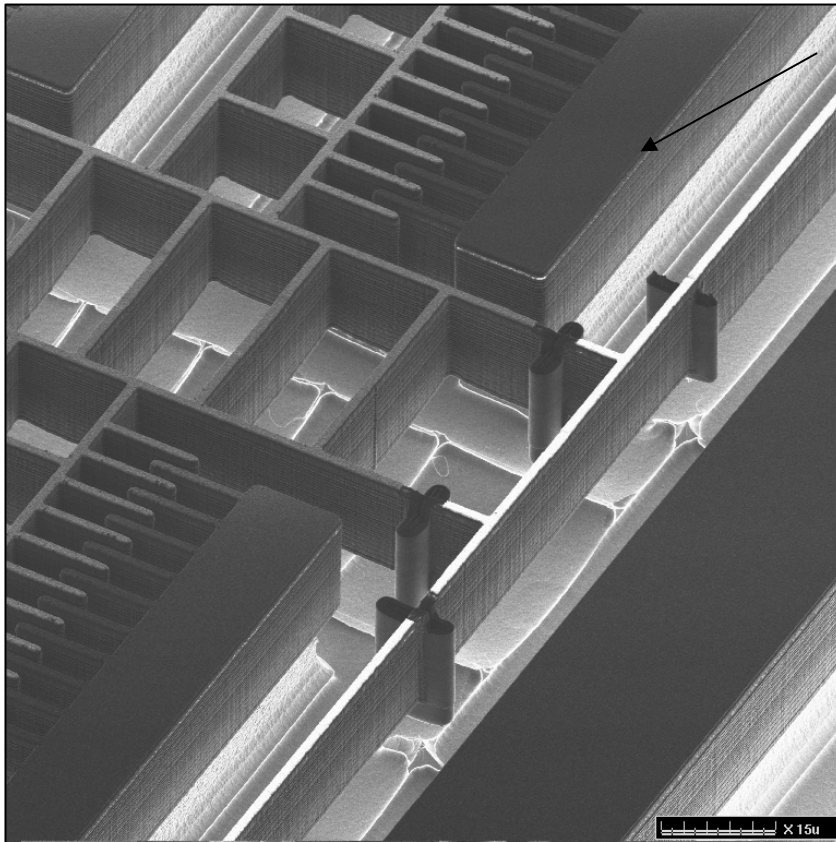

Bulk Titanium MEMS

Noel C. MacDonald
Fred Kavli Chair for MEMS
Professor Departments of
Mechanical Engineering, and Materials,
University of California,
Santa Barbara, CA

Outline

- **Brief Introduction: Silicon – based MEMS: Micro-Electro Mechanical Systems**
- **Introduction: Titanium- based MEMS: **Ti MEMS****
- **Ti Deep Etch Processes**
- **3D Ti MEMS: Multiple Wafers Gold – Gold bonding**
- **Ti MEMS Devices**
- **Nano – Structured Titania**
- **3D, Ti MEMS for Bio Chips: Dielectrophoresis**
 - **Molecular/ cellular Collection and Separation**
 - **Molecular-scale Mixing**
 - **Nm-scale molecular assembly**
- **Summary**

Si Comb Drive Actuator: SiO₂ Electrical Isolation



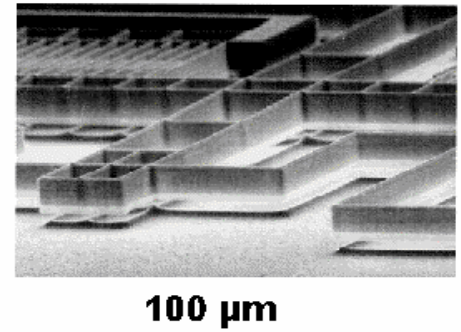
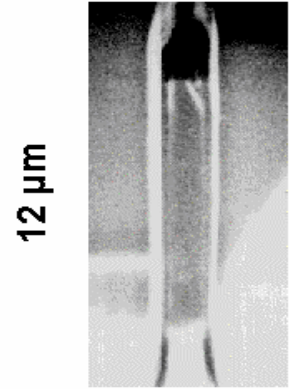
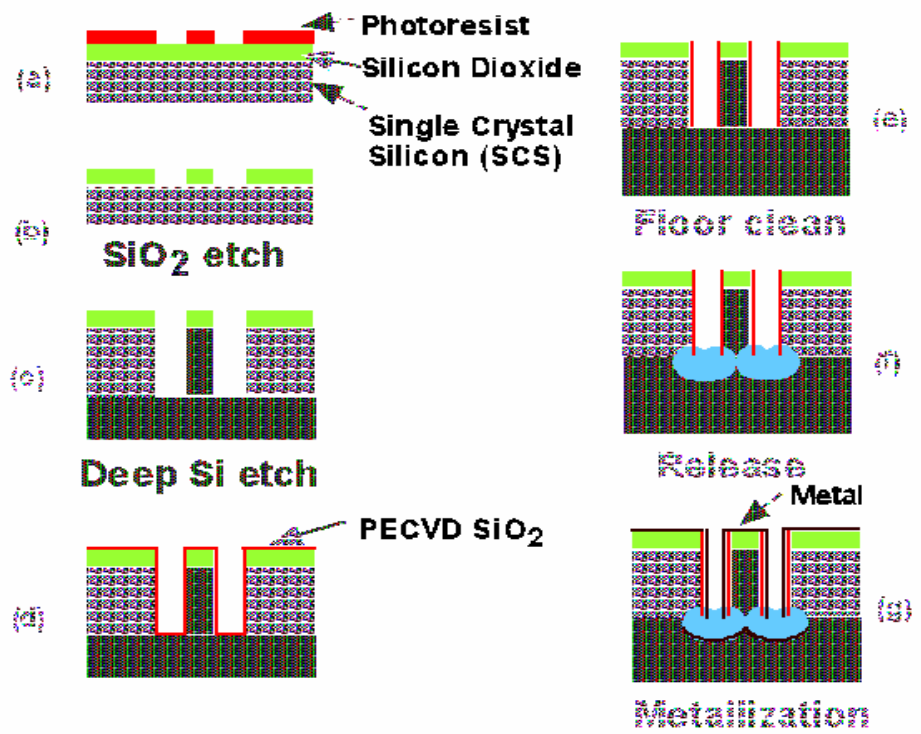
$$C_{\text{isolation}} = 350 \times 10^{-18} \text{ F}$$

$$V_{\text{breakdown}} > 400 \text{ V}$$

$$R_{\text{isolation}} = 10 \text{ P}\Omega @ 100 \text{ V}$$

SCREEN

(Single Crystal Reactive Etching And Metallization)



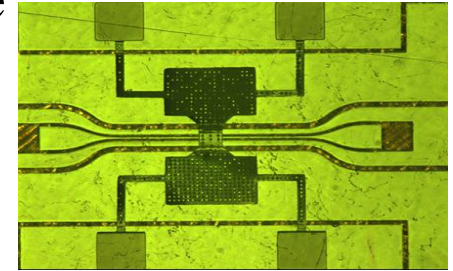
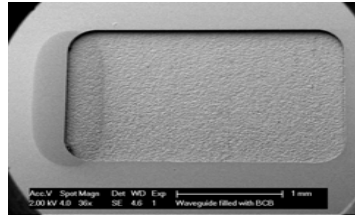
M. T. A. Saif and N. C. MacDonald

HERMIT: Bulk Titanium MEMS

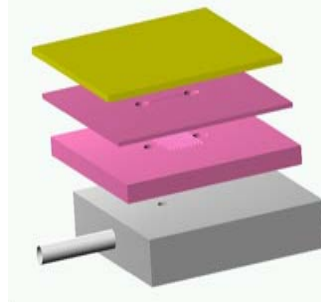
Introduction

- Ti Sheet Metal to Ti Polished wafers
- Deep Etch Processes for High-Aspect Ratio Ti MEMS
- 3 D Integration: Ti/Au - based Bonding
- Wafer-scale Packaging
- Nano-structured TiO_2

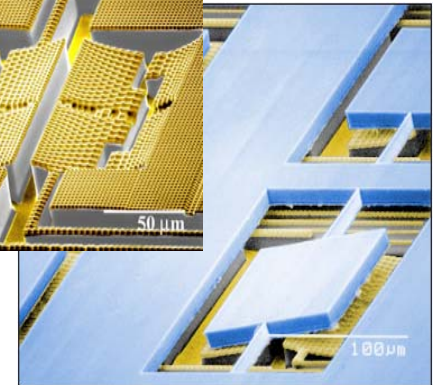
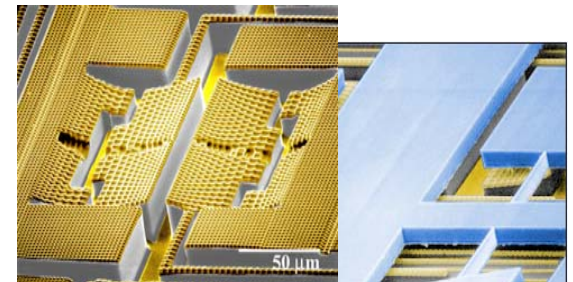
Titanium Waveguide/relay & Wafer –scale package



Ti/Au -Bonding



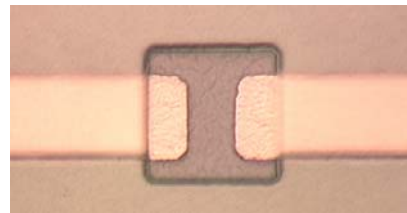
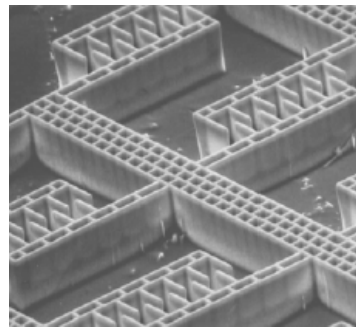
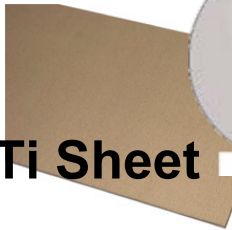
Ti Micro-mirror Array



Ti Wafer



Ti Sheet



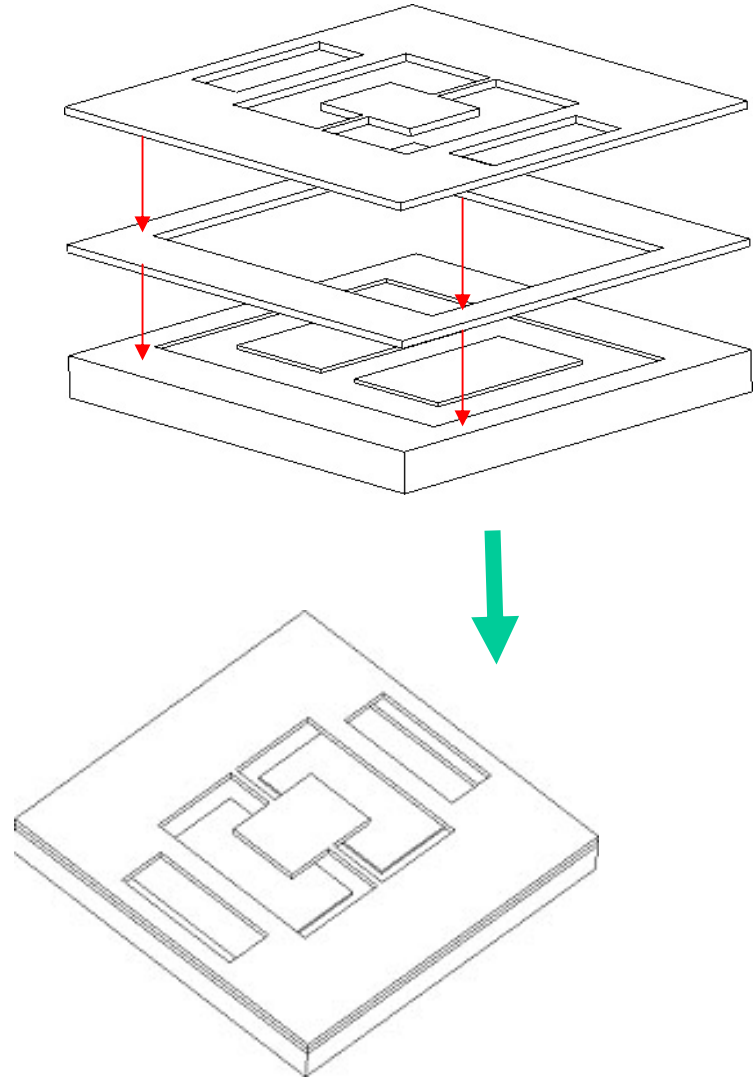
Nano-Structured TiO_2 H_2 Gas Sensor

Titanium MEMS Key Attributes

- Miniaturization
- Released, Moving and Fixed Microstructures
- Ti Fracture Toughness 60X Silicon
- Sensors, Actuators, 3D Microstructures
- Micro-Fluidics/ Bio-Chips
- Batch Fabrication
- Microelectronic Integration
- Use of Silicon and Titanium Infrastructures
- Wafer Scale Packaging

3D Ti MEMS

- **Through-etch titanium wafer**
- **Deposit gold on mating surfaces**
- **Bond Wafers**
- **Typical Wafer Thickness**
25 μm – 1 mm.



Titanium as a structural material

- **Ti is corrosion resistant and bio compatible**
- **Ti may be forged or wrought by standard techniques**
- **Ti can be cast; investment casting preferred method.**
- **Ti may be processed using power metallurgy.**
- **Ti can be joined by fusion welding, brazing, adhesives, diffusion bonding and fasteners.**
- **Titanium is formable and readily machined.**
- **Titanium is available in a wide variety of types and forms.**

Titanium: a technical guide Matthew J. Donachie Jr. 2nd ed. 2000

MACRO-Machining Titanium

- Cincinnati ARROW Series VMC-500 Accuracy
 - Positioning (X,Y) +/- 3 microns
 - Positioning (Z) +/- 4 microns
 - Repeatability +/- 1 micron
 - Dynamic Contouring +/- 15 microns

www.cinmach.com

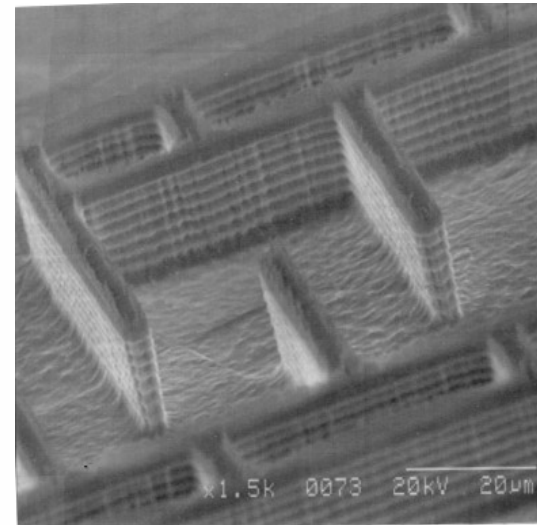
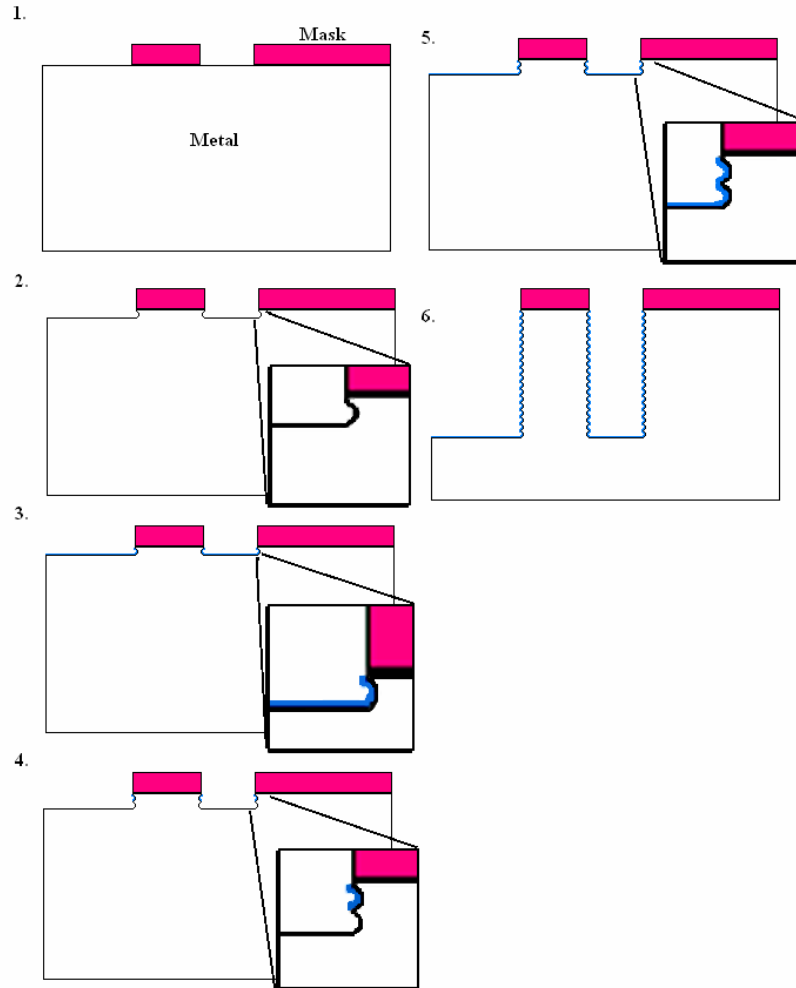
- Can be machined using similar techniques as 316 stainless steel.

