Introduction to Nanotechnology and Nanoscience – Class#7

Liwei Lin

Professor, Dept. of Mechanical Engineering
Co-Director, Berkeley Sensor and Actuator Center
The University of California, Berkeley, CA94720
e-mail: lwlin@me.berkeley.edu
http://www.me.berkeley.edu/~lwlin

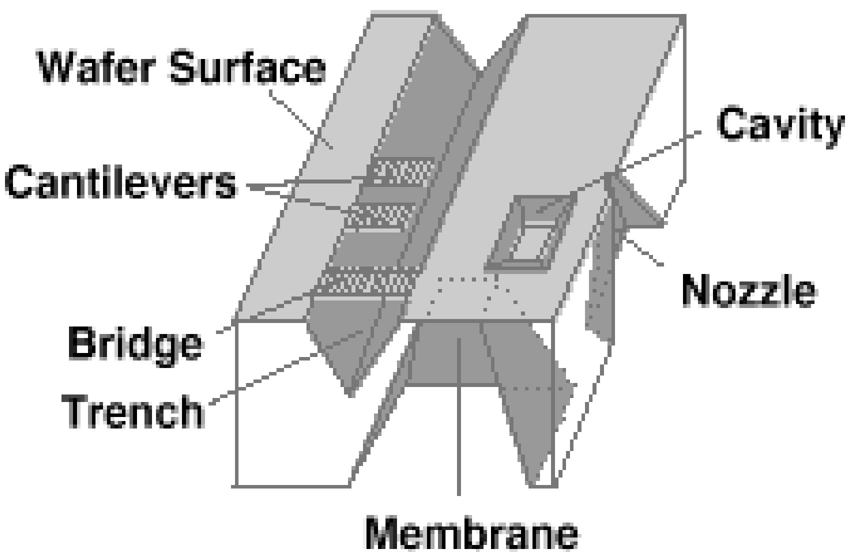
Outline

- Recap
- TEM
- SOI process – an example (HW#2 problem 2)
- Paper 1_1 last session

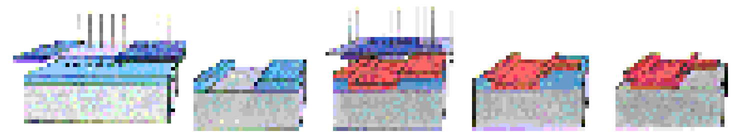
MEMS Fabrication

- MEMS collocate sensing, computing and actuating to change and control physical world

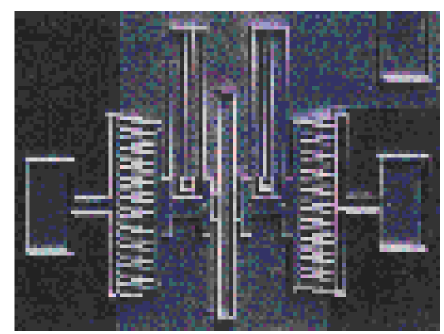
Common MEMS Fabrication Processes



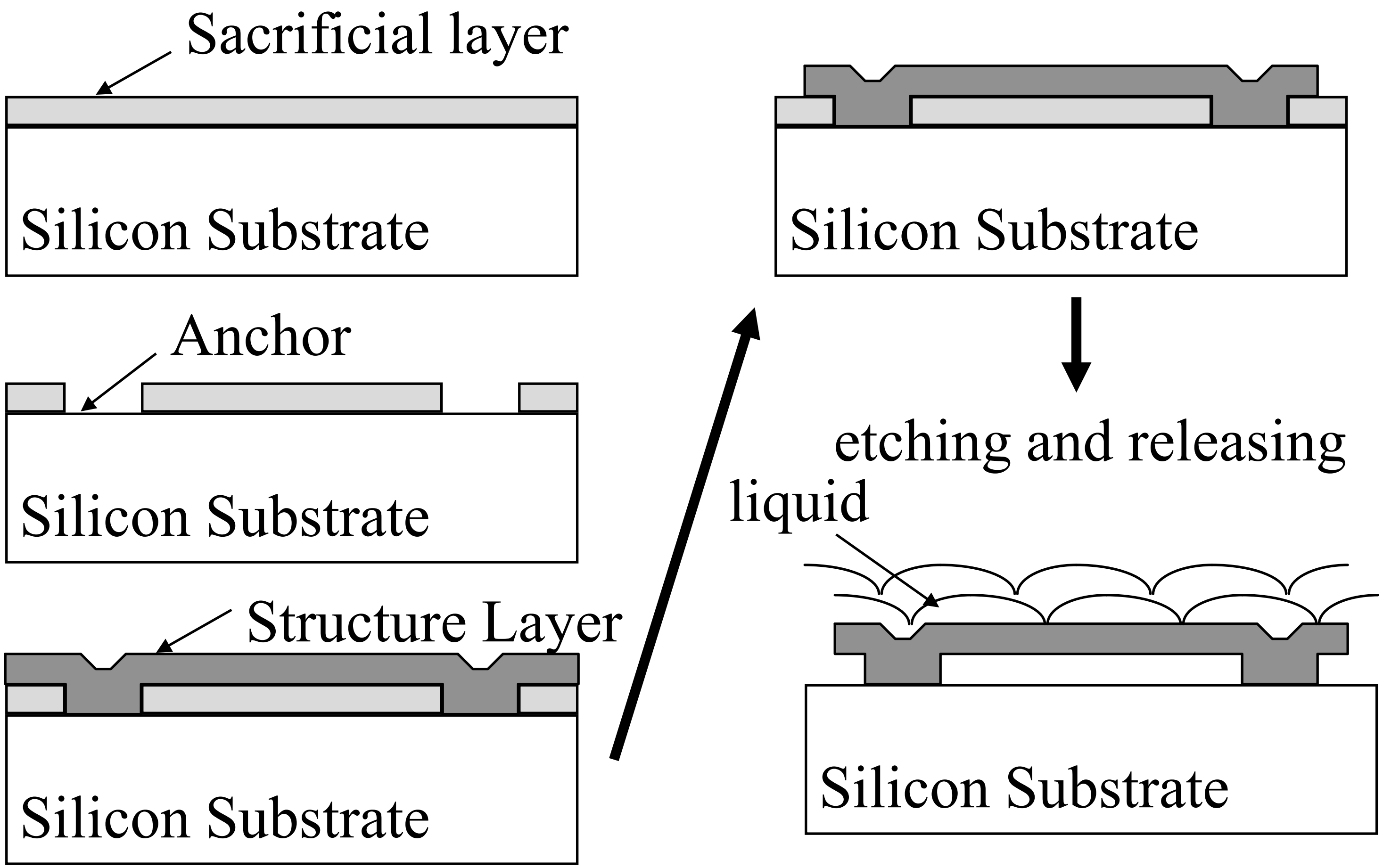
Bulk Micromachining



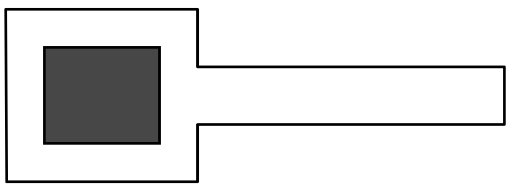
Surface Micromachining



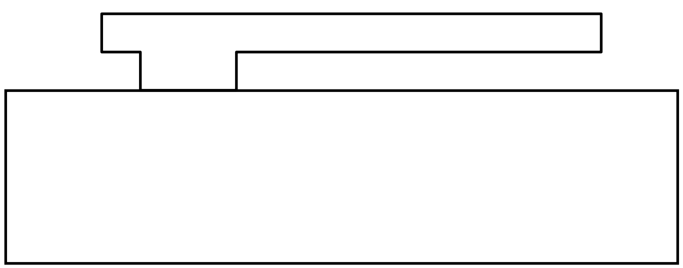
Surface Micromachining



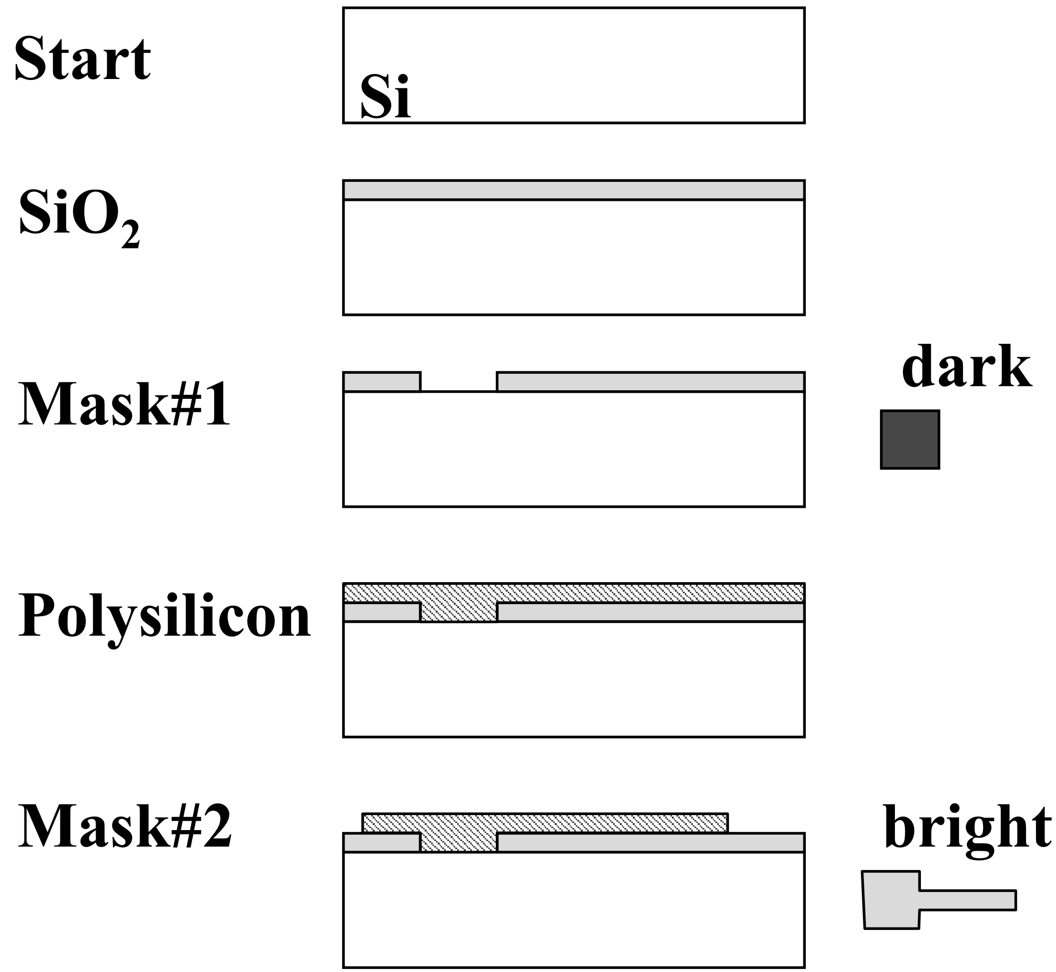
How to Make Cantilever?



Top View



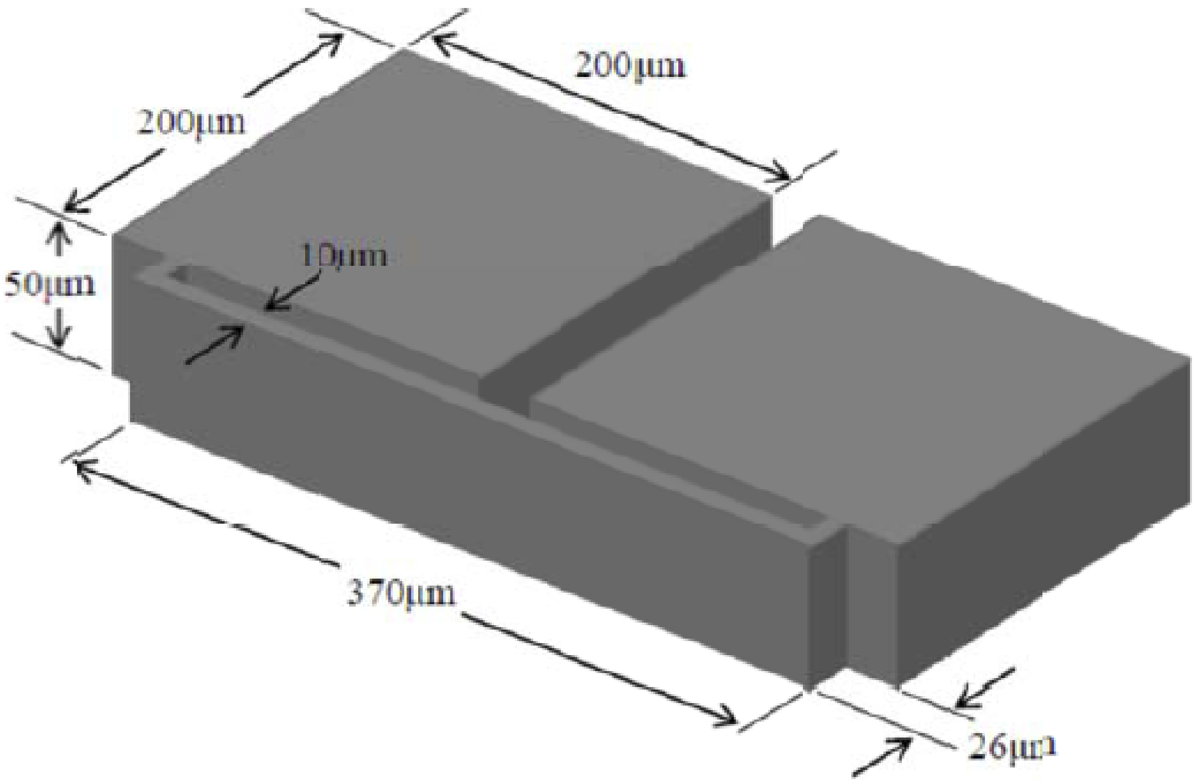
SideView



Problem #2

Problem 2 (Top-Down Process – MEMS)

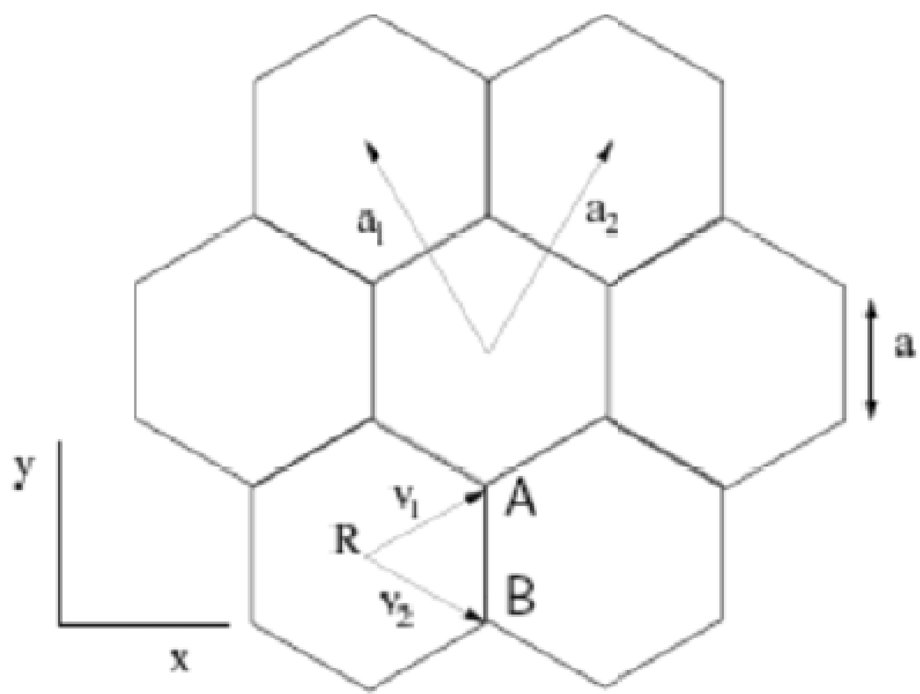
The figure shows an old experimental sample we used in previous ME 118 class for laboratory. The structure is made from the SOI (Silicon on Insulator) substrate using the top silicon layer as the structural layer. Please design a process flow chart to make this device – cross sectional view figures on the left and concise process explanations on the right. Please also draw the "mask(s)" to make the device.



Problem #3

Problem 3 (Graphene & CNT)

A graphene sheet is a honeycomb lattice of carbon atoms (see figure). Let the distance between carbon atoms be a. A good model for graphene is to consider a single plane in which there is one valence electron per carbon atom. We will use the tight-binding approximation, in which this electron can occupy a single p_z orbital at each carbon site. Let **R** denote the centers of the hexagons in the honeycomb: these form the underlying hexagonal Bravais lattice. Please notice that the latter is indeed a Bravais lattice differently from the graphene honeycomb lattice. The unit cell spanned by a_1 and a_2 contains two carbon atoms conventionally labeled as A and B atom, located at $\mathbf{R}+\mathbf{v}_A$, $\mathbf{R}+\mathbf{v}_B$, as shown in the figure.



