Methods and Techniques of Experimental Physics (MTEP)

a compulsory course for

Semester-I, M. Phil in Physics
Department of Physics, University of Sargodha

Session 2020-22

Instructor: Dr. Bilal Rasul Warraich

PHYS 9202 Methods and Techniques of Experimental Physics Cr.H-3

Basics of X-ray diffraction, X-ray spectra, Bragg's law and importance, construction and operation of diffractometer, data analysis, Qualitative (Hannawalt method), Quantitative (matrix flushing methods), Vacuum techniques, Production of vacuum (Vacuum pumps), Measurements of vacuum (Gauges), Leak detection, Thin film Physics, Methods of preparation of thin films, Methods of thickness measurement, Characterization techniques, Basics of spectroscopy and importance, Lambert-Beer's law, Construction and Operation of spectrophotometer, Radiation detection (Detectors), Data analysis, Error theory.

Recommended Books:

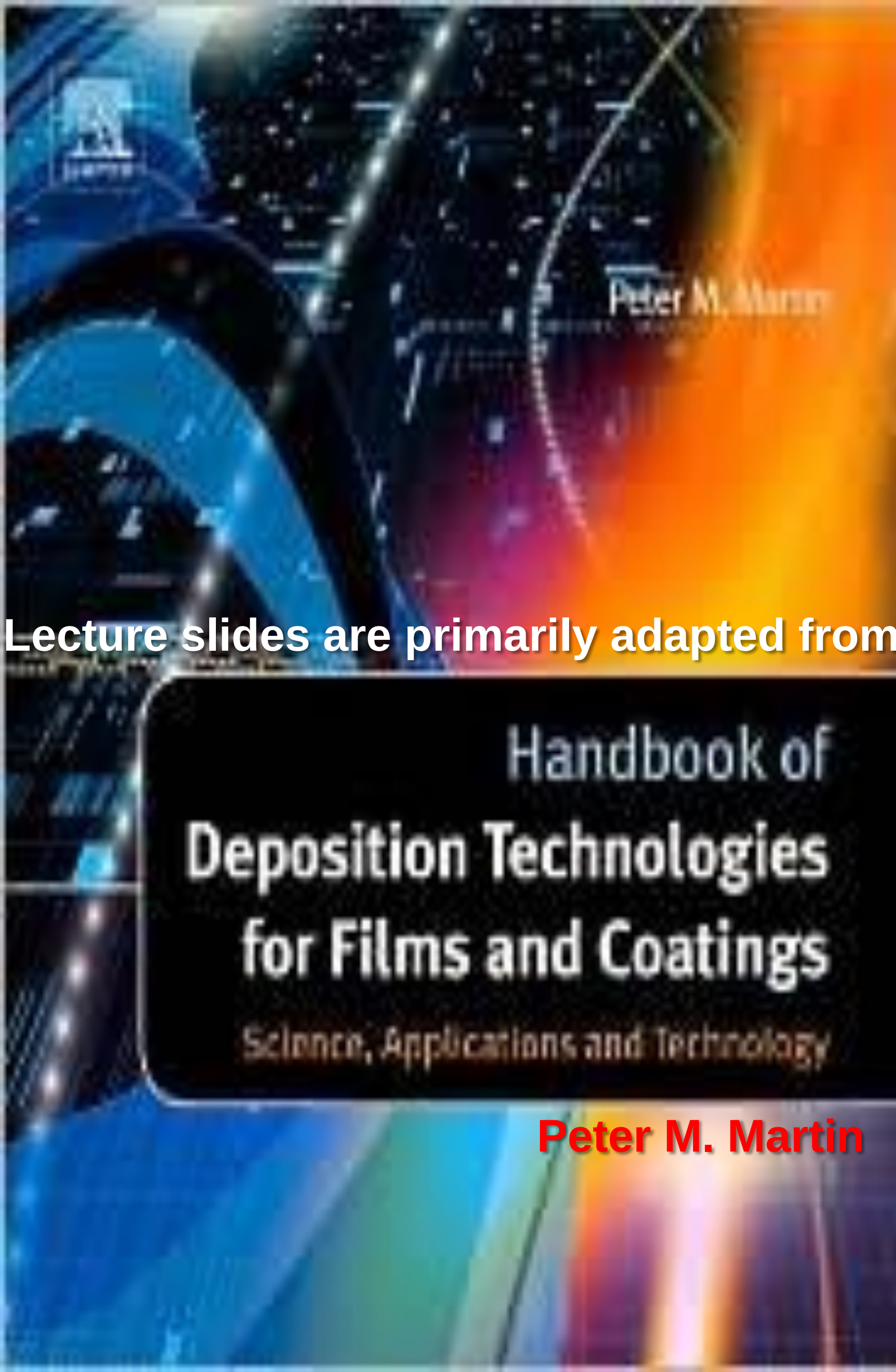
1. Physics for Scientists and Engineers, by Richard Wrolfson and Jay M. Pasachoff, Published by Addison Wesley / Longman Inc. (1999).
2. Elements of X-ray Diffraction, by B.D. Cullity, Published by Addison-Wesley Publ. Co. Inch. Reading MA, USA (1978). York, USA (1978).
3. Methods of Experimental Physics, by R.L. Horovitz and V. A. Johnson, Acadmic, (Latest Edition).
4. Methods of Experimental Physics, by D. Williams, Acadmic (Latest Edition).
5. Porous Silicon Science & Technology, by Jeane Claude Vial and Jacques Derrian published by Springer Verlag, Holland (1999).
6. Electron Microscopy and Microanalysis of Crystelline Materials, Edited by J.A. Beck, Published by Applied Science Publishers Ltd., London, England (1979).
7. Electron Microscopy, by S.Amelincex, Dirk Van Dyck, J.Van Landuyt, Gustaff Van Tendeloo, Published by J.Wiley & Sons, Inc. USA (1997).

Handbook of Deposition Technologies for Films and Coatings: Peter M. Martin

The Basics of Crystallography and Diffraction: Hammond

Scientific Foundations of Vacuum Technique" by Saul Dushman

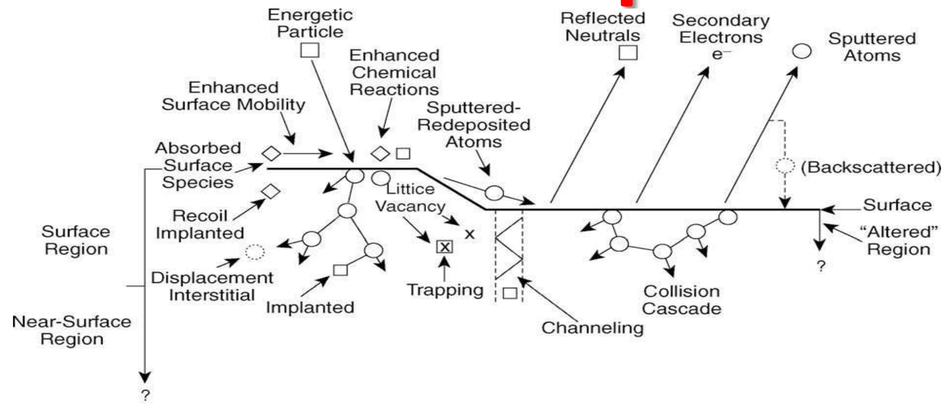
Lecture slides are primarily adapted from



Peter M. Martin

Chapter 3

Surface Preparation for Thin Film Deposition Processes



ENERGETIC PARTICLE BOMBARDMENT OF A SURFACE

Introduction

External Cleaning

Evaluating and Monitoring of Cleaning

Recontamination in the Ambient Environment

In-Situ Cleaning

Recontamination in the Deposition System

Some Surface Modification Processes

Deposition Methods

Chemical Vapor Deposition (CVD)

