

Nanoscale < ~100 nm

1 grain of sand $\phi \approx 1\text{ mm}$ (10^{-3} m)
 human hair $\sim 50\text{ }\mu\text{m}$ ($50 \times 10^{-6}\text{ m}$)

Smoke particle $\sim 4 \mu\text{m}$ ($4 \times 10^{-6} \text{ m}$)

transistors ~ 2 - 20 μm (wide)

DNA ~ 2 nm wide (2×10^{-9} m)

1 nanometer = $\frac{1}{10^6}$ of a mm

$$1 \text{ ATOM} \sim \frac{1}{10} \text{ nm} = 10^{-10} \text{ m} = 1 \text{\AA}$$

We can now build quantum structures $\leq 20\text{nm}$ wide

3 main areas:

(not comprehensive list) Electronics → faster electronics
computers, memory

Materials → optoelectronics, catalysts
high performance materials
for applications such as the
auto industry
SENSOR DEVELOPMENT

**Medicine → DNA chips, drug delivery
toxic properties
BIO COMPATIBLE**

NEED TO CHARACTERISE MATERIALS ON THE SMALLEST SCALE POSSIBLE

- QUANTUM PHYSICS

NANOSCALE RESOLUTION NOT EASILY OBTAINED

\Rightarrow need a new breed of microscopes (pre 1980)

Why? Conventional optical microscopy has fundamental limit **DIFFRACTION LIMIT**

Numerical Aperture (NA)

- brightness depends on light gathered by objective
- depends on NA of objective

$$NA = n \sin \mu$$

Can also change n to improve NA
for example by using oil.

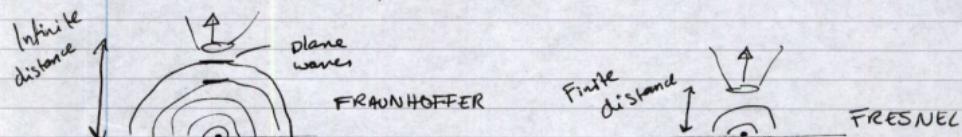
n = refractive index of medium b/w specimen & objective

μ = half angle of max. cone of light picked up by objective (angular aperture)

Theoretical max. NA of lens w/ air is 1.0, in practise < 0.95
Use of 'immersion oil' $n=1.51$ NA up to 1.4

NA determines resolving power of objective Resolution.
determines smallest observable structure.

Optical systems affected by diffraction of light.
Fresnel & Fraunhofer



Resolution in optical microscopes

TWO GOVERNING APPROACHES

Rayleigh Criterion & Abbe limit

Diffraction exists in two general traditional forms
FRESNEL & FRAUNHOFFER DIFFRACTION

- wavelike properties of light

Huygen's principle states that each point on a propagating wavefront is an emitter of secondary wavelets. Wavelets from propagating waves which can interfere & produce fringe pattern → DIFFRACTION

optical resolution: Abbe

- fundamental limit determined by wavelength of light & NA.
Known as Abbe limit, accurately describes resolution of optical microscope system

$$d = \frac{\lambda}{2 \times NA}$$

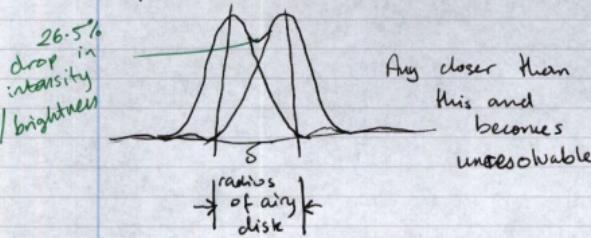
Rayleigh Criterion

When point source imaged by lens, diffraction pattern produced, Airy pattern.