Micromachining

Bulk titanium etching for MEMS

- High aspect ratio Etch
- High etch rates
- Release Method
 - Undercut
 - Through Wafer Etch
- Compatible with semiconductor processes and Equipment
- Etch Tool is AMAT Centura Platform

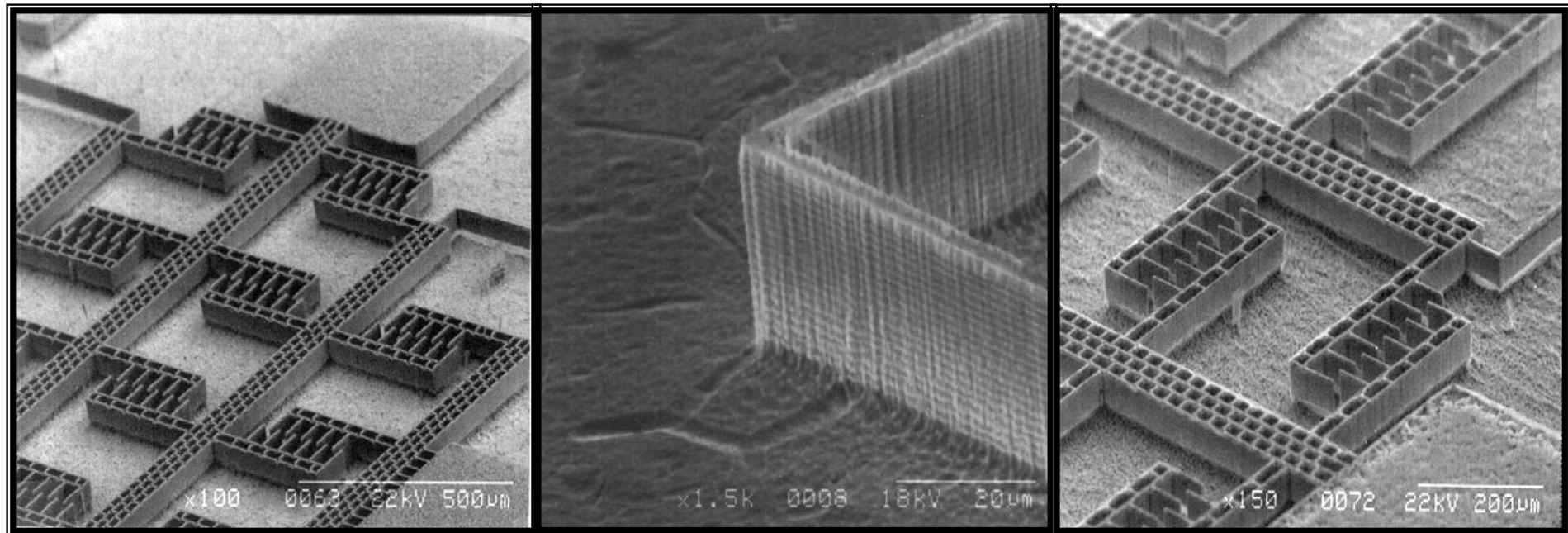
Metal Anisotropic Reactive Ion etching with Oxidation (MARIO)



Nature Materials, 3, pp. 103-105, Feb. 2004.

Titanium Deep Etch

MARIO Process

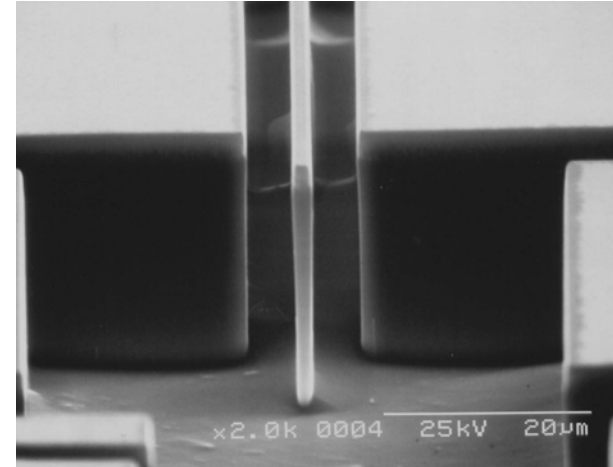
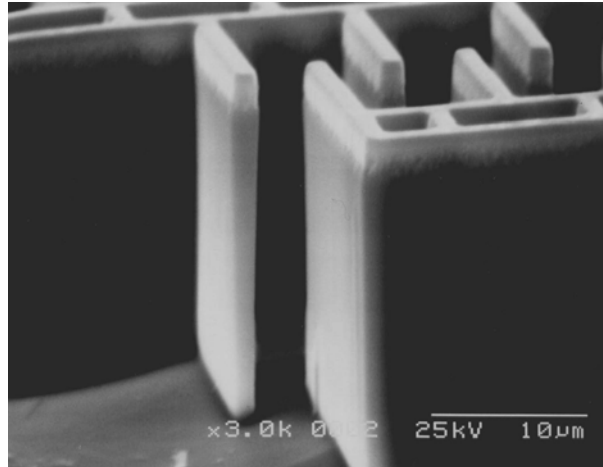
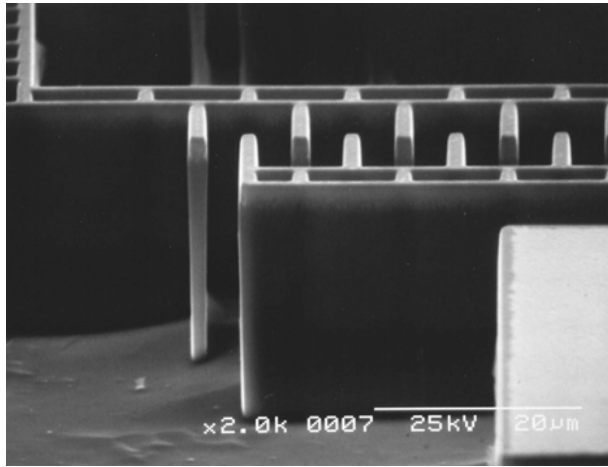


Deep etched comb drive

Deep etched 5 micron line

Deep etched comb drive

Titanium ICP Deep Etch

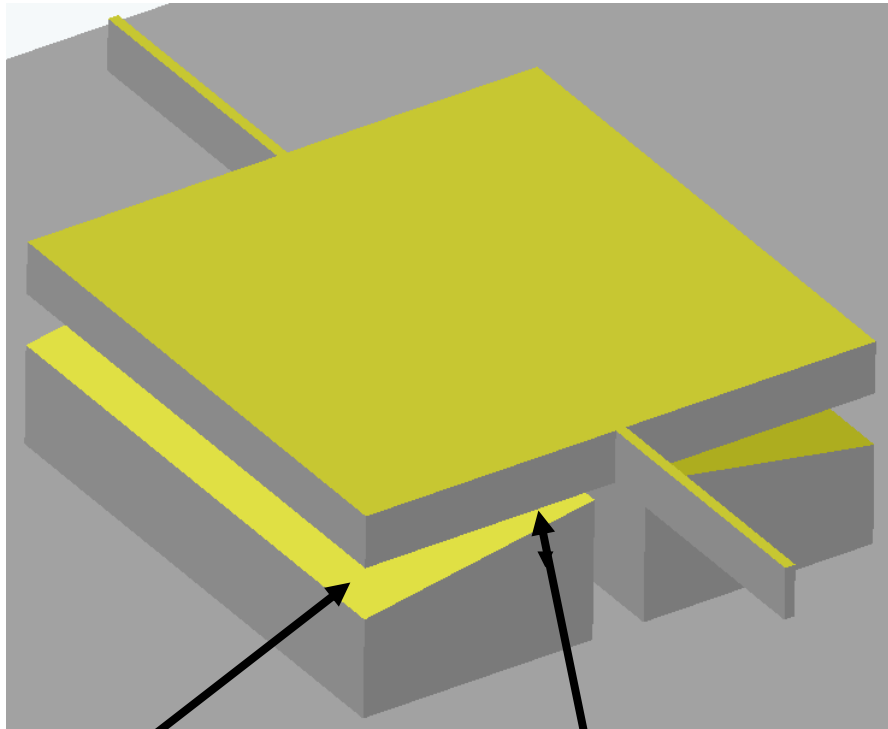


TIDE Process Considerations:

- Optimized parameter set: ICP Source = 400 W, RF (Bias) 150.
- Pressure = 2 Pa;
- Chlorine = 100 sccm, Argon = 5 sccm.
- Etch rates > 2.0 μm/min.
- Selectivity of 40:1 (Ti:TiO₂).
- Non-cyclic etch: exceptionally smooth sidewalls.

Sloping Electrode Driven Micromirrors

Sloping Electrode Micromirror



Rationale:

Reduced gap near rotational axis increases capacitive force without sacrificing tilt angle or switching speed

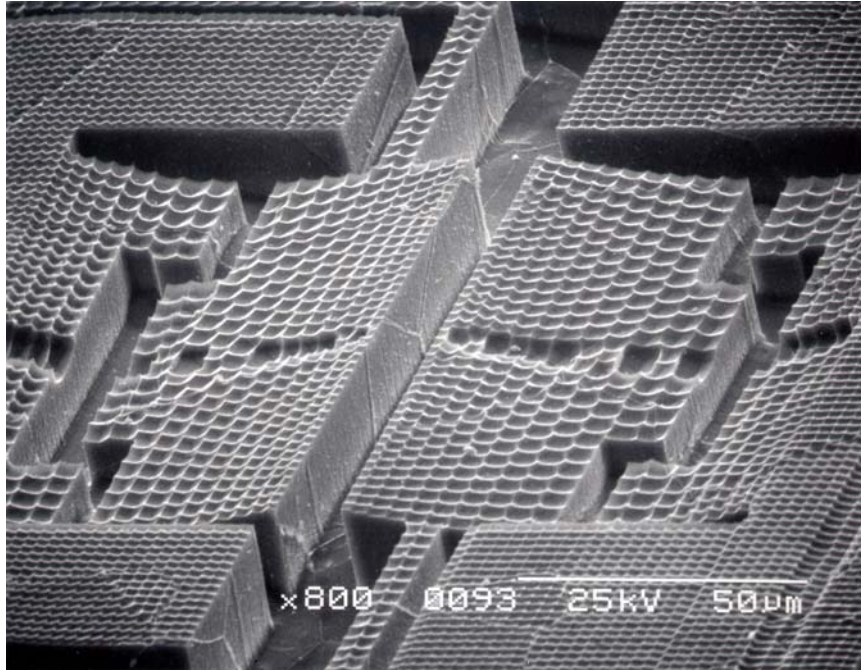
Advantage:

Maintains simplicity of parallel plate design without significant design modification,

Small GAP Large Force at Lower Voltage
Large GAP Large Angular Displacement at Lower Voltage

Applied Physics Letter, Dec. 2005

Fabrication: Titanium Sloping Electrodes



Bulk Ti Sloping Electrodes

*Single Mask 3-D
Micromachining Process*



Optical Profilometric Scan

~8° electrode slope

Bonded Electrode / Micromirror Array



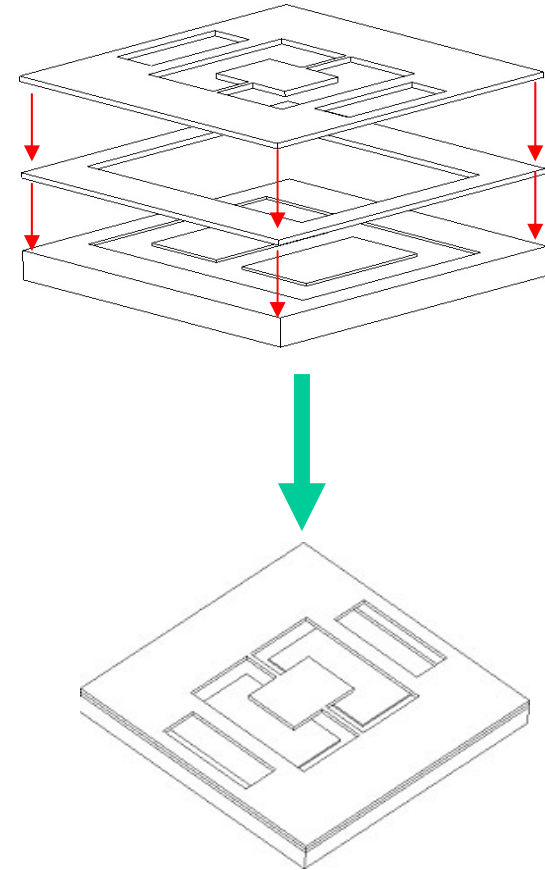
Actuated Mirror (snapped down)



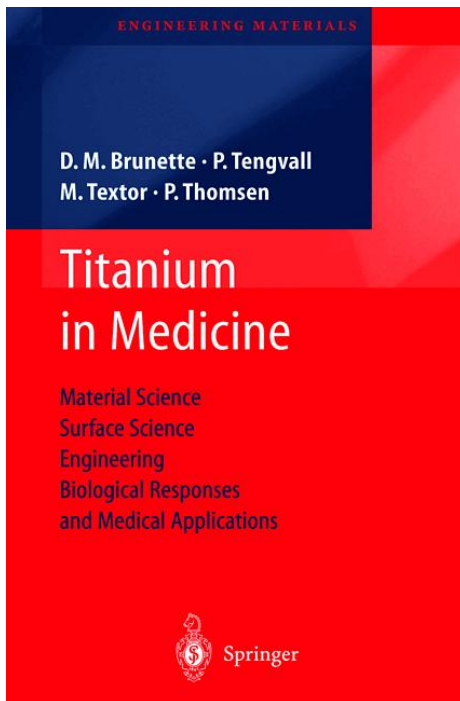
Interconnects on Lower Wafer

Motivation: Why Titanium?

1. Excellent biocompatibility
2. High Fracture toughness (60 times that of silicon)
3. 3-D structures formed by stacking of Ti foils
4. Micromachining AND Macromachining



Bio response

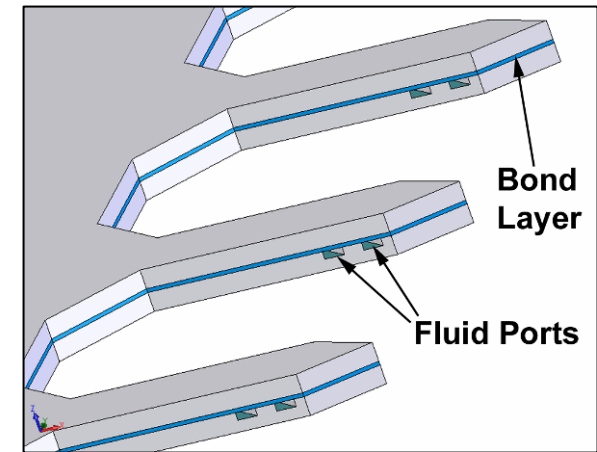
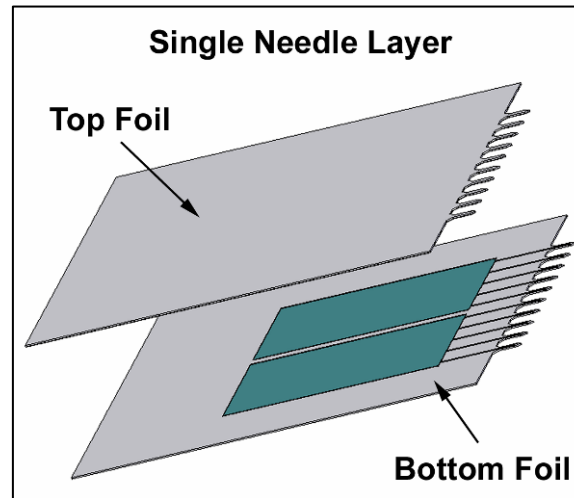
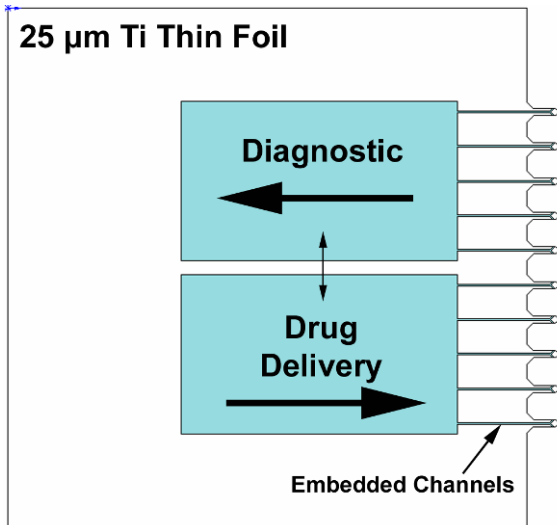


Oxide	Dielectric constant	Solubility at pH 7 [mol/L]	Typical tissue response
TiO ₂	86-170	3x10 ⁻⁶	Inertness
Al ₂ O ₃	9.3-11.5	10 ⁻⁶	Sequestration
V ₂ O ₅	13.8	>1	Toxicity
V ₂ O ₄	13.8	~10 ⁻⁴	Toxicity
ZrO ₂	12.5	<10 ⁻⁶	Inertness
Ta ₂ O ₅	24-65	~10 ⁻⁵	Inertness
Fe ₂ O ₃	14.2	<10 ⁻¹⁰	Sequestration
Cr ₂ O ₃	11.9-13.3	~10 ⁻¹¹	Toxicity
Co ₂ O ₃	12.9	~10 ⁻¹²	Toxicity

