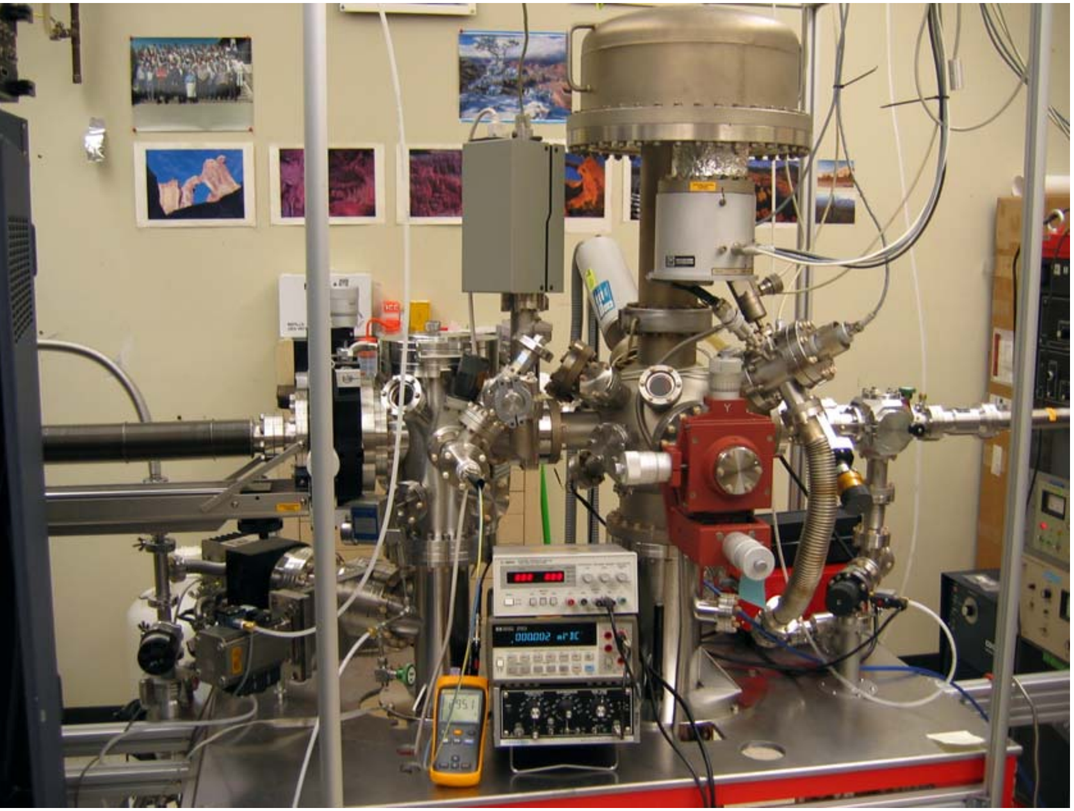
Vacuum Science Techniques and Applications
Dan Dessau
Adv. Lab 2007

Vacuum increases the ***mean-free-path* of gas molecules.**
Vacuum prevents ***chemical reaction*.**
Vacuum removes ***contaminants*** from surfaces.
Vacuum reduces the ***particle flux*** on a surface.
Vacuum allows the ***operation of sensitive*** components, high voltages, etc.



X-ray Photoelectron Spectrometer
- operates in UHV conditions
- for surface-sensitive spectroscopy for elemental analysis, chemical analysis, etc.

Units

1 atm = 760 mm Hg = 760 torr

MKS   1 N/m^2 = 1 Pa (Pascal)
cgs   1 dyne/cm^2 = 1 microbar (barometric)

1 bar = 750 torr ~ 1 atm
100 Pa = 1 millibar = .75 torr ~ 1 torr

1 millitorr = 1 micron (of Hg)

Pressure Fractions of Atm	Pressure	Number Density (cm^{-3})	Mean Free Path	Particle Flux ($cm^{-2}\ sec^{-1}$)	Time - One Monolayer to stick
1	760 torr	2.5×10^{19}	65 nm	2.9×10^{23}	3 nsec
10^{-3}	.76 torr	2.5×10^{16}	65 microns	2.9×10^{20}	3 µsec
10^{-5}	7.6 mtorr	2.5×10^{14}	6.5 mm	2.9×10^{18}	300 µsec
10^{-8}	7.6×10^{-6} torr	2.5×10^{11}	6.5 m	2.9×10^{15}	30 msec
10^{-13}	7.6×10^{-11} torr	2.5×10^{6}	650 km	2.9×10^{10}	3.5 days

Rough pump

HV

UHV

6.5 mm is a **shorter distance** than any dimension that characterizes almost any vacuum system:

The length or diameter of any tube between chamber and pump, or to a pressure gauge.
The length between a deposition device and the substrate to be deposited on.