Two sources just resolved when separated by distance equal to radius of Airy disk

$$d = \frac{0.61\lambda}{NA}$$

For High NA objective, resolution approx. ~~approximately~~ $\frac{\lambda}{2}$
practical limit ($\lambda \sim 350\text{nm}$, $NA \sim 1.4$) is $\sim 175\text{nm}$



Rayleigh

$$d = \frac{1.22\lambda}{2 \sin \mu} = \frac{0.61\lambda}{NA}$$

$$\begin{aligned} \text{Abbe limit} &= \frac{\lambda}{2 \sin \mu} \\ &= \frac{\lambda}{2NA} \end{aligned}$$

use $\lambda = 350\text{nm}$

$$NA = 1$$

$$\text{Abbe limit} = \frac{350}{2} = 175\text{ nm}$$

FRESNEL DIFFRACTION

concerned with what happens in the immediate vicinity of a diffracting object
 \Rightarrow light observation plane is at a finite distance (pin-hole camera)

FRAUNHOFFER DIFFRACTION

plane waves (collimated \rightarrow infinite) illumination, lens is focussing at finite plane

SCANNING PROBE Microscopy (SPM)

\rightarrow used to overcome the limits of optical microscopy
 BASED UPON THE INTERACTIONS OF A PROBE WITH THE AIM OF GENERATING RESOLUTIONS $< \mu\text{m}$ (10^{-6}m)

$$\sim < 100\text{ nm}$$

Range of instruments available - scan $10^{-10}\text{ m} \rightarrow \mu\text{m}$

1st development of an instrument for these type of measurements was the

SCANNING TUNNELLING MICROSCOPE (STM)
 (not scanning electron microscopy SEM or TEM transmission electron microscopy or variant on these two STEM)

An STM, Scanning tunnelling microscope uses the principle of quantum mechanical tunnelling
 Electron tunnelling was 1st demonstrated by Giaever in the early 1960's (shared a Nobel prize in 1973) (in superconductors)

Esaki also demonstrated tunnelling in diodes tunnel diodes, or Esaki diodes (V. FAST)

Young and co-workers produced vacuum tunnelling & electron field emission scanning (topographiner) in 1970+

Decade later (1981/1982) Binnig & Rohrer (IBM scientists) produced the STM \rightarrow Clear atomic imaging

how does it work?

what equipment does it require?

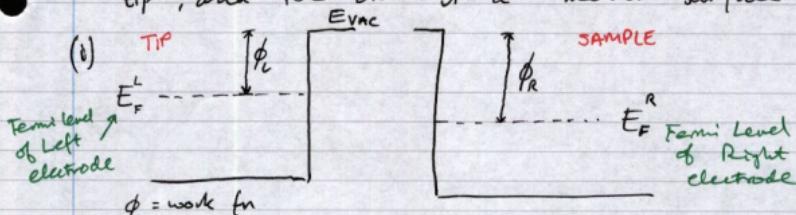
What is it that you actually measure?

Generated further developments in SPM such as atomic force microscopy (AFM)

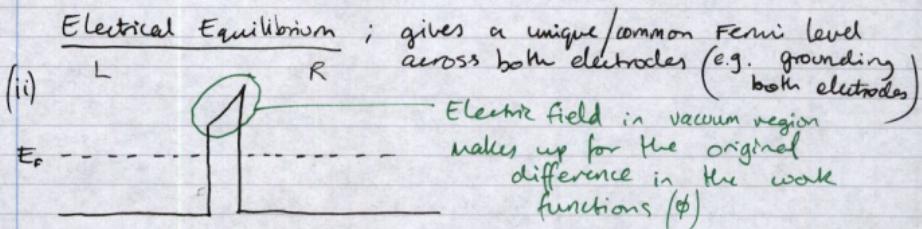
STM - how does it work?