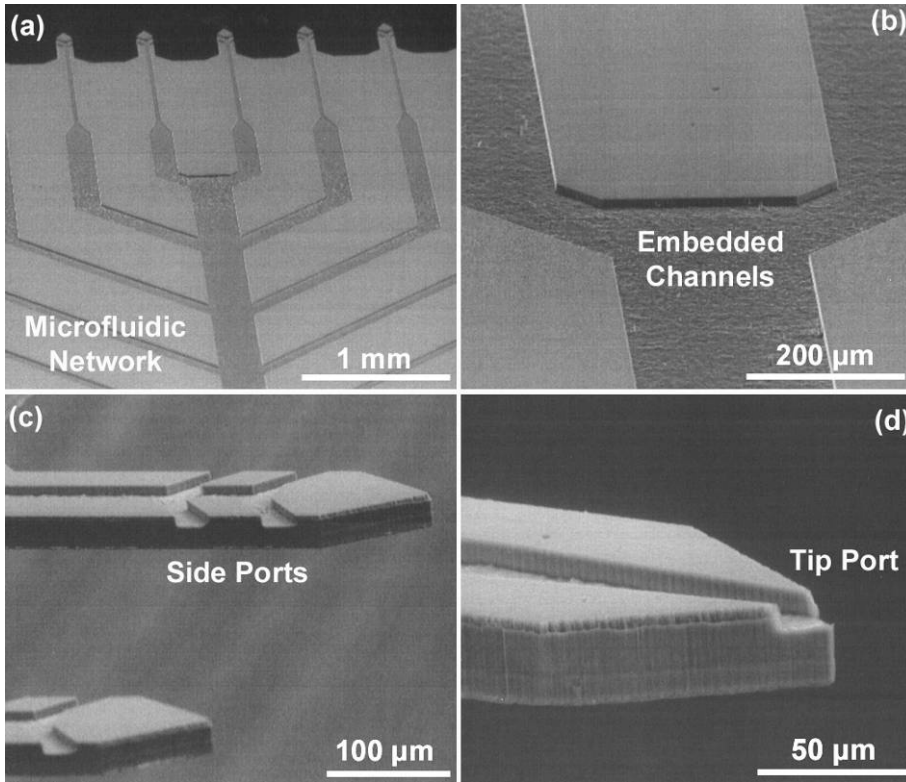
Bulk Titanium Microneedles

Design Concept:

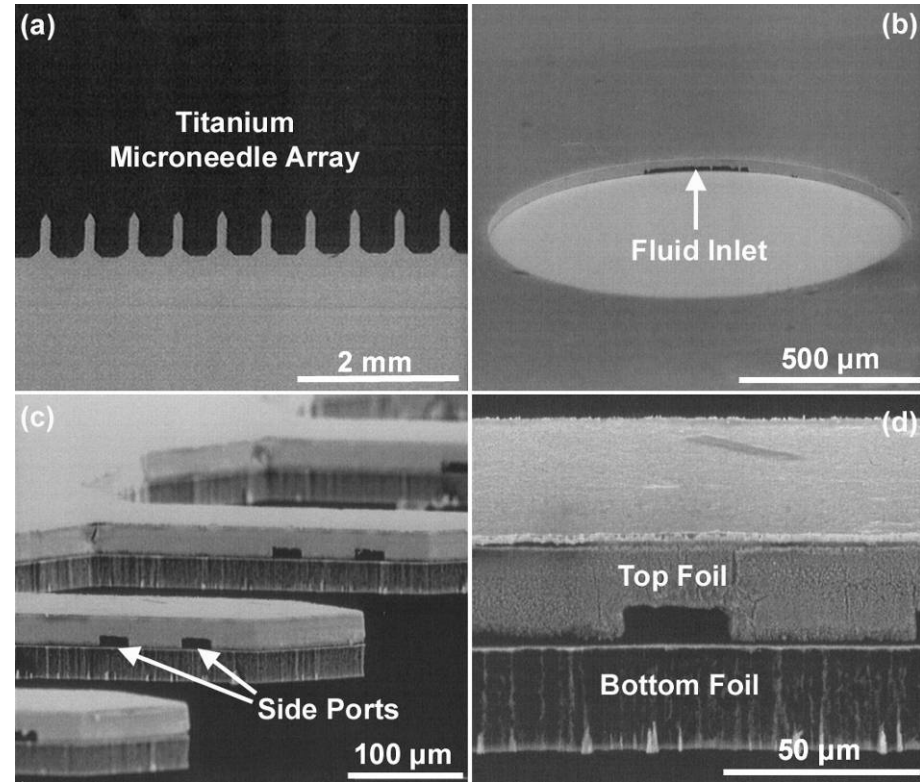
- Utilize titanium thin foil multilayer lamination technology
- Eliminate fracture induced failure
- Improve material biocompatibility
- Simplify fabrication
- Integrate drug delivery and diagnostic capabilities on single titanium chip



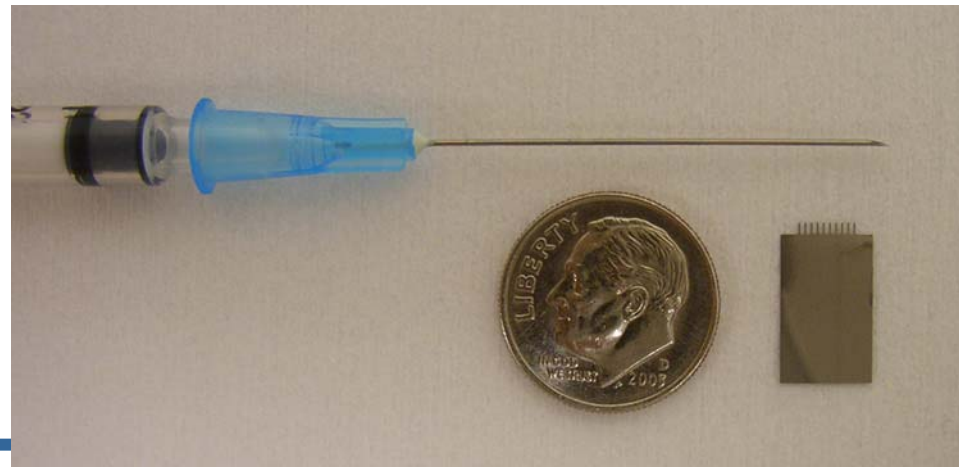
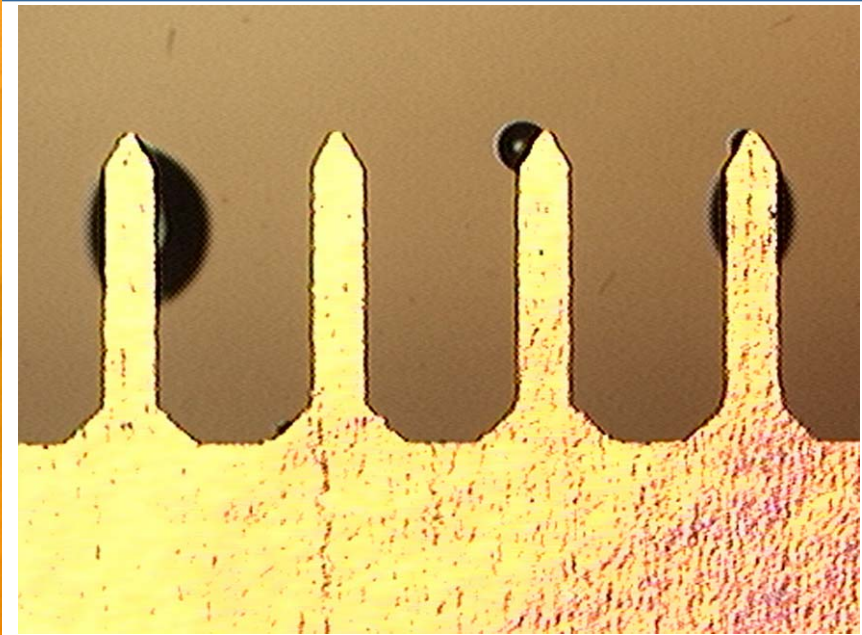
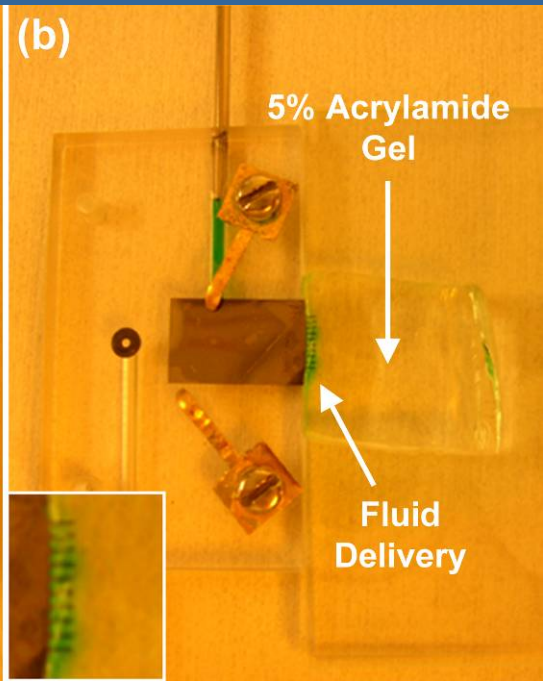
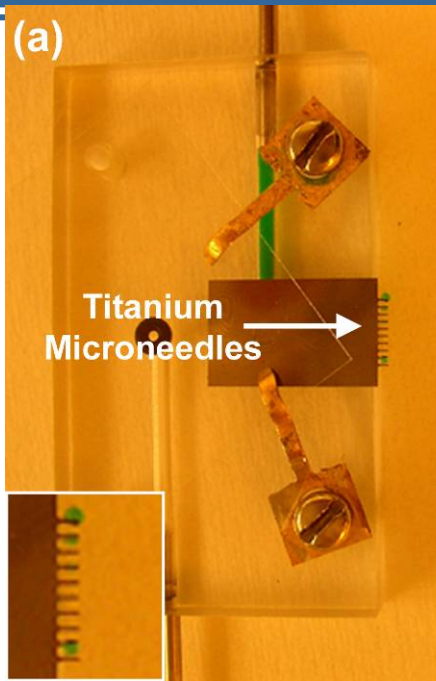
Embedded Microfluidics



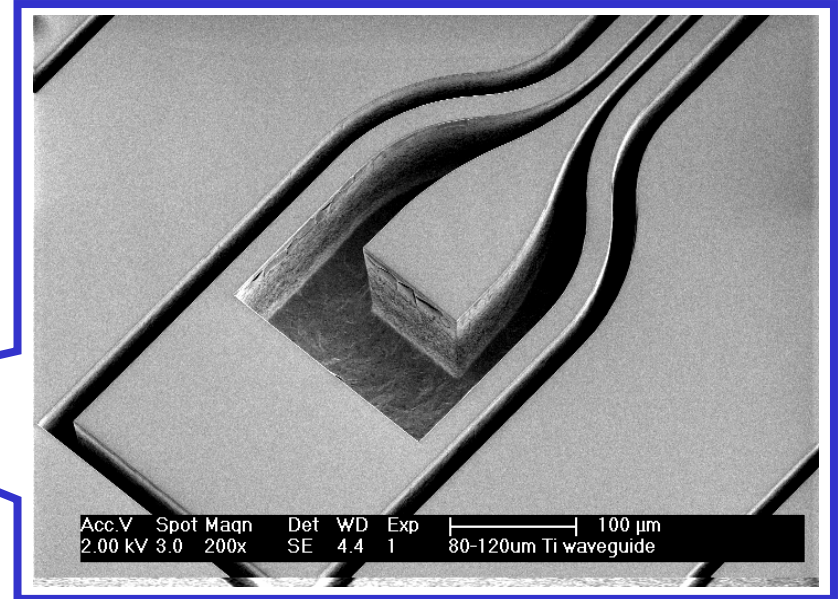
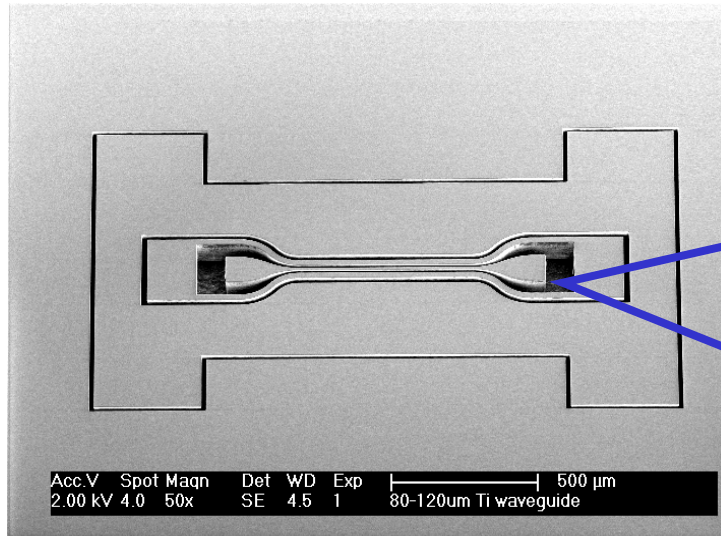
Bilayer Microneedles



Titanium Microneedle Device

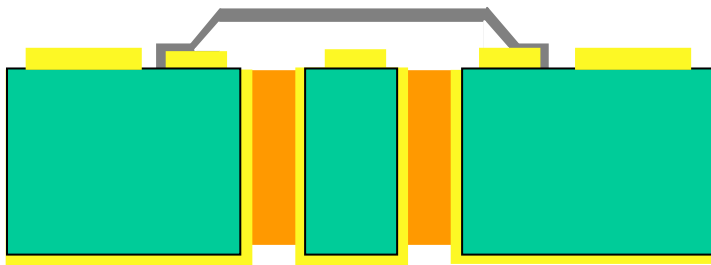
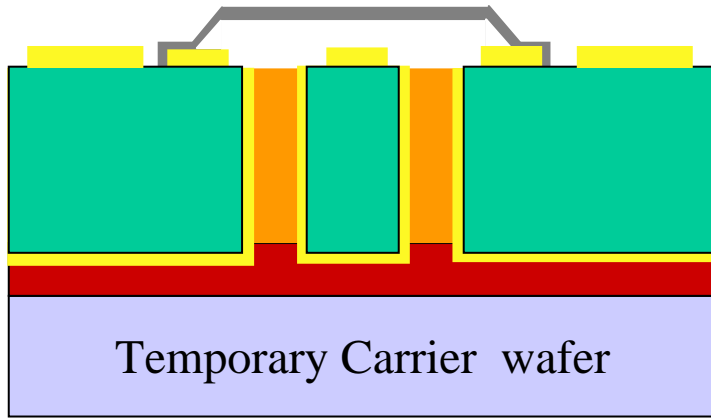


High aspect ratio Ti Waveguide etching

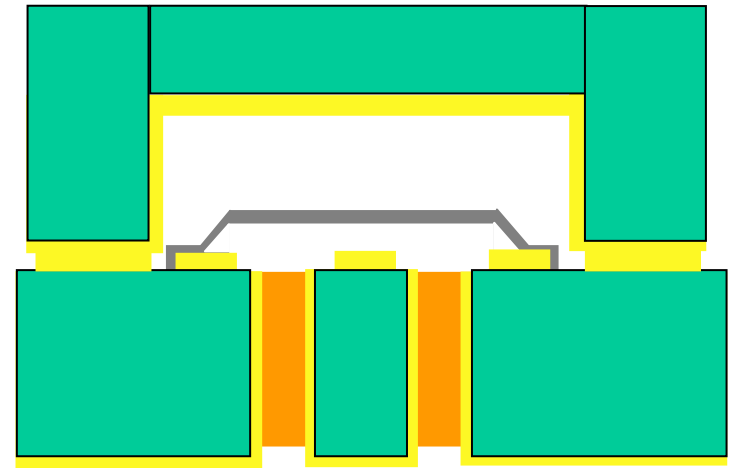


- High-aspect-ratio, ‘out-of-plane waveguide’
- Panasonic ICP etching of Ti using Cl_2 and Ar.
- Mask: $3\mu\text{m}$ SiO_2 deposited at 250°C .
- Etch rate for open areas $> 180\mu\text{m} / 50 \text{ min} = 3.1\mu\text{m} / \text{min}$.
- Selectivity is about 90:1: etch $180 \mu\text{m}$ of Ti with $2\mu\text{m}$ SiO_2 mask.