-Process Family

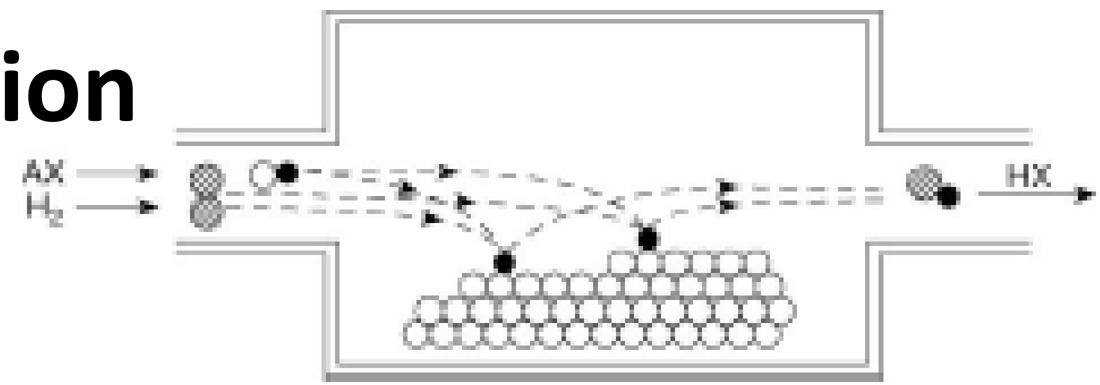
-Important Reaction Zones

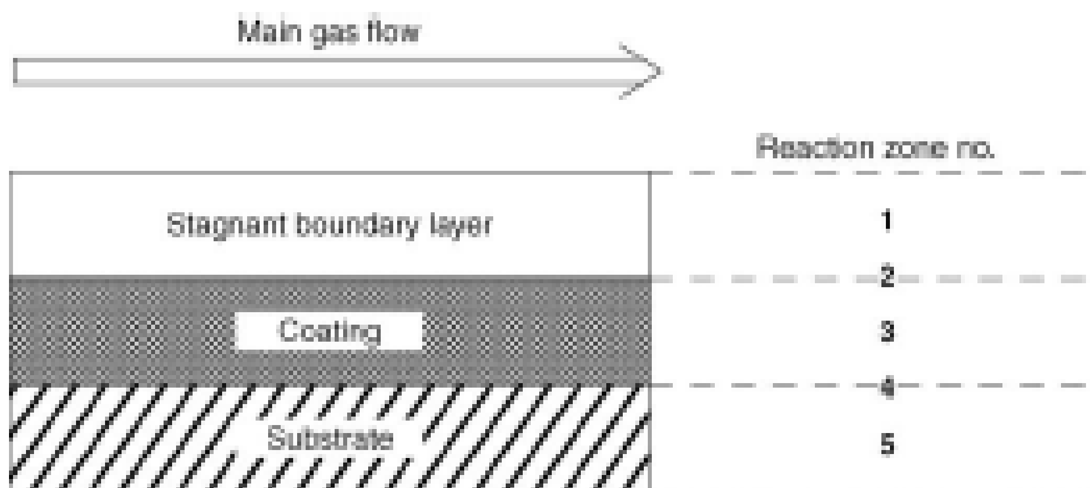
-Classifications

-CVD System

-Applications

$$CH_4 \rightarrow C(s) + 2\ H_2(g)$$





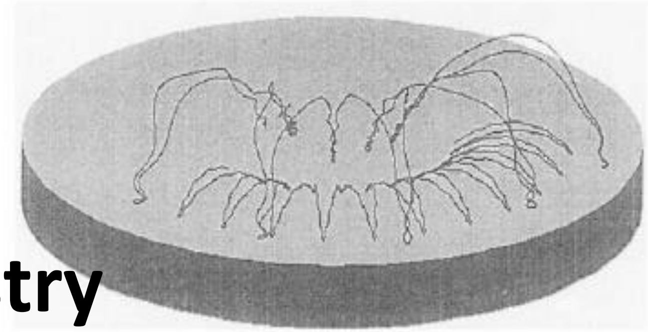
Deposition Methods

Magnetron Sputter Deposition

-How are energetic Particles Generated?A DC Glow Discharge in Gases

-Trapping the e^{-}s(Magnetrons)

-Applications in Research and Industry



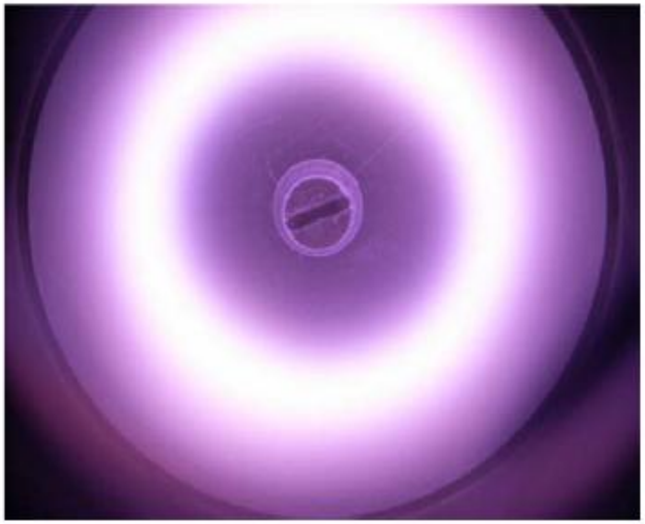


Figure 5.9: Simulated part of a trajectory of an electron on a circular planar magnetron (a) and the resulting torus-shaped argon plasma (b).

Deposition Methods

Ion Plating

- -Bombardment Effects
- -Sources of Depositing Particles
- -Sources of Bombarding Particles
- -Applications

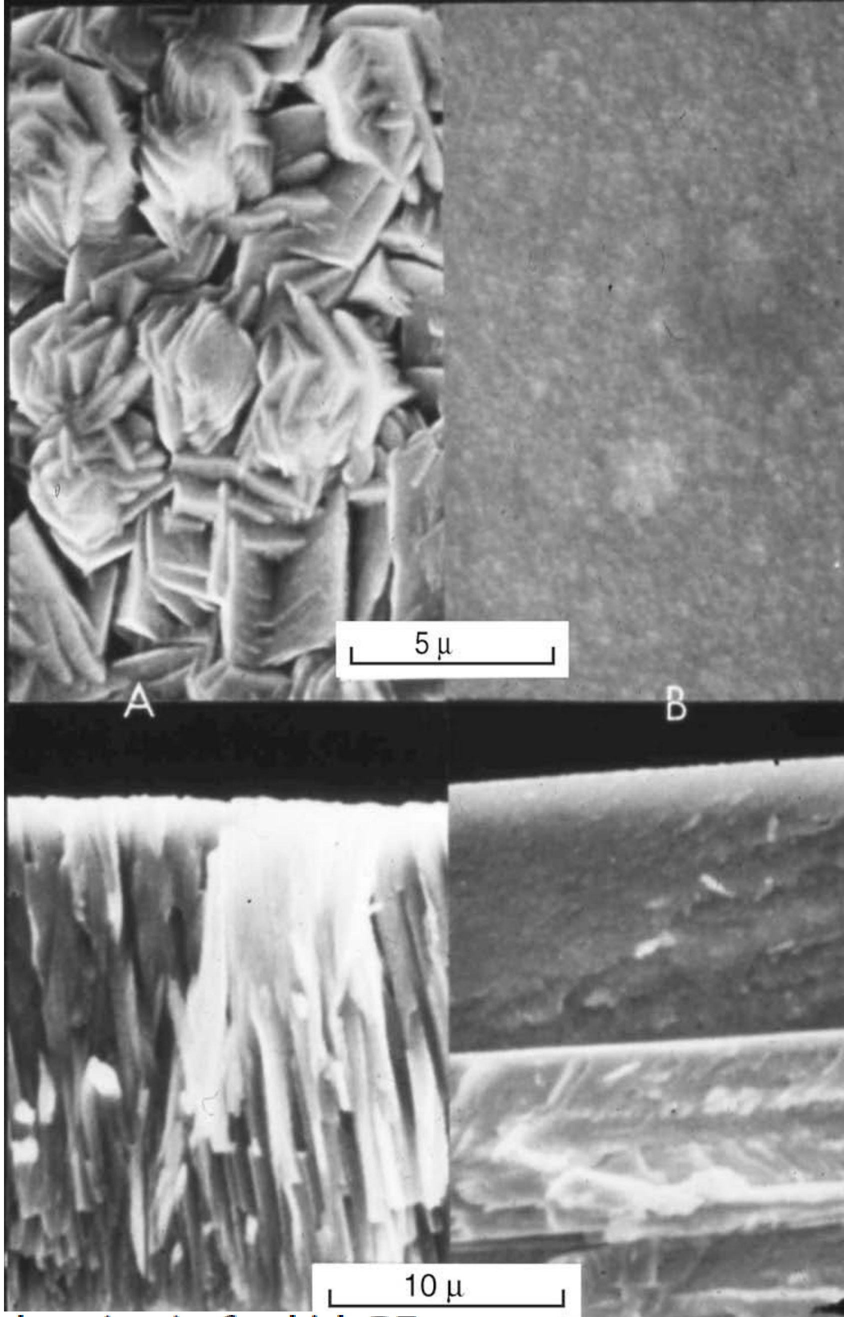


Figure 6.3: Fracture cross-section (bottom) and surface morphology (top) of a thick RF sputter deposited chromium deposit: (A) without a bias (no bombardment) and (B) with concurrent bombardment (−500 V bias on the substrate) [13].

Deposition Methods

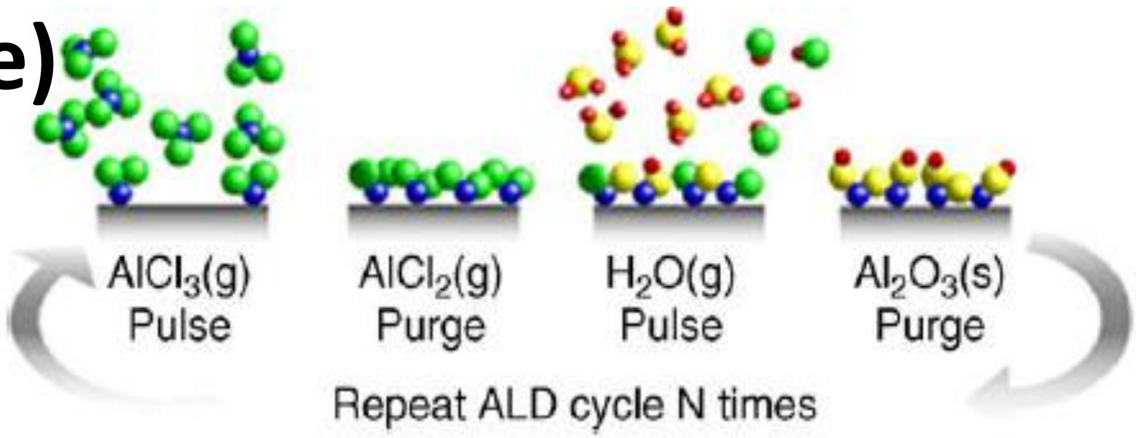
Atomic Layer Deposition (ALD)

-Principle(ALD Cycle)