At 7.6 x 10^{-3} torr we expect many gas phase collisions before a gas molecule bounces from one surface to the next. **Continuum or Viscous Flow.**

By contrast, **6.5 m is a longer distance** than characteristic dimensions of most vacuum systems. Following a molecule desorbing from a surface at 7.6 x 10^{-6} torr will show, on average, that the molecule will hit another surface before it hits another gas phase molecule. **Molecular Flow**

In **Viscous Flow** the conductances increases linearly (and sharply) with pressure. At atmospheric pressure, a particular component's conductance may be 10^4 times higher than its Molecular Flow (minimal) value.

Base Pressure (of a vacuum system)– capped all leaks, stopped all flow of gases, and waited a long time. It is asymptotically approached.

Ultimate vacuum (of a pump)- cap the pump's inlet and measure the equilibrium pressure after operating the pump for many hours.

Outgassing rates = amount of gas coming out (off) something in a given time.
PRESSURE x VOLUME per unit AREA per unit TIME = Torr-liters/cm^2-sec

Wall Desorption. A spherical chamber, 1 ft diameter at 1 x 10^{-6} torr, with one monolayer of adsorbed water vapor on inner walls, has ~7000 times more molecules on the surface than in the gas-phase.

Other issues –

- Virtual leaks.
- High vapor pressure materials   - sulphur, Pb, or Zn containing. Brass (bronzes are OK), Common solders (Ag solder OK).
- Hydrocarbons and fingerprints.
- Bake-outs (limits gaskets and other materials).

Decreasing Gas Load

- Use only the lowest outgassing materials in construction
- Use appropriate vacuum design and welding techniques
- Physically and chemically clean the components and chamber walls
- Thoroughly leak check components prior to initial assembly
- Bake chamber walls to desorb gas molecules
- Freeze molecules to the wall with cryogen cooling
- Use a pump with minimum backstreaming and/or operate in a range where it will be minimal.

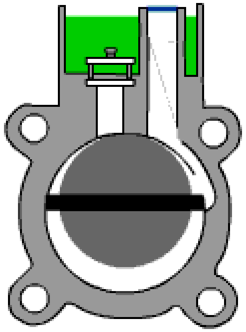
Increasing Pump Throughput

- Replace existing pump with another of larger pumping speed (or add more pumps)
- Increase effective pumping speed by increasing tubing diameter and reducing number of bends
- Choose proper pump for type of gas being pumped

  Turbo pump bad for Hydrogen.
  Ion pumps bad for “noble” gasses. Triode variant better.
  Cryo pumps bad for Helium, Hydrogen.
  Hydrogen, ozone, etc. may make some pumps explode!

Rotary Vane pumps (oil lubricated). From atm to ~ 10 mtorr. Oil vapor may backstream for P<1 torr.

Dry pumps (piston, diaphragm, etc.) becoming more popular. Typically they cost more and have higher ultimate pressures.

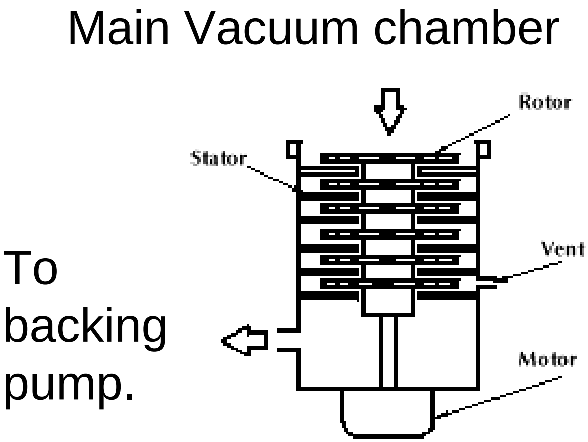


Turbo pumps. 10^{-3} to 10^{-11} torr range.

Blades rotate at ~ 50,000 RPM.

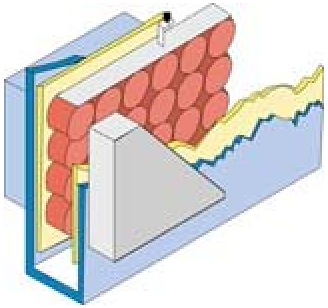
Gas molecules, hit by the underside of the angled blades, move with momentum in the direction of the higher pressure exhaust.

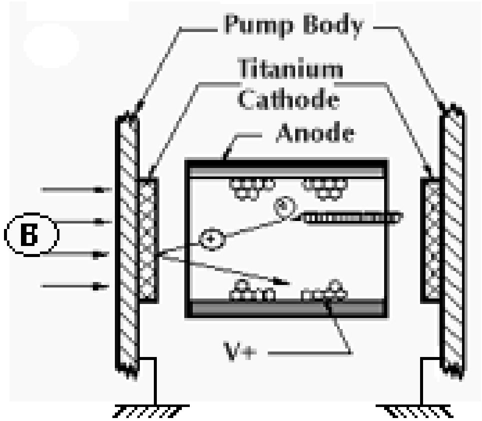
Must “back” these by a rotary vane or a dry pump. Be careful not to vent too quickly, nor to back-stream oil into the chamber! (This will happen if you vent the main vacuum while keeping the rotary vane pump under vacuum.)