In vacuum tunnelling, the potential in the vacuum region acts as a barrier to electrons. And if two metal electrodes are placed close together, the barrier for electron transfer is the potential in the vacuum region

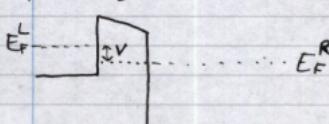
In STM, one of the electrodes is a sharp metal tip, and the other is a "metal" surface



electrodes, separated by vacuum
→ non-interacting

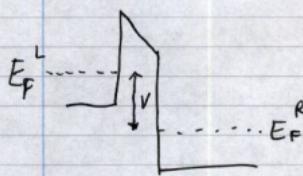


(iii) Apply voltage



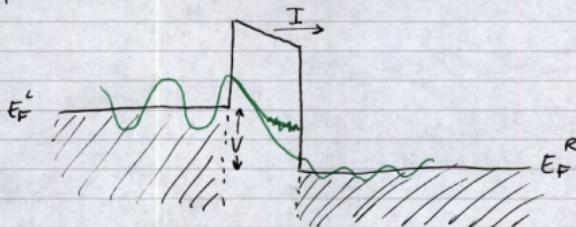
Field in vacuum barrier region is now both a contribution of $\Delta\phi$ and the applied V .

In a Classical sense the electrons need to overcome the barrier (field emission). But in STM, quantum mechanical (QM) tunnelling through the barrier is made possible.



field in the vacuum region is both
 $V \neq \phi$ contributed

QM allows for penetration of barrier due to the wavelike nature of the electrons. If the barrier is ~~small~~ narrow enough then the current can flow due to tunnelling of electrons. QM treatment predicts an exponential decaying solution for the electron wave function in the barrier.



I (the tunnel current) is proportional to the probability of the electrons tunnelling

[Rigorous treatment will follow]

indicates that $I \propto e^{-2kd}$

$$d = \text{barrier width (air or vacuum)}$$

$$k \approx \frac{\sqrt{2m\phi}}{\hbar}$$

$$\phi = \phi_{AV} = \frac{1}{2}(\phi_{sample} + \phi_{tip})$$

For QM tunnelling d has to be small
If $\phi = 5\text{eV}$ $I \propto e^{-1.025 \frac{\text{eV}}{\text{Å}} d}$

then a change of 1 Å ($1 \times 10^{-10}\text{ m}$) causes a change of nearly one order of magnitude (~ 10) in I .

\therefore very sensitive to change in tip-sample distance (d)

ALSO Equally important is its sensitivity to the local density of electronic states (DOS)
(hence posⁿ of the tip in relation to the atoms)

$$I \propto \rho(r_t, E_F)$$

density of states at the tip at Energy E_F

So how does the STM scan?

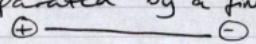
- Sharp tip to ensure lateral resolution

- Control tip sample distance for tunnelling (d is ~~approximately~~ typically $\sim 8\text{ \AA}$)

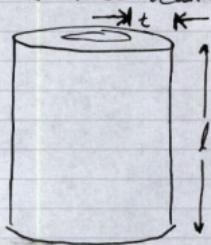
\Rightarrow ~~Answers~~ Answers are key to the STM, developed by Binnig & Rohrer

1. Piezo electric materials

aka piezo ceramics \rightarrow made from molecular dipoles (+/-ve charges) separated by a finite distance



e.g. a tube scanner
hollow tube



Voltage can be applied b/w the inner wall & outer wall

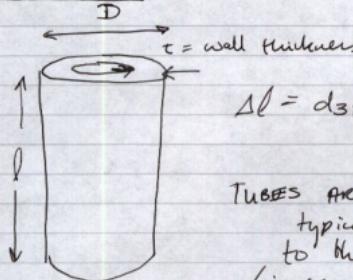
Lateral mode; displacement is perpendicular to the applied field.

$$\Delta l = d_{31} \frac{V \cdot l}{E}$$

V is equal to the voltage applied
 l is original length
 d_{31} is the piezo electric coefficient

A common piezoceramic used is PZT ($\text{Pb}(\text{ZrTi})\text{O}_3$)

TUBES ARE POLARIZED AT ELEVATED TEMPERATURES WITH TYPICALLY +ve voltage on the outer electrode (inner at ground)