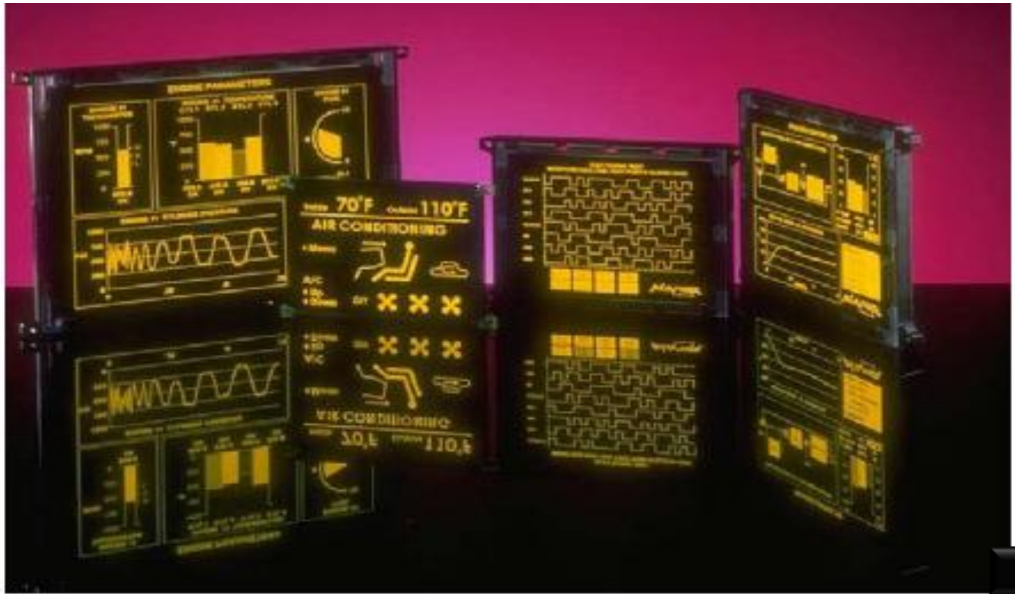
-Advantages

-ALD Precursors

-Applications

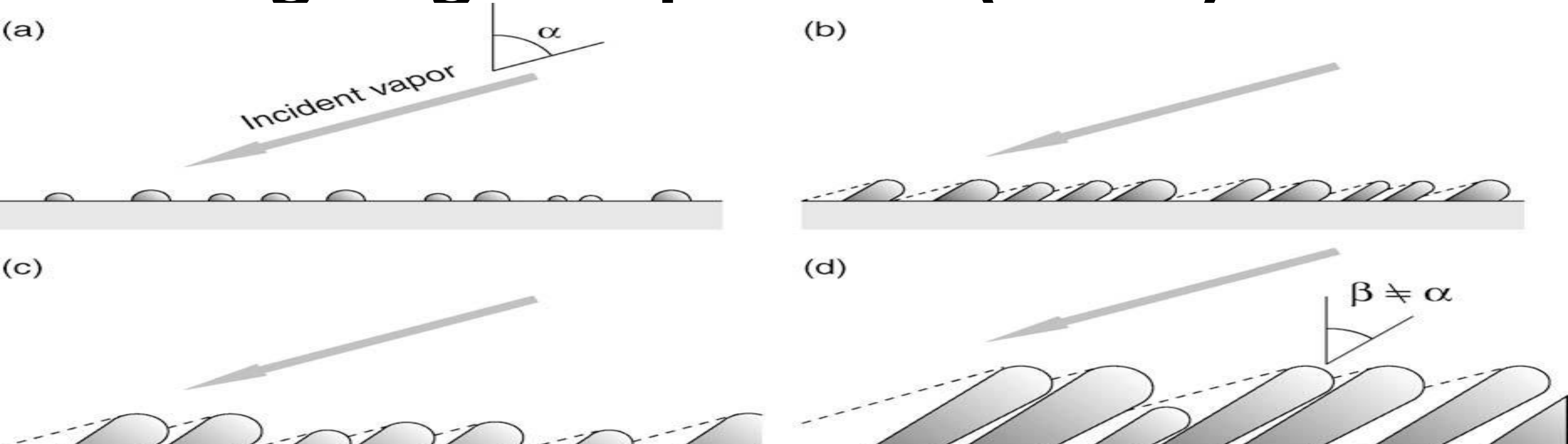


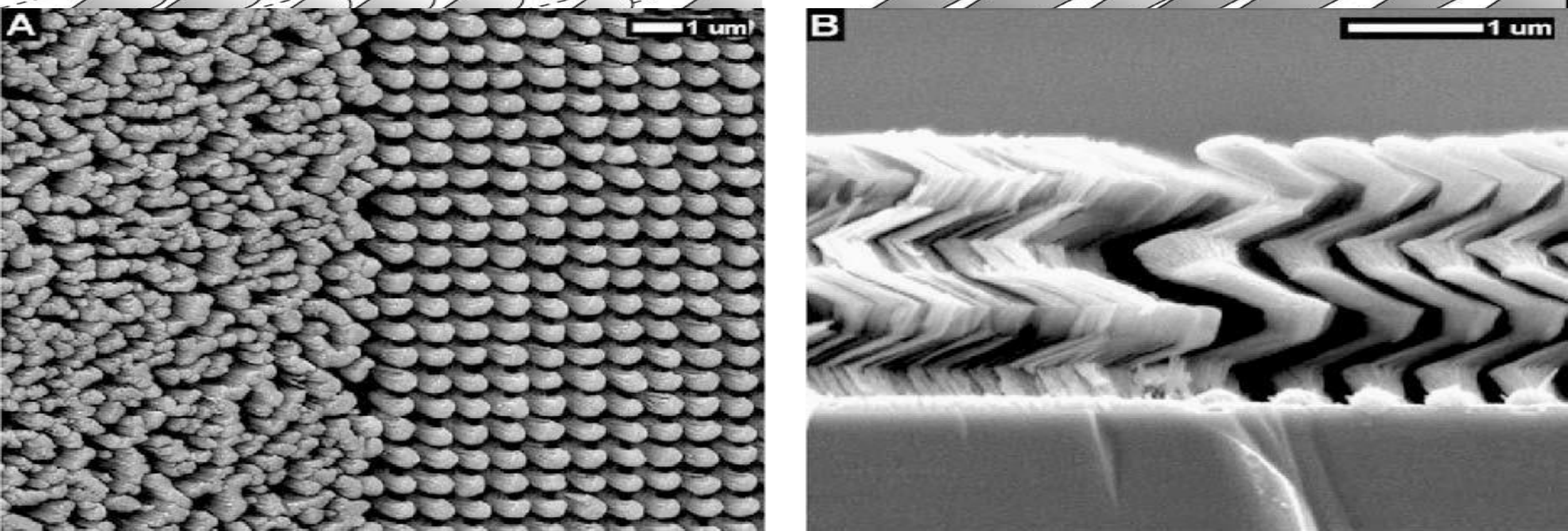
Schematic illustration of one ALD cycle.



Deposition Methods

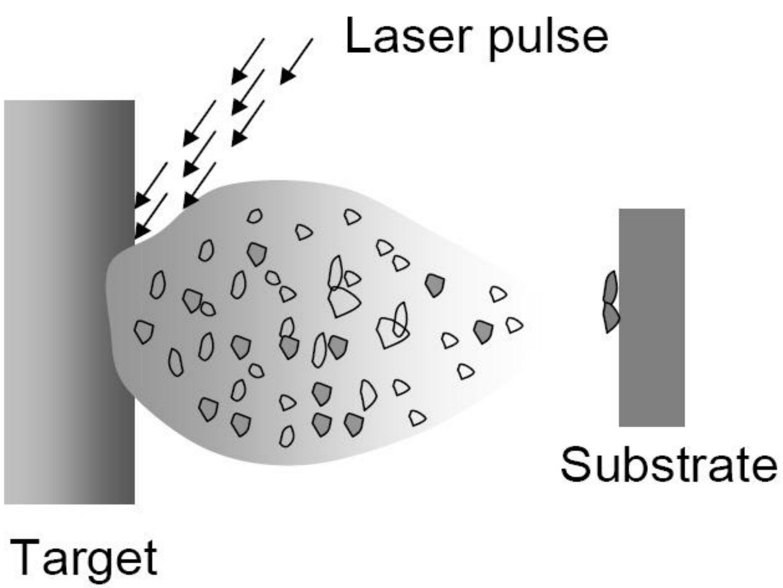
Glancing Angle Deposition (GLAD)

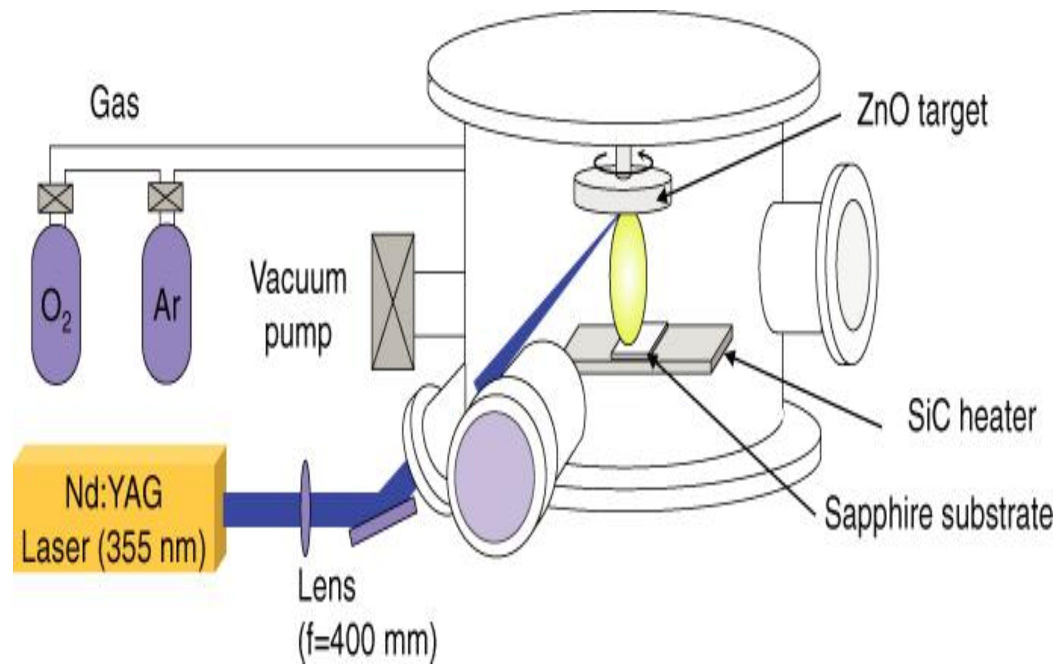




Deposition Methods

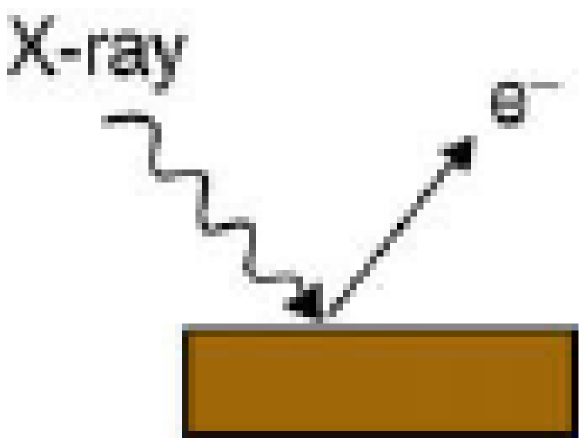
Pulsed Laser Deposition (PLD)



