing, pumping and ejections realized
- Biodetection demonstrated
- FBAR is extremely sensitive to most of physical parameters, and can be used for physical sensors
- FBARs have been used for biosensing with sensitivity improved by 3 orders of magnitudes compared to QCM.