Relay with Wafer-scale Package



1. Handle wafer/adhesive removal



2. Package wafer fabrication and bonding

Ti

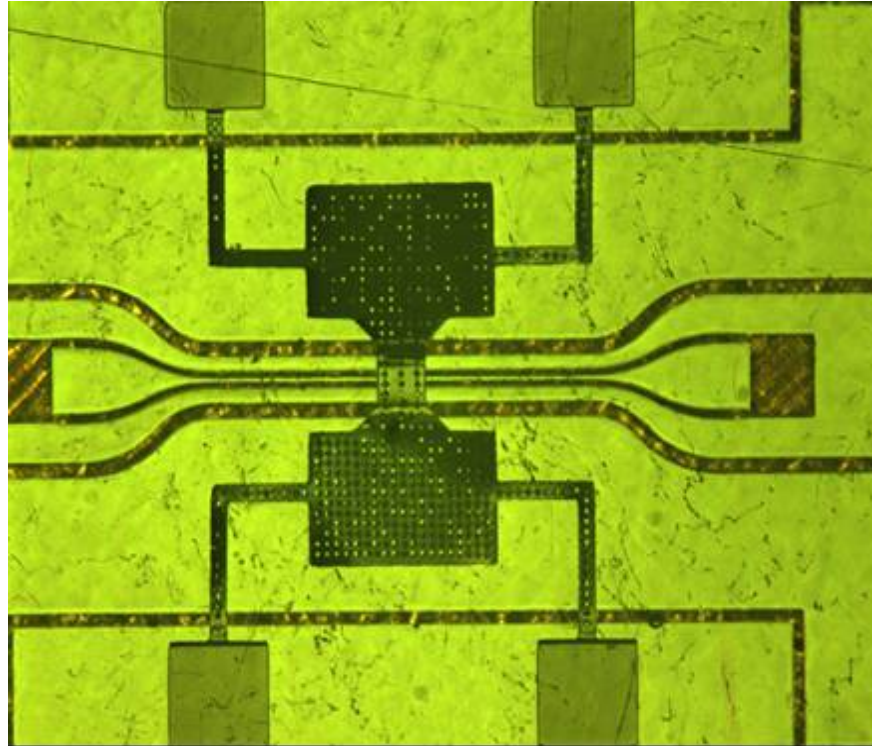
Au

BCB

Alumina

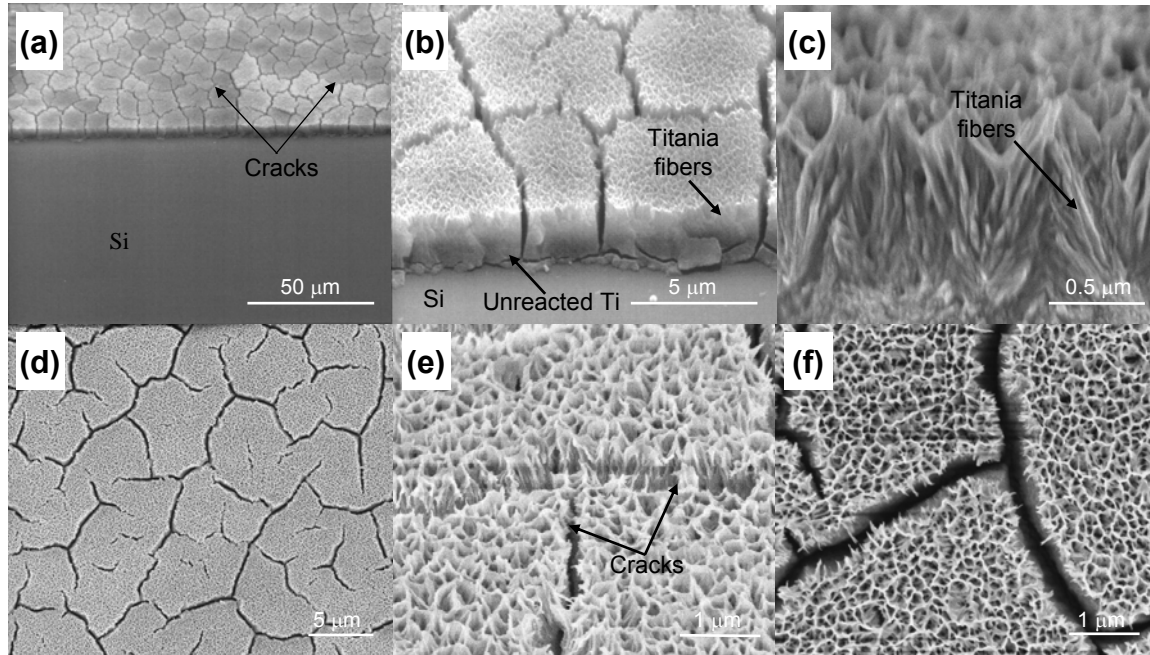
CrystalBond

Surface switch on bulk waveguide



- Crab-Leg, surface Ti switch. Double layered Ti - 0.3 μm (compressive) sputtered Ti and 0.8 μm (tensile) evaporated Ti.
- Waveguide is 40 μm thick. Both sides of the waveguide are lapped and Polished (CMP).

Nano-structured Titania on Ti

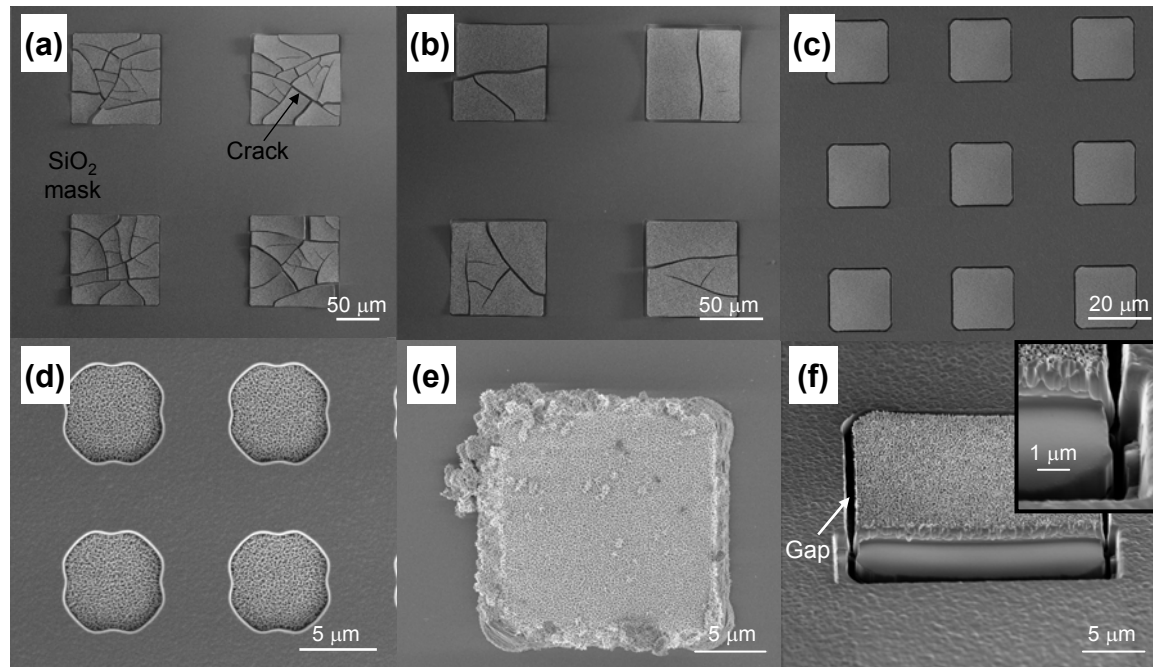


Cracks in titania layers formed on blanket Ti films.

- **a-c**, Aged sputtered Ti film showing **a**, high crack density; **b**, cracks propagating through film; **c**, nanofibers with diameters about 50 – 90 nm.
- **d-f** Aged evaporated Ti film showing

Advanced Functional Materials, March 2005

Nano-structured Titania on Ti



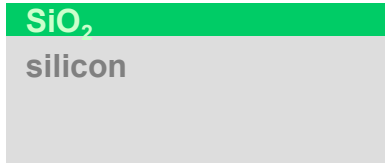
Crack reduction and elimination in titania layers formed on patterned Ti films:

- **a-d and f**, Ti pads formed by selective masking technique
- **a**, 100 μm; **b**, 70 μm; **c**, 20 μm ;
- **e**, 15 μm Ti pad formed by lift-off technique.

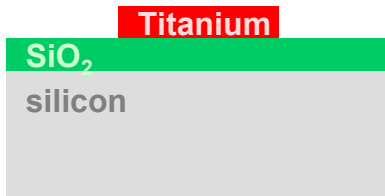
Advanced Functional Materials, March 2005

Arrayed Thin Film NST Gas Sensor

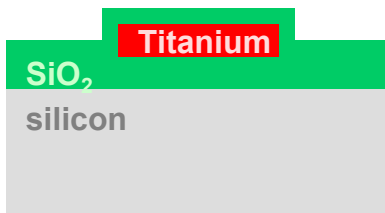
Wafer Scale Processing



SiO₂ Si Wafer



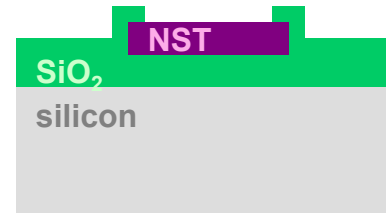
E-beam Ti (500nm)
Patterned via liftoff



PECVD SiO₂



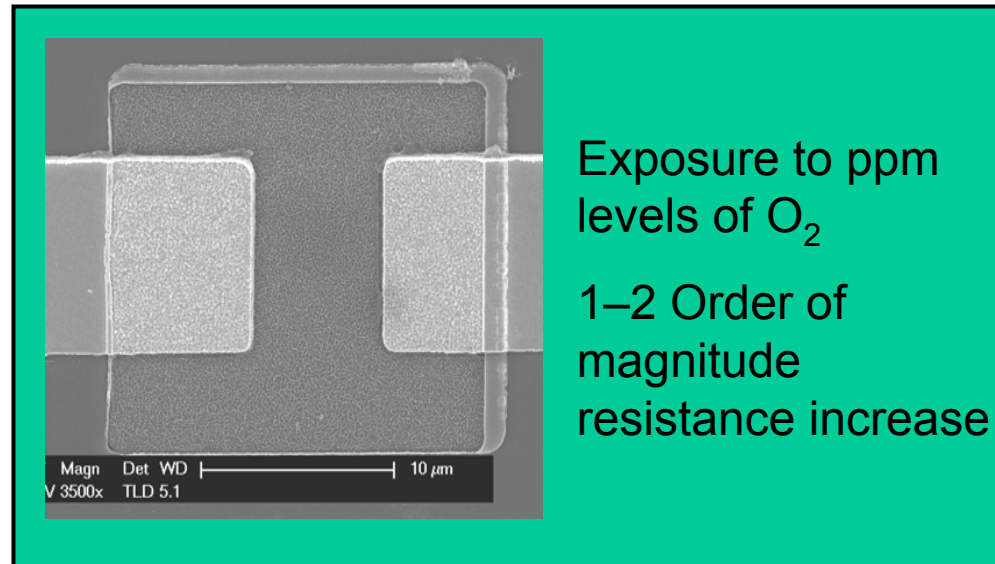
SiO₂ Patterned
and Dry etched



Oxidized in H₂O₂ to
create Nano-
Structured-Titania



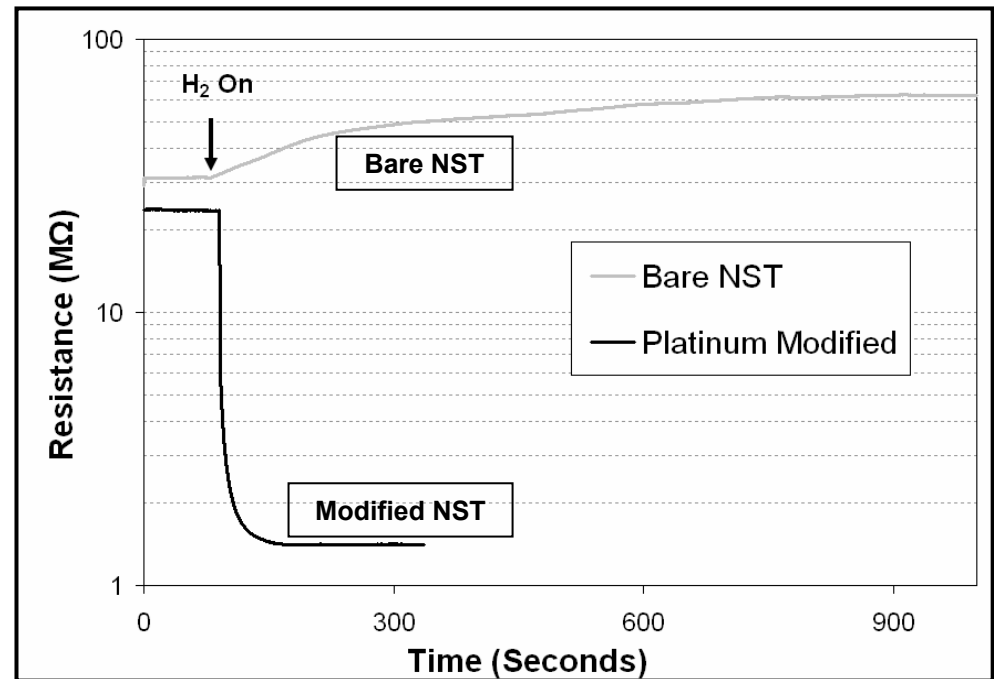
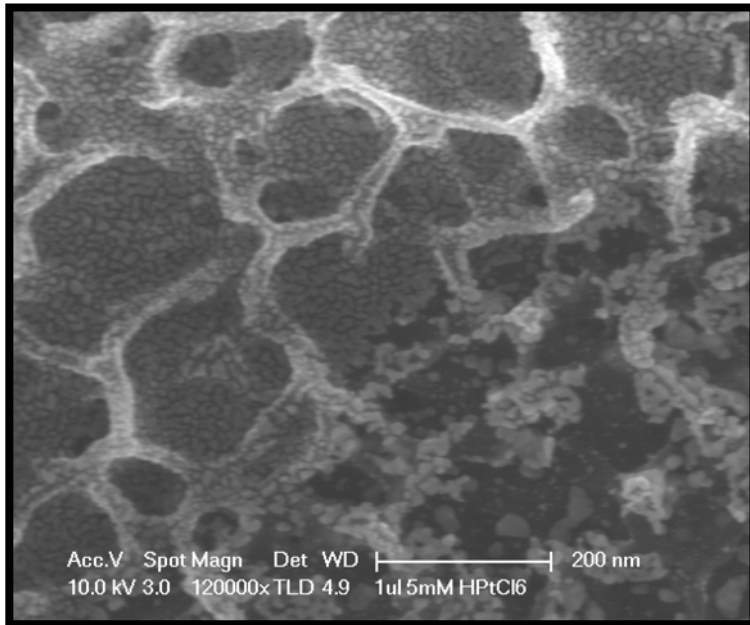
E-beam Au
electrodes



NST Hydrogen Sensor

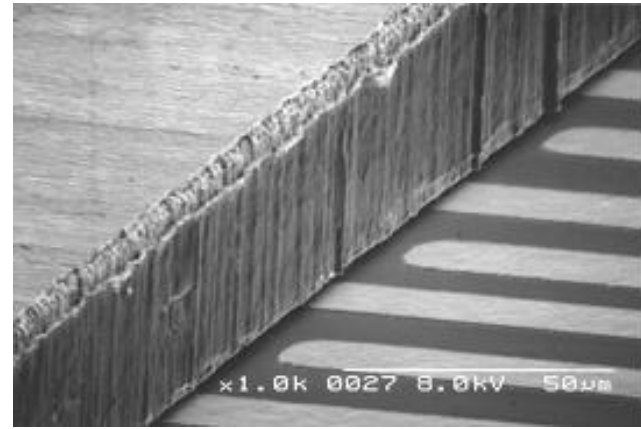
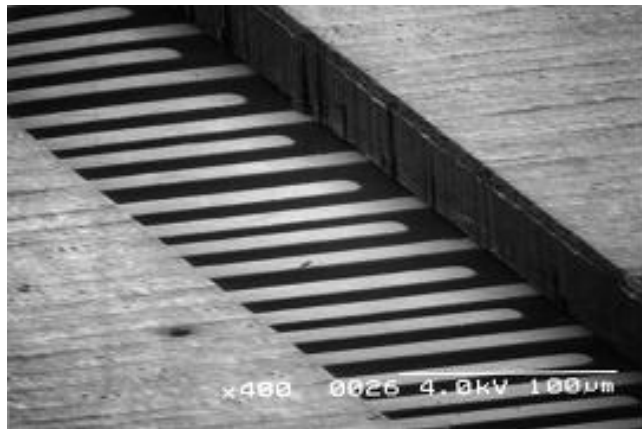
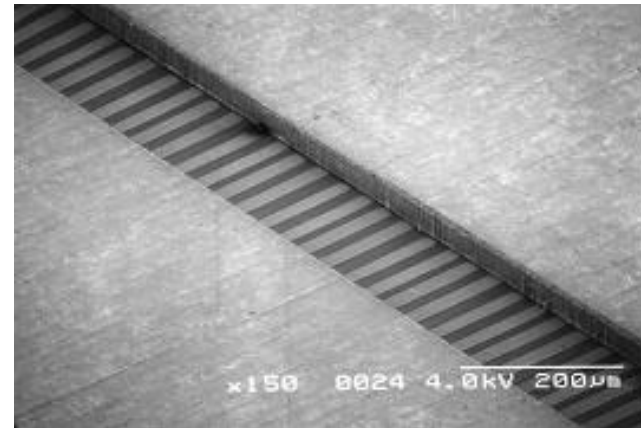
- NST Gas sensor functionalized with platinum
- Thermal decomposition of platinum salt on NST
- High sensitivity–fast response H₂ sensor

Platinum Nanoparticles on NST



Investigating additional functionalization

Ti Dielectrophoresis Device



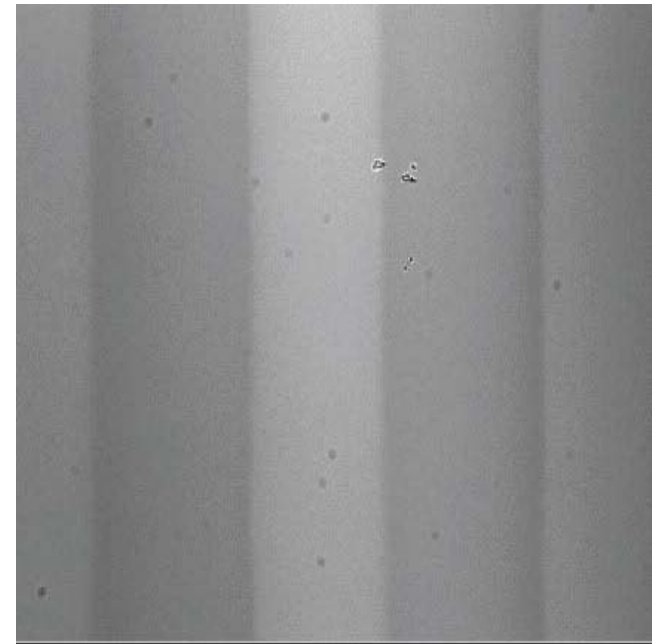
Low magnification view of the reservoir and channel with 24 electrodes located at the bottom of the channel

3D,Ti MEMS for Bio Chips: Dielectrophoresis

- **Molecular/ Cellular Collection**
- **Molecular/ Cellular Separation**
- **Molecular-scale Mixing**
- **Molecular Assembly**
- **Nm-scale Assembly**

70 nm Polystyrene Spheres are Collected on Ti Electrodes When Two 10 Volt Peak to Peak Voltages with Two Characteristic Frequencies are Applied to the Ti Electrodes.