Ion pumps. 10^{-5} to 10^{-11} torr range.
Gas molecules ionized, accelerated to titanium plates. Ions chemically react and/or buried.
Clean/zero vibration.

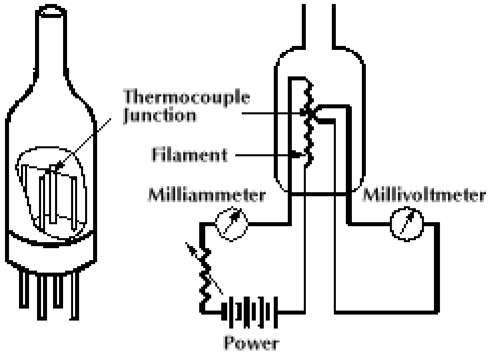
Ti lifetimes short for high pressures.



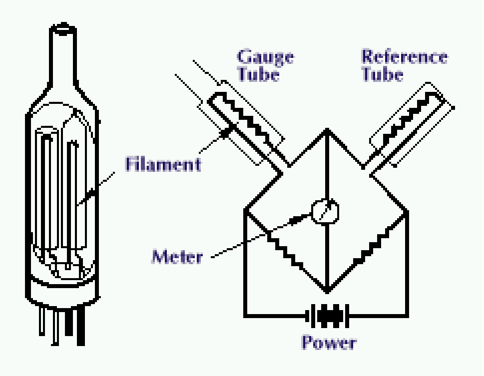


Pressure Measurement

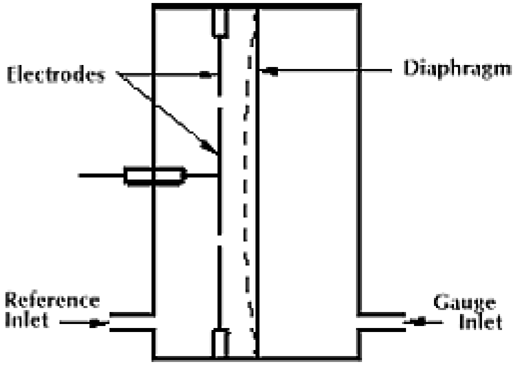
Thermocouple or TC guage. 10 to 10^{-3} torr range.
A hot filament loses heat to gas in system. TC measures temperature of filament.



Pirani. 20 to 10^{-5} torr range. Same as TC but compares system to a reference.



Capacitance Manometer. ~ 4 decades with varying max pressure (.1 – 1000 torr)
Most accurate for high pressure range.

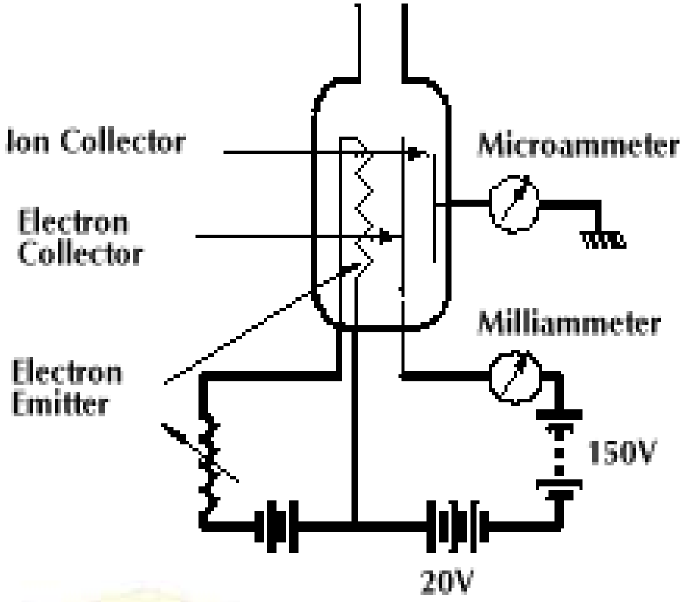


Pressure Measurement

Ion or Bayard-Alpert.
Glass tubulated 10^{-4} to 10^{-9} torr
Nude 10^{-4} to $2\text{x}10^{-11}$ torr

Low pressures dominated by “x-ray limit”
-Electrons striking electron collector give off soft X-rays. These strike the ion collector giving off photoelectrons, affecting measured current.

Different gas species have different sensitivities. Easy to ionize acetone, difficult to ionize H.



Residual Gas Analyzers (RGA’s).
Quadrupolar electric field – measures e/m ratio of ionized gas molecules. Most sensitive of all for UHV conditions.

Great for helium leak detection and vacuum diagnostics.