Problem #3

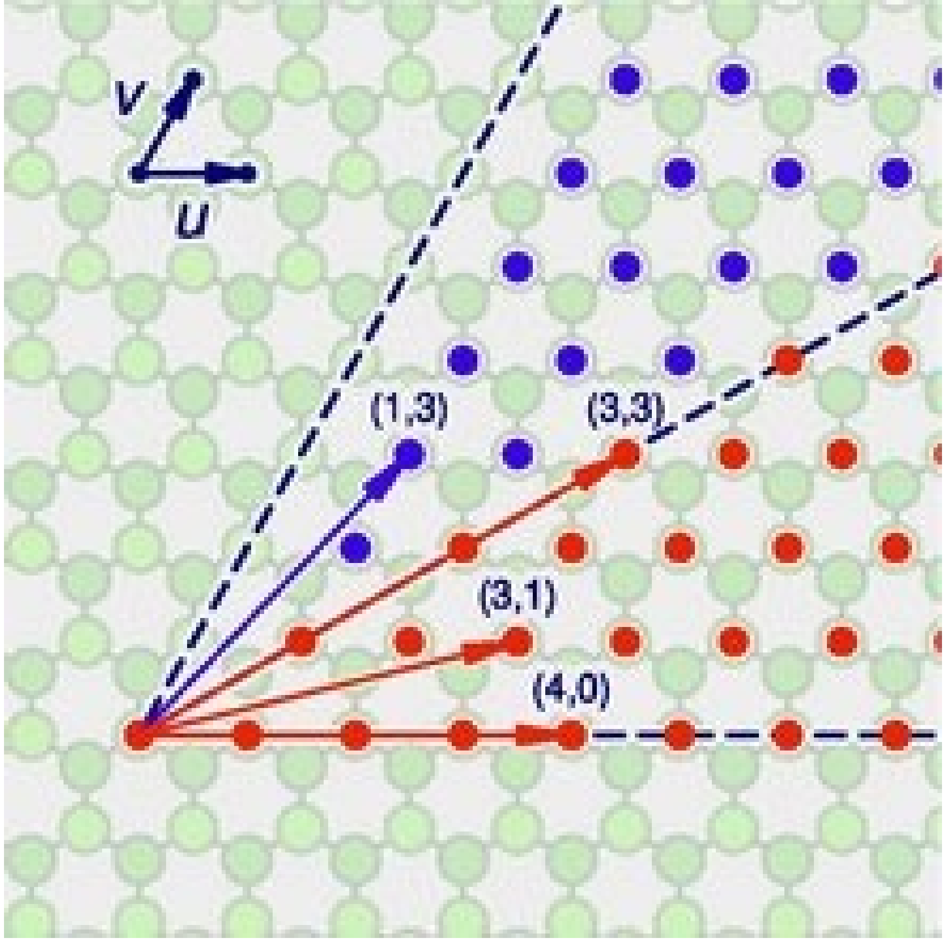
Carbon nanotubes are made up of a section of the graphene lattice that has been wrapped up into a cylinder. You can specify the way the lattice is wound up by identifying the winding vector **W**. The winding vector must be a Bravais lattice vector, and so can be specified by two integers:

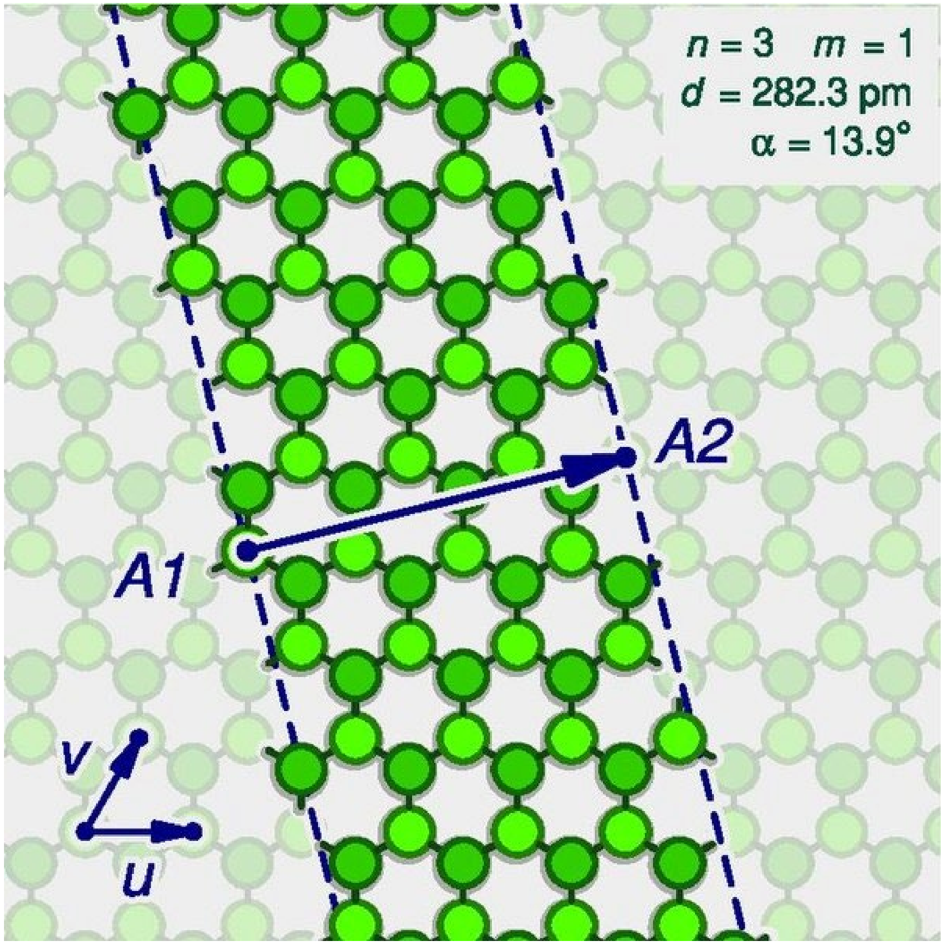
$$\mathbf{W} = n\, a_1 + m\, a_2;$$

where n and m are integers. To construct a nanotube, take a graphene lattice and mark one atom (either A type or B type) as the origin. Shift the origin of the vector **W** on the chosen atom. The new vector $\mathbf{W_n}$ will point to another atom of the same type. Roll up the sheet perpendicular to $\mathbf{W_n}$ so that the second atom sits exactly on top of the first. You have constructed a (n;m) nanotube! Nomenclature: we can specify some special tubes said **achiral**: they are (n; n) tubes which are called **armchair tubes**, and (n; 0) **zig-zag tubes**. All other tubes are said **chiral**.

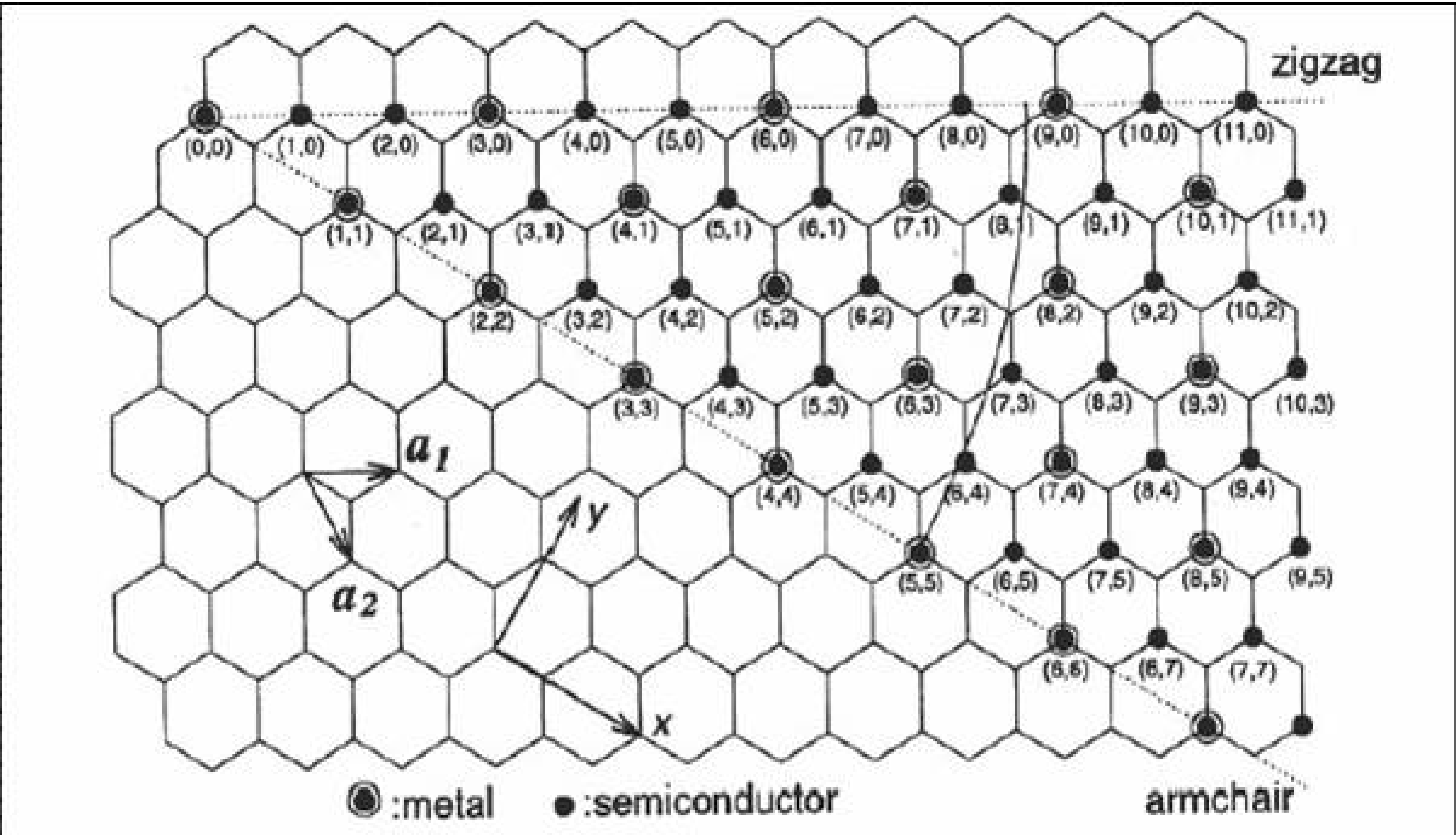
a. Build a (5; 5) armchair tube (i.e. with scissors and adhesive tape!) by making use of transparencies - You can download this sheet from course homepage and print it out. The easy way to submit your homework is to take and print out a photo with finished structure on a white paper with you name on the paper as the evidence.
b. Construct a (8; 0) zig-zag tube.
c. Build a chiral (7; 3) tube.
d. Create and name a new tube of your own.

More Examples

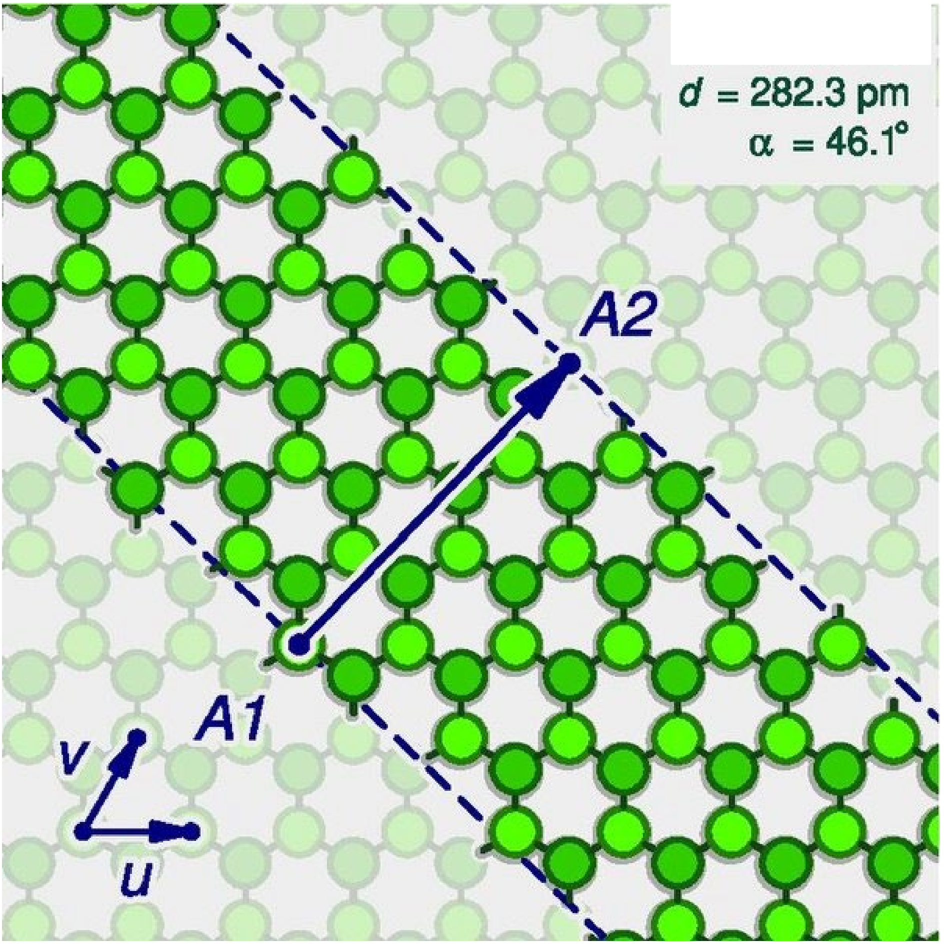


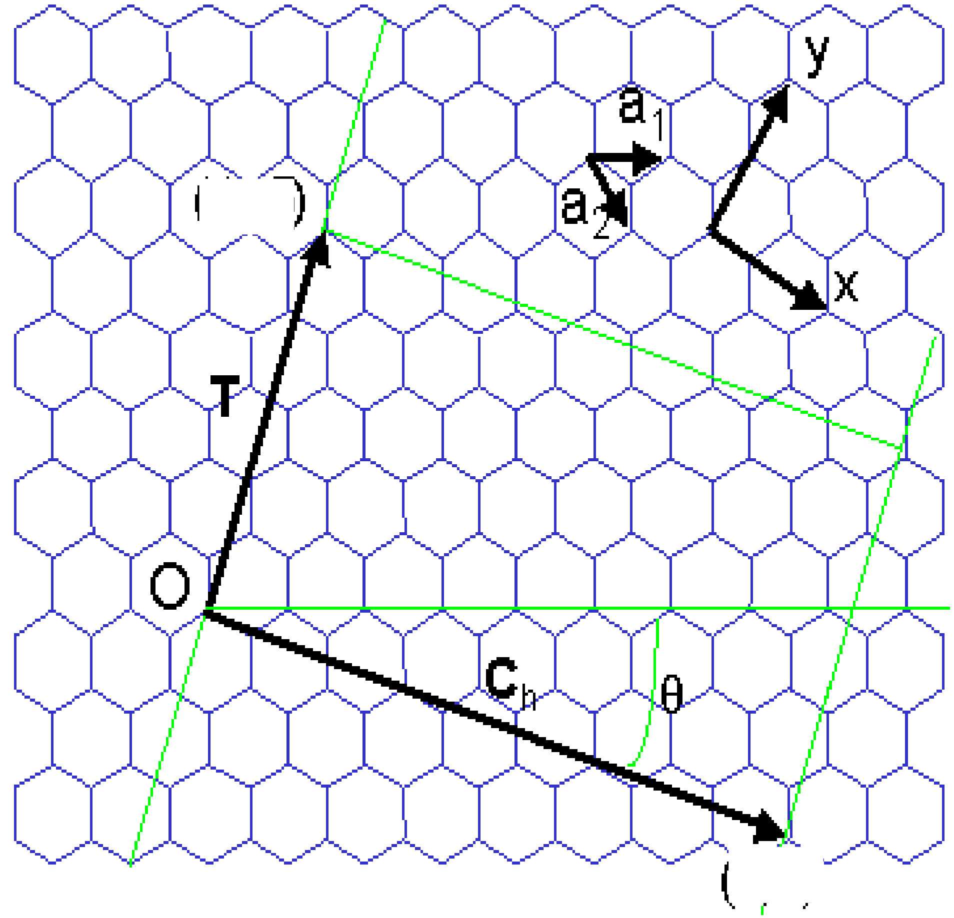


Examples


zigzag
(0,0) (1,0) (2,0) (3,0) (4,0) (5,0) (6,0) (7,0) (8,0) (9,0) (10,0) (11,0)
(1,1) (2,1) (3,1) (4,1) (5,1) (6,1) (7,1) (8,1) (9,1) (10,1) (11,1)
(2,2) (3,2) (4,2) (5,2) (6,2) (7,2) (8,2) (9,2) (10,2)
(3,3) (4,3) (5,3) (6,3) (7,3) (8,3) (9,3) (10,3)
(4,4) (5,4) (6,4) (7,4) (8,4) (9,4)
(5,5) (6,5) (7,5) (8,5) (9,5)
(6,6) (6,7) (7,7)
a_1
a_2
y
x
:metal
:semiconductor
armchair