Movie



Summary: Bulk Titanium MEMS

- Introduced a New, **SHOCK RESISTANT**, Wafer-Scale Titanium MEMS Process
- 3D MEMS, BioMEMS,
- Wafer Scale Packaging: “System-on-a-Ti-Chip”
- Process Development
 - Polished wafers
 - Deep Etching Process for High-Aspect Ratio Bulk Ti MEMS
 - 3D Integration through Ti-based Bonding Techniques
 - High Density Through Wafer Interconnects
 - Integrated process for Nano-structured TiO₂
- Devices: Mirror Array, Waveguide/Relay, Solar Cell, H₂ and O₂ Sensors Dielectrophoresis Chip

Ti MEMS Support

We Acknowledge the support
from DARPA/MTO, NSF and
The Kavli Chair in MEMS

Properties Table

	SC Silicon	Poly Silicon	SiO2	Si3N4	SiC	TEOS
Elastic Modulus (Gpa)	63-170	134	90	300	410	74
Density (kg/m3)	2329	2330	2200	3440	3160	2150
Resistivity (ohm m)			1.00E+16		1.00E+02	1.00E+16
Specific modulus (E/p)	7.30E+07	5.75E+07	4.09E+07	8.72E+07	1.30E+08	3.44E+07
Acoustic Velocity (m/sec)	8.54E+03	7.58E+03	6.40E+03	9.34E+03	1.14E+04	5.87E+03
Thermal Expansion (K-1)	2.60E-06		5.00E-07	8.00E-07	3.30E-06	5.00E-07
Thermal Cond. (W/m K)	124	29	1.4	29	120	0.9
Dielectric Constant	11.9	11.8	3.9	8	9.7	4.3
Melting Temperature (C)	1414	1414	1722	1900	2830	1722
Hardness Mohs (knoop)	7		6.5		9.3 (2500)	
Fracture Toughness (Mpa m ^{1/2})	0.8		0.95	6	3.5	
Structure	Diamond	Amorphous	Quartz	Amorphous	Wurtzite	Amorphous
	Ti	TiO2	TiB2	TiC	TiN	AlN
Elastic Modulus (Gpa)	108	282	400	100-500	600	394
Density (kg/m3)	4506	4230	4380	4900	5210	3200
Resistivity (ohm m)	3.90E-07	0.1	9.00E+04	0.005	2.05E-07	
Specific modulus (E/p)	2.40E+07	6.67E+07	9.13E+07	6.12E+07	1.15E+08	1.23E+08
Acoustic Velocity (E/p) ^{1/2}	4.90E+03	8.16E+03	9.56E+03	7.82E+03	1.07E+04	1.11E+04
Thermal Expansion (K-1)	8.60E-06	7.50E-06	5.60E-06	6.40E-06	6.30E-06	4.15E-06
Thermal Cond. (W/m K)	21.9	6.7	25	25	29	280
Dielectric Constant		86-170				4.7
Melting Temperature (C)	1668	1830	3225	3140	2950	3000
Hardness Mohs (knoop)	5.7	6.2	(2850)	(2470)	9 (1770)	(1225)
Fracture Toughness (Mpa m ^{1/2})	50	5	4	3	5	3
Structure	Hexagonal	Rutile	Hexagonal	Cubic	Cubic	Wurtzite

Thermal Expansion

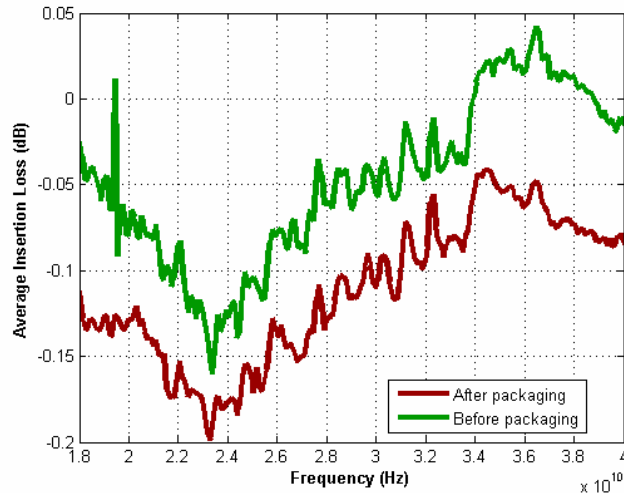
Fracture Toughness

Dielectric Constant

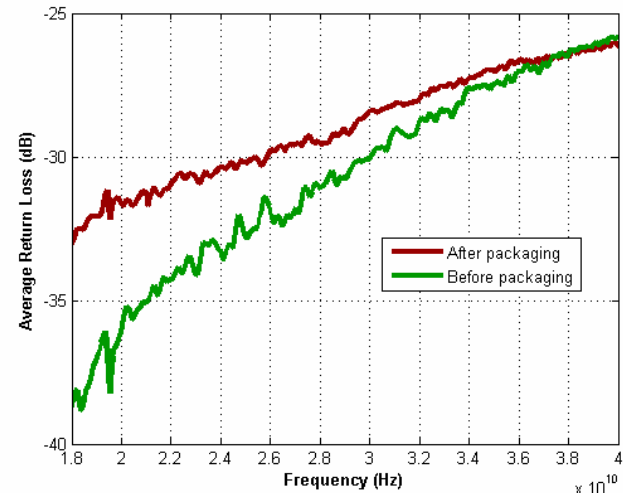
Hardness

Acoustic Velocity

Microwave Measurement Results



(A)



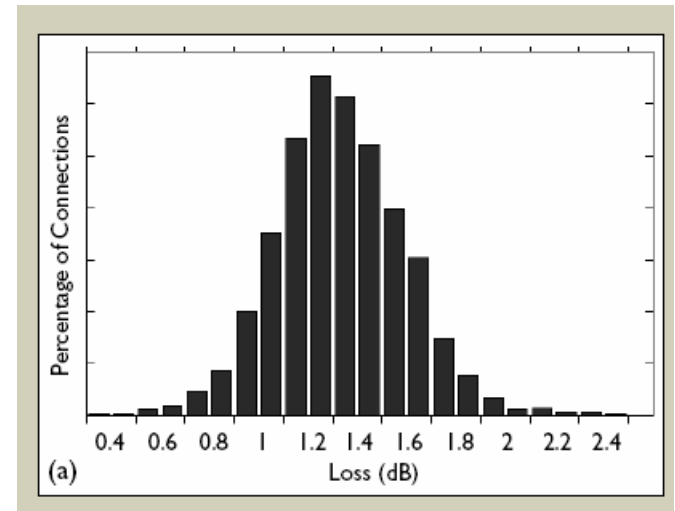
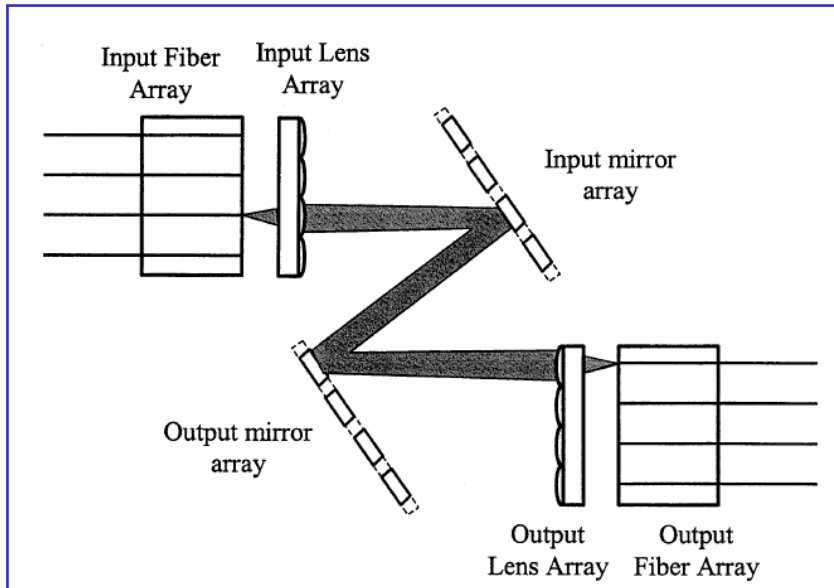
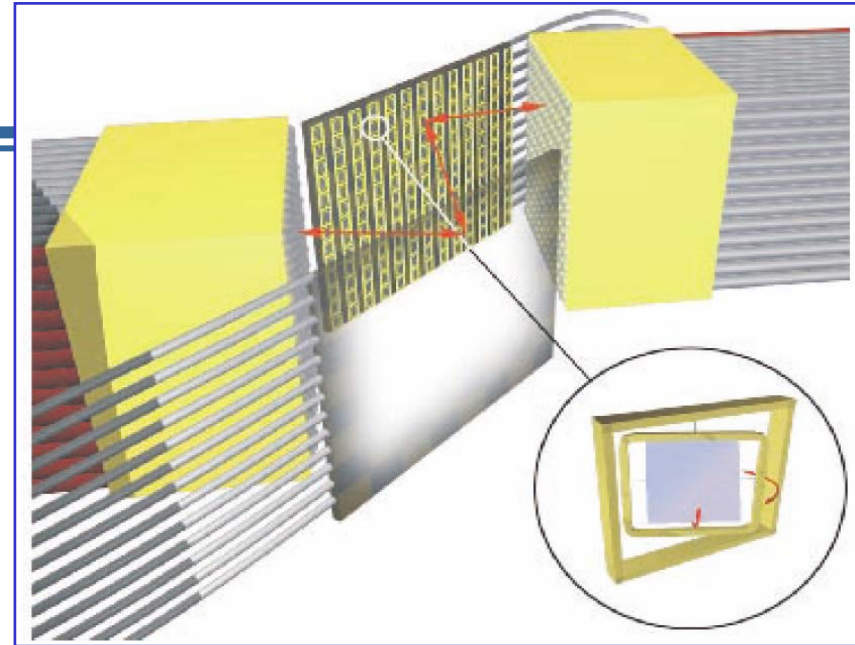
(B)

Measured S parameters of Ti waveguides: (A) before packaging; (B) after packaging

- **Measurement setup:**

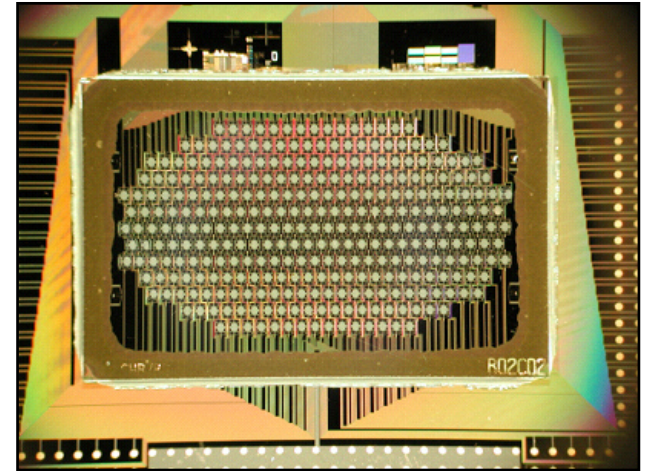
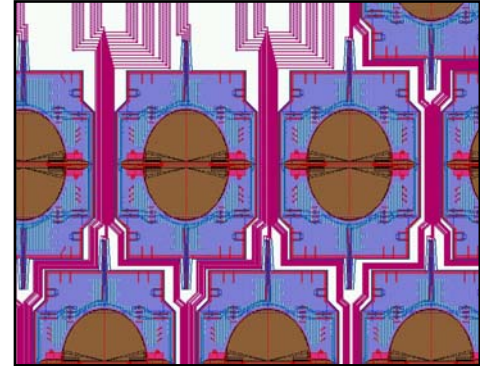
- **TRL calibration with short, ~ 1.5 ps through, ~ 5 ps line standards**
- **Agilent Technologies E8364A PNA (45MHz-50GHz), IF bandwidth: 50Hz**
- **Cascade Microtech RF-1 Microwave Probe Station with Infinity Probes I40-A-GSG-150**

All OPTICAL Fiber Optical Switch: 320 Fibers IN; 320 Fibers OUT



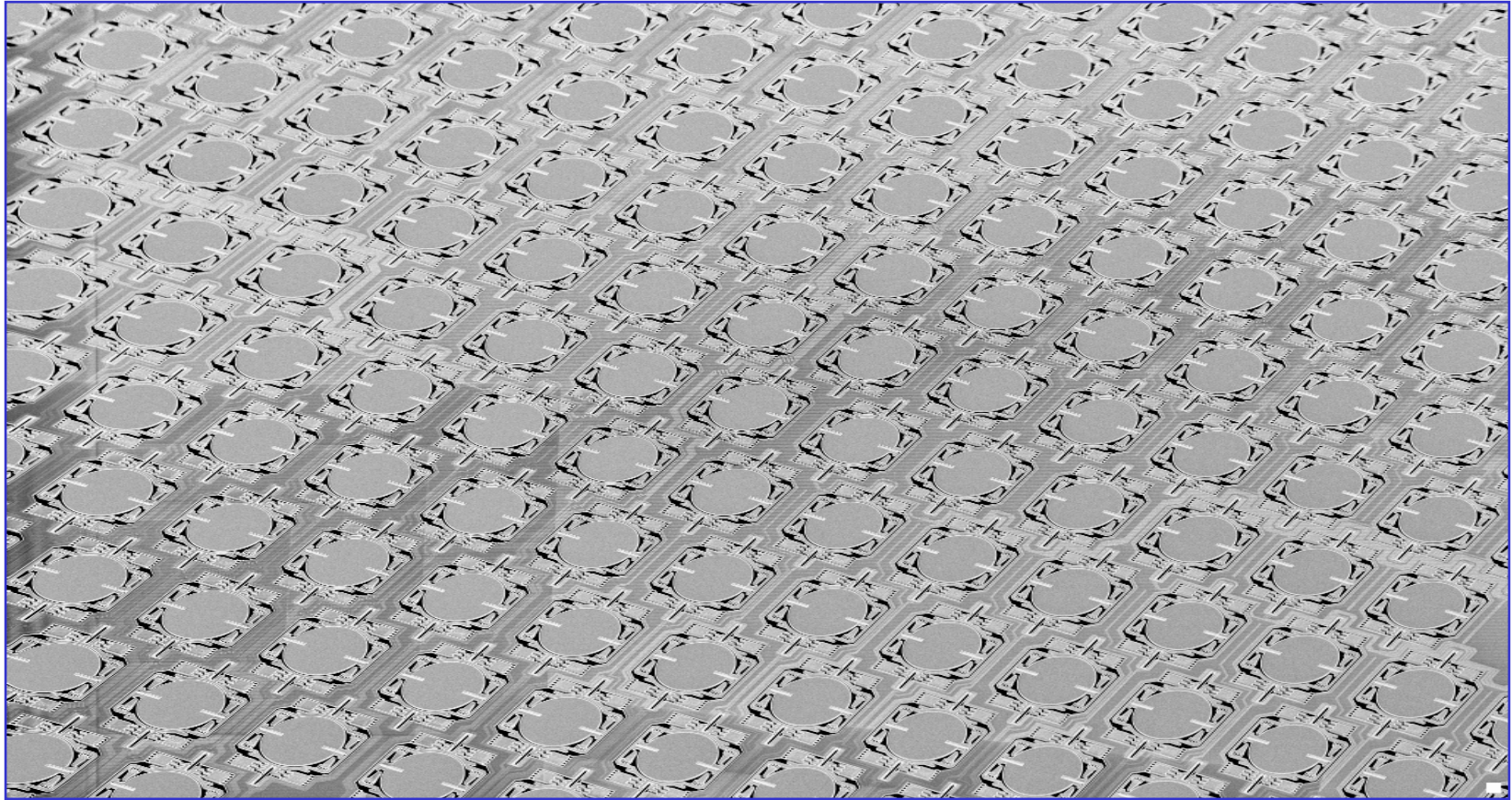
Mirror properties

- Two-axis gimbaled mirrors
 - Each mirror surface is supported by a pair of orthogonal torsional flexures
 - allowing independent rotation about the x and y axes.
 - Each mirror has actuation drives for the x and y axes.
 - The drives are electrostatic and allow roughly constant torque across the entire rotational range.
 - Continuous mirror angles
 - Not discrete, continuous