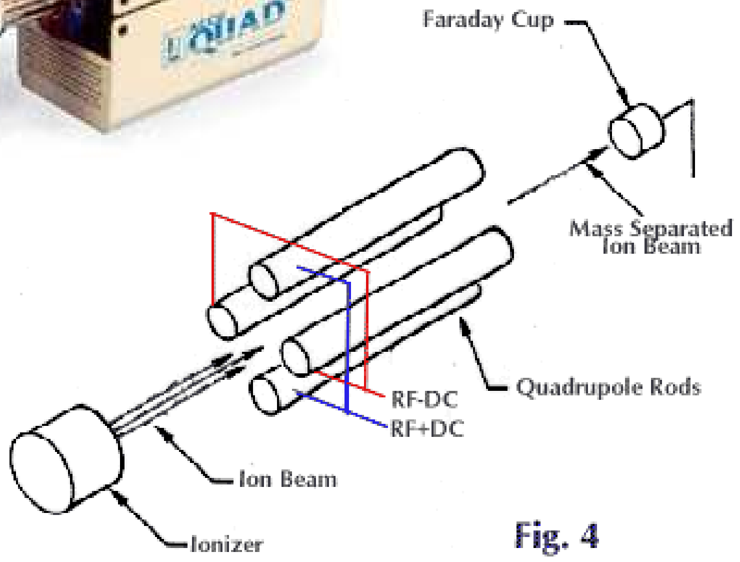
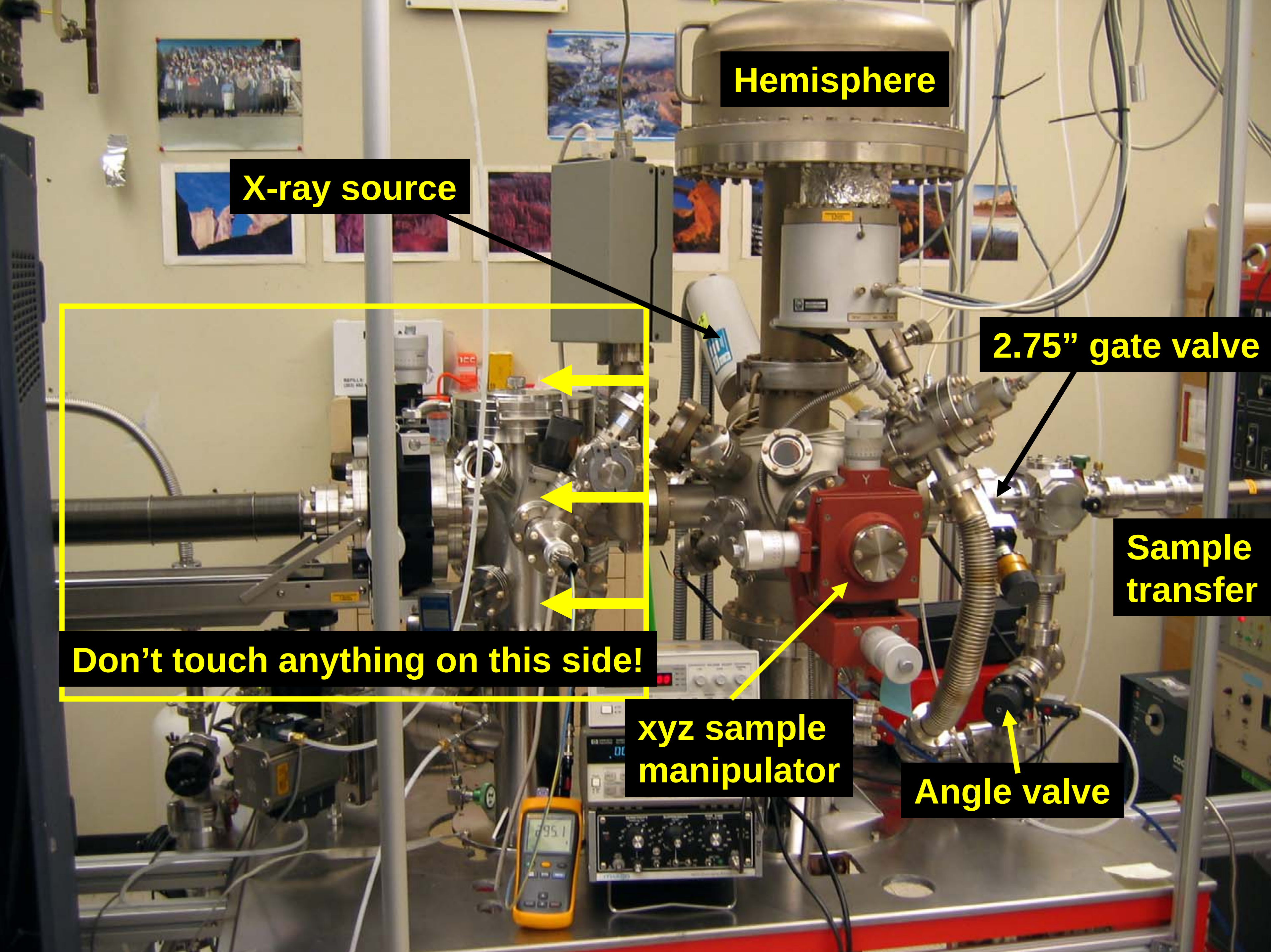