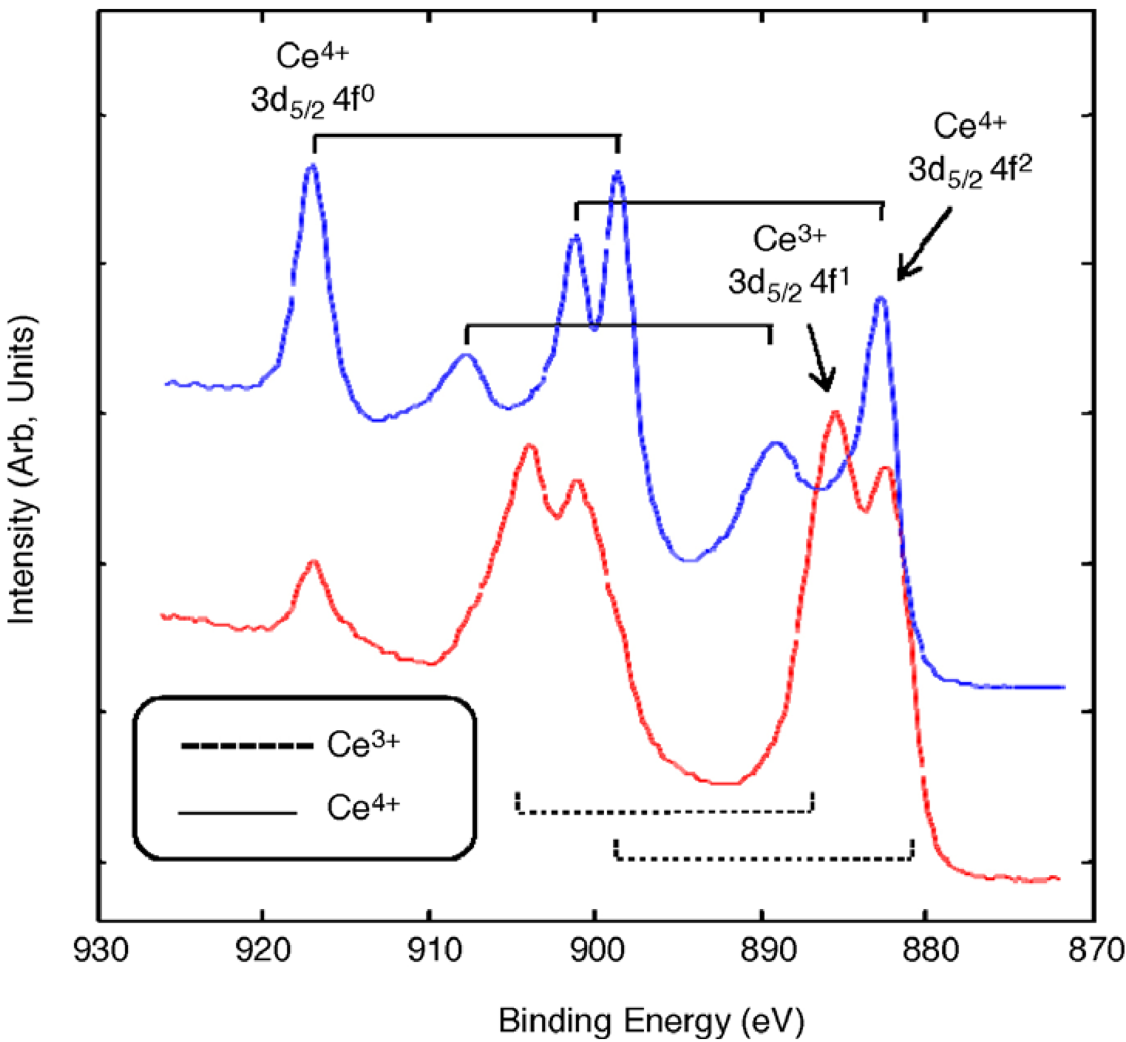


Spectroscopy and Spectrometry

X-Ray Photoelectron Spectroscopy (XPS)



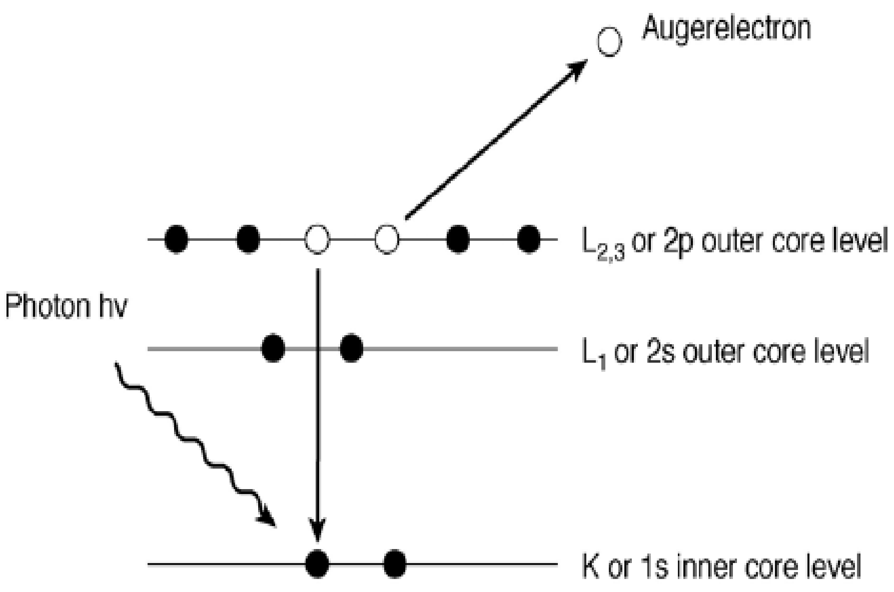
$$KE = h\nu - BE - \varnothing$$

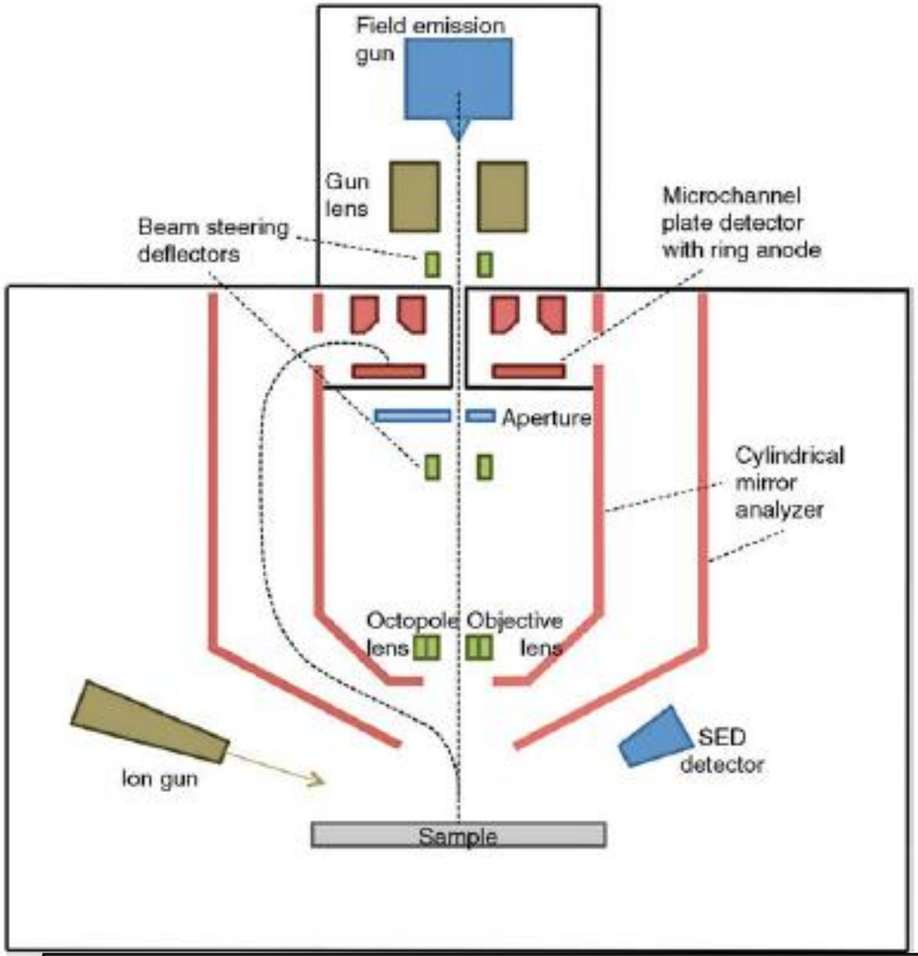


Spectroscopy and Spectrometry

Auger Electron Spectroscopy (AES)