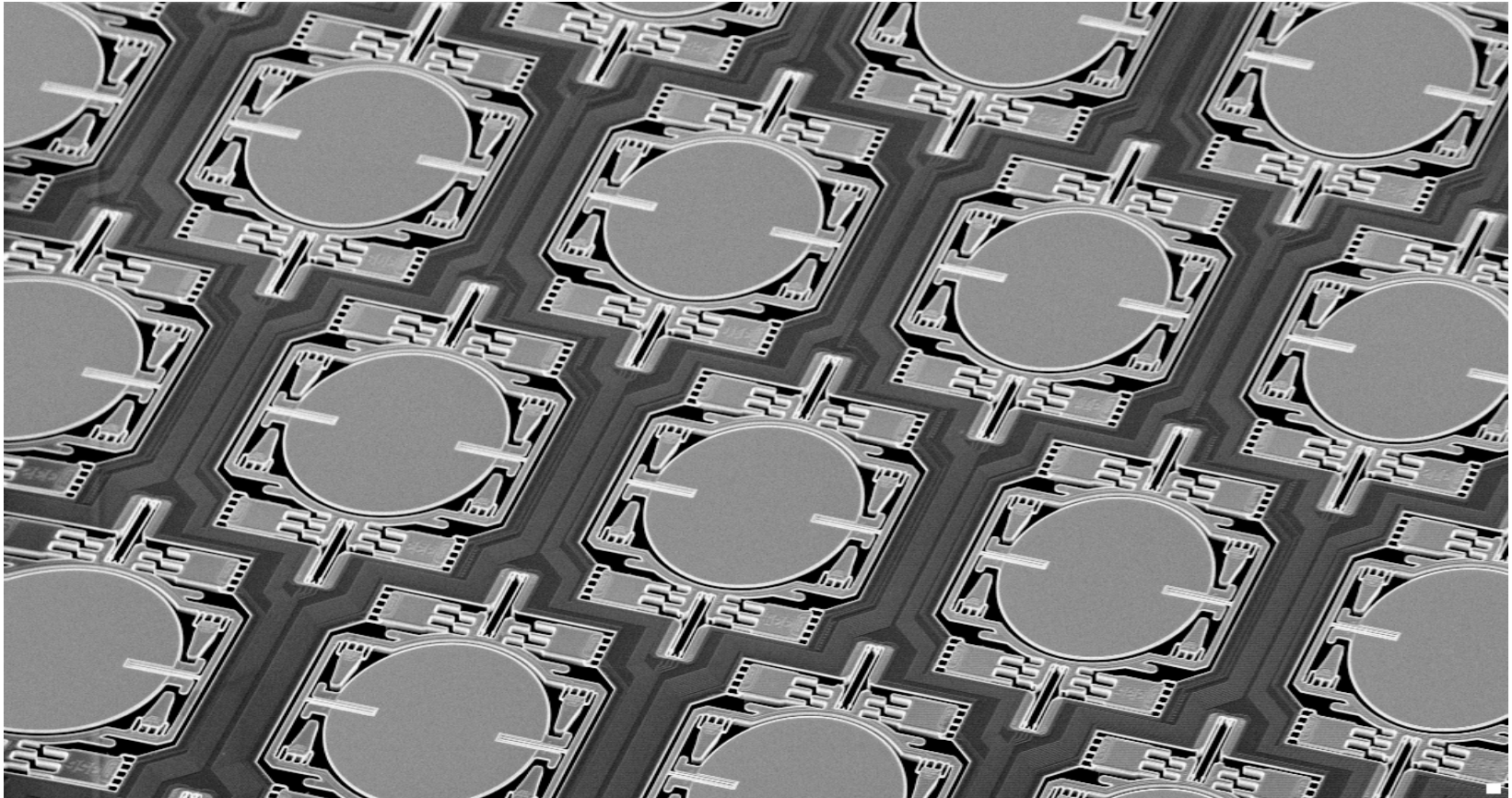
Calient Networks, Inc.

> 320 Mirror Arrays

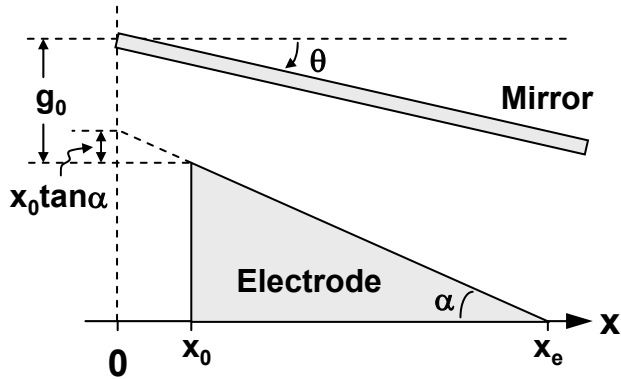


Dual Axis mirror design

Calient Networks, Inc.



Analysis



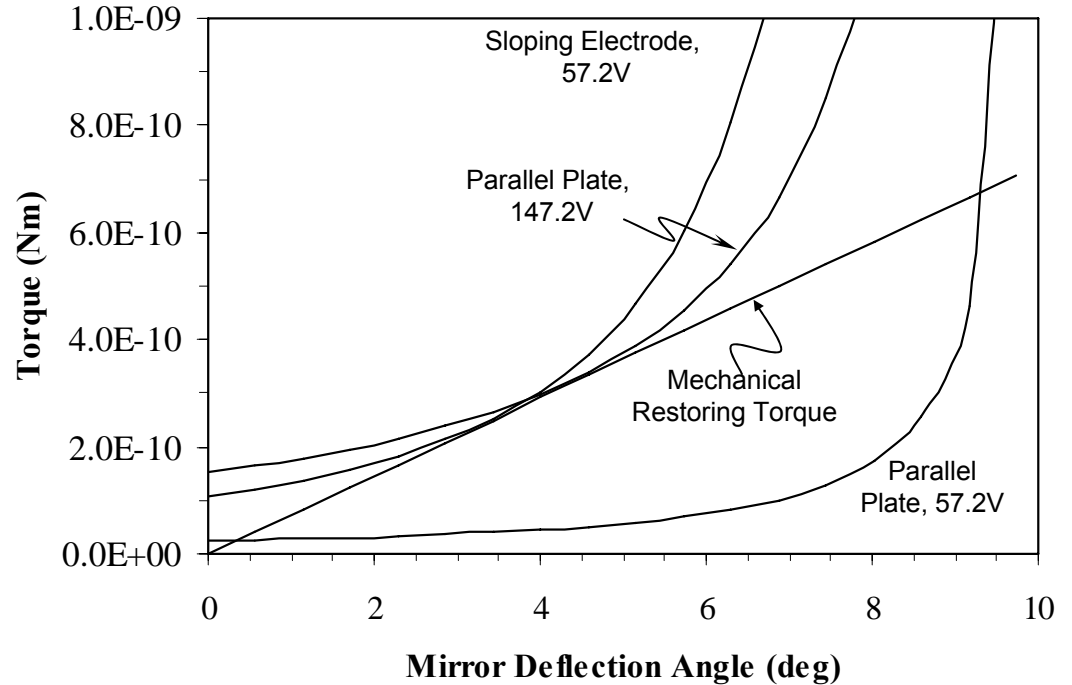
$$T_E = \int_0^{x_e} x F_E(\theta, x) dx - \int_0^{x_0} x F_E(\theta, x) dx$$

where:
$$F_E = \frac{\epsilon_0 w V^2}{2g(\theta, x)^2}$$

$$g = g_0 - x_0 \tan \alpha + x \tan(\alpha - \theta)$$

Device dimensions modeled:

100x100x10μm mirror, 1x10x60μm springs
 $x_0=5\mu\text{m}$, $x_e=49.24\mu\text{m}$, $\alpha=10^\circ$, $g_0=0.88\mu\text{m}$
 $f_0=16.7\text{kHz}$, $\pm 10^\circ$ tilt

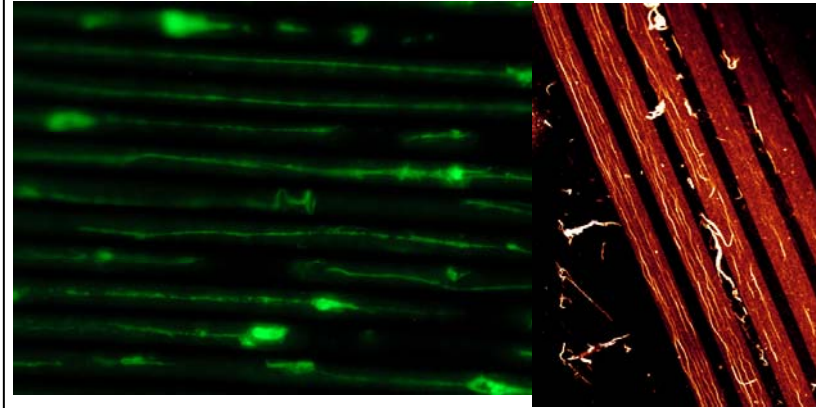


Sloping electrode yields significant reduction in drive voltage relative to comparable parallel-plate design without sacrificing switching speed or displacement

Titanium Bio-Channels

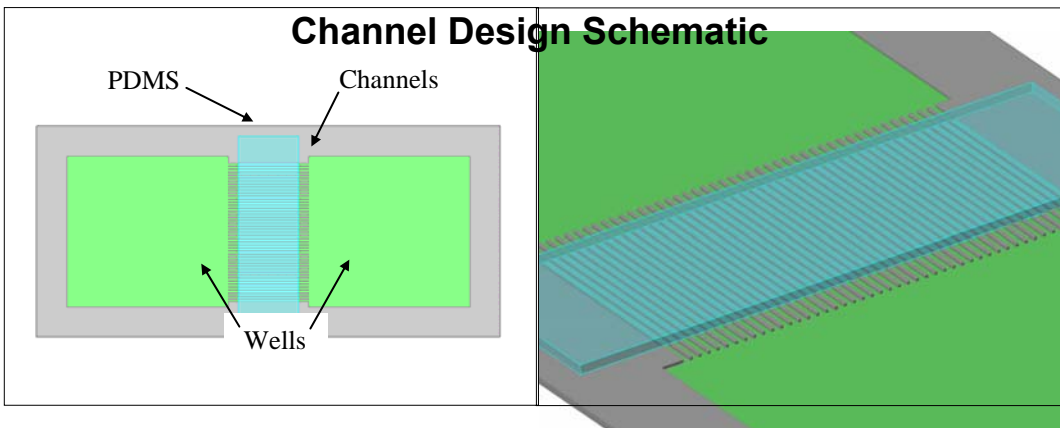
- Thin (25 μm) Ti foil for transmission x-ray analysis.
- Backside etch to 3.8 μm to reduce attenuation
- Sputter SAM TiO_2 for hydrophilic layer
- Prevent protein adsorption (PLL-g-PEG)
- Biomolecules assemble and align in channels

Fluorescence Microscopy

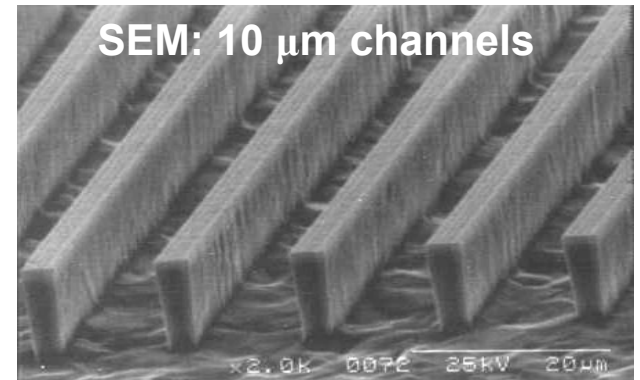


F-actin - introduction of α -actinin
Vary Channel Width with
biomolecule persistence length

Channel Design Schematic



SEM: 10 μm channels



Titanium Multi-frequency Traveling Wave Dielectrophoresis Device

Given electrical potential as

$$\phi(x,0,t) = \phi^c(x,0) \frac{1}{\sqrt{2}} (\cos(\omega_1 t) + \cos(\omega_2 t)) + \phi^s(x,0) \frac{1}{\sqrt{2}} (\sin(\omega_1 t) + \sin(\omega_2 t))$$

Get the DEP force

$$\langle F_{DEP} \rangle = \frac{1}{8} \text{Re}[G(j\omega_1) + G(j\omega_2)] \nabla (|E^c|^2 + |E^s|^2) + \frac{1}{4} \text{Im}[G(j\omega_1) + G(j\omega_2)] \nabla \times (E^c \times E^s)$$

where

$$G(\omega) = 4\pi r^3 \epsilon_m \frac{(\epsilon_p + \frac{\sigma_p}{j\omega}) - (\epsilon_m + \frac{\sigma_m}{j\omega})}{(\epsilon_p + \frac{\sigma_p}{j\omega}) + 2(\epsilon_m + \frac{\sigma_m}{j\omega})}$$

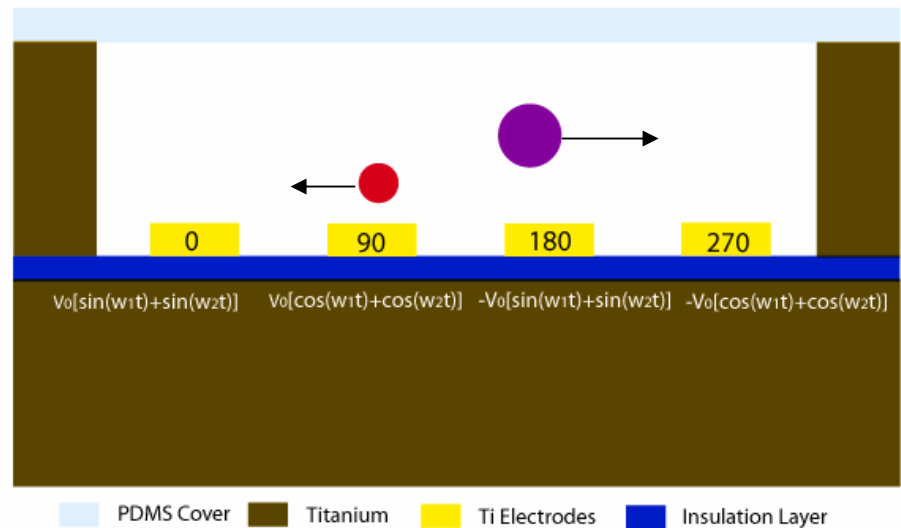
Vertical translation

Horizontal motion

Separation Strategy

$\text{Re}[G_A(j\omega_1) + G_A(j\omega_2)] < 0$
 $\text{Re}[G_B(j\omega_1) + G_B(j\omega_2)] < 0$ → Lift the particles above the electrodes

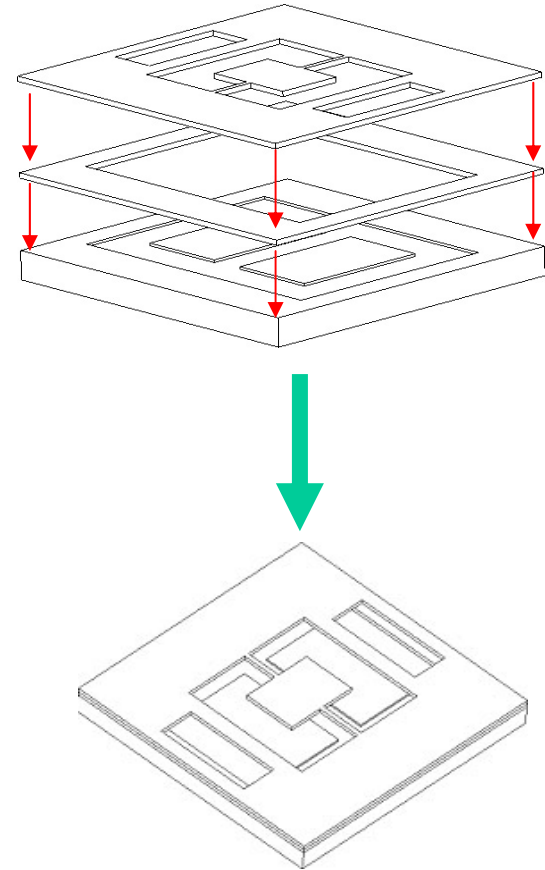
$\text{Im}[G_A(j\omega_1) + G_A(j\omega_2)] > 0$
 $\text{Im}[G_B(j\omega_1) + G_B(j\omega_2)] < 0$ → Drive the particles towards two different directions



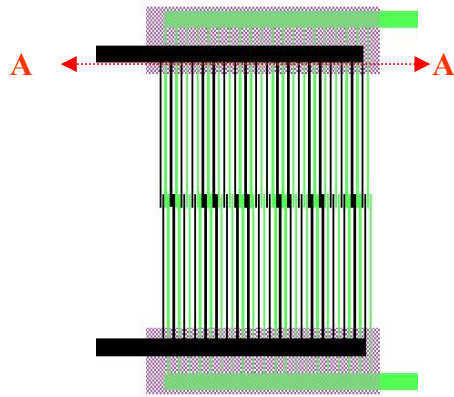
Schematic of the device setup and working principle

Motivation: Why Titanium?