Fig. 4



Hemisphere

X-ray source

2.75” gate valve

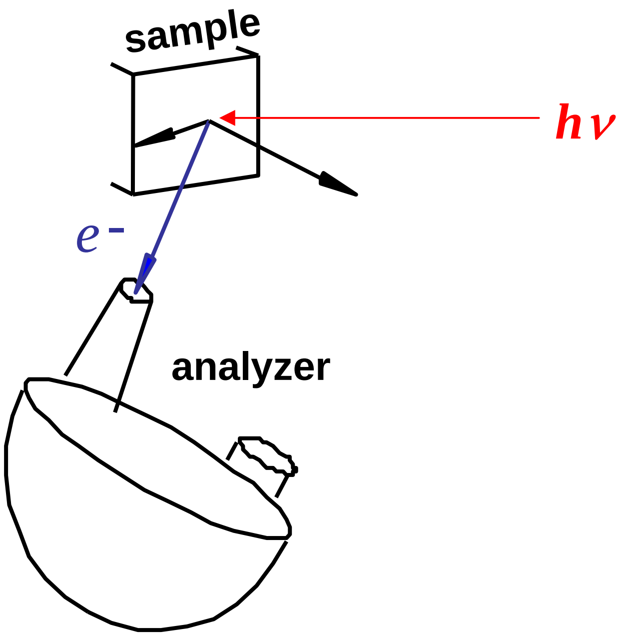
Sample transfer

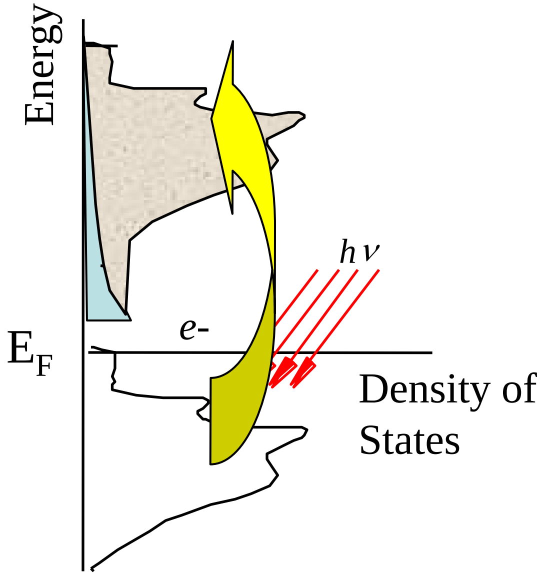
Don’t touch anything on this side!

xyz sample manipulator

Angle valve

Photoemission Spectroscopy





High K.E.   Low B.E.

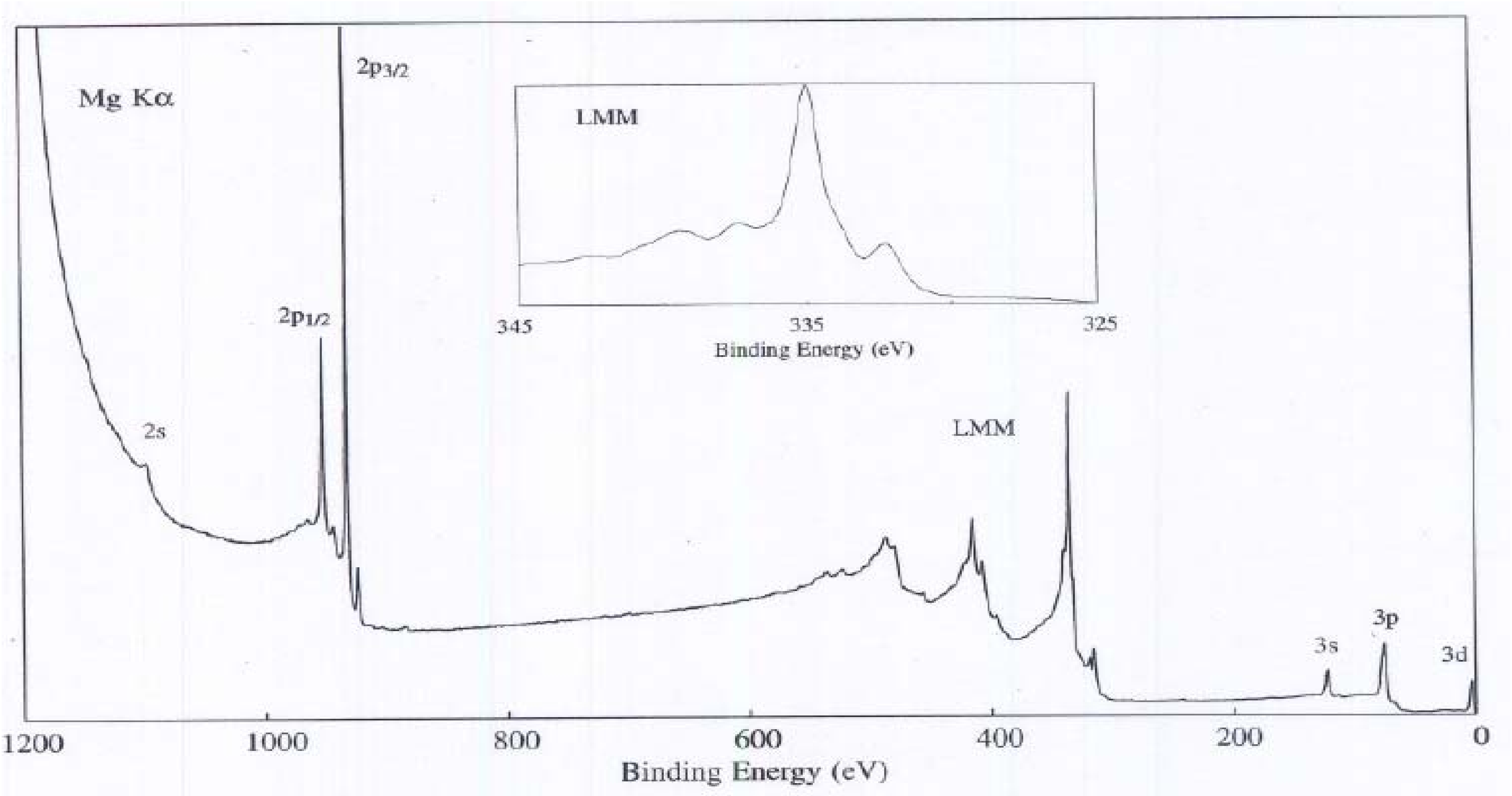
Low K.E. High B.E.