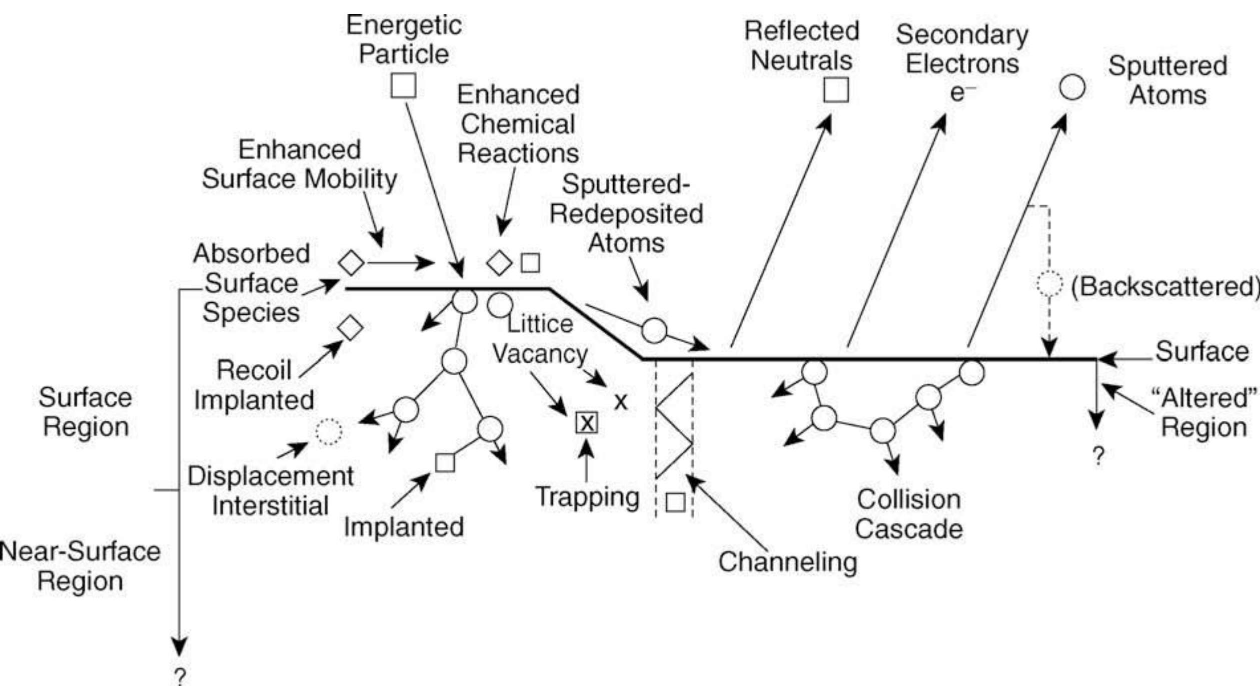
$$E_{KLL} = E_K - E_L - E_L$$

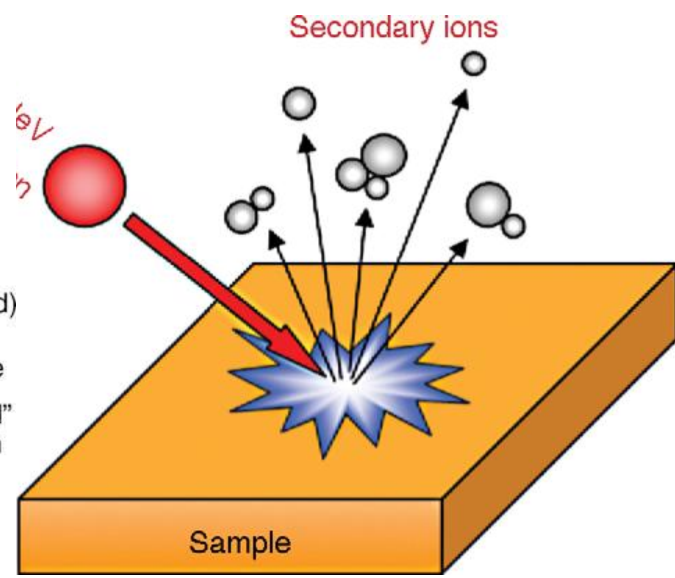




Spectroscopy and Spectrometry

Secondary Ion Mass Spectrometry (SIMS)

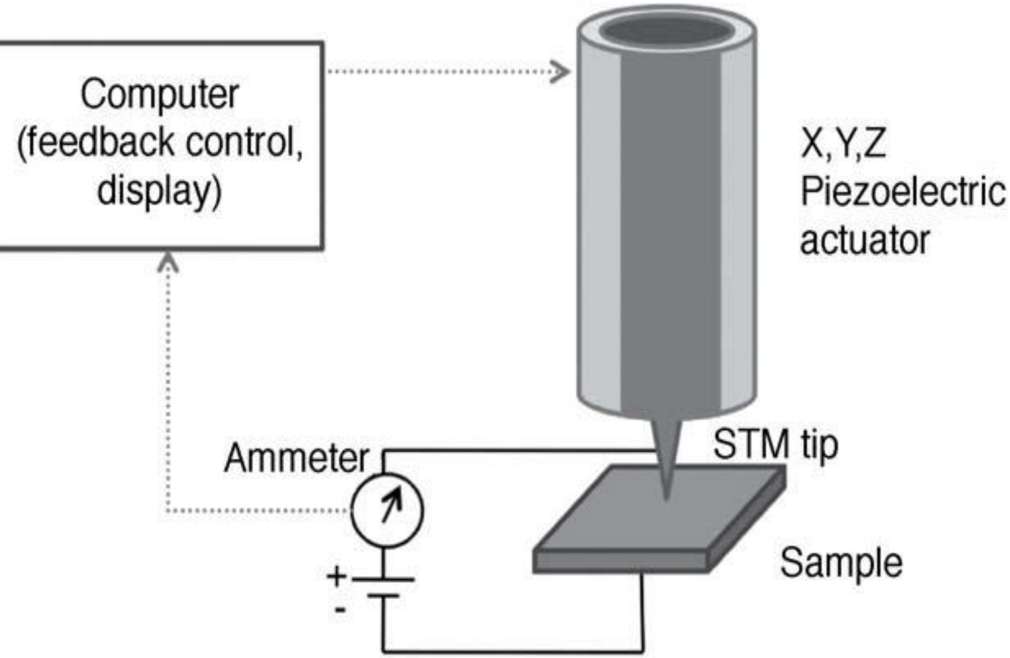




Spectroscopy and Spectrometry

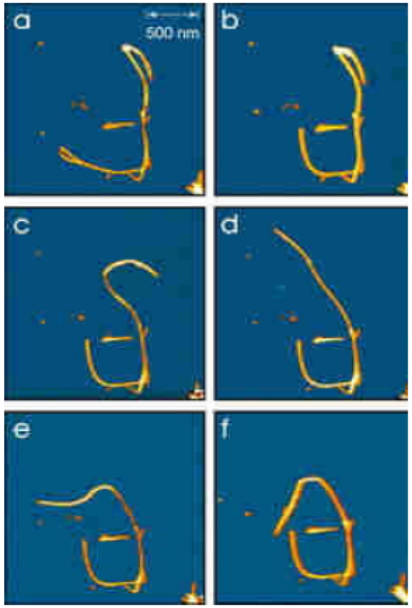
Scanning Tunneling Microscopy (STM)

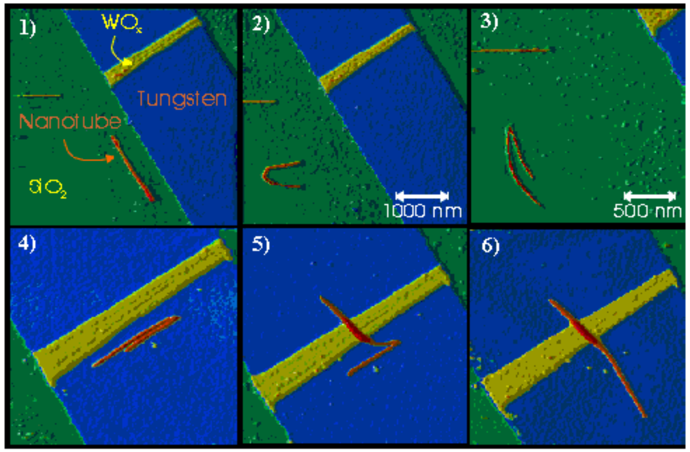
$$I \alpha e^{-2\kappa z}$$



Spectroscopy and Spectrometry

Atomic Force Microscopy (AFM)





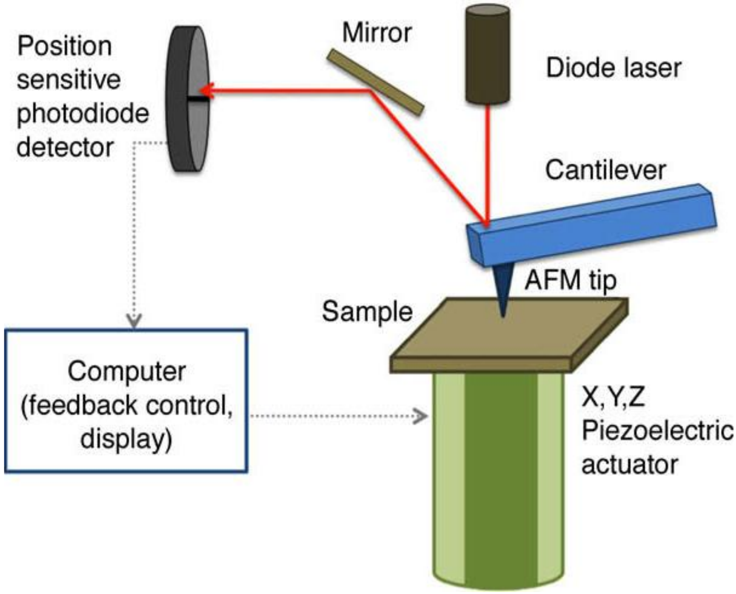
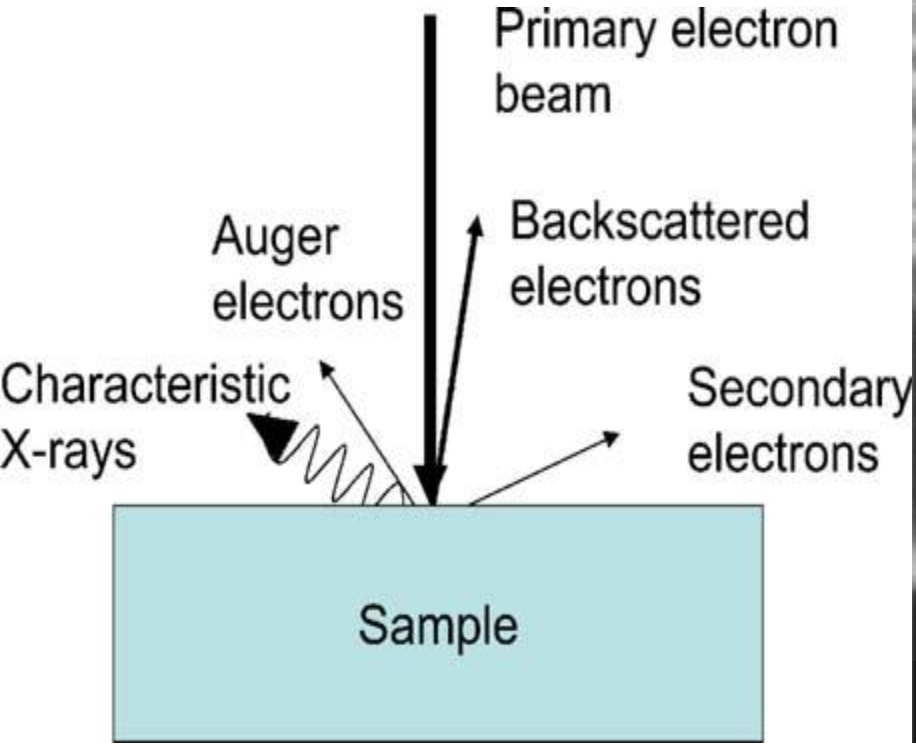