1. Excellent biocompatibility
2. High Fracture toughness (60 times that of silicon)
3. 3-D structures formed by stacking Ti Wafers/ foils
4. Micromachining AND Macromachining



Device Processing Ti Electrode Substrate



Isolate the Ti substrate



First layer Ti electrode



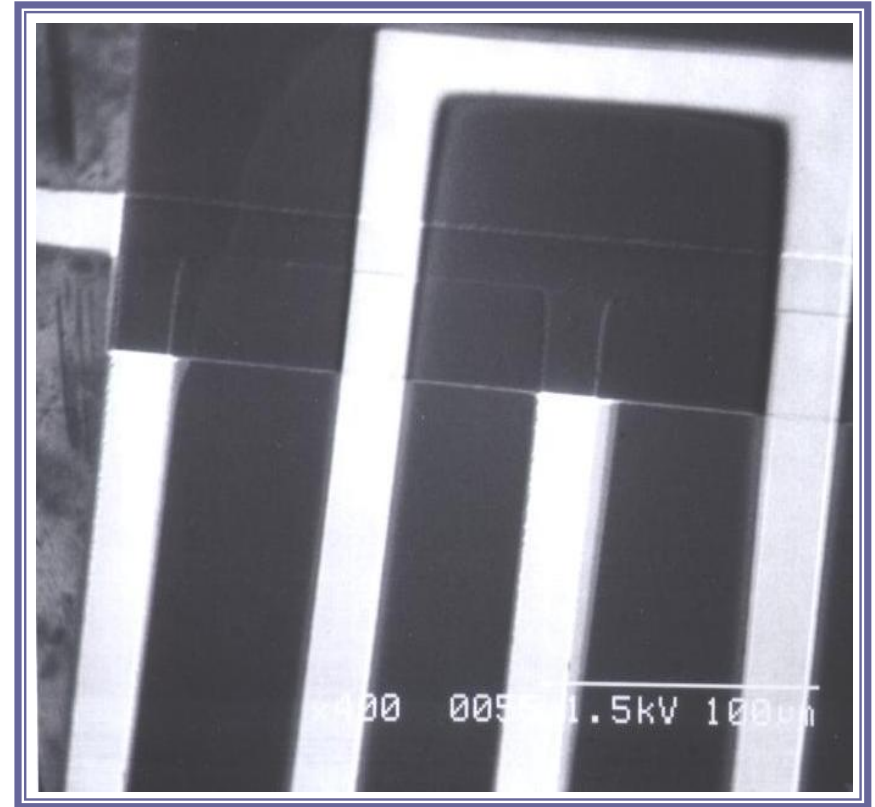
400nm SiO₂



Second layer Ti electrode

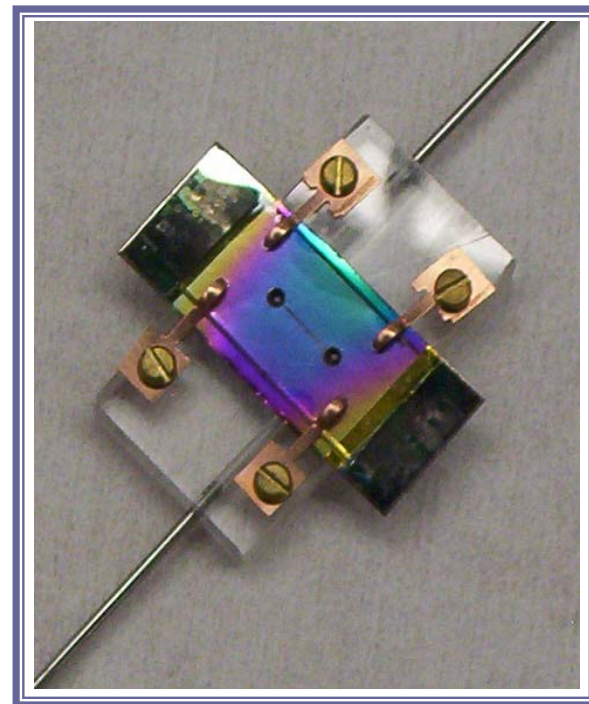
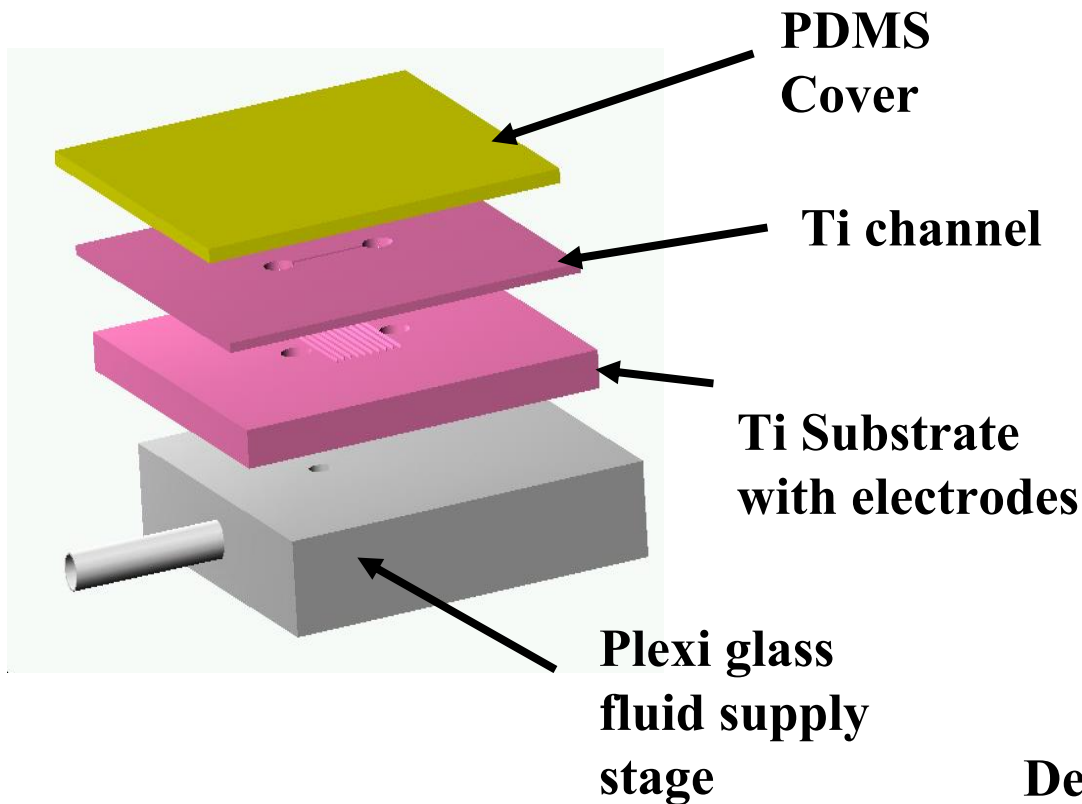


■ Ti substrate ■ Thin film Ti
■ Thin SiO₂ ■ Isolation layer



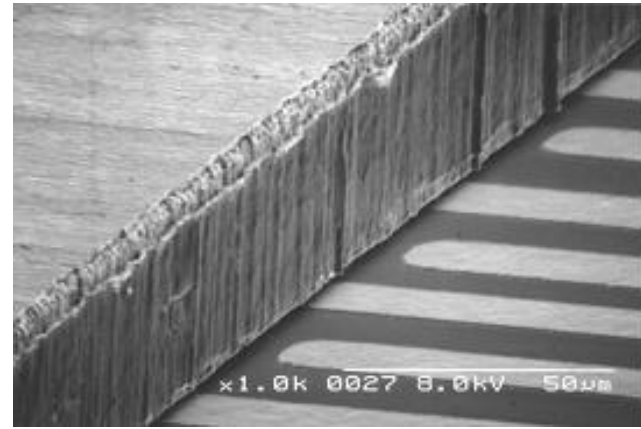
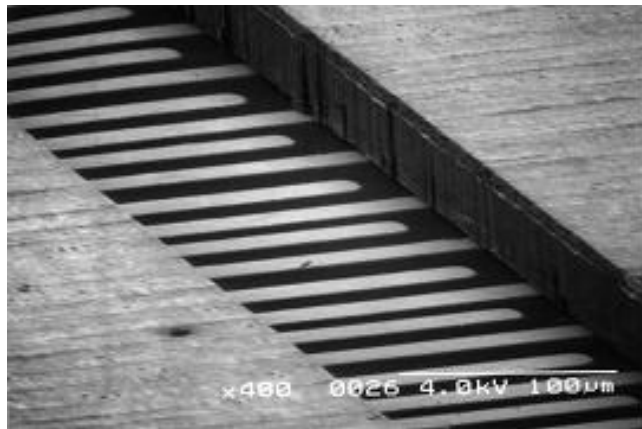
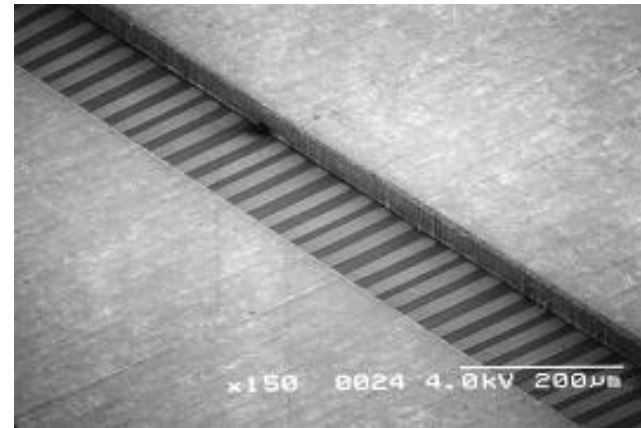
SEM image of a 300nm silicon dioxide layer sandwiched between two layers of titanium electrodes

Ti Dielectrophoresis Device Test Fixture



Device Channel 200 μm wide)

Ti Dielectrophoresis Device

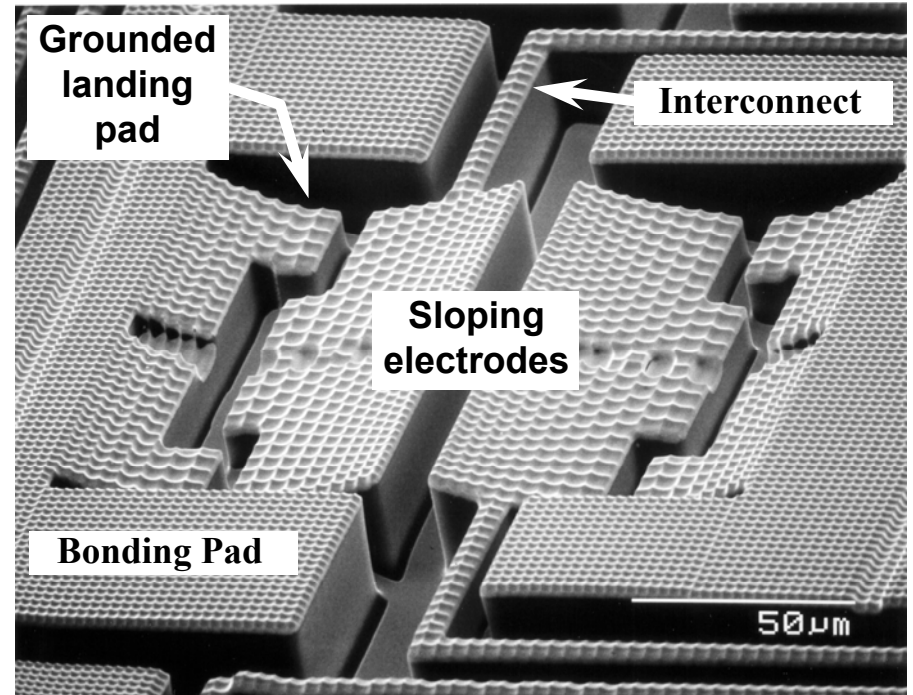
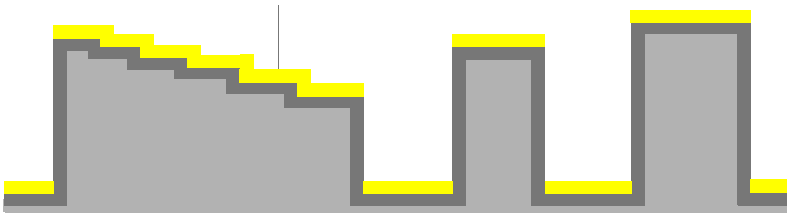


Low magnification view of the reservoir and channel with 24 electrodes located at the bottom of the channel

Fabrication: Silicon Sloping Electrodes

Process flow:

- 1) PR pattern
- 2) Deep etch
- 3) Oxidization
- 4) HF strip
- 5) Oxidization
- 6) Au deposition



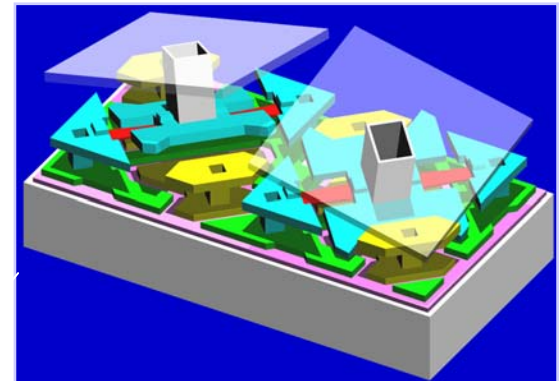
Process demonstrates simplified, single mask method for 3D microfabrication relative to previous methods

Picture on Cover of Applied Physics Letter, Dec. 2005

Commercial Examples:

‘Chip-scale’ Heterogeneous Integration

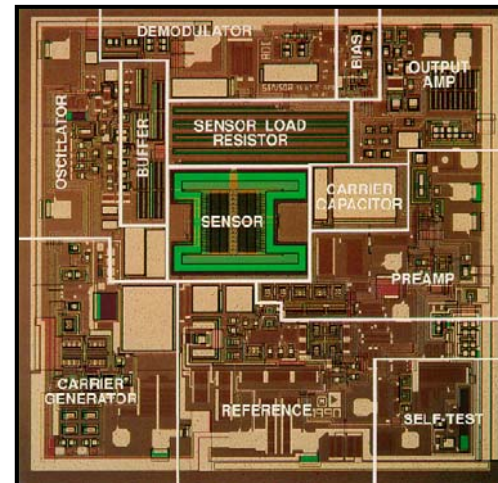
Texas Instruments Digital Light Modulator:
(Microelectronics/MEMS to direct photons)



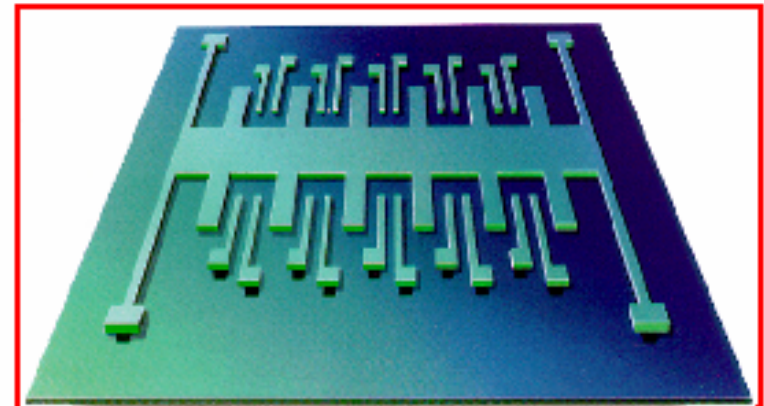
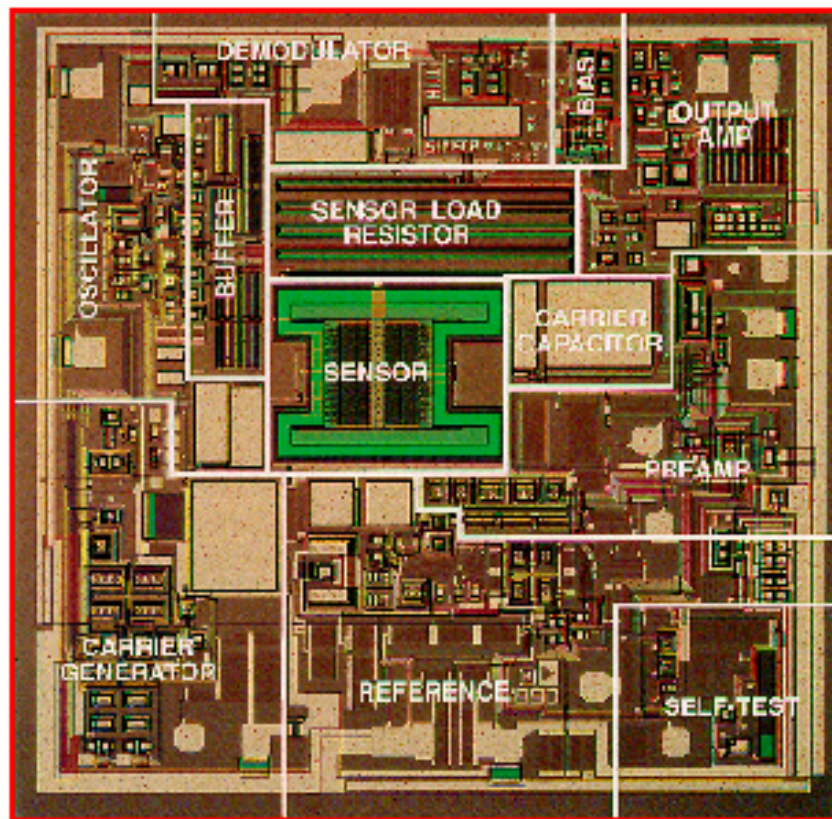
Commercial Examples:

‘Chip-scale’ Heterogeneous Integration

Micro Accelerometer (Airbag Deployment):
(Microelectronics/MEMS)



Accelerometer for Airbags (Analog Devices)



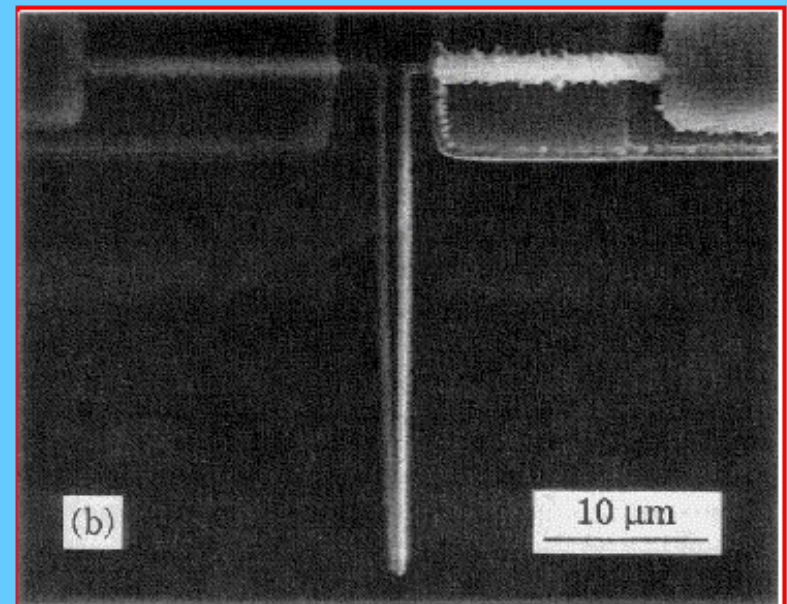
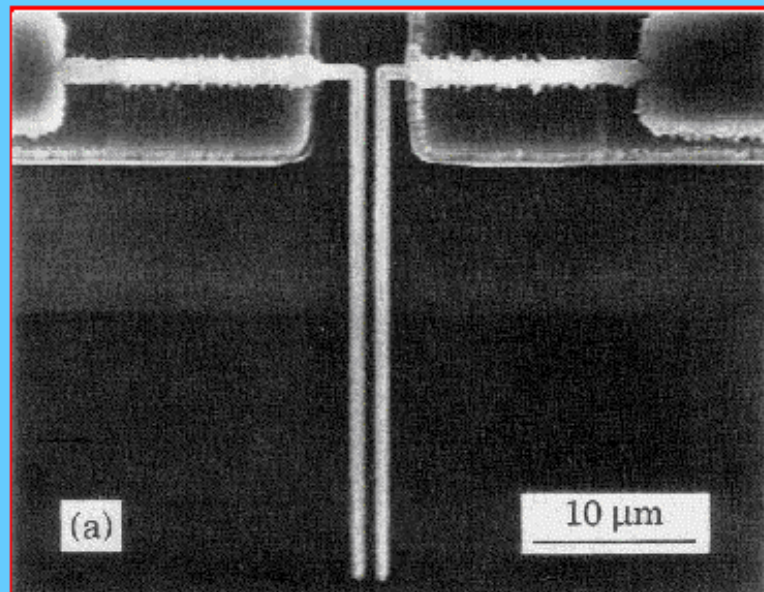
DiamondWave Product Line

Calient Networks, Inc.