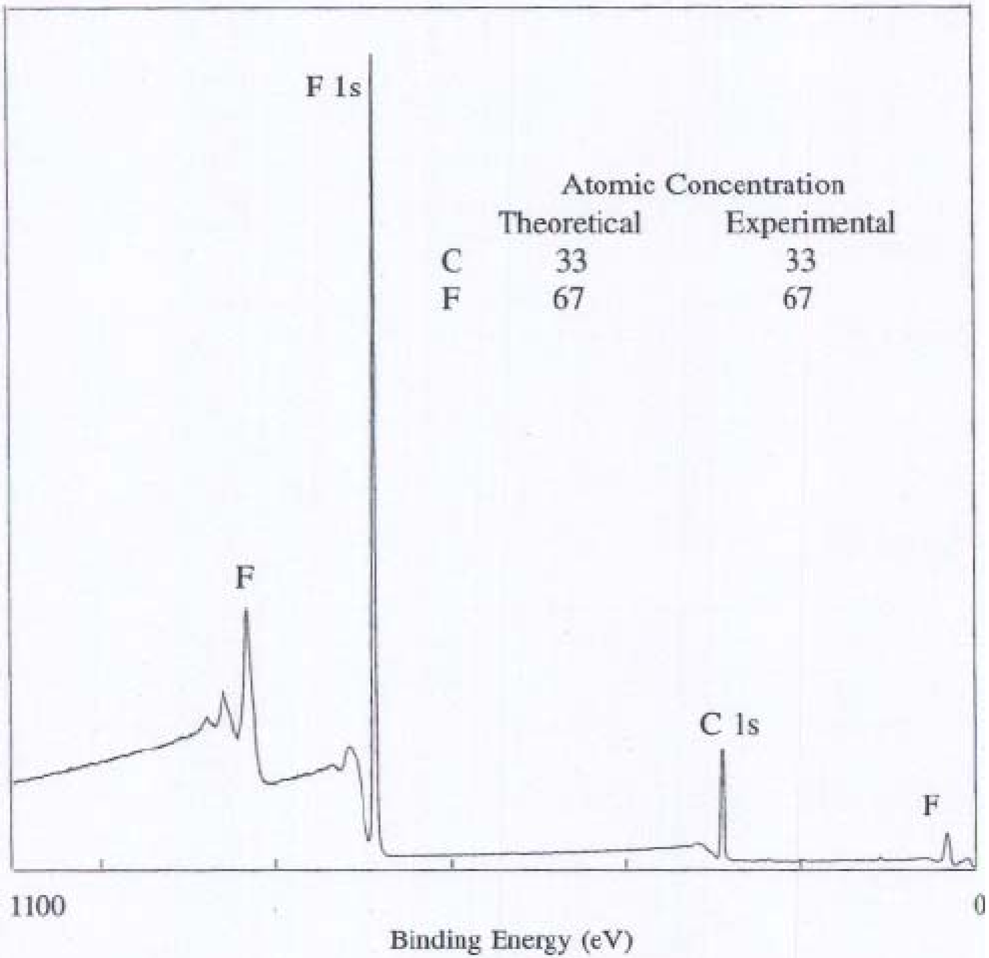
Primary electrons – no scattering events.
Contain information of density of states

Secondary electrons (inelastic background) – increases with decreasing kinetic energy.

$$E_{kin} = \hbar\omega - \Phi \ - |E_B|$$

XPS of copper metal





Atomic Concentration

	Theoretical	Experimental
C	33	33
F	67	67

Figure 13. Quantitative analysis of poly(tetrafluoroethylene).