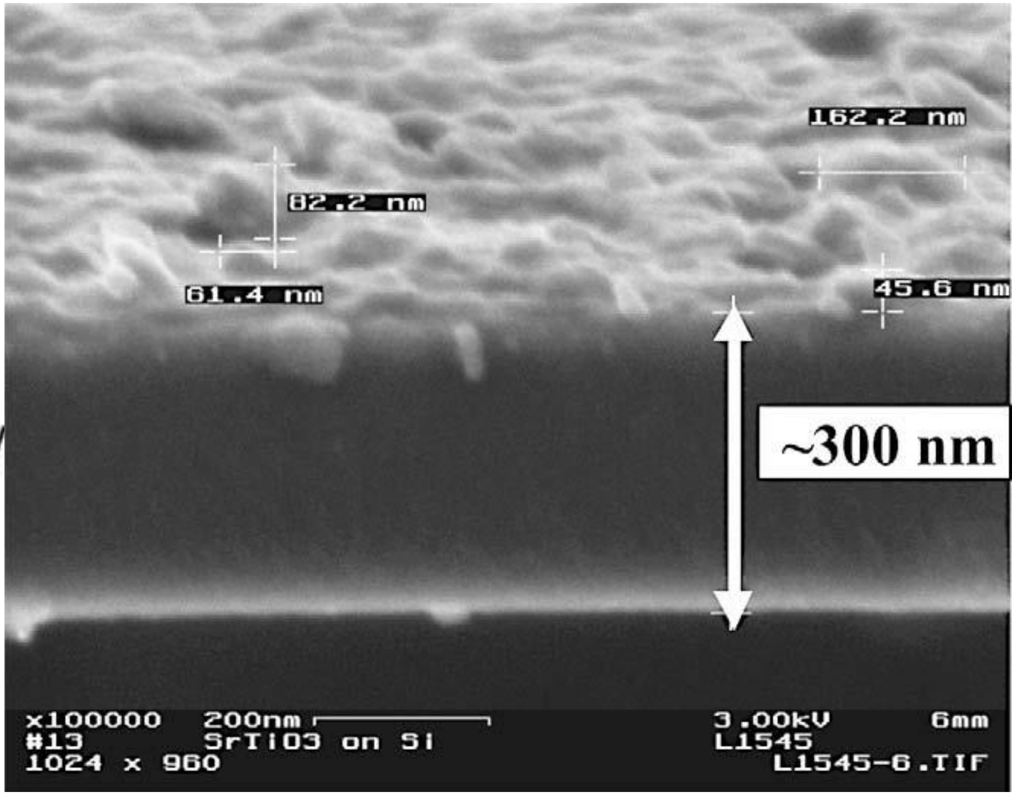
Figure 11.

Left image: Manipulation of a nanotube on a silicon substrate. The AFM tip is used to create the Greek letter "theta" from a 2.5 micron long nanotube.
Right image: A single nanotube (in red) originally on an insulating substrate (SiO_2, shown in green) is manipulated in a number of steps onto a tungsten film thin wire (in blue), and finally is stretched across an insulating tungsten oxide barrier (in yellow).

Spectroscopy and Spectrometry

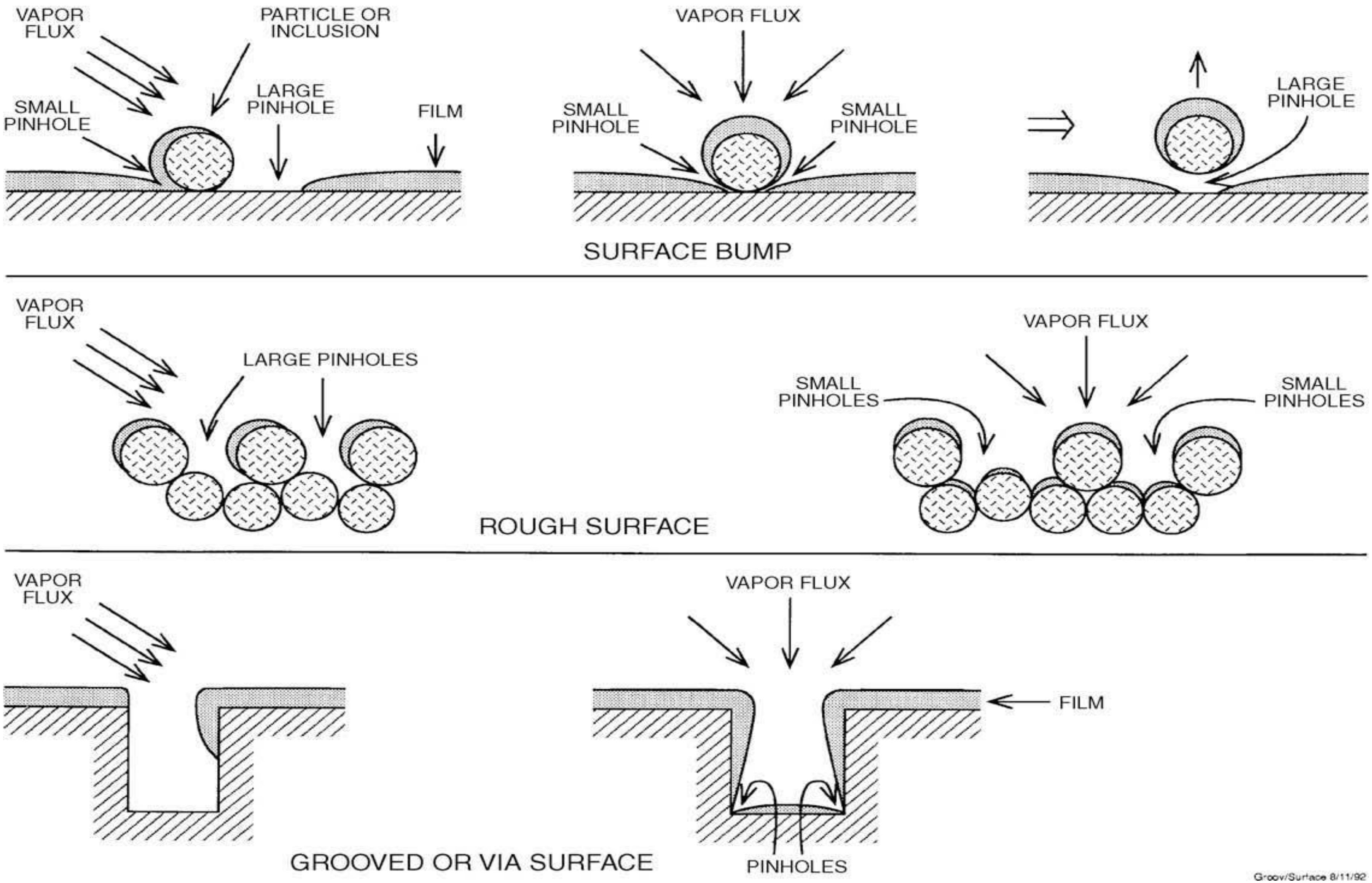
Scanning Electron Microscopy (SEM)





Chapter 3

Surface Preparation for Thin Films Deposition Processes



Introduction

- Surface Preparation= as Homogeneous as possible by Modification and Cleaning
- Modification
  - Roughening or Smoothing
  - Making a surface harder by plasma treatment
  - Activating a surface by plasma treatment
- Cleaning
  - No undesirable material
  - e.g. HC layers cause poor adhesion and probably poor electrical contacts
  - Porosity, embedded particles, steps, roughness that effect film properties, produce pinholes, local loss of adhesion

External Cleaning: Gross Cleaning

Outside the deposition system as a separate process in a controlled environment

i- Gross cleaning: removal of large amount of contaminants often by removing some substrate surface

ii- Specific cleaning: removal of a specific contaminant

- Stripping by mechanical abrasion or chemical etching
- Abrasive cleaning
  - Abrasive surfaces e.g. sand paper, Scoth Brite™
  - Abrasive powders e.g. diamond paste
  - Particles in a gas or liquid stream i.e. Grit Blasting
  - Al_2O_3, diamond, glass beads, silica sand, SiC, CeO etc.

External Cleaning: Gross Cleaning

- Wet Chemical Etching

Removes surface layers e.g oxides, eliminates or blunts surface cracks in brittle materials and removes difficult-to-remove contaminants

Surface material along with contaminants

Pickling

Removal of oxides formed on metals during fabrication

Al and Alloys by sulfuric acid, nitric acid etc.

Cu and Alloys by comb. of Sulfuric and oxidizing acids

Fe and SS by sulfuric and HCL

HF acid for Si, leaves -H or -OH terminated

May change the surface chemistry e.g.

i- glass-bonded alumina ceramic in HF, removes a specific glass phase and weakens the ceramic; poor adhesion

ii- acid etching of soda lime glass, removes Na and surface is acidic i.e. wetting properties vary

Sequential etching: Cu-Al alloys with NaOH leaves Cu and/or Si. Cu is removed by HNO_3 and Si by HF.

External Cleaning: Specific Cleaning

- Solvent Cleaning
  - Polar solvents for polar contaminants e.g. water for ionic materials
  - Nonpolar e.g. CFC for grease-mixtures also used
  - Solubility parameter: max. of specific contaminant that can be dissolved in a specific amount of solvent

External Cleaning: Specific Cleaning

- Alkaline Cleaners
  - Are saponifiers that convert organic fats to water-soluble soaps
  - Clean oxide surfaces strongly adsorb HCs that can't be removed by normal detergents
  - Carbonized HCs can be removed by saturated water solution of KOH
- Soap Cleaners
- Solution Additives
  - Surface energies are important both of solid and liquid as well as their interfacial energy
  - Wetting agents reduce the surface energy
  - Surfactants (surface active agents that reduce the interfacial energies) used with water are both Hydrophobic and Hydrophilic, can reduce the S.E. of water by 30 mJm^{-2}, interface b/w oil and water.

External Cleaning: Specific Cleaning

- Solution Additives
- Basics are better than Acidic solutions, PH adjusted NH_3 or NH_4OH
- Wet Reaction Cleaning
  - Volatile or soluble product by liquids, plasma, vapors or gases.
  - Acid-Base solutions as oxidants Piranha Solution i.e. conc. H_2SO_4 and $(NH_4)_2S_2O_8$……atomic O
  - $K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3O$ (i.e. Free O)
  - H_2O_2 is a good oxidizing agent for cleaning glass 8 (30% H_2O_2):1 (NH_4OH):1 (H_2O)
  - RCA for cleaning Si (organic clean, Ox. Strip and ionic clean)
  - Modified RCA

External Cleaning: Specific Cleaning

- Reactive Gas Cleaning

Formation of volatile reaction products of the contaminants i.e. Oxidation Cleaning using O, Cl, F, O3, NO etc.

Non-volatile as residue

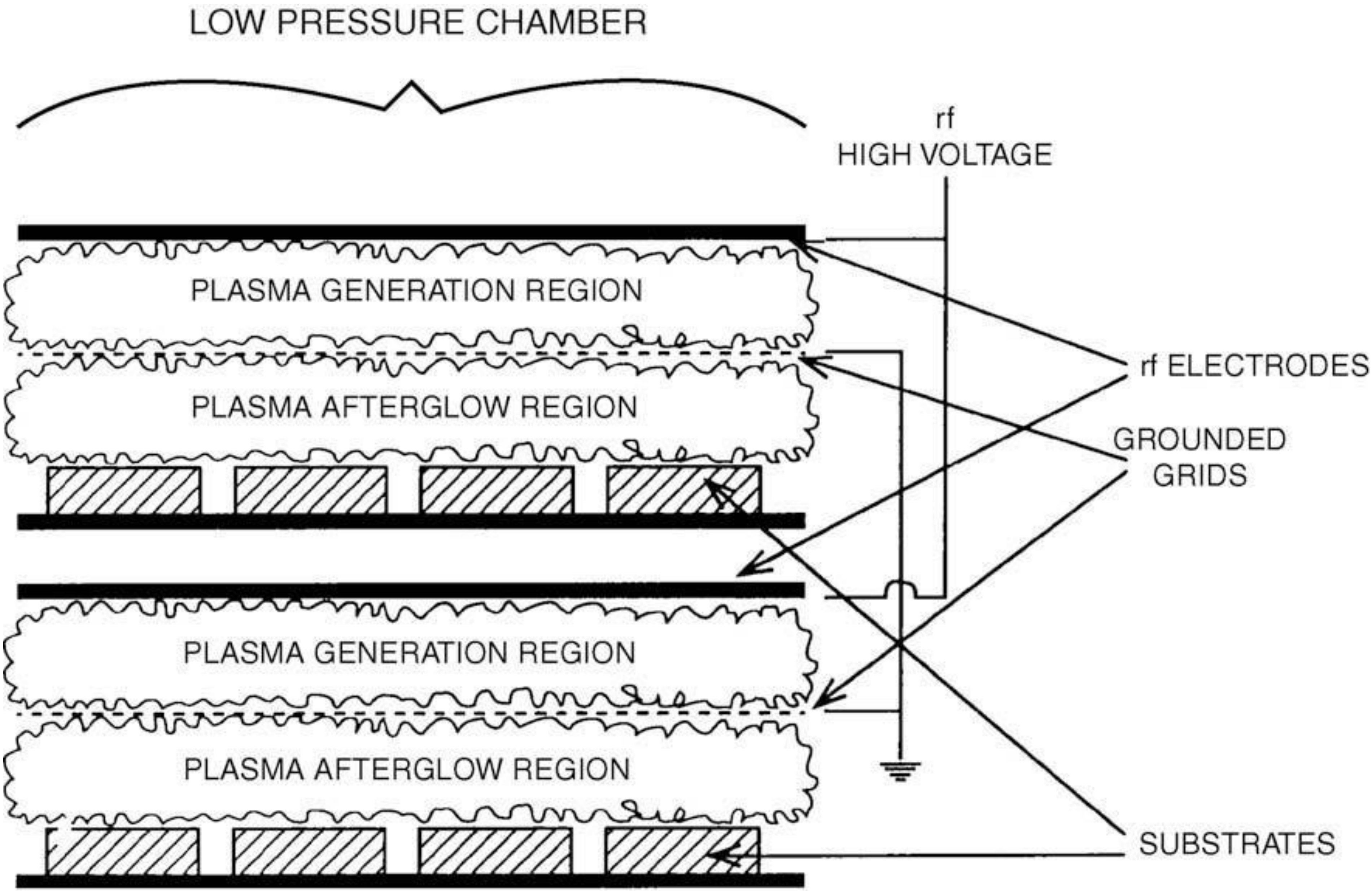
Reaction with a gas at high temperature e.g.

Air Firing-to oxidize HCs e.g. Alumina is cleaned of HCs by heating to 1000 ^{o}C in air e.g. Kitchen ovens are cleaned by Oxidation at 425 ^{o}C

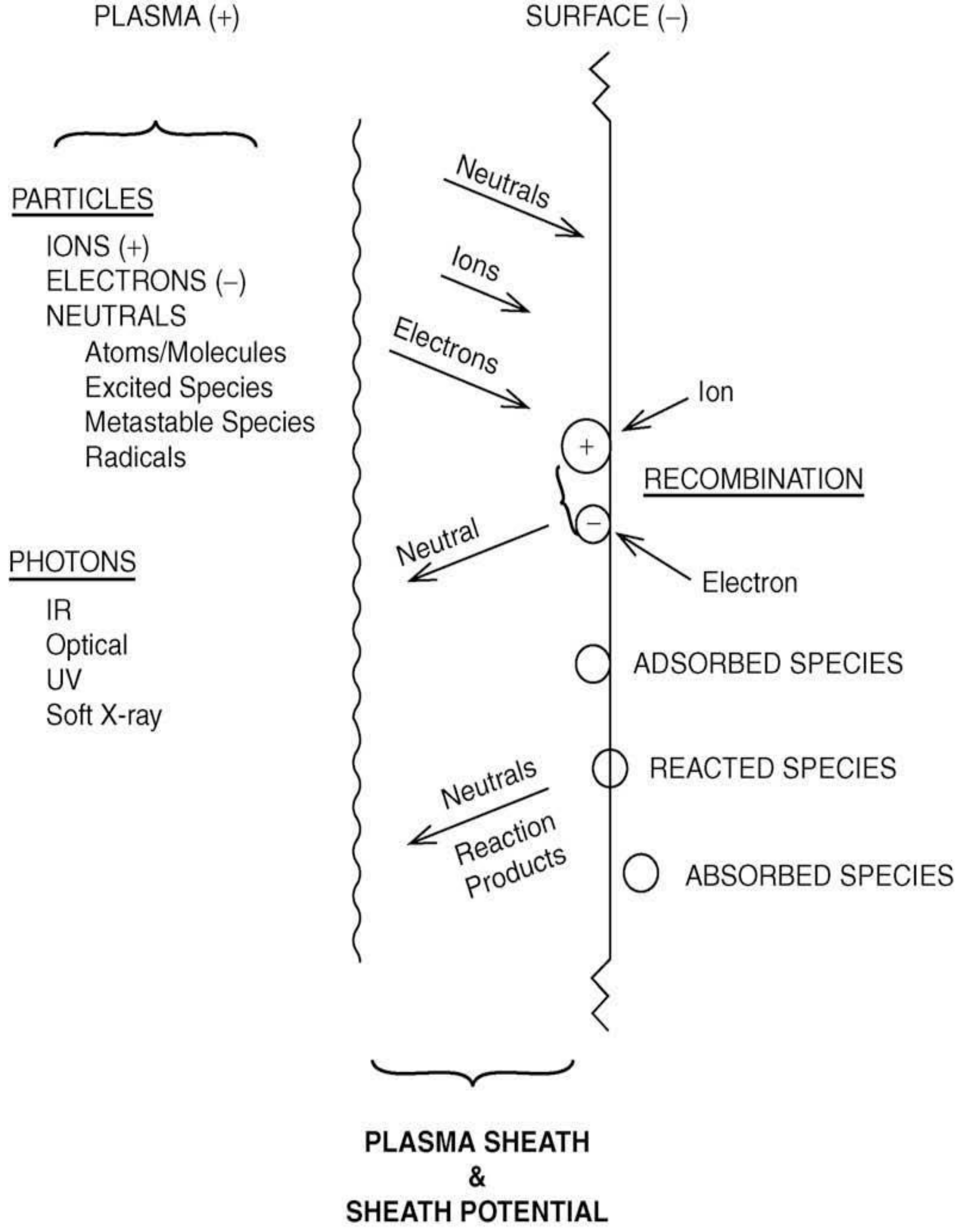
UV/O_3 method-oxidation at at. pressure results the bond scission of HC layers with UV radiation intensity of about 1-10 $mWcm^{-2}$ at the substrate

External Cleaning: Specific Cleaning

- Reactive Plasma Cleaning
  - At lower temp. than RGC, uses reactive species in plasma to react with surface and form volatile species that leave the surface
  - no residue
  - low etch rate of substrate and removes oxide layer



PLASMA CLEANER



PLASMA — SURFACE INTERACTION
(LOW ENERGY IONS)

External Cleaning: Specific Cleaning

- Reactive Plasma Cleaning
  - O_2, H_2, F and Cl as gases. Metals cleaned with F.
  - O_2 very effective for HCs
  - Ion Scrubbing
  - Mixture of gases for improved etching e.g. for Si, CF_4 96% with O_2 4%, SiO_x from Si is removed by mixture of HF and H_2O.

Use He as diluent

  - Compound gases leave products e.g. B, C or Si change the chemical composition of surface e.g. C from CCl_4 or CF_3.

External Cleaning: Specific Cleaning

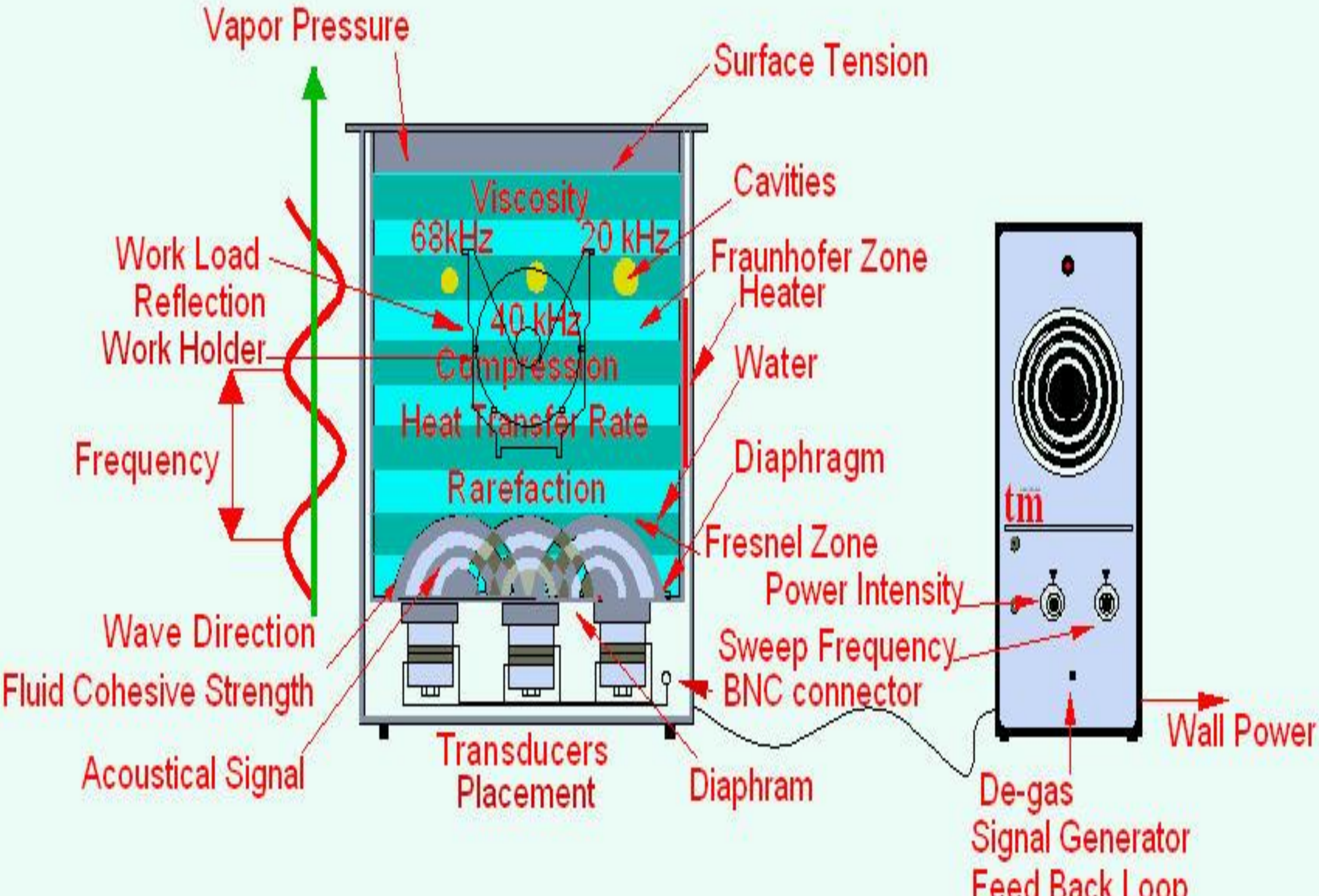
- Reactive Plasma Cleaning
  - RF plasma at pressures of 100-500 mtorr
  - O_2 for those that can withstand oxidation or H_2 for those that require non-oxidizing environment.
  - Surface placed outside plasma that leaks from the grid electrode.
  - For HCs, H plasma is useful. To clean vacuum surfaces in Nuclear Fusion Reactors.

External Cleaning: Specific Cleaning

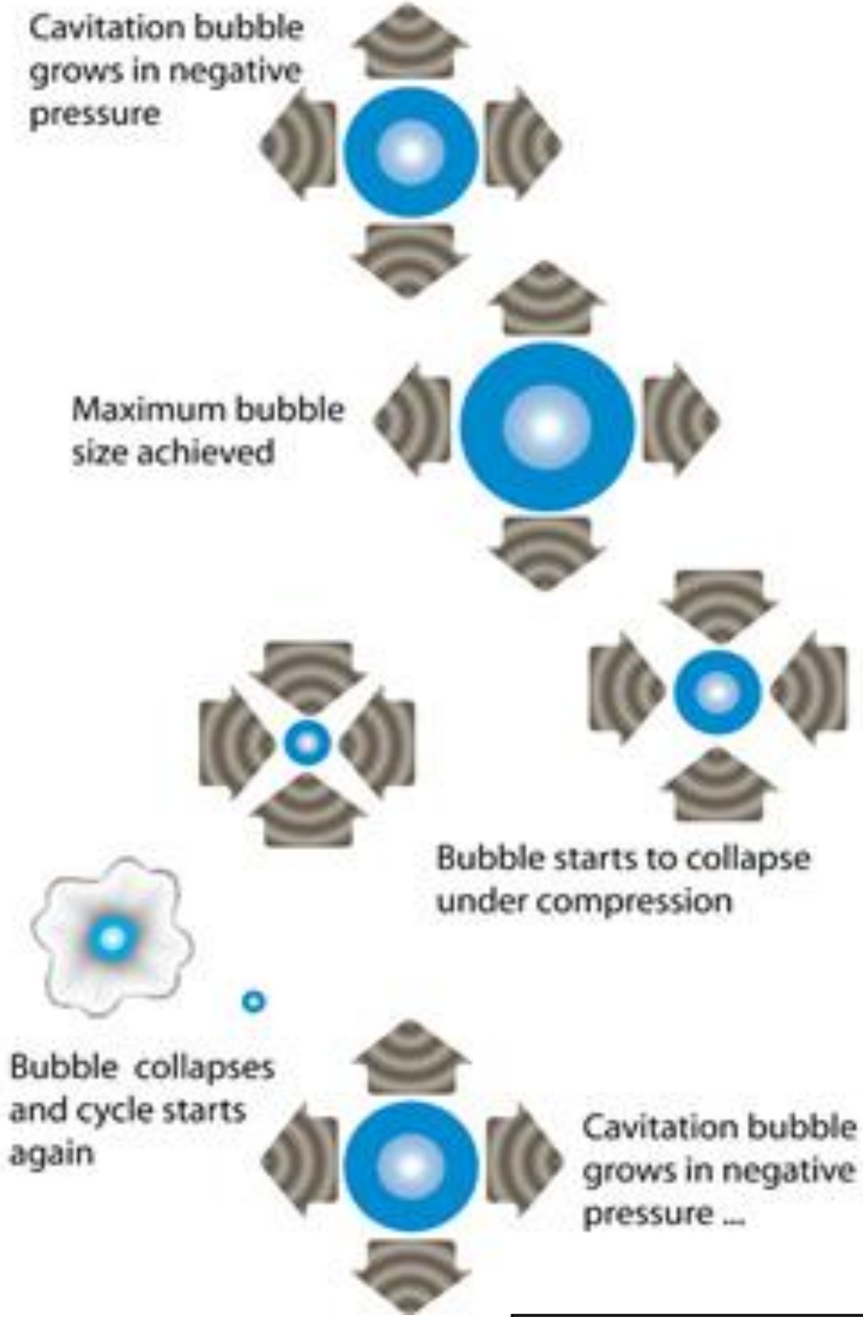
- Application of Fluids
- Ultrasonic Cleaners
  - Collapsing of bubbles in contact with surface provides high pressure
  - After grinding and abrasive procedures
  - Ultrasonic wave is produced by Electrostrictive or Magnetostrictive Transducers at 18-120 kHz and energy density of about 100W/gallon of fluid
- Size of Bubbles

  vapor pressure, surface energy, temp.

  H_2O @ 60^{o}C, 40 kHz---3 µm, jet pressure-300 psi


Vapor Pressure
Surface Tension
Viscosity
68kHz
20 kHz
Cavities
Work Load
Reflection
Fraunhofer Zone
Heater
40 kHz
Work Holder
Compression
Water
Heat Transfer Rate
Frequency
Diaphragm
Rarefaction
tm
Fresnel Zone
Power Intensity
Wave Direction
Sweep Frequency
Fluid Cohesive Strength
BNC connector
Wall Power
Acoustical Signal
Transducers
Placement
Diaphram
De-gas
Signal Generator
Feed Back Loop

CAVITATION AND IMPLOSION



External Cleaning: Specific Cleaning

Variables:

- Amplitude and Frequency of pressure wave(i.e. energy density, standing wave pattern)
- Nature of the transducer fluid(density, viscosity, surface tension, vapor pressure)
- Flow and filtering of fluid
- Temperature, for water, 55-65 ^{o}C
- Gas content (Degasing of fluids to enhance cleaning)
- Geometry of the system

External Cleaning: Specific Cleaning

- Application of Fluids
  - Ultrasonic Cleaners
    - Energy required for cavitation is proportional to surface tension and vapor pressure e.g. ST of water 70 dynes/cm. Using a surfactant, 30 dynes/cm.
    - Fixturing is important. Area in cm^2 < Volume in cm^3.
    - May cause Erosion and fractures e.g. glass observes increase in light scattering.
    - High Frequency ultrasonic baths 400 kHz uses a train of wave fronts that sweeps across the surface. Viscosity plays the role here. 50 psi, for smaller area of impact.
    - Megasonic > 400 kHz for Si wafer processing.