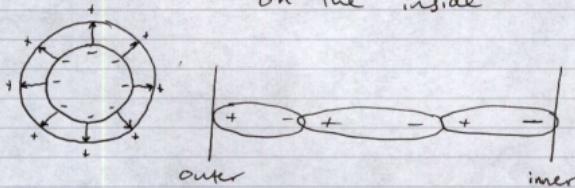


$$\Delta l = d_{31} \cdot V \cdot \frac{l}{t}$$

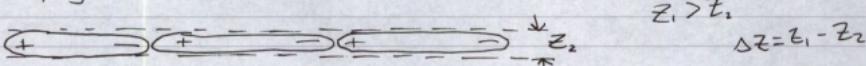
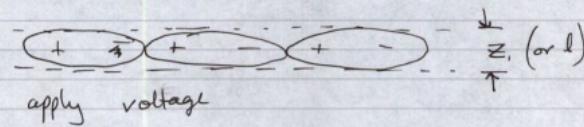
TUBES ARE POLARISED AT ELEVATED TEMPS.

typically apply a high +ve voltage to the outer electrode on the tube (inner at ground)

- dipoles align \rightarrow radial polarisation
 - ceramic is cooled \rightarrow permanently polarised
- can be ruined with high temp. and with high voltages
- +ve charge on the outside, -ve charge on the inside



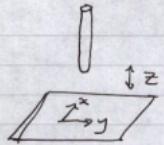
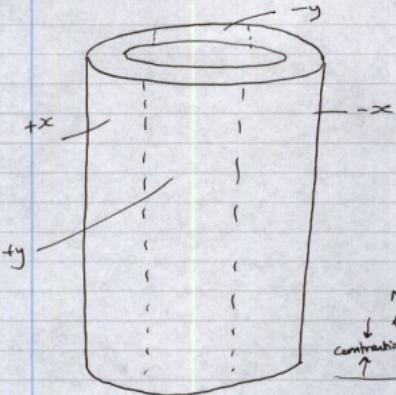
When the same sign voltage is applied (as was used to generate the polarisation) i.e. +ve on the outside
 \Rightarrow you get expansion in the polarising direction.



radius expands with expansion in polarising direction
 \Rightarrow contraction in perpendicular direction, i.e. Z.

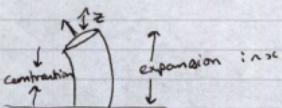
Opposite occurs if -ve voltage on the outside of the tube.

$\frac{1}{4}$ sections of $+x, -x, +y \neq -y$



same voltage but opposite polarity
on the x -directions ($+x, -x$)

- gives orthogonal x -movement
i.e. z does not change



to change z -displacement, apply a voltage
to all outer electrodes (the same), get
displacement in z .

Three main types of scanner:
original tripole \rightarrow bar or rod



~~Resonant~~ simplest system
polarisation in three orthogonal
directions.

TUBE SCANNER (BINNIG & SMITH, 1986)



most popular, low noise

Bimorph: stacked layers of piezo ceramic disks.

BENEFITS of TUBE SCANNER:

Compact
Simple

high sensitivity
high resonant freq.

(less interference)
mechanical vibrations

REIM: z -lateral mode displacement applied voltage

$$\Delta l = d_{31} \cdot \frac{V}{t} \cdot l$$

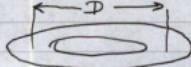
piezoelectric coefficient original length
thickness of wall

x, y lateral tip displacement

$$\Delta x = d_{31} \frac{l^2}{D.t} \cdot V_x \cdot \frac{2^{3/2}}{\pi}$$

These apply formulae only to
tube scanner

D = tube diameter (from $\frac{1}{2}$ wall thickness)



note; if D is small & t is small we get more displacement as l increases, range increases with the square.

Typical Tube values

$$d_{31} = \sim -2 \times 10^{-10} \text{ m/V}$$

contracts when the voltage applied

$$D = 6.3 \text{ mm}$$

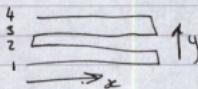
$$t = 0.56 \text{ mm}$$

$$l = 12.7 \text{ mm}$$

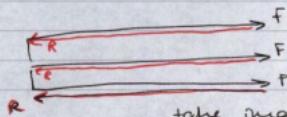
- BEWARE temperature effects, thermal expansion/contraction.

Non-linearities in the scan tube

hysteresis: return path differs to the extension path



suffer from hysteresis



take image from both forward & reverse path.
images will be same but offset.

non-linear deflection at high deflections.

i.e. need more voltage to create anticipated deflection.

Creep: driving voltage on the ceramic causes movement quickly but final 5% slow!

\therefore creeps the extension/deflection
use modified driving voltages
to overcome this effect.

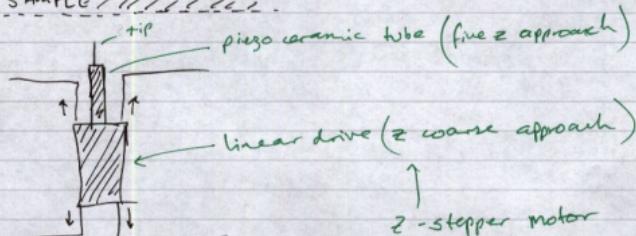
Tunnelling → How do you achieve it?

TUBE HOUSES THE TIP

WE NEED ~~THE~~ TIP TO APPROACH SAMPLE

DUAL MECHANISM OF COARSE and FINE APPROACH

/SAMPLE// / / / / / / / / .



So tunnelling is achieved by a combination of piezoceramic extension/retraction followed by a coarse step
Coarse step is still small < full extension of piezoceramic

1, extend piezo fully → If detected?
If no, turn ~~motor~~ worm

2, retract piezo fully

3, Move forward one small step (coarse)

4, extend piezo — If detected?

If no, go to step 2.

If yes, how do we maintain?

DEVICES FOR COARSE APPROX.:

- 'Stepper motor — fine pitched screw thread, $\frac{1}{2}$ turn < full extended piezo ($\sim 0.1 \mu\text{m}$)

- 'Inch worm — 'walks' with two feet, body is a piezo can extend & retract with ~~clamped~~ feet clamped

- 'stick-slip — uses frictional forces & inertia

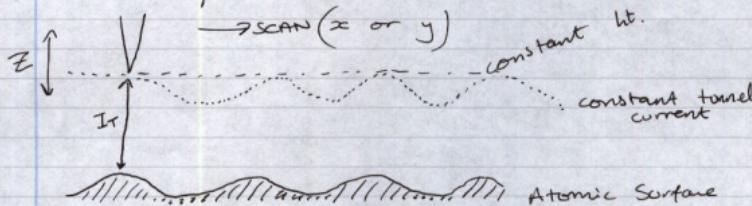
sample holder — the sample itself can also be moved by similar methods
— so tip can be positioned on areas of interest.

Control Electronics:

- to produce a scan, control x, y , & z .
- to maintain a tunnel current, I_T
- to not crash the tip → control z -gap

Modes of Operation

Two MAIN/DISTINCT MODES



Modes

- constant current, I_T , must follow the topography of the sample
- constant height mode, I_T varies as a function of dz → also reveals topography!

REMEMBER: STM maps the variation in the local density of states and is not just an atomic surface profiler.

requires good feedback controls!
unless pure constant height is used
once I_T established
(always need feedback to establish I_T) .

z -control

x, y -control

voltage & current measurement

Control Electronics

R_s = reference signal (set by user $\sim 1\mu A$)

E_s = error signal

$G(s)$ = gain section

$H(s)$ = mechanical section

$V_{piezo} \rightarrow piezo control element (H, G)$

{
tunnel current @ junction

{
↓

PRE-AMP (I/V CONVERTER)

↓
log amp. (to produce a linear relationship $\ln I \propto V_s$)

Aim of feedback is to minimise E_s , \propto i.e. maintain the pre-defined current (Reference current)

Gain Section is to optimise this response

→ quickly but controllably → steady fashion.

TIP CONSIDERATIONS

Material - depends upon conditions

FOR AIR : Pt-Ir

platinum-iridium wire

Pt wire

Au wire (gold)

reasons : ductile, non-oxidising

can be "cut" to form useful tips

drawn / cut & broken

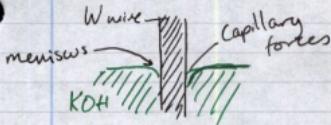
FOR UHV : (required for ^{"most"} atomically clean samples)

(ultra high vacuum) the oxidising issue is not important
Physically breaking a tip is not reliable
so move to electrochemically etching a tip.

etch allows for control of tip parameters
(aspect ratio) ↗ ↘

Electrochemical etching is an ion exchange process

e.g. etching Tungsten, W, in potassium Hydroxide (KOH) 10% by volume
meniscus determines the shape of the tip.



Key parameters,

- immersion depth of wire
- how wire was "introduced" into etchant
- rate of etching, voltage parameter
- cut-off circuit to "stop" etch

W is best material (so far) for reliable & reproducible etching for ~~3~~ STM applications

wet oxide covers tip apex, to get good atomic imaging in UHV, we need to remove contamination

No. of ways, most common

→ ion bombardment, Ar⁺ gun

or

→ tip conditioning, using I/V characteristics of tunnel junction to "heat" up tip & deposit tip material on the sample (reverse voltage pulses)

Tip needs to be sharp,

- so as to follow atomic scale features

Tip is defined by aspect ratio

$$= \frac{\text{tip height (length)}}{\text{tip diameter}} = 10:1 \text{ is a high ratio}$$

OR, defined by the radius of curvature at the end apex.

TIP SHAPE & RESOLUTION

→ scan-resolution concept of step size is important (see figure!) in the raster scan.

→ minimum tip radius of your STM probe

Consider an image with 512×512 pixel (points)

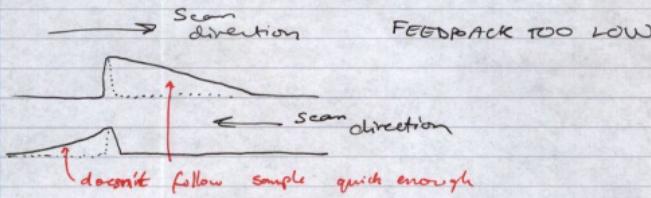
a $1\text{ }\mu\text{m} \times 1\text{ }\mu\text{m}$ scan would have

a resolution of $\sim 20\text{ \AA}$

⇒ SCAN LIMITED RESOLUTION
(STEP SIZE LIMITED)

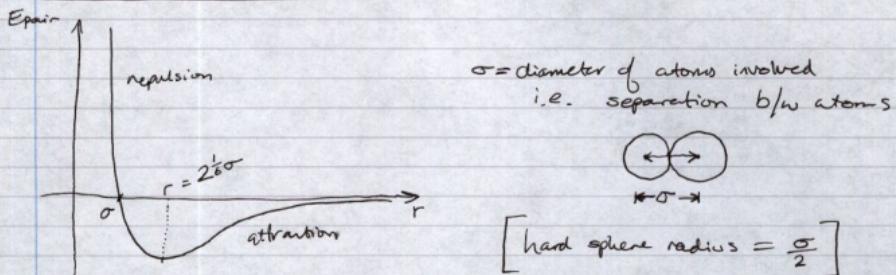
Figure two shows resolution limited by the size of the tip
19% is a bit like Airy radius seen earlier
— Rayleigh criterion

Figure 3, shows the effect of tip shape
STM dominated by the nearest atoms
to the surface.
However in "blunter" tips, more atoms
contribute to tunnelling



STM only useful in conductors and low band gap semiconductors.
Need new technique for insulators & high band gap semiconductors.

ATOMIC FORCE MICROSCOPY



Variation in the pair-potential energy [Epair] with

separation r b/w two atoms (identical) is described by the Lennard-Jones potential or (12:6 potential)

AFM seeks to exploit the forces of attraction & repulsion b/w atoms & molecules.

Forces of attraction & repulsion due to charge & in balance of charge (dipoles) is often termed generally Van der Waals forces.

$$E_{\text{pair}}^{(r)} = 4\epsilon \left[\left(\frac{\sigma}{r}\right)^{12} - \left(\frac{\sigma}{r}\right)^6 \right]$$

σ & ϵ are constants
 \rightarrow material dependent

σ = hard sphere diameter of atoms involved

when r is small, $\left(\frac{\sigma}{r}\right)^{12}$ dominates

$E_{\text{pair}} \rightarrow$ repulsive regime
Pauli exclusion principle (electron clouds overlapping)

When r is large, $\left(\frac{\sigma}{r}\right)^6$ dominates, E_{pair} is $-ve$ cohesion forces, attraction between atoms.

Van der Waals forces are also known as dispersion forces, despite attraction. Can be long range.

AFM takes a probe and "looks" at the forces of interaction b/w the sample material & the probe apex - exploiting the L-J potential.

Other forces:

electromagnetic interactions forces can be detected

ionic bonds (+ve & -ve ions, strong attraction)

$$F = \frac{q_1 q_2}{4\pi \epsilon_0 r^2}$$

Covalent bonding forces: (electron exchange)
charge density b/w atoms

Metallic adhesion forces: free valence electrons interacting b/w ionic cores

Repulsive forces: nuclear charge, repulsion,
electrons screened

Pauli exclusion principle

2 electrons can't have same state (space)

Van der Waals: dipole-dipole interactions
(permanent dipoles)
dipole-induced dipole
(dipole induces dipole in non-polar molecule)
dispersion forces
→ instantaneous dipole formed
in non-polar molecules

An AFM uses a probe at the end of a cantilever to sense the force interaction with the sample.

The Cantilever is "like" a record stylus, it can move up & down.

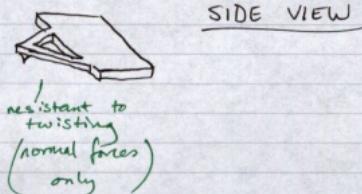
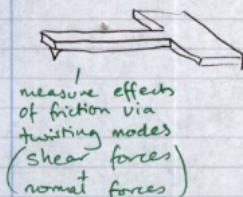
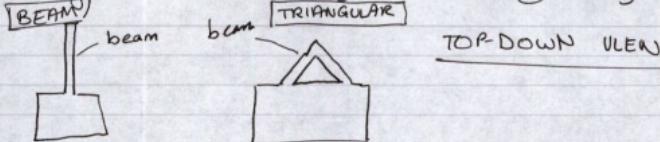
Cantilever has a low spring constant and can control force between tip & sample to great precision.

Can be made to many different designs.

Two main types using Si-based technologies

- Cantilevers usually made from Si or Silicon Nitride

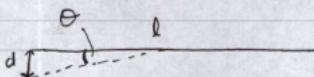
design is either ① Straight beam or ② triangular geometry



Hooke's Law defines the action,

$$F = -kd \quad \begin{matrix} \text{displacement (deflection)} \\ \text{Spring constant} \end{matrix}$$

Force minus sign indicates force acts in opposite direction to displacement.



$$\sin \theta = \frac{d}{l} \quad \text{for small angles } \sin \theta \approx \theta \quad \therefore \theta \approx \frac{d}{l}$$

$\therefore \theta$ is greater if l is small - i.e. short cantilevers give large change in θ .

How can θ be measured? (see handouts)

1 Optical lever

laser beam focussed on the back of the cantilever
reflections are directed onto a position sensitive detector
deflection allows detection by an "optical lever"
[↑]
amplifying the motion

Alternative methods (not commonly found) include:

2 Interferometer: light path changes, interference occurs,
changes in pattern reflect displacement.

3 Tunneling detector; metal coated cantilever forms part of
an STM system to monitor motion of
cantilever

Optical lever method is the one found in commercial
instruments — 4 segmented photodiode detection.

By measurement of displacement for a cantilever, the
force can be mapped.

A force-distance curve can be generated to study the
forces between the tip & the sample as a fn of
their separation.

Diagram (Handout) sequences 1-6
Cantilever deflection ($\Rightarrow F$) Vs distance b/w Tip & Sample

1 Large separation ($> 100\text{ nm}$)
 \Rightarrow spring like nature of cantilever, no deflection

2 Decrease separation \Rightarrow attractive regime, cantilever starts
to bend (measured deflection)

3 The tip is now v. close to contact, and can snap-into-contact
with the sample (rapid change)
due to forces \rightarrow tip is now in contact (large deflection)

4 As sample is pushed closer, cantilever deflection straightens
(less deflection, can vary somewhat if sample is soft!)

(Cantilever deflection diagram)

4 As the sample is pushed closer to the tip, the cantilever deflection changes and it straightens out (unless the sample is soft).

5 Cantilever deflection is zero, balancing the attractive & repulsive forces when in contact

→ low force is good for "contact" imaging
minimum

6 Repulsive forces take over, deflection upwards, Forces can damage sample.

AFM modes of operation → main types!

↳ CONTACT MODE : tip tracks the surface like a record stylus
i.e. the tip is in contact mode imaging
- also referred to as d.c. mode.

(Can be implemented in a no of ways:

- (i) constant-force mode ; piezo scanner drives the sample into the tip to a pre-defined deflection.
i.e. set up an imaging force ($F = -kd$)
• → maintains this force when scanning by z-motion of the piezo tube
⇒ we have to have feedback.

or (ii) variable deflection mode ; deflection signal used to show displacement

⇒ no feedback, forces can be quite variable & therefore high.

(e.g. can be used to 'cut up' material using high forces)

↳ NON-CONTACT MODE (a.c. mode) uses the long-range attractive forces which are weaker, more gradual & thus harder to exploit

two main types: tapping & true non-contact

(i) Tapping ; excite beam type cantilever, resonates

Amplitudes of $\sim 100\text{ nm}$

A ↴  so it can start to tap the surface

How

Stick a cantilever tip to a piezo electronic material and apply an a.c. voltage

- tip vibrates with a frequency of the a.c. voltage
- tune to resonance of the cantilever - max. deflection response

(ii) TRUE NON CONTACT: oscillation amplitude is damped by the long range forces

monitor A as a fn of distance - measure the van der Waals forces directly.

- Signals can vary too much & system loses feedback
- frequency or phase is more sensitive
use this in feedback.

We have considered the forces that exist between atoms & molecules

- forces in relation to scanning force microscopy

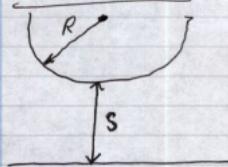
- need to consider environment
(forces change depending on whether measured in air, vacuum or liquid)
- scanning is dynamic → lateral forces/shear forces
e.g. frictional forces
- deformation of the sample → even destruction
- bonding b/w tip and sample
→ re-arrangements on surface
- many bodied problem, rather than simple two-bodied Lennard-Jones Potential

Van der Waals (non bonded): dipole-dipole, dipole-induced dipole or transitory (instantaneous) dipoles (London forces)

range of a few Å's to a few 100 Å's.
They can be modelled - simple power law with a specific Hamaker constant for the materials.

Force between tip (sphere) and a plane (sample)
is $\propto \frac{1}{s^2}$ Hamaker constant

$$F(s) = -\frac{AK}{6s^2} \quad \text{radius of curvature of the tip (sphere)}$$

V. der Waals