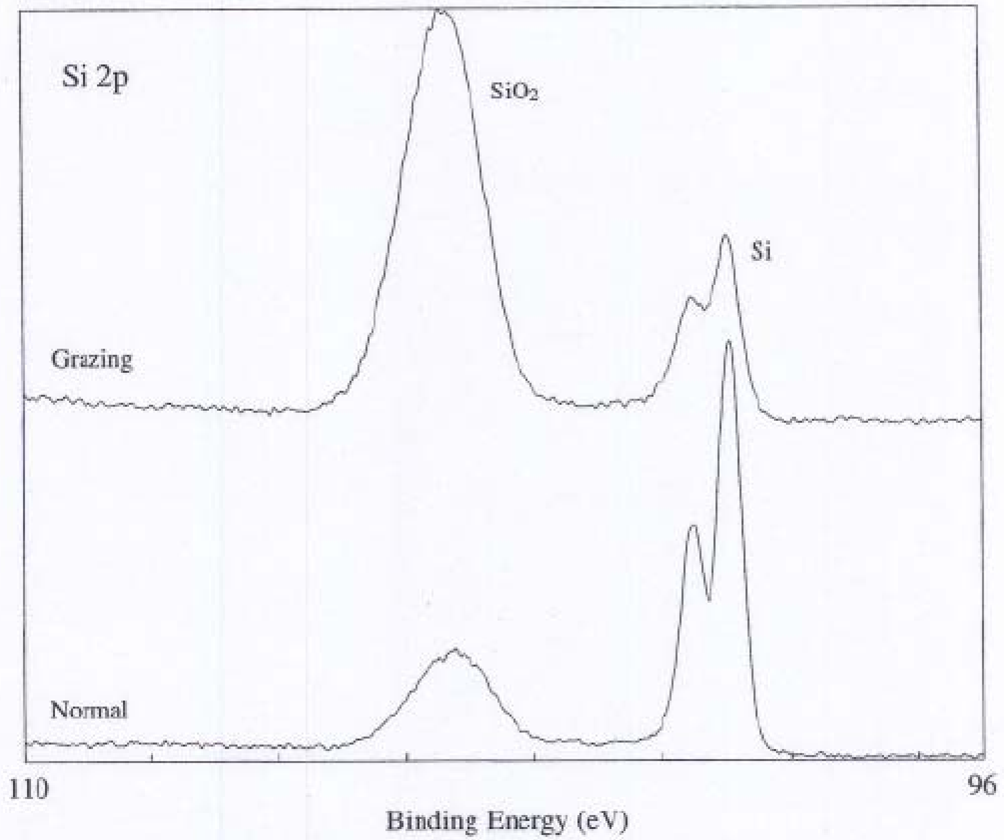
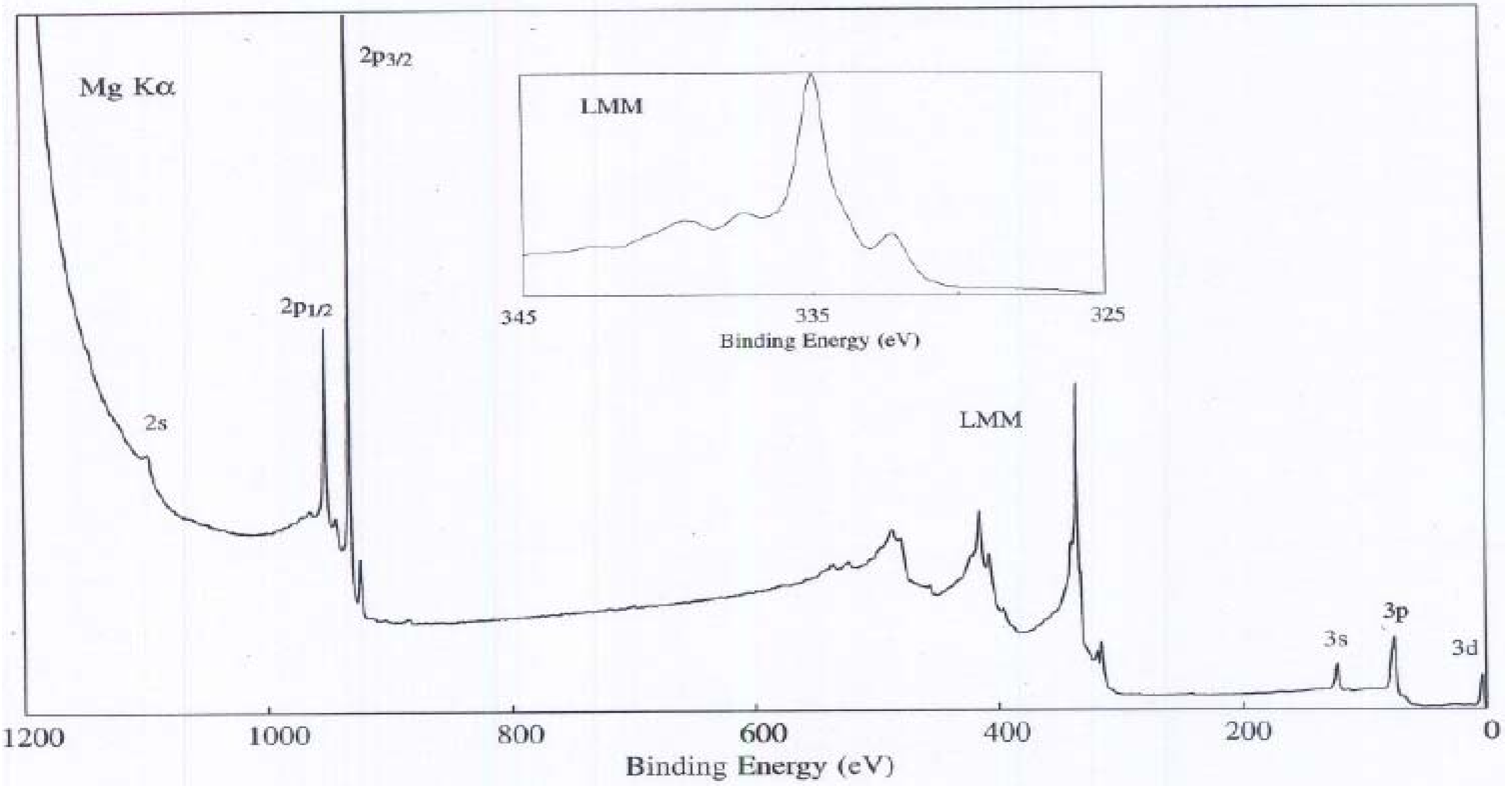
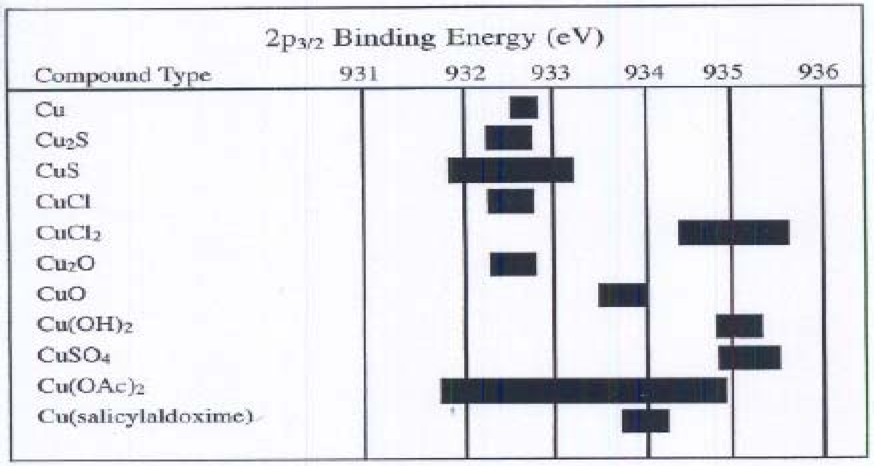
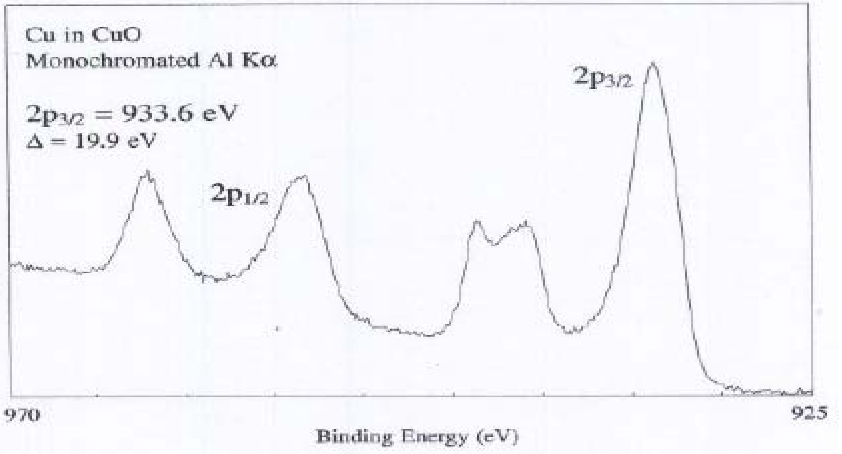


Figure 14. An example of the enhanced surface sensitivity achieved by varying the electron take-off angle. A thin oxide on silicon is enhanced at the low take-off angle.


Mg Kα
2p$_{3/2}$
2p$_{1/2}$
2s
LMM
3s
3p
3d
1200
1000
800
600
400
200
0
Binding Energy (eV)
LMM
345
335
325
Binding Energy (eV)


2p$_{3/2}$ Binding Energy (eV)
Compound Type
931
932
933
934
935
936
Cu
Cu$_2$S
CuS
CuCl
CuCl$_2$
Cu$_2$O
CuO
Cu(OH)$_2$
CuSO$_4$
Cu(OAc)$_2$
Cu(salicylaldoxime)


Cu in CuO
Monochromated Al Kα
2p$_{3/2}$ = 933.6 eV
Δ = 19.9 eV
2p$_{1/2}$
2p$_{3/2}$
970
925
Binding Energy (eV)