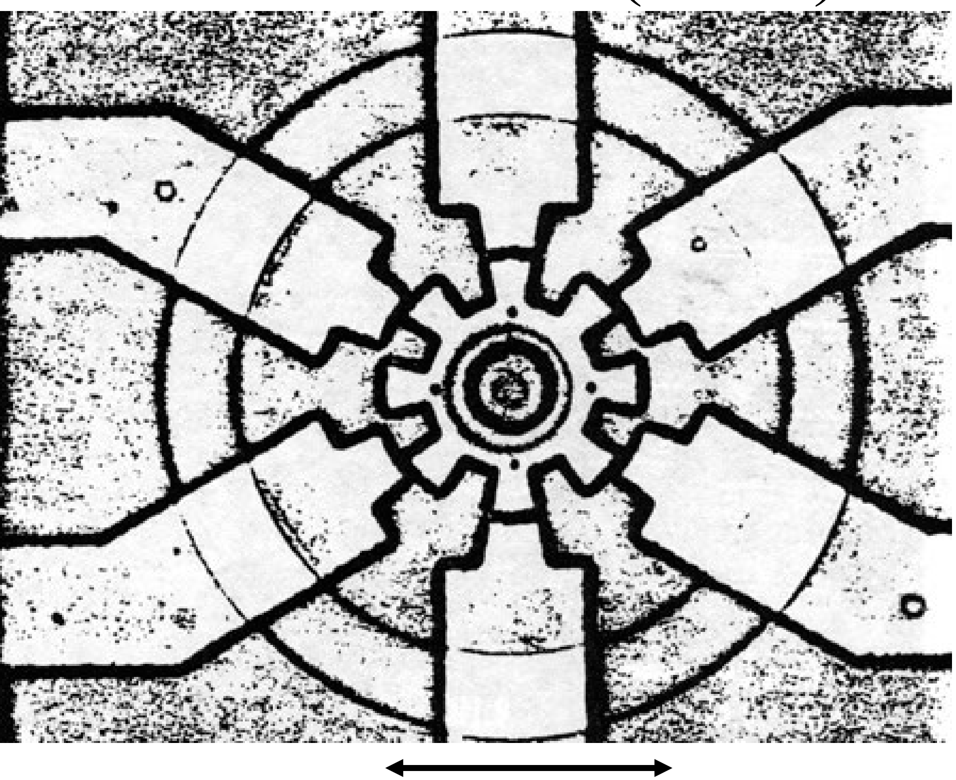
More Examples



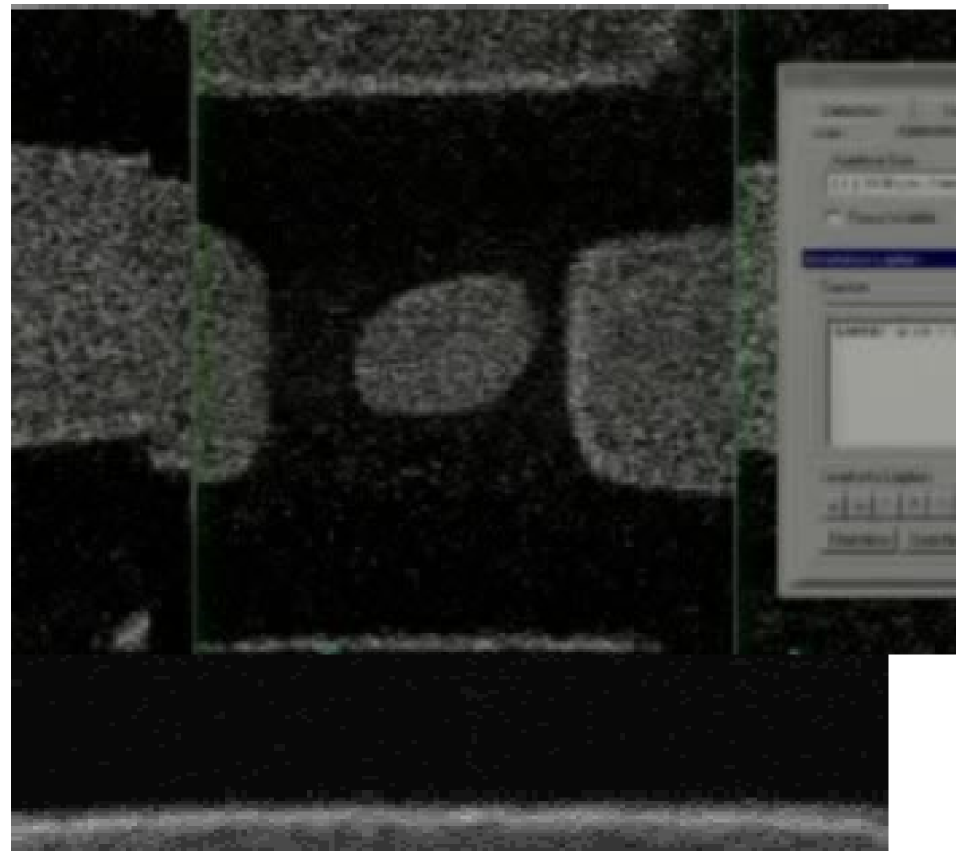


Nano Motor

- http://sciencematters.berkeley.edu/archives/volume2/issue11/story1.p - (2003)
- Micro motor (1988)



100 µm



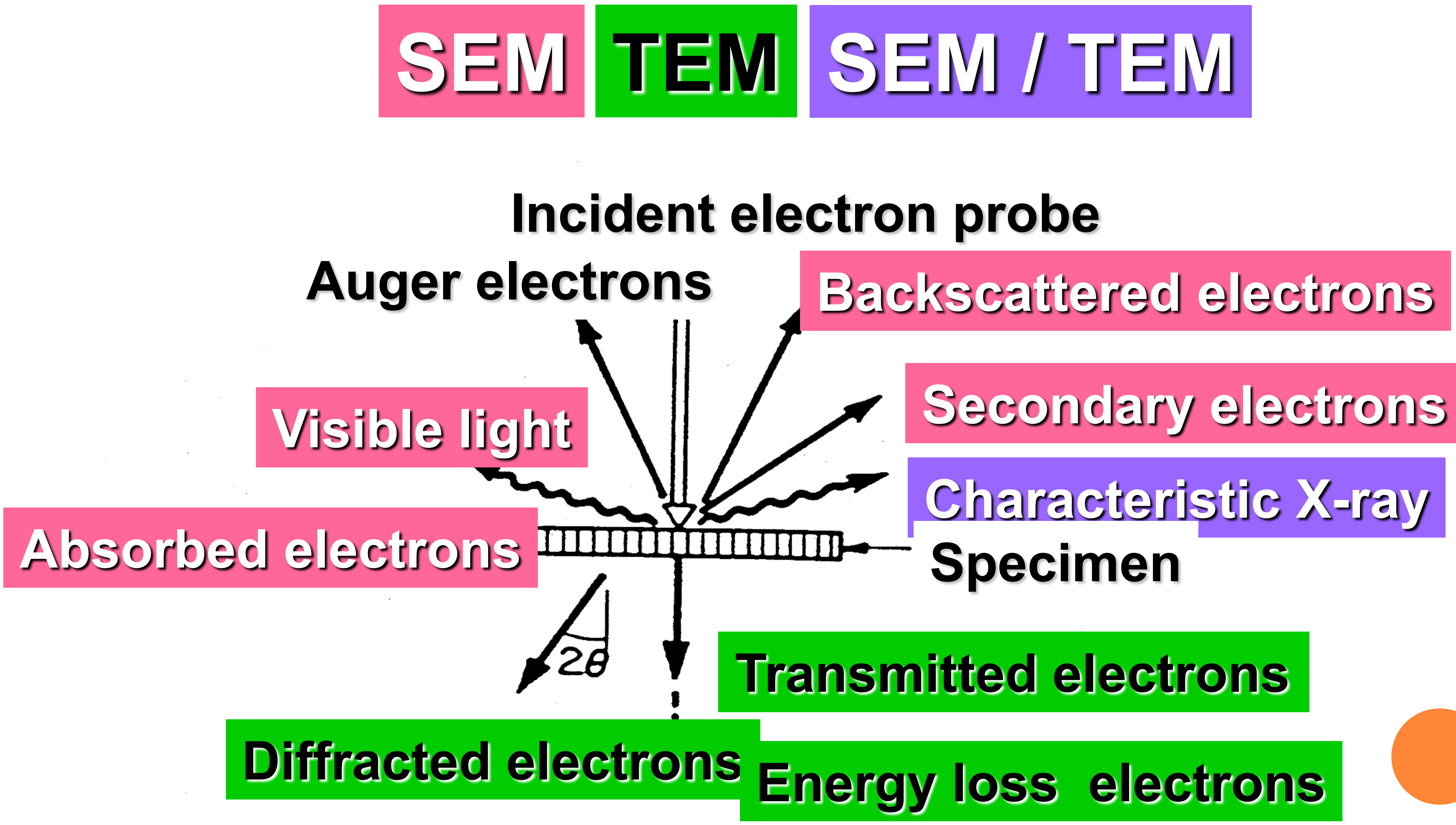
TEM ANALYSIS

ME118/218N
JongYoon HA

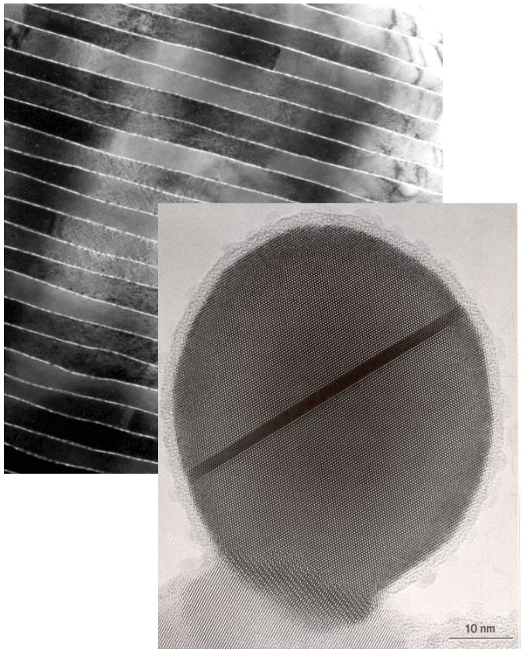
WHAT IS TEM

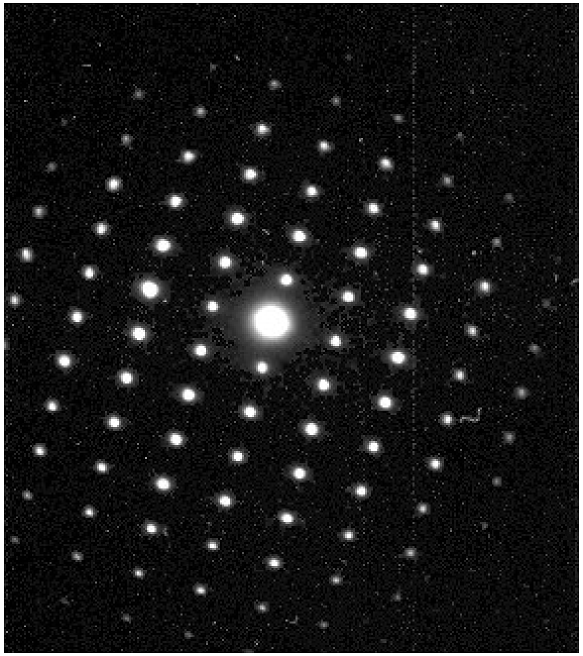
- **T**ransmission **E**lectron **M**icroscopy
- A microscopy technique whereby a beam of electrons(100 – 400 kV) is transmitted through an ultra thin specimen(10 – 500 nm), interacting with the specimen as it passes through it.
- Image resolution of 0.2 nm
- Electron probe size of about 0.5 nm

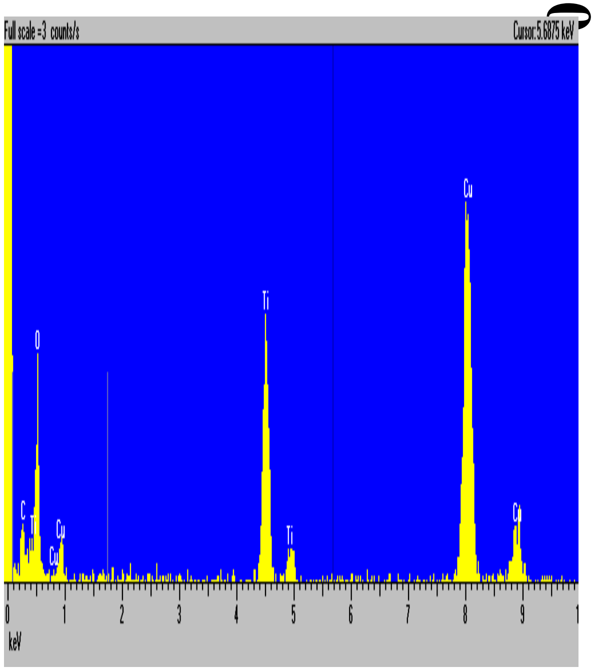
INTERACTION OF ELECTRONS WITH SPECIMEN



TEM IMAGING & DIFFRACTION







Imaging

- Bright field imaging
- Dark field imaging

Diffraction

Structure analysis

- Diffraction patterns
- Convergent beam electron diffraction
- Kikuchi diffraction

Spectroscopy

Chemical analysis

- Energy dispersive spectroscopy
- Electron energy loss spectroscopy

SAMPLE PREPARATION

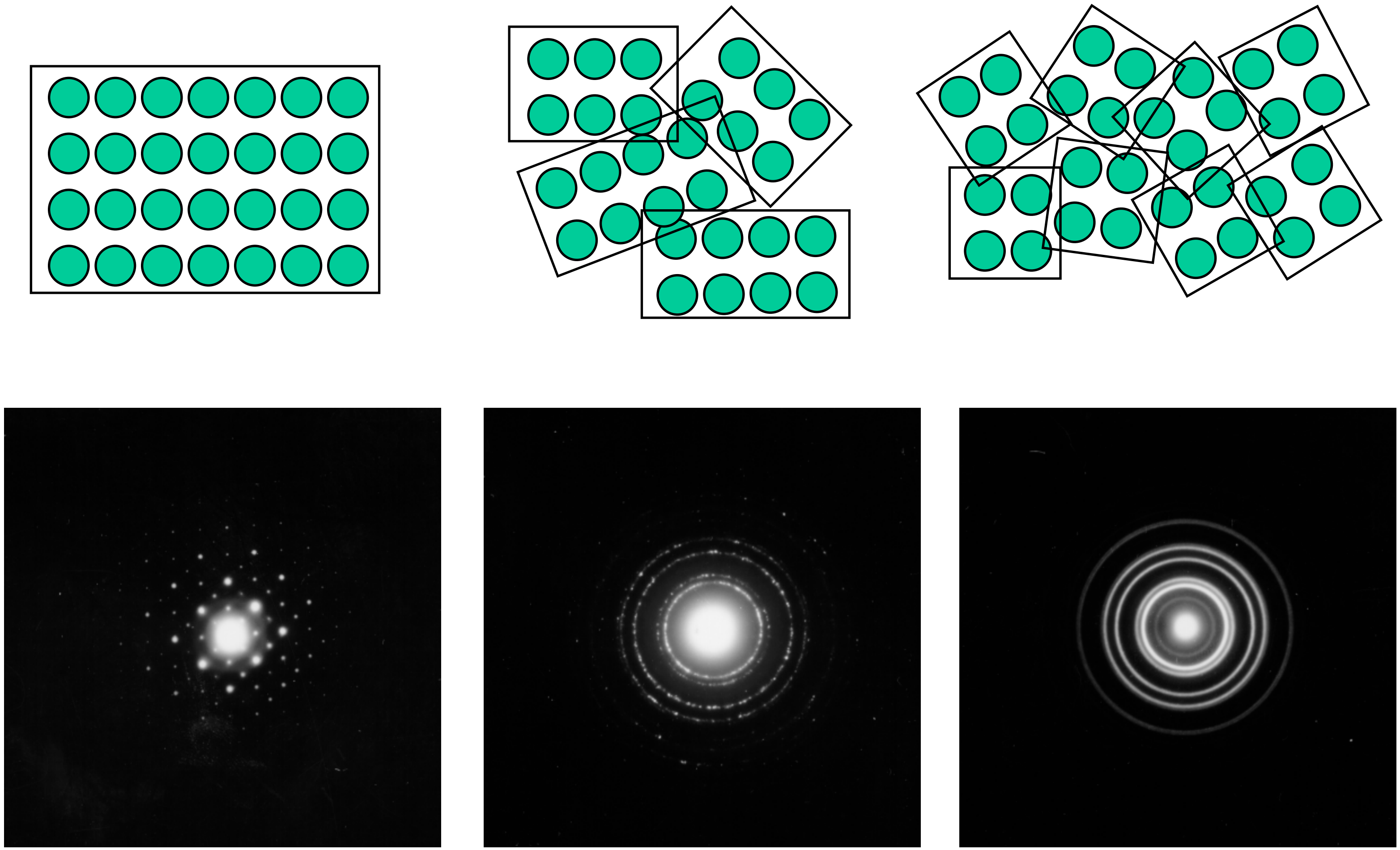
- Cut/Grinding/Polishing
- Electropolishing
- Ion beam milling
- Jet thinning
- Cleaving / crushing
- Replication
- Floating/Lift-off

CRYSTAL?

- In chemistry, mineralogy, and materials science, a **crystal** is a solid in which the constituent atoms, molecules, or ions are packed in a regularly ordered, repeating pattern extending in all three spatial dimensions.
- A **single crystal**, also called monocrystal, is a crystalline solid in which the crystal lattice of the entire sample is continuous and unbroken to the edges of the sample, with no grain boundaries.

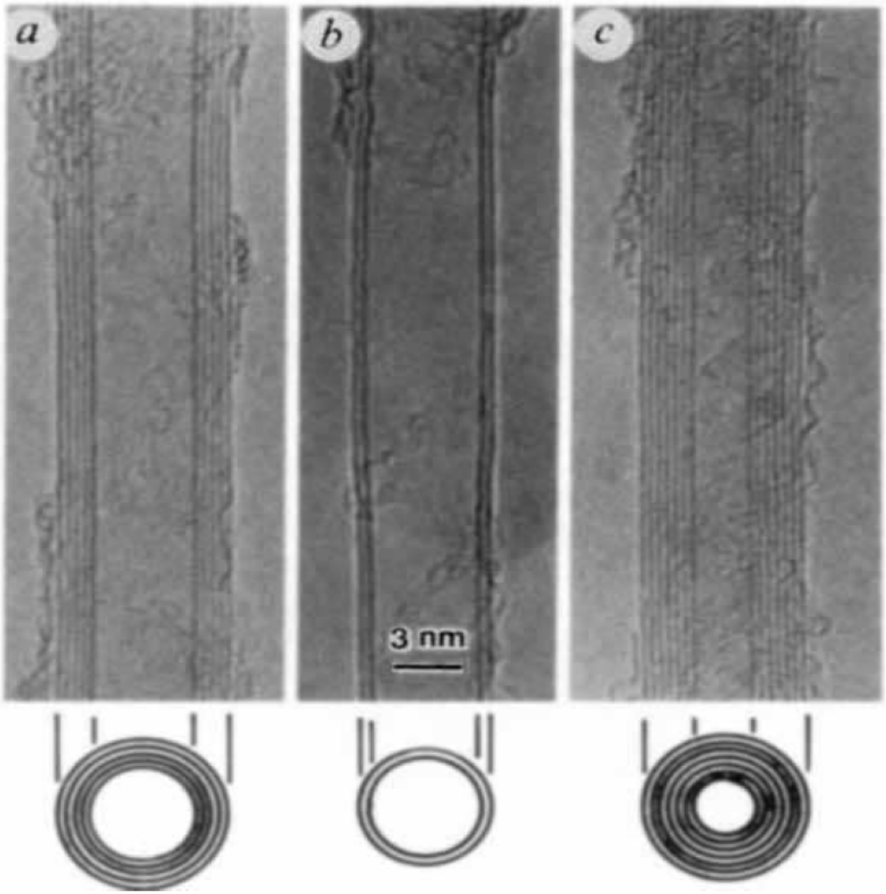
Ref : Wikipedia

DIFFRACTION METHODS IN THE TEM

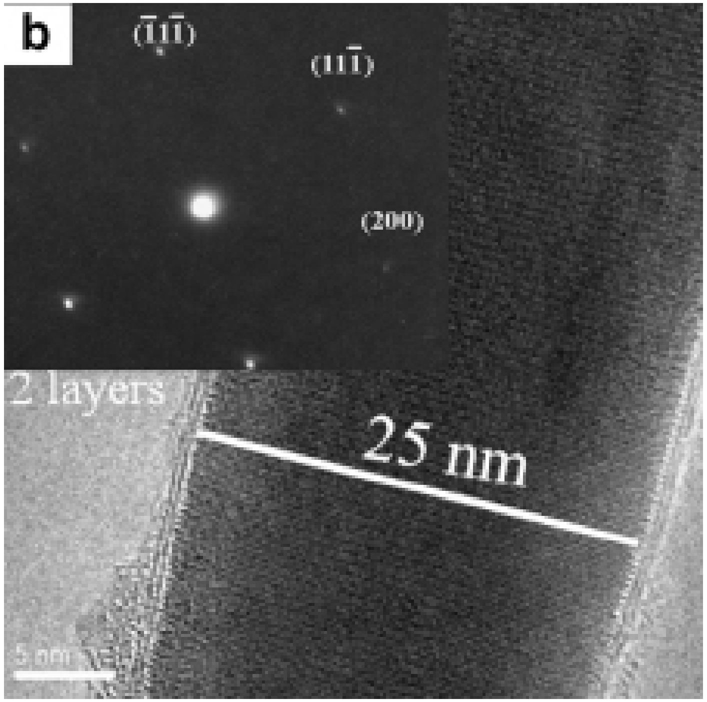


EXAMPLES

- Carbon Nano-Tubes



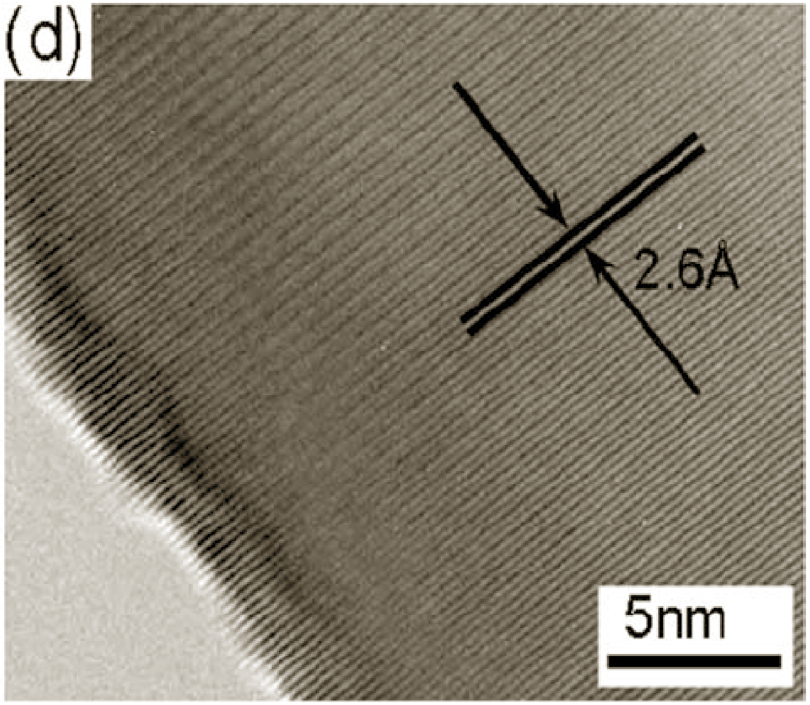
Ref : Sumio Iijima, Nature 354 56 (1991)

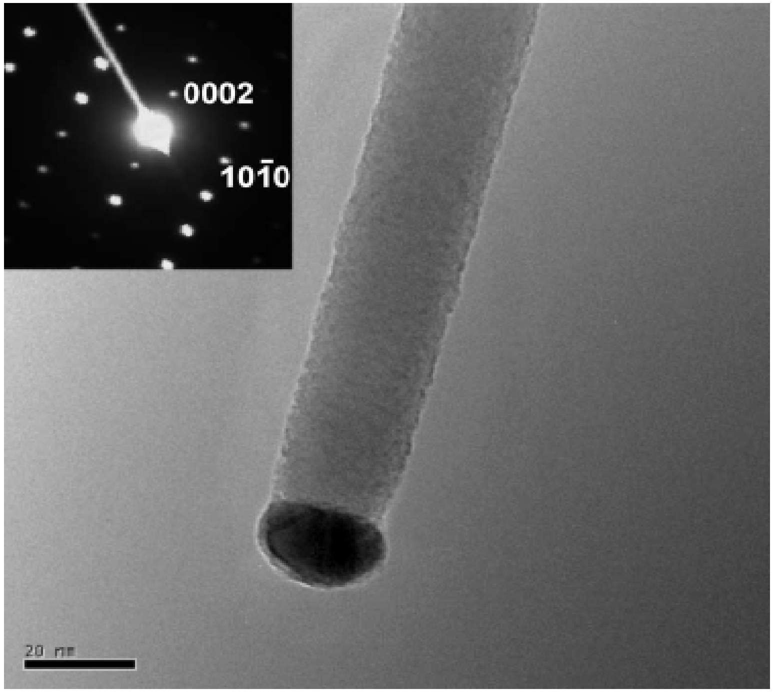


Ref : Wenxiang Wanga *et al*, Carbon 45 1127 (2007)

EXAMPLES

- Zinc Oxides



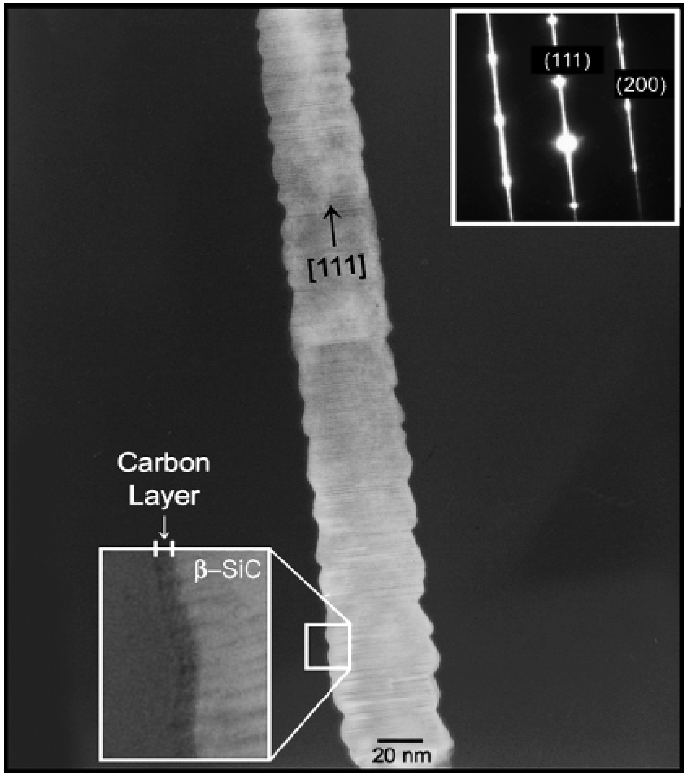


Ref : Lei Luo *et al*, APL 90 093101 (2007)

Ref : Y.W. Heo *et al*, Materials Science and Engineering R 47 1 (2004)

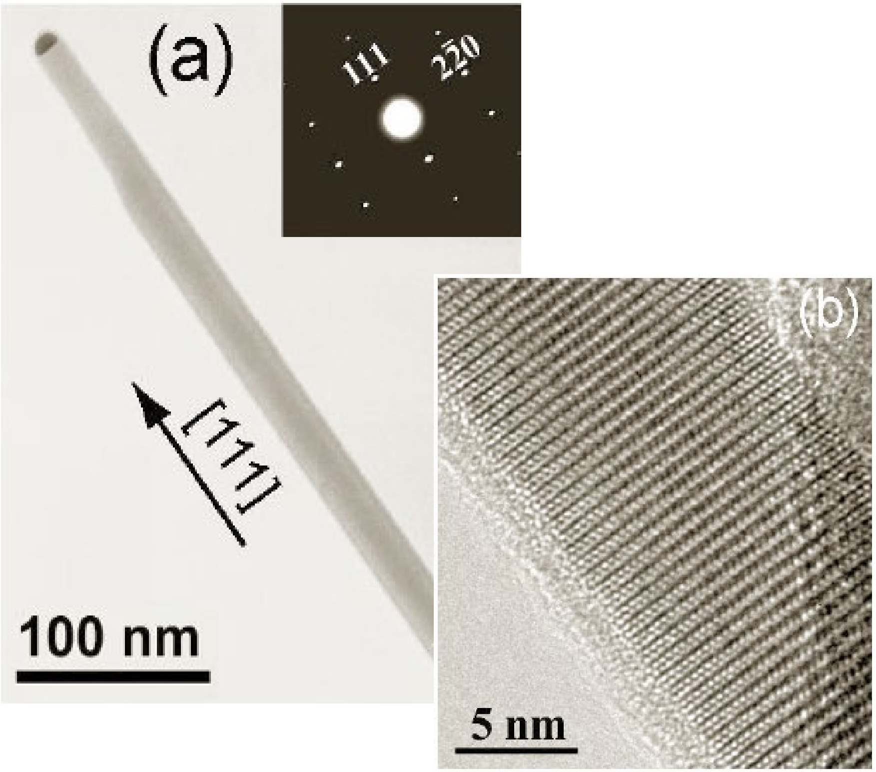
EXAMPLES

- SiC



Ref : B.-C. Kang *et al*, Thin Solid Films 501 181 (2006)

- GaAs



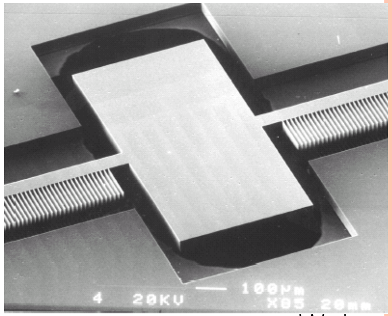
Ref : GaAs nanowires grown by molecular-beam epitaxy

CONCLUSION

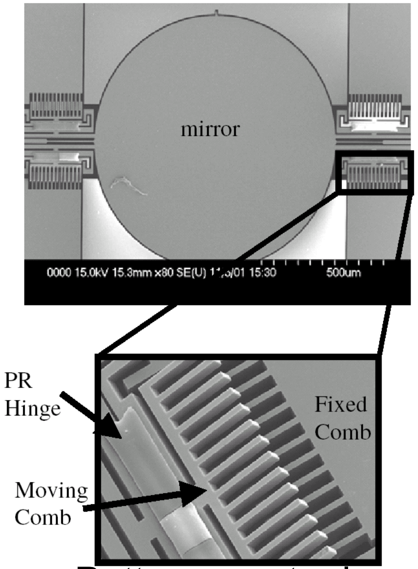
TEM, high spatial resolution, capability of electron diffraction and spectroscopy, is an essential tool for the characterization of nanostructure materials.

SOI – Vertical Actuator Example

- Vertical Comb Actuators have the following advantages:
  - Larger force
  - Longer actuating distance
  - Linear driving force (No Pull-in)
- Our objectives are:
  - Angular Vertical Comb actuator fabricated by silicon plastic deformation process
    - All the structures are robust and reliable single crystal silicon

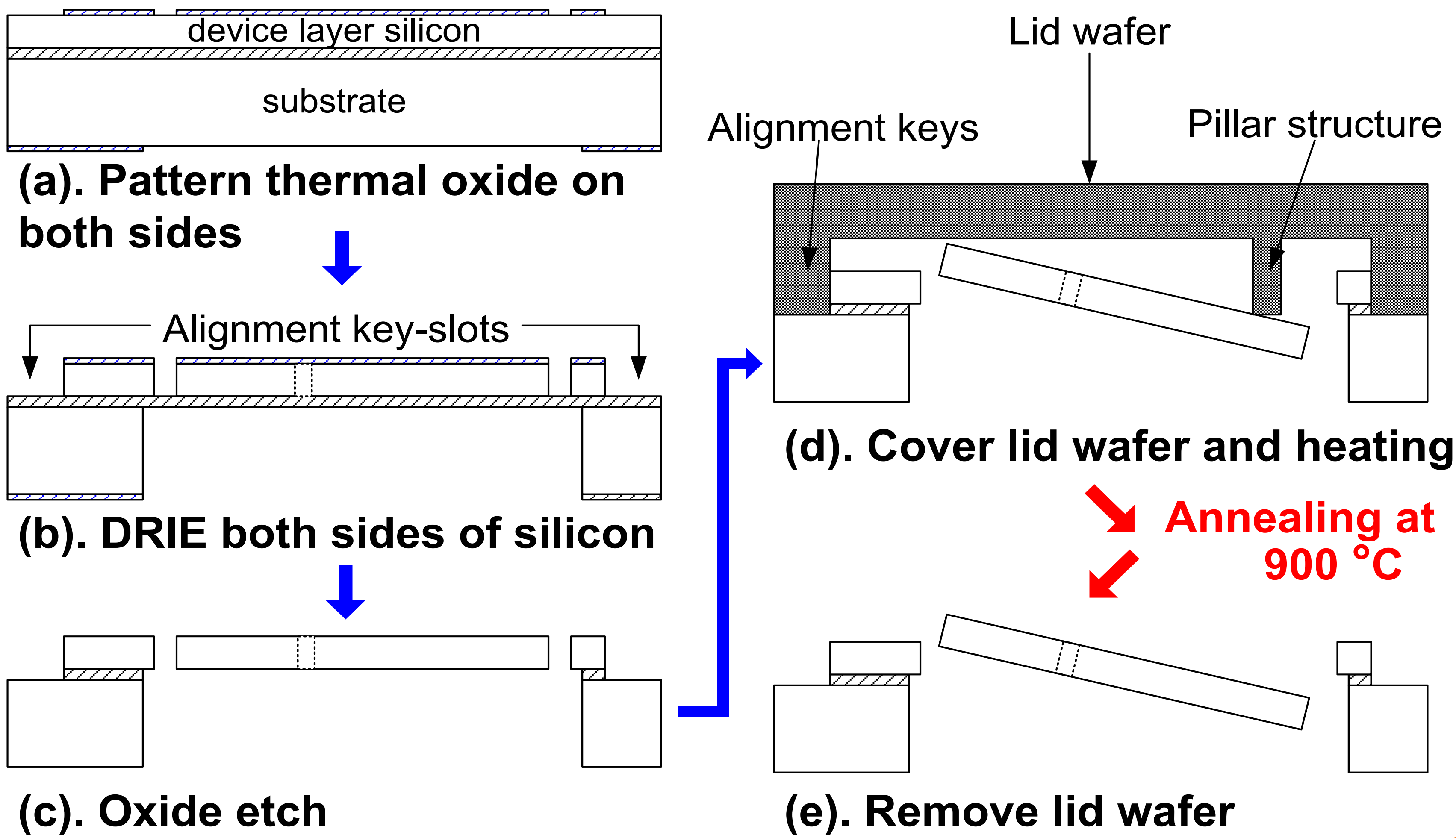


Conant, et.al.
Hilton Head 2000

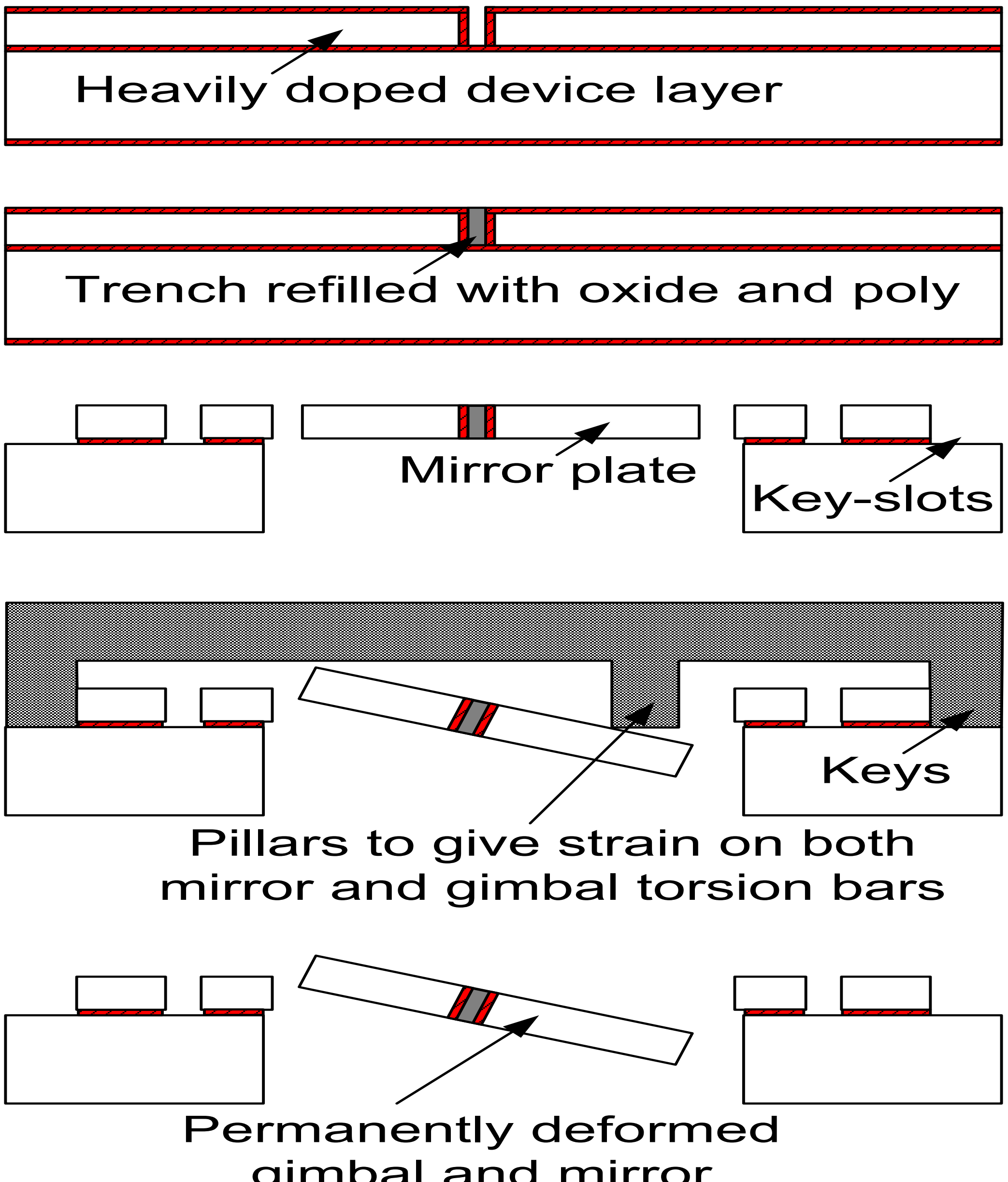


Patterson, et. al.,
MEMS 2002

Fabrication Process (1)



Fabrication Process (2)



(a) Trench etch and oxidation (for electrical isolation)

(b) Poly deposition (for mechanical connection) etch-back

(c) Pattern and DRIE on both side followed by Etch oxide to release the structure

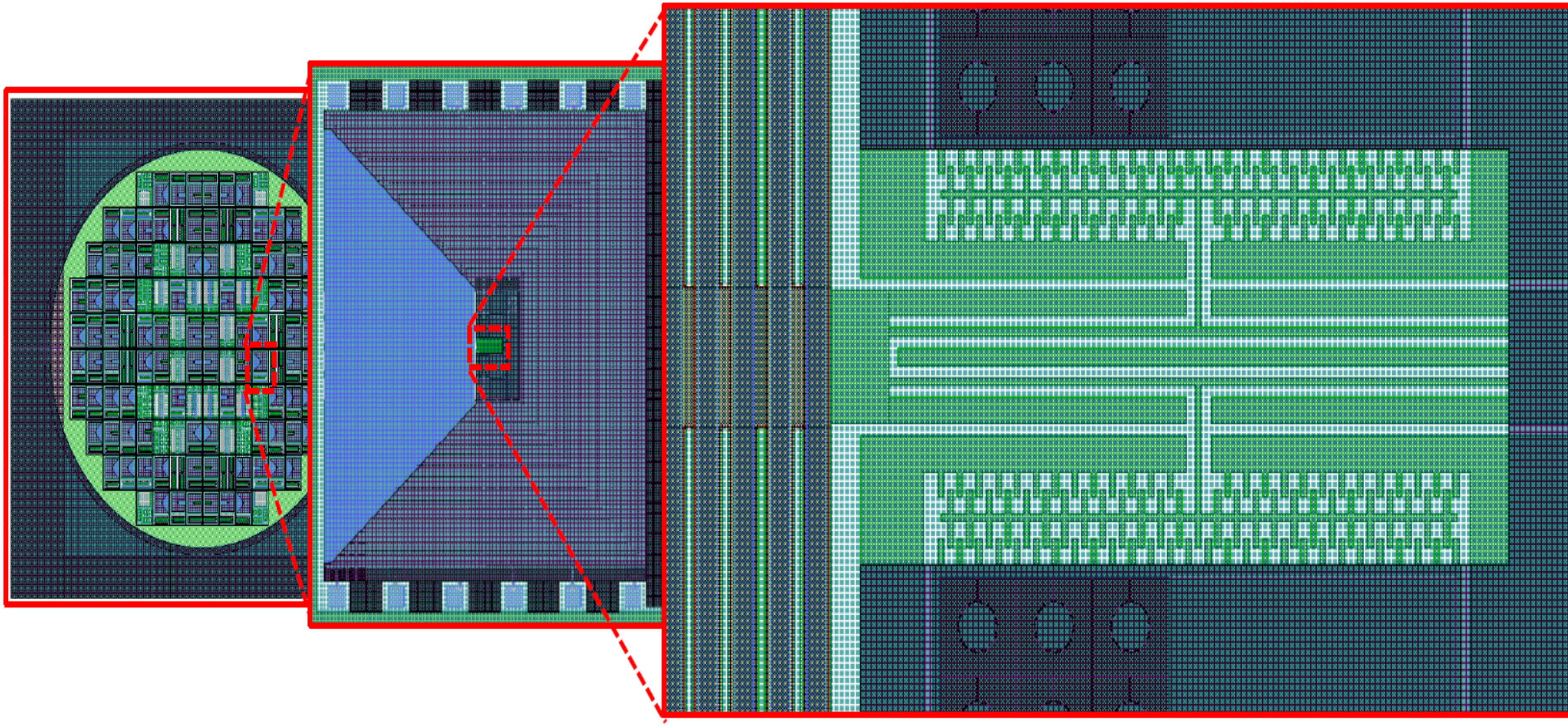
(d) Place separately prepared lid wafer on top of device wafer and anneal at high temperature (900 °C)

(e) Separate the device wafer and the lid wafer (flexures are deformed permanently)

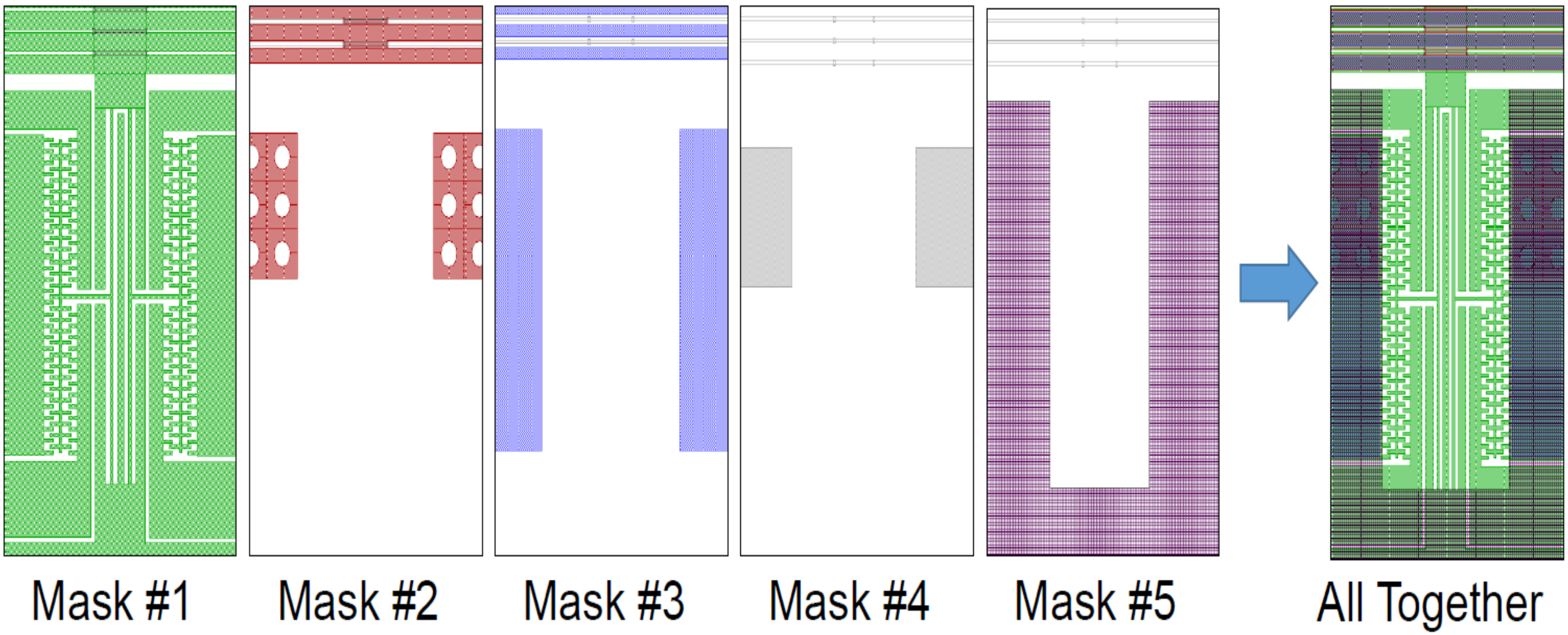
2-D Scanning Mirror

2D-Scanning Results

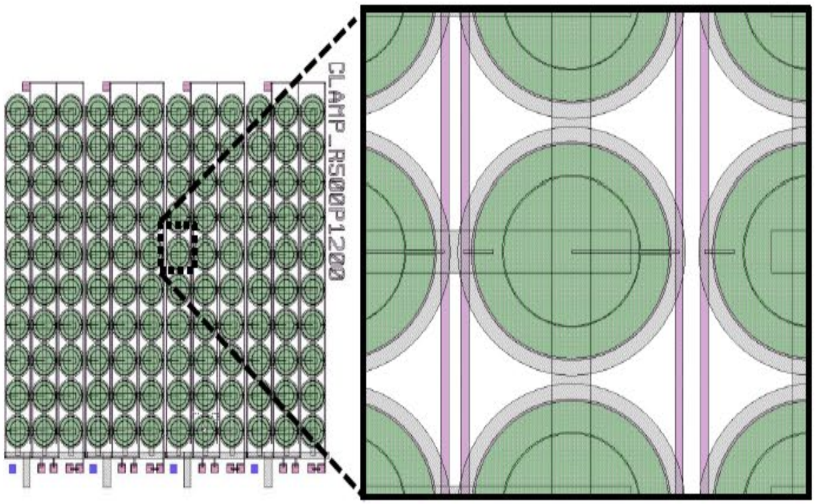
Microfabrication – Mask Layout



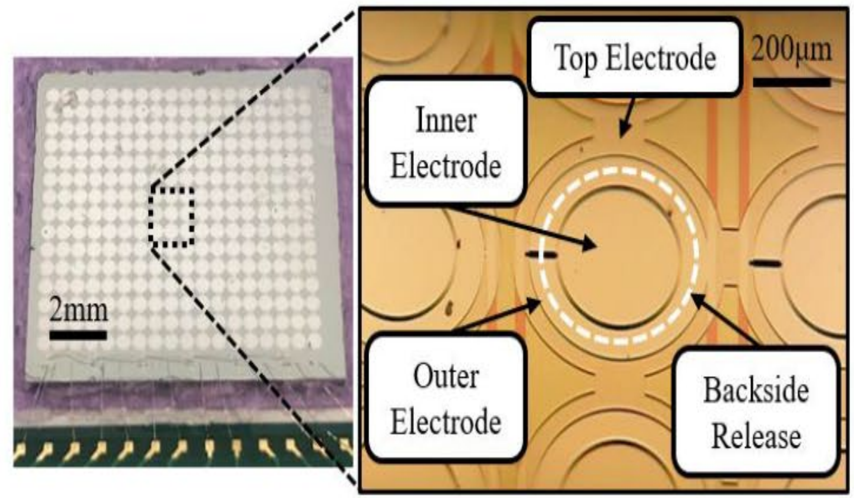
Microfabrication – Mask Layout



Microfabrication

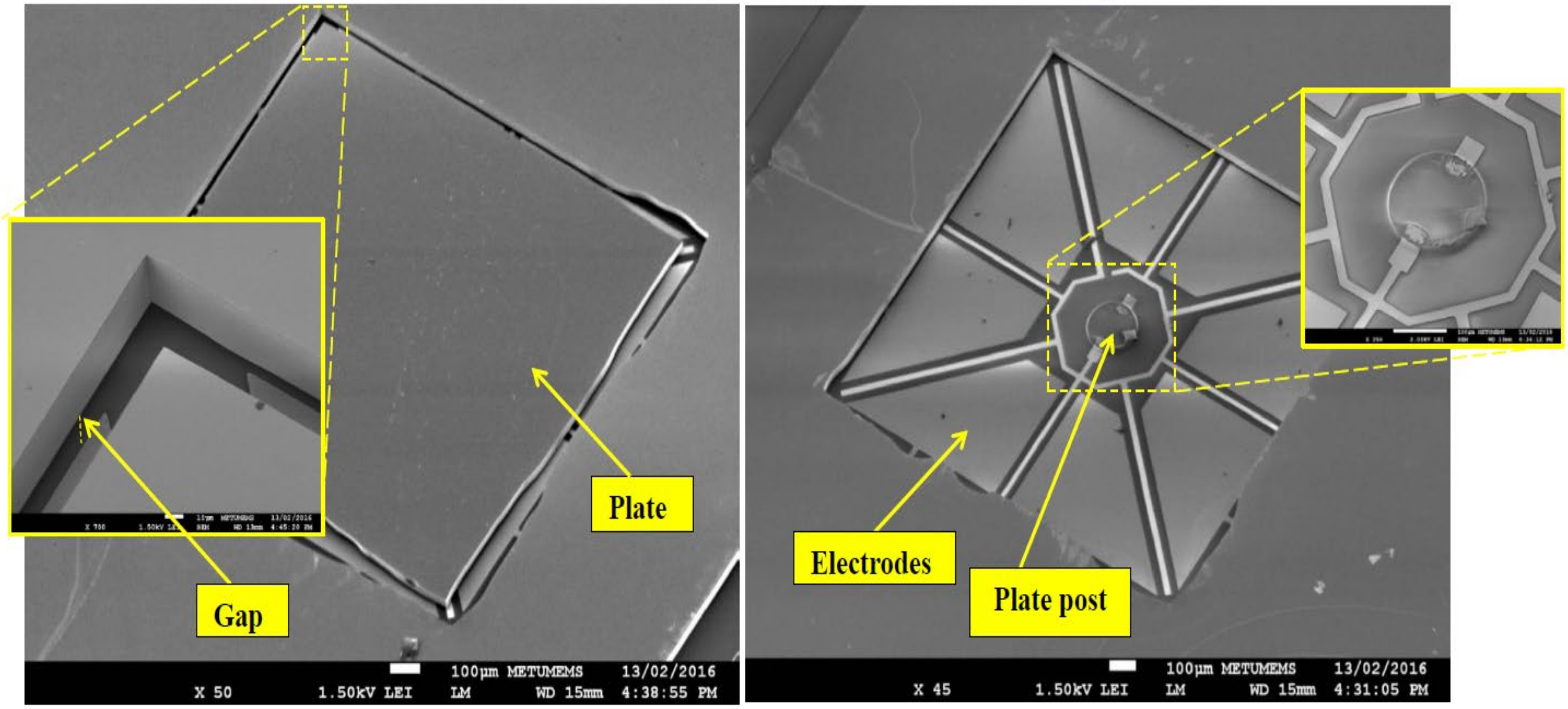


Design



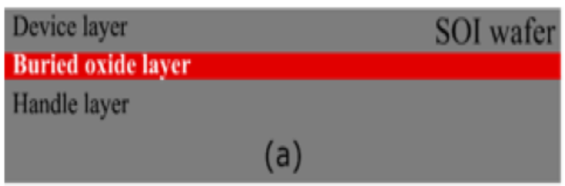
Fabricated

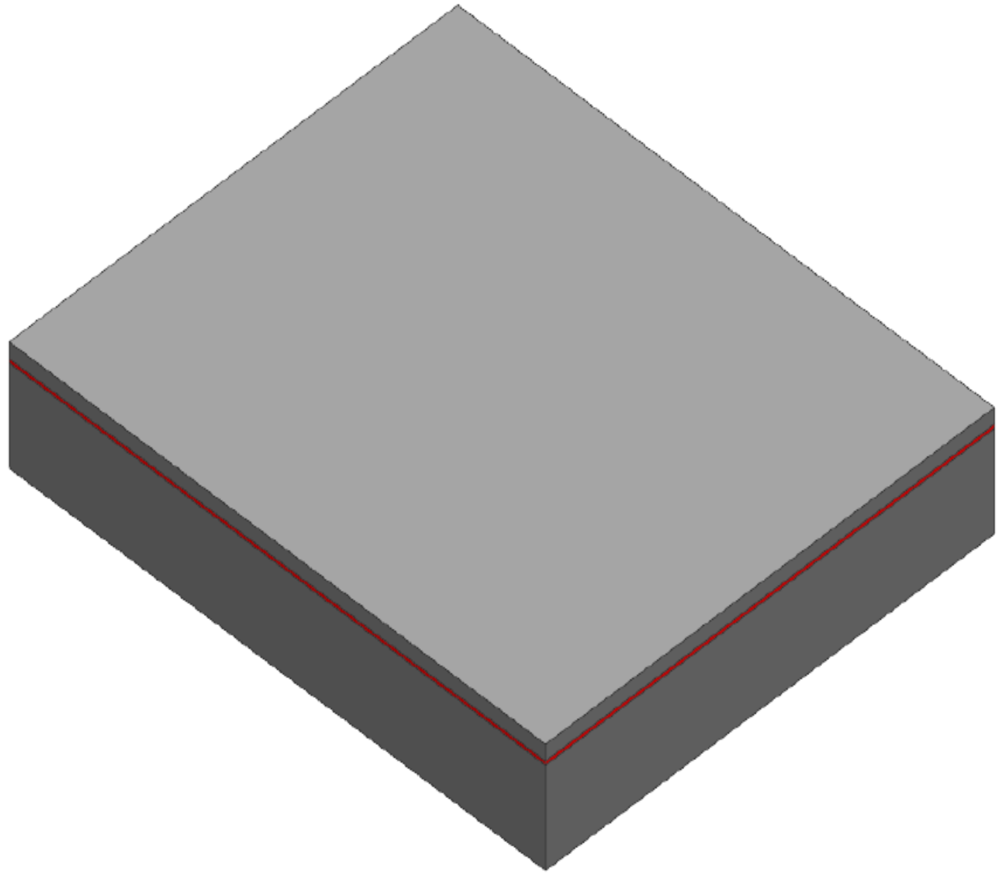
Microfabrication – MEMS Fabrication Steps



Scanning Electron Microscope (SEM) images.

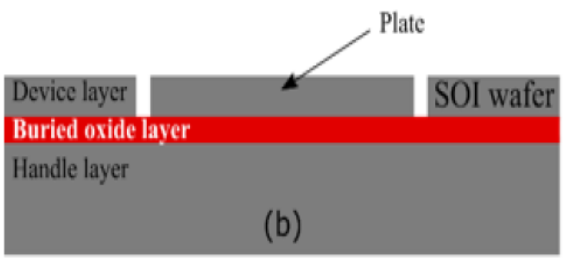
Microfabrication – MEMS Fabrication Steps

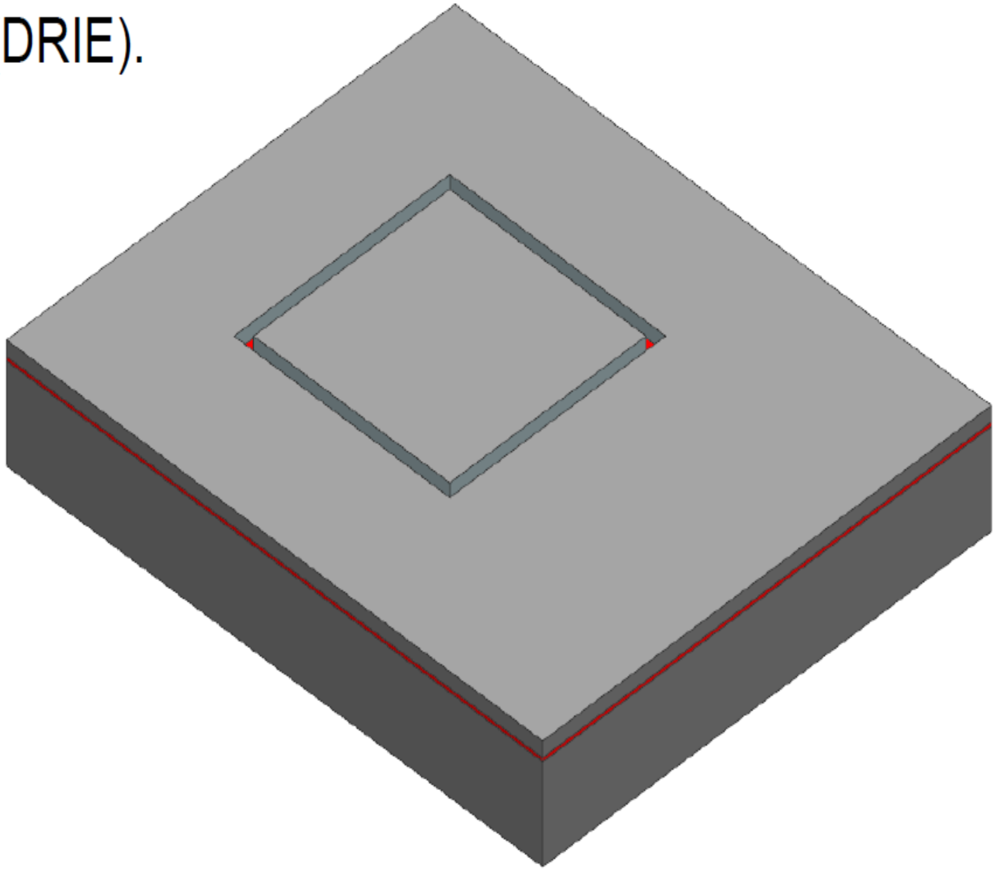




Microfabrication – MEMS Fabrication Steps

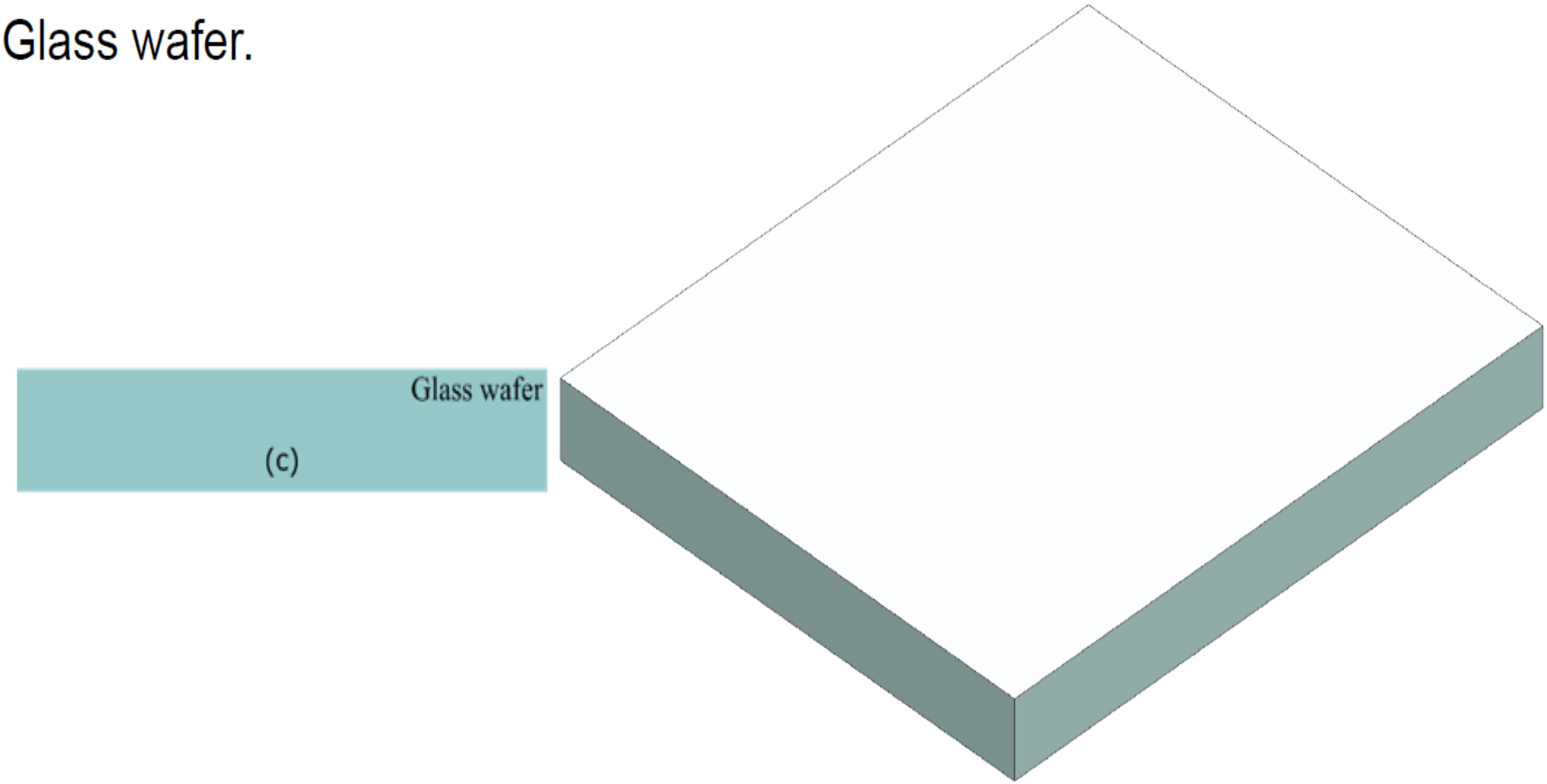
Deep Reactive Ion Etching (DRIE).





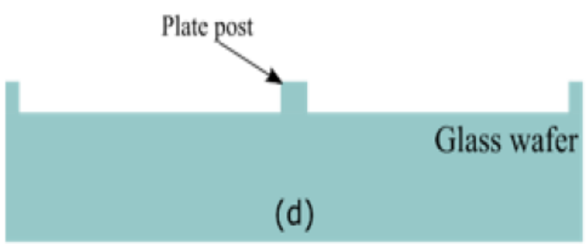
Microfabrication – MEMS Fabrication Steps

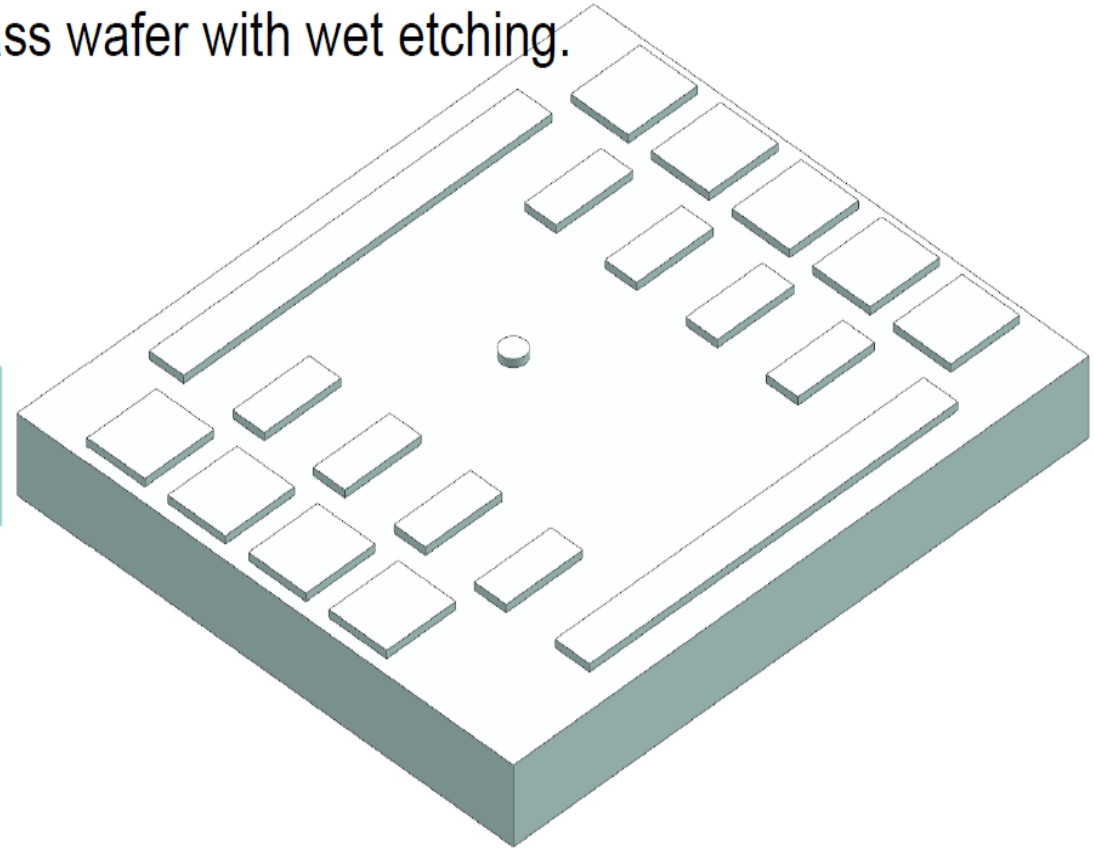
Glass wafer.



Microfabrication – MEMS Fabrication Steps

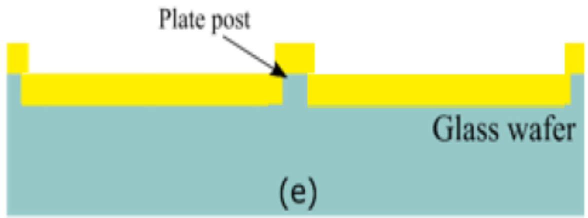
Anchors defined on the glass wafer with wet etching.

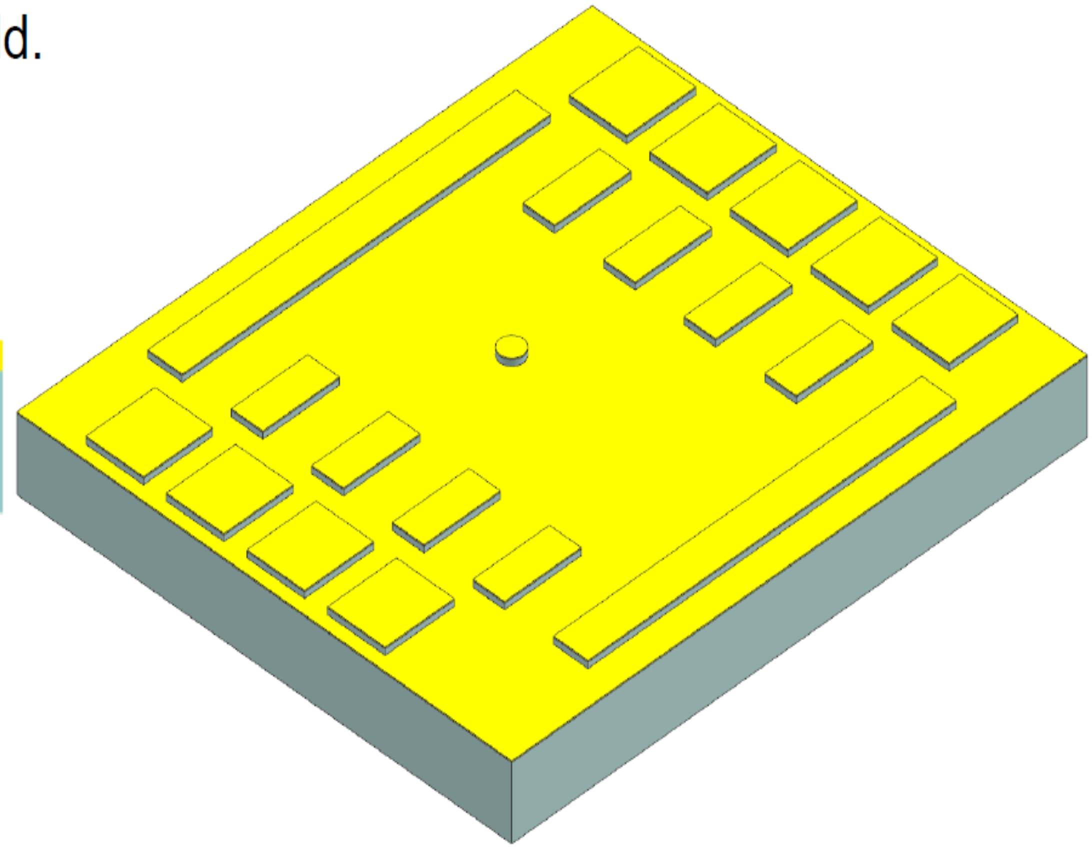




Microfabrication – MEMS Fabrication Steps

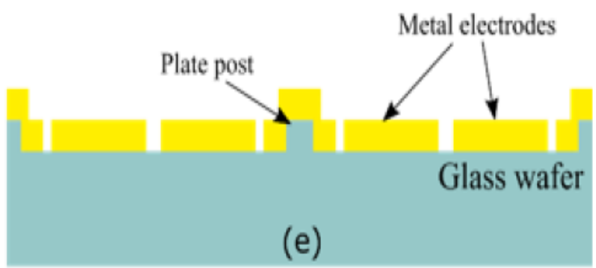
Thermal evaporation of gold.

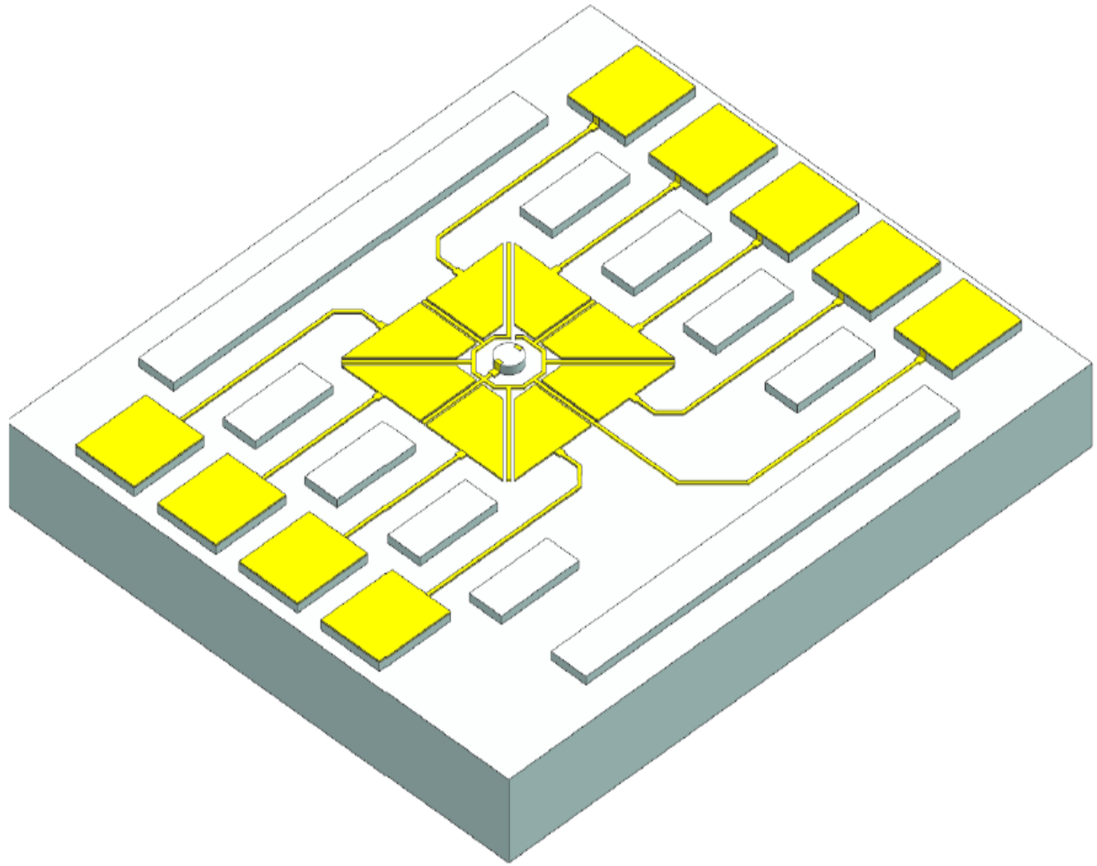




Microfabrication – MEMS Fabrication Steps

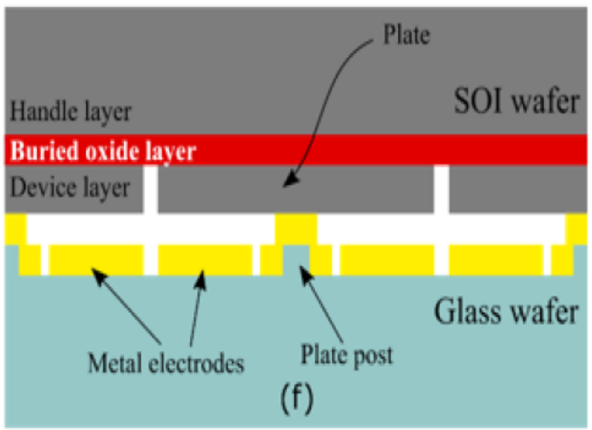
Metal etching.

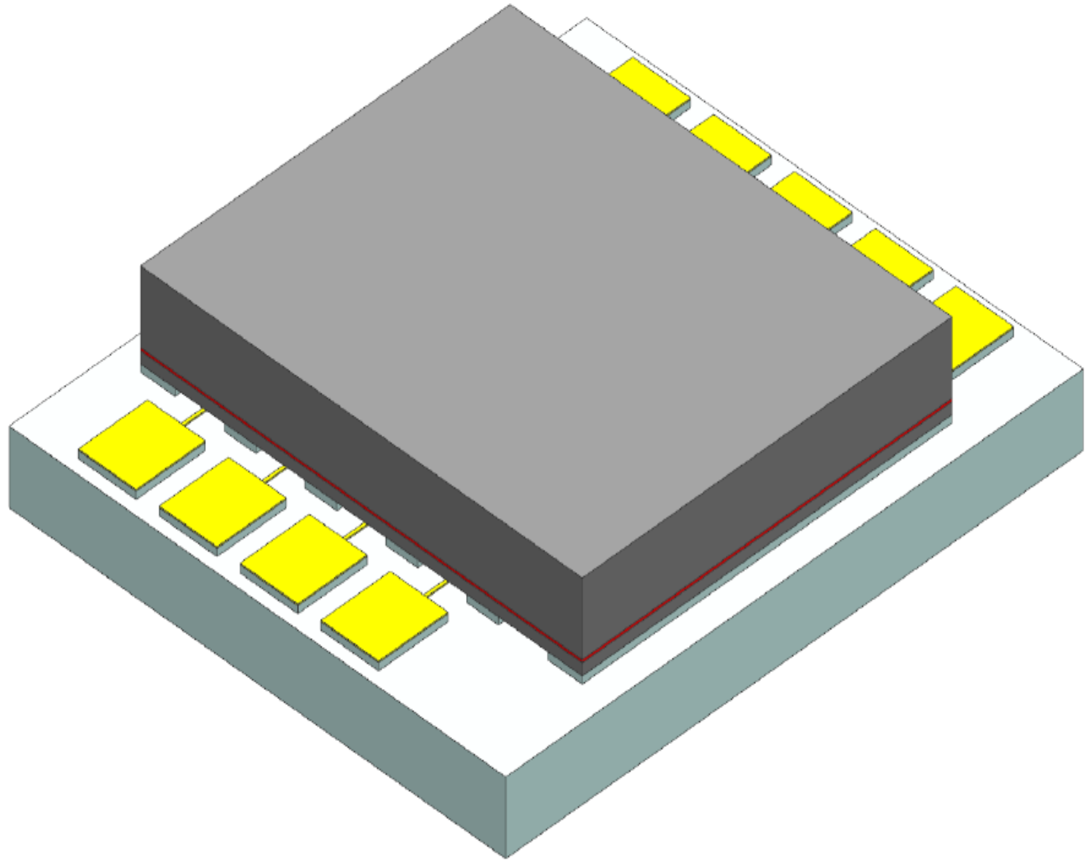




Microfabrication – MEMS Fabrication Steps

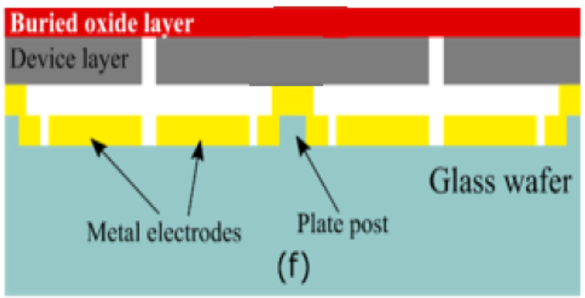
Anodic bonding.

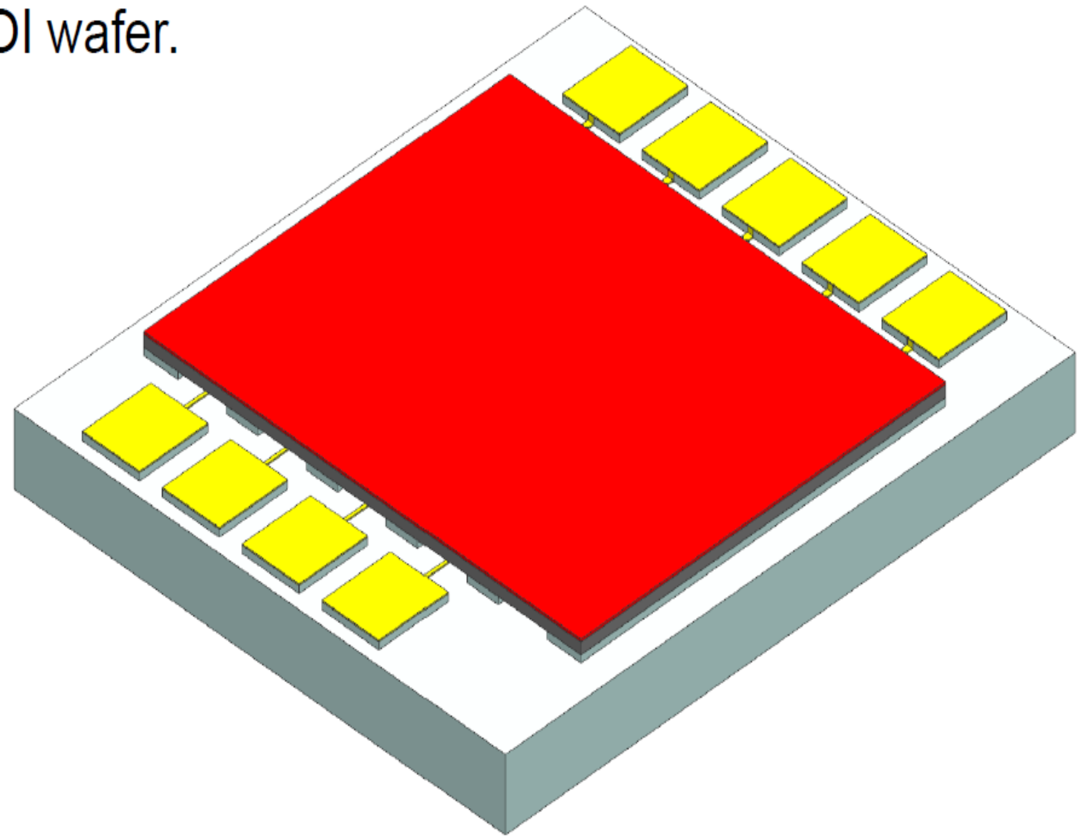




Microfabrication – MEMS Fabrication Steps

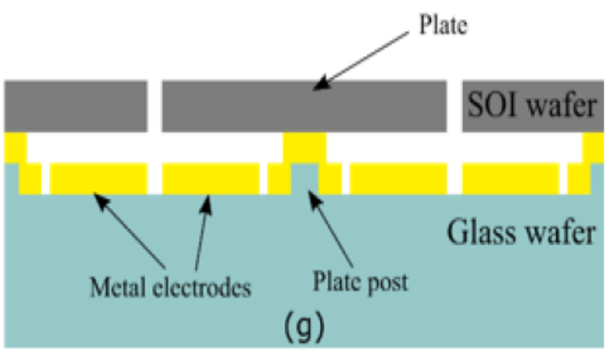
Handle layer removal of SOI wafer.

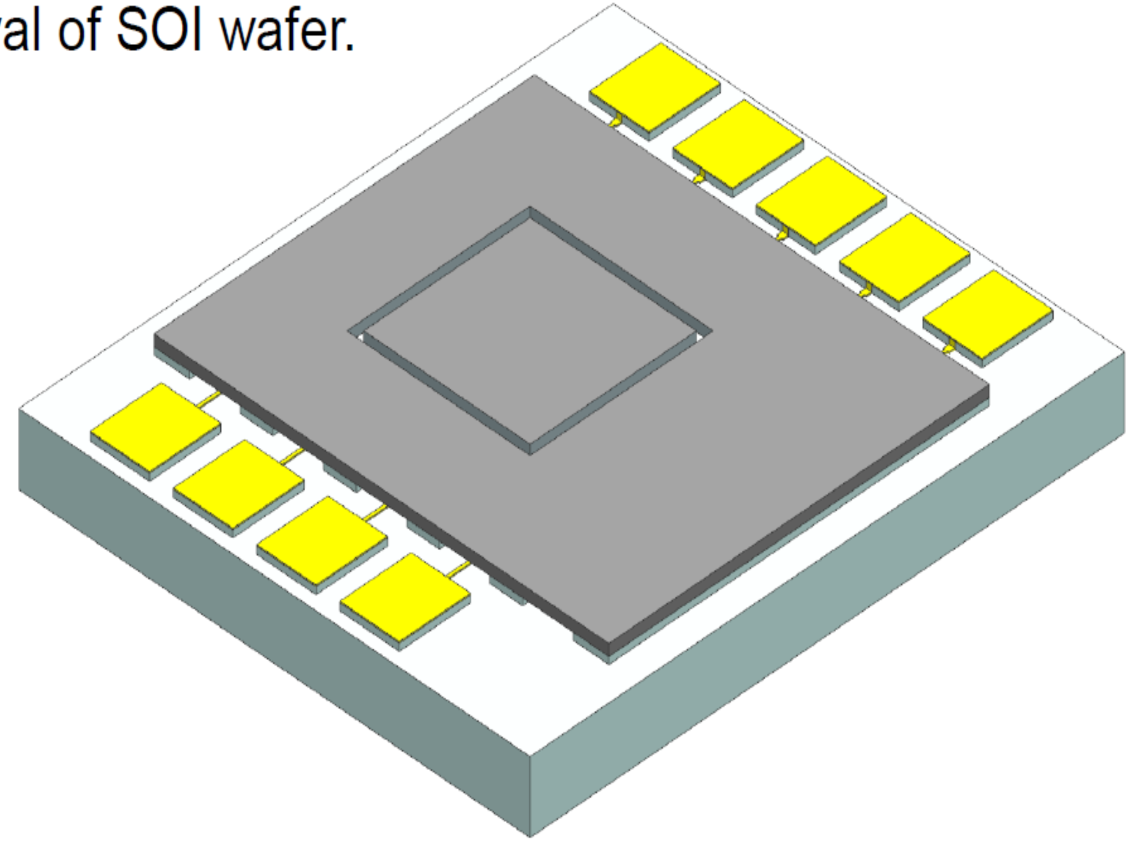




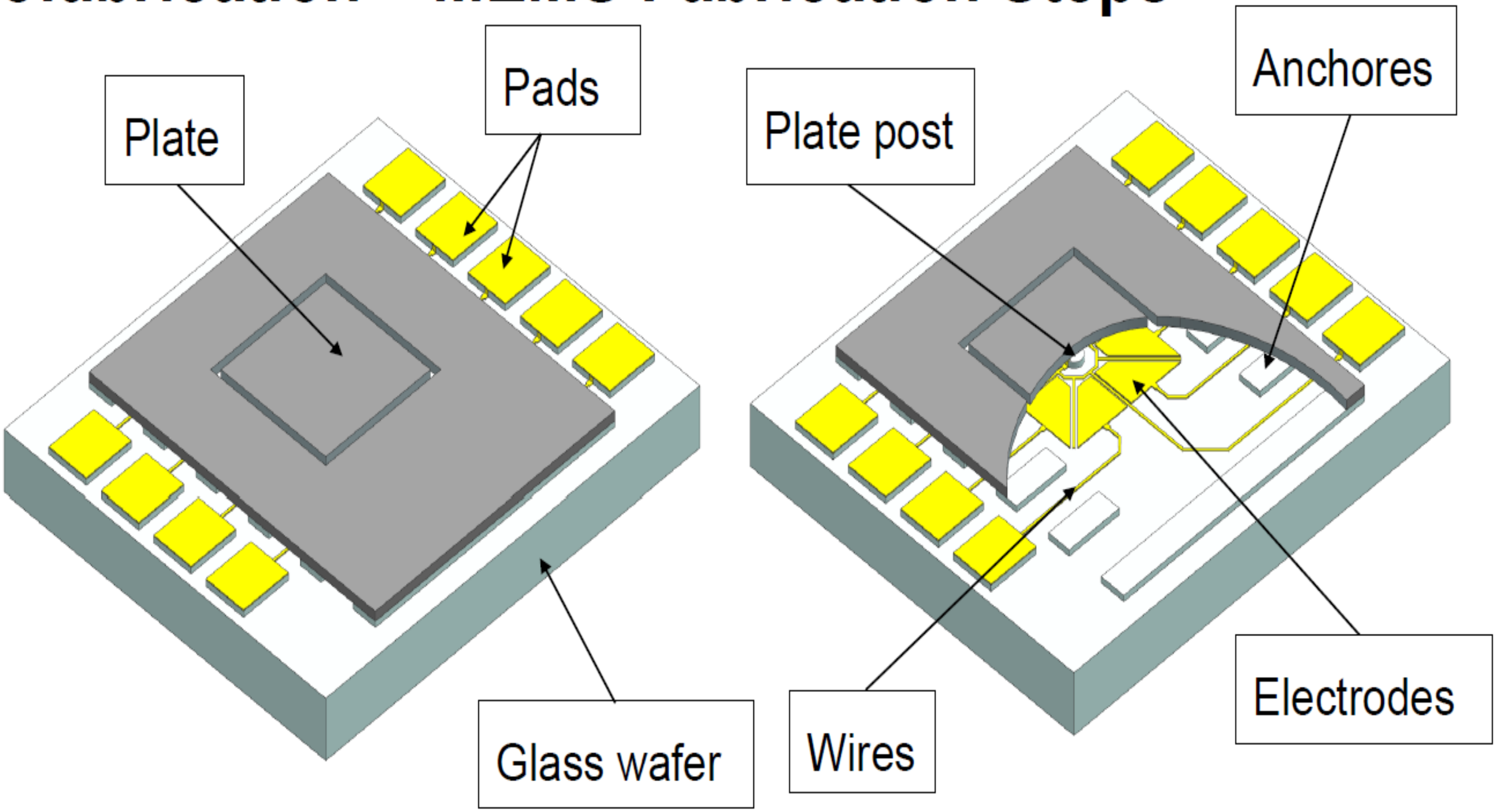
Microfabrication – MEMS Fabrication Steps

Buried oxide layer removal of SOI wafer.

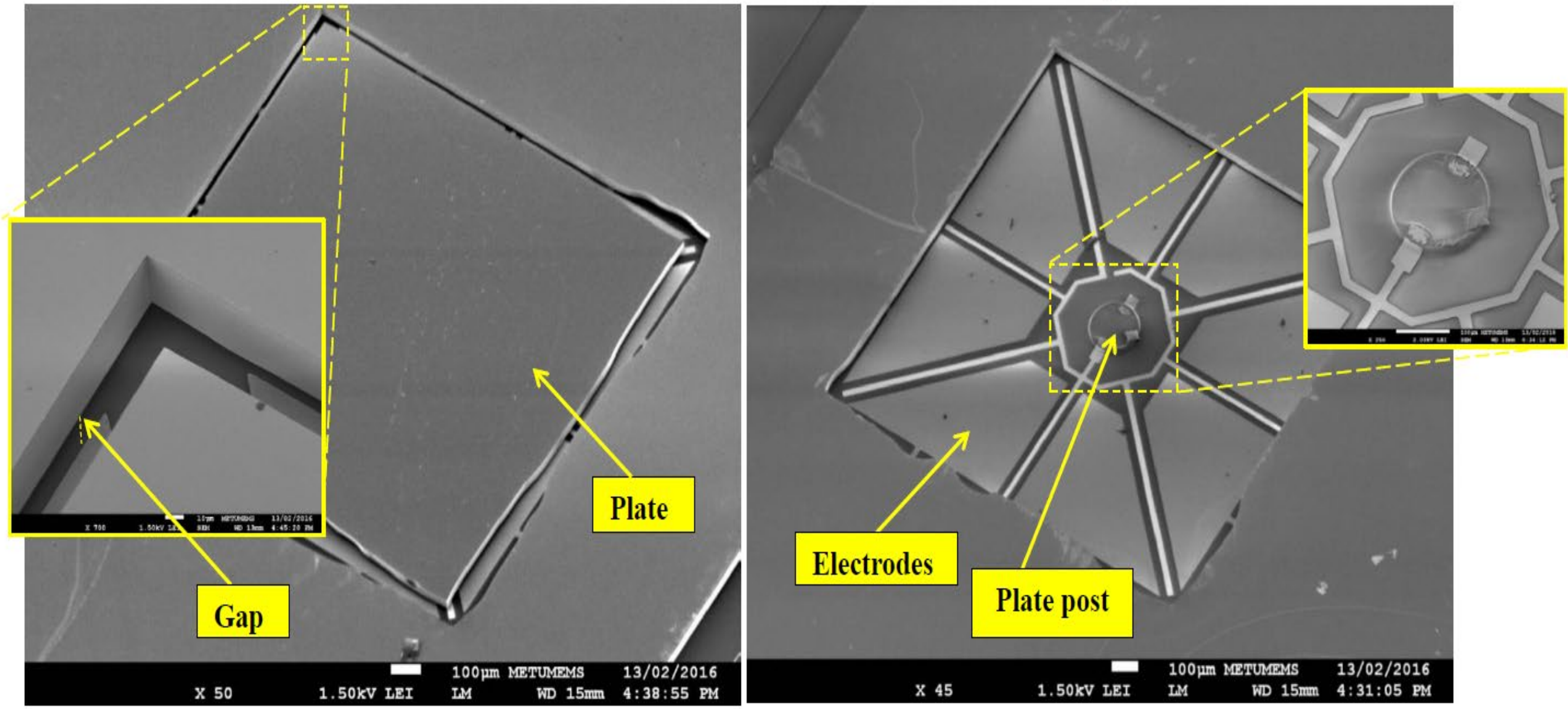




Microfabrication – MEMS Fabrication Steps



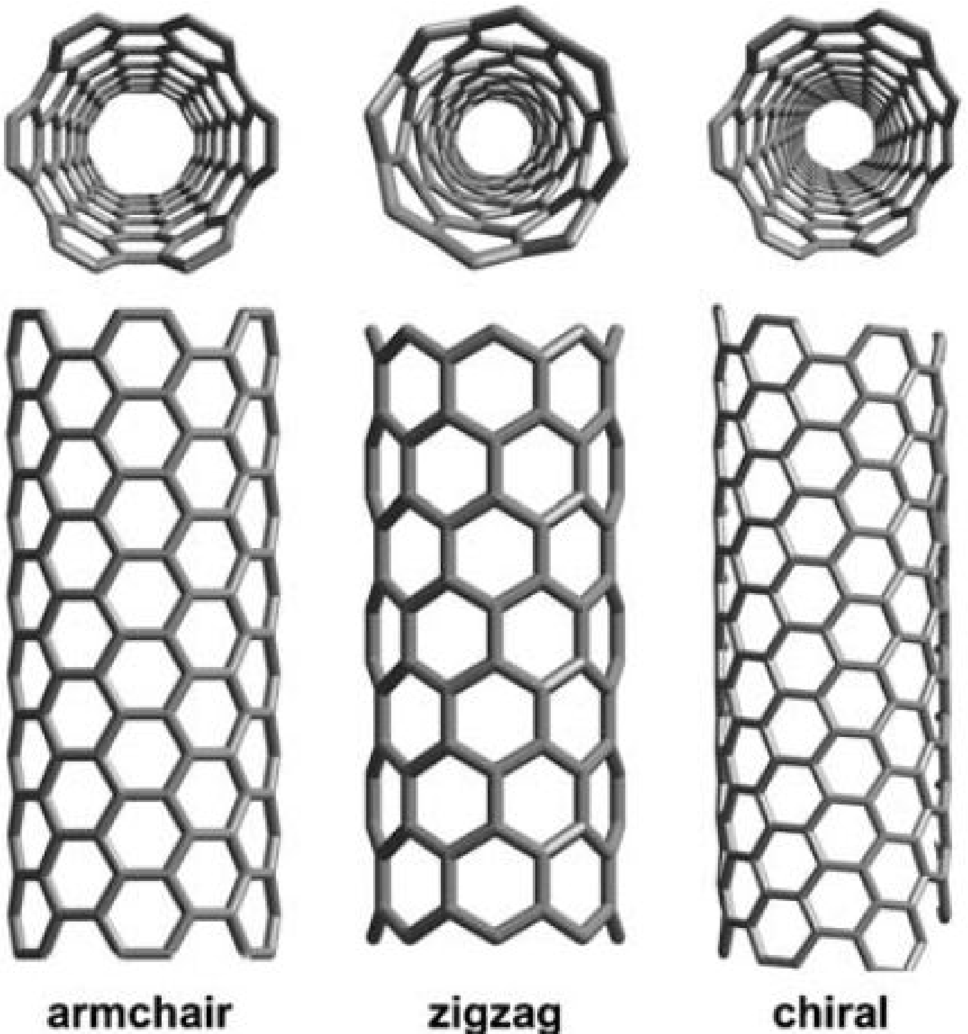
Microfabrication – MEMS Fabrication Steps



Scanning Electron Microscope (SEM) images.

Tubule Structure

http://carbonlab.roma2.infn.it/



armchair	zigzag	chiral
$\alpha = 30°$	$\alpha = 0°$	$0° < \alpha < 30°$
Metallic	**Semi-conductor**	**Semi-conductor**

How do we Study the Structure of Carbon nanotubes ?

Electron Micrograph	Electron Diffraction
An image obtained by bombarding the specimen with a finely focused (<10 nm diameter) electron beam with an acceleration voltage under vacuum, and detecting the transmitted, secondary, backscattered and diffracted electrons, and characteristic X-rays emitted.	Electron diffraction is a technique that allows determination of the crystal structure of materials. When the electron beam is projected onto a specimen, its crystal lattice acts as a diffraction grating, scattering the electrons in a predictable manner, and resulting in a diffraction pattern.
Source : Royal Society of Chemistry 2024	Source : Science Direct| Electron Diffraction - An Overview

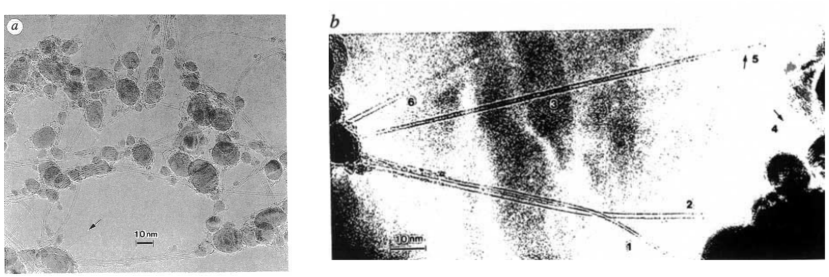


Figure 1 : Electron micrograph showing bundles of single-shell carbon nanotubes which are curved and entangled.

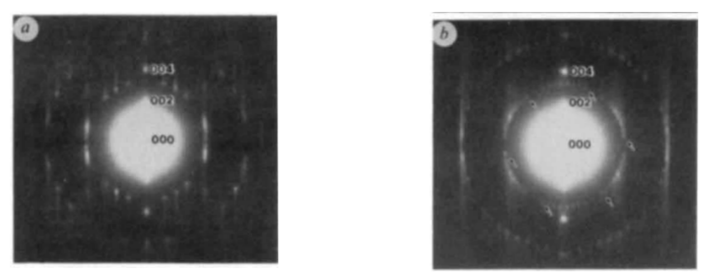


Figure 2 : Electron diffraction patterns from individual microtubules of graphitic carbon.

Source: Iijima, Sumio, and Toshinari Ichihashi. "Single-shell carbon nanotubes of l-nm diameter." Fundamental Research Laboratories, NEC Corporation, 34 Miyukigaoka, Tsukuba, Ibaraki 305, Japan.

Structure of Carbon nanotubes

- Electron Microscopy reveals
  - Coaxial tubes of graphitic sheets
  - Ranging in number from 2 up to about 50
- Diameter of the Needle Ranges from 4 to 30nm and up to 1 micrometre in length
- High Resolution Micrographs of typical Needles Shows that its Seamless and Tubular Structure (Same number of Lattice Fringes from both the sides of the needle)
- The tip of the Needles are usually closed by caps that are curved , polygonal or cone shaped
- The last of these have specific opening angles of about 19 degrees or 40 Degrees

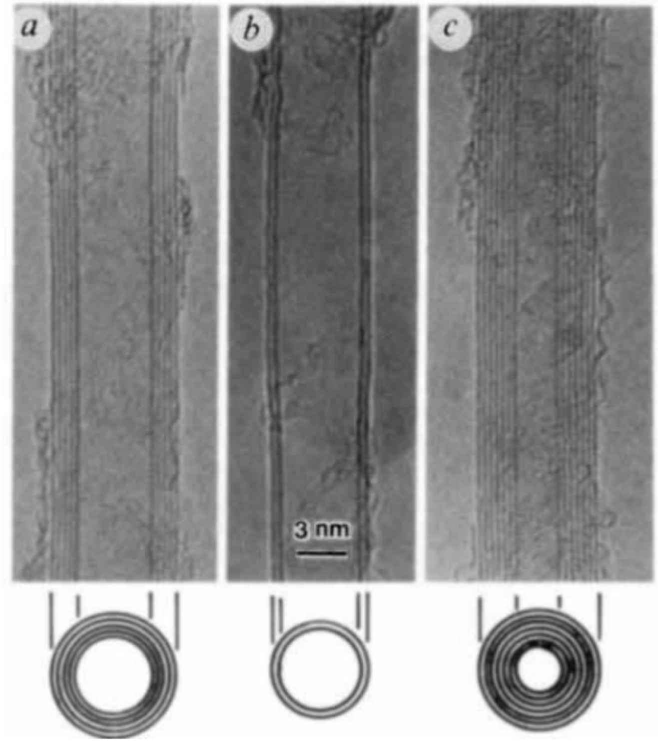
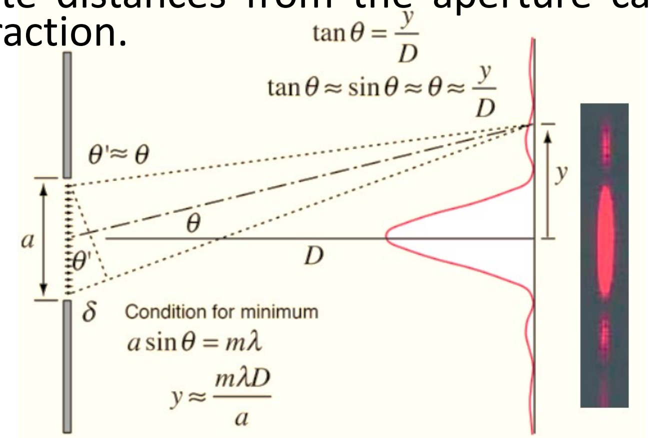


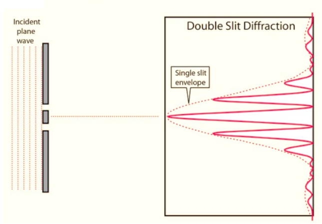
Figure 3 : Electron Micrographs of Microtubules of Graphitic Carbon

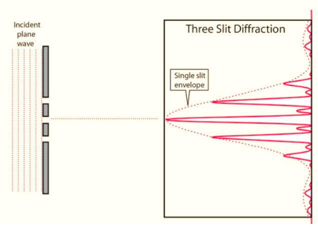
Source: Iijima, Sumio, and Toshinari Ichihashi. "Single-shell carbon nanotubes of l-nm diameter." Fundamental Research Laboratories, NEC Corporation, 34 Miyukigaoka, Tsukuba, Ibaraki 305, Japan.

Fraunhofer Diffraction

Fraunhofer diffraction deals with the limiting cases where the source of light and the screen on which the pattern is observed are effectively at infinite distances from the aperture causing the diffraction.







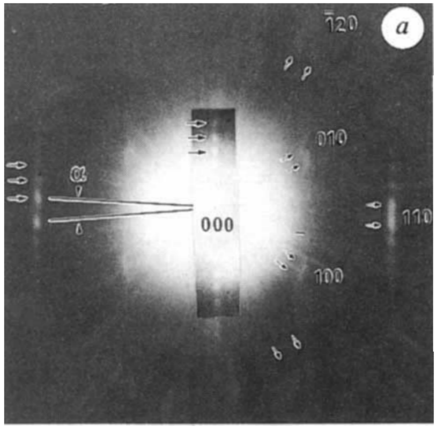


Figure 1 : Electron Diffraction Pattern from a Single Shell Nano Tubule

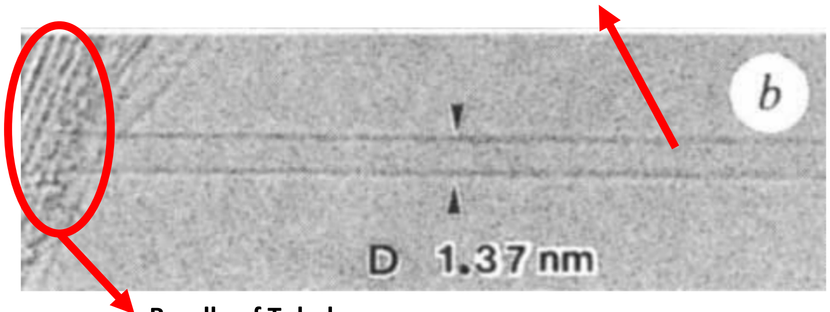
Fraunhofer Diffraction from the two portion of the tubules Conveys

- Each streaked spot has intensity maxima appearing with a period of 0.73 nm-1.
- The value corresponds to the diameter measured on the tubule (1.37 nm).
- Alpha Measured = 7 Degrees

Source: Iijima, Sumio, and Toshinari Ichihashi. "Single-shell carbon nanotubes of l-nm diameter." Fundamental Research Laboratories, NEC Corporation, 34 Miyukigaoka, Tsukuba, Ibaraki 305, Japan.

Structure Of Carbon Nanotubes

Electron Beam of 20 nm Diameter was focused on to the Single Tubule so that the area comprised about 2000 Carbon Atom



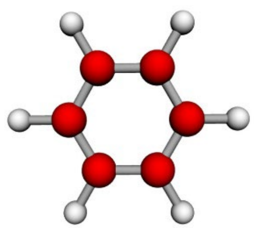
Bundle of Tubules

Figure 4 : Electron Micrograph of Single-Shell nanotubule

Reason for the Extremely weak and Diffused Diffraction

- Small Scattering Volume
- Cylindrical Structure

Carbon Atom Hexagons are arranged in Helical Fashion

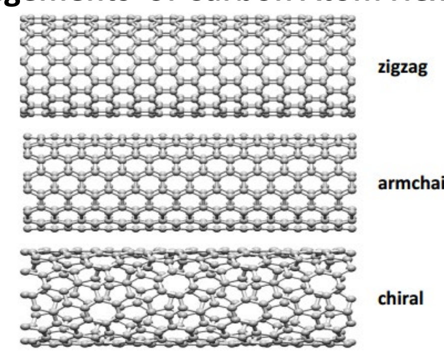


Two hexagonal diffraction pattern Confirms Helicity

There are two hexagonal (hk0) diffraction patterns rotated ± a/2 from perfect alignment along the tube axis, and these patterns show mirror symmetry both along and perpendicular to the tube axis. The mirror symmetry confirms helicity in the single-shell tubule structure

Helical Pitch Varies from Needle to Needle and tube to tube Within Single Needle

Other Arrangements of Carbon Atom Hexagons



Source: Iijima, Sumio, and Toshinari Ichihashi. "Single-shell carbon nanotubes of l-nm diameter." Fundamental Research Laboratories, NEC Corporation, 34 Miyukigaoka, Tsukuba, Ibaraki 305, Japan.
Online Open-Source Images

What Makes Each Carbon Nanotubes Unique ?

- Tubule Diameter (D)
- Pitch Angle (Alpha) With Respect to the Fibre Axis
- Helicity

Author’s Speculation

- Single Shell Tubules Might be the Embryo for the Multi Shell Tubules
- In the Proposed Model

Tubule Ends are Open → Dangling Bonds →Carbon Atoms are Captured

- In Single Shell Tubes , Author assumes that axial growth dominates over layer growth
- Iron present in the vapour Phase act as a Homogeneous Catalyst

Source: Iijima, Sumio, and Toshinari Ichihashi. "Single-shell carbon nanotubes of l-nm diameter." Fundamental Research Laboratories, NEC Corporation, 34 Miyukigaoka, Tsukuba, Ibaraki 305, Japan.