Microtweezer

- ¥ Single Crystal Silicon beams
- ¥ Applied Electric-field produces attractive force between plates



Bonded Electrode / Micromirror Array



Actuated Mirror (snapped down)

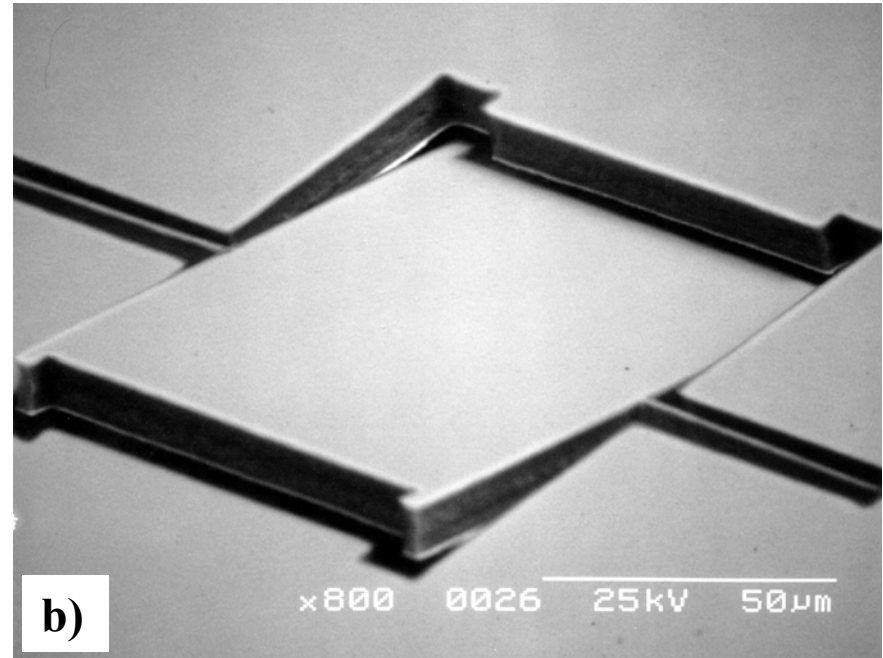


Interconnects on Lower Wafer

10x10 Array of Titanium Torsion Mirrors

Digital operation mode

- Pull-in voltage $\geq 50V$
- Mirror tilt $\pm 10^\circ$

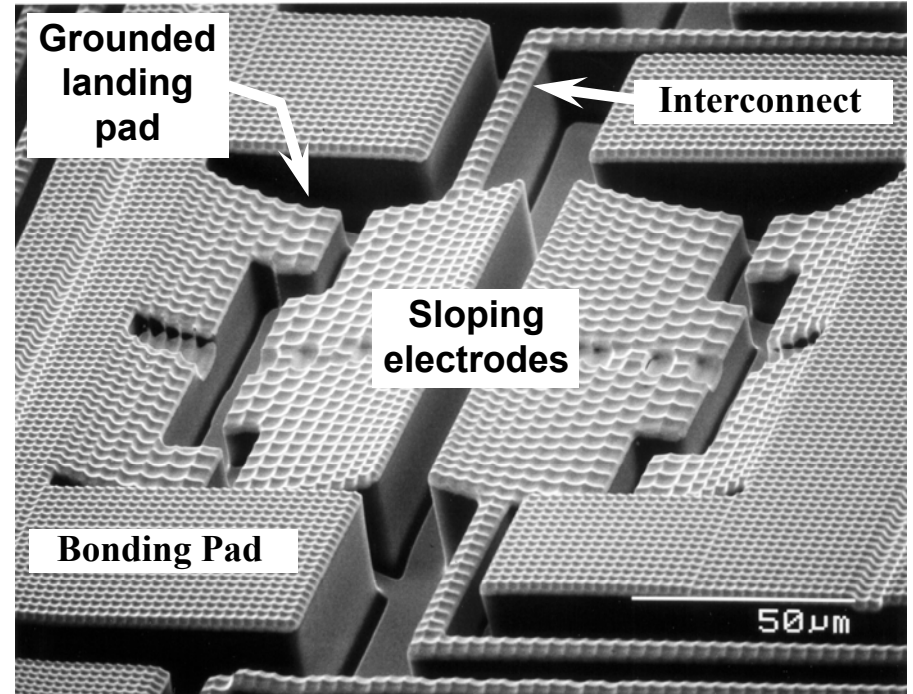
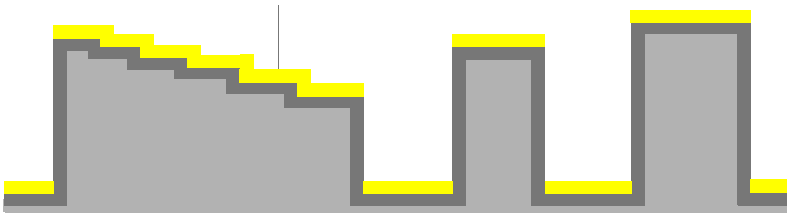


Applied Physics Letter, Dec. 2005

Fabrication: Silicon Sloping Electrodes

Process flow:

- 1) PR pattern
- 2) Deep etch
- 3) Oxidization
- 4) HF strip
- 5) Oxidization
- 6) Au deposition



Process demonstrates simplified, single mask method for 3D microfabrication relative to previous methods

Picture on Cover of Applied Physics Letter, Dec. 2005

Packaged device/ Microscope picture

Fig. 1: backside of a packaged device.

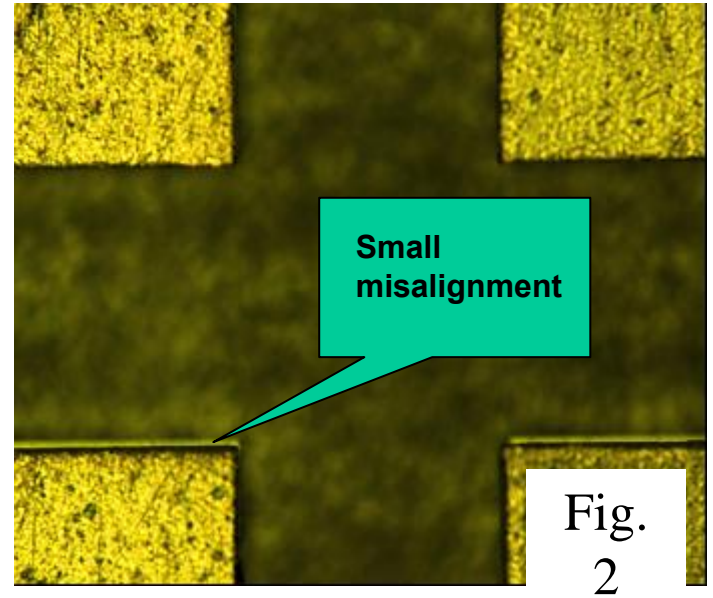
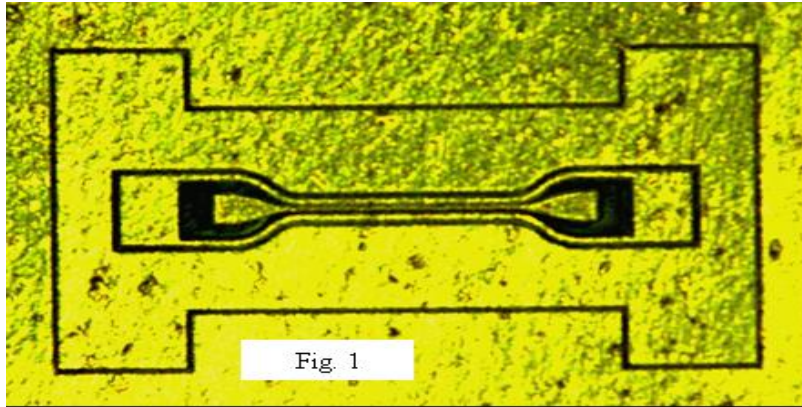


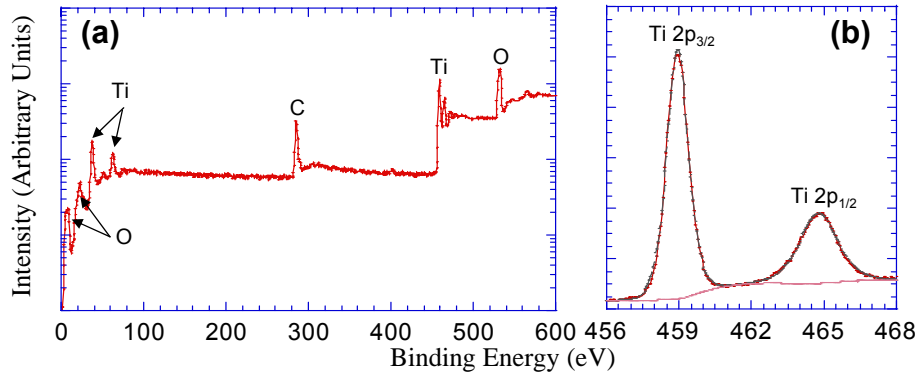
Fig. 2: The small miss-alignment shows the two Au-bonded layers.



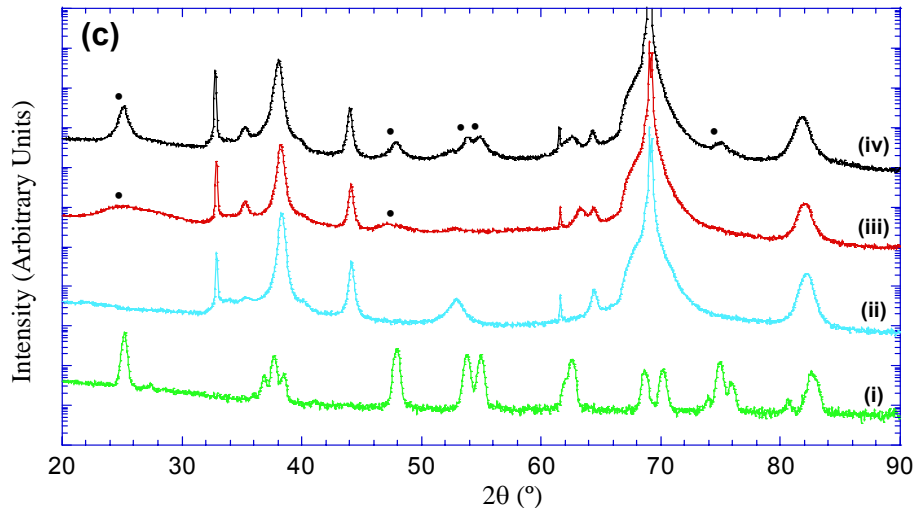
Fig. 3 shows (black) the waveguide with dielectric.

Joining of titanium

• **Characterization of titania layers formed after aging in aq. H_2O_2 .** XPS spectra of titania layer formed on evaporated blanket Ti film:



• **a**, survey scan;
• **b**, high resolution Ti 2p scan. **c**, XRD spectra of



• **(i)** reference anatase powder;
• **(ii)** as evaporated Ti film;
• **(iii)** as-formed titania layer on evaporated blanket Ti film;
• **(iv)**, titania layer in iii after annealing for 8 hr at 300 °C. (Anatase peaks in films are represented by •).

Advanced Functional Materials, March 2005

Outstanding Issues: Stiction

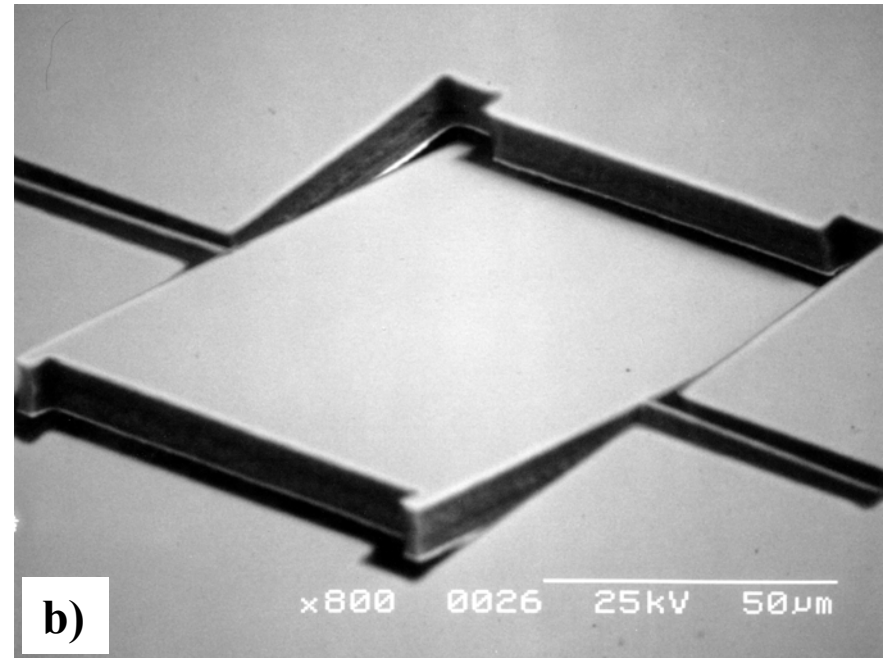
Digital operation mode

- Pull-in voltage $\geq 50V$
- Mirror tilt $\pm 10^\circ$

→ PROBLEM: Stiction

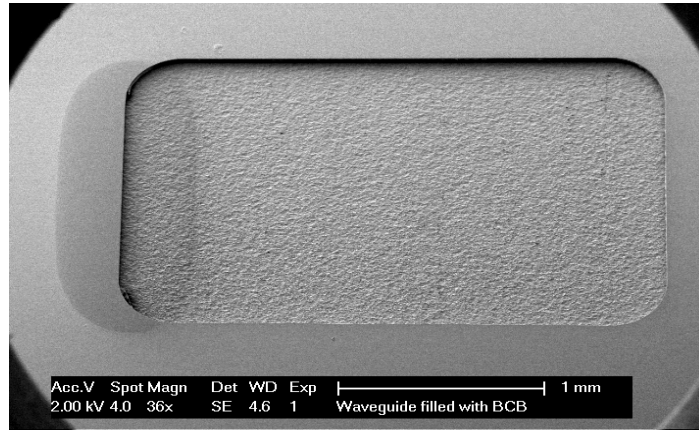
→ PROBABLE CAUSES:

- gold-gold contact
- adsorbed molecules

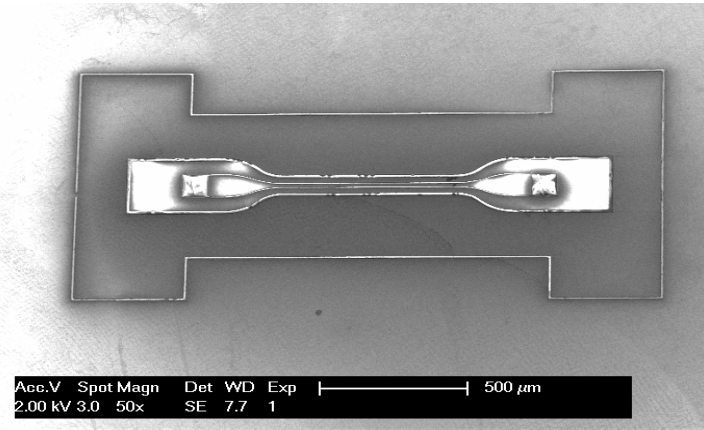


Stiction problem was expected and can be mitigated by minor design modification and testing in controlled environment

Device sample before packaging



(A)



(B)

- (A) Titanium package (lid) 1.3 mmX2.6 mm. The cavity which covers the relay is 100 μ m deep.
- (B) BCB/CVD Dielectric-filled waveguide (10 μ m thick) after lapping and CMP. The SEM charging highlights the dielectric. The Ti surfaces of the waveguide and the chip perimeter are coated with Au for bonding.
- Au is deposited (4 μ m) on the Ti package (lid), and the lid is bonded to the 'relay chip' using thermal compression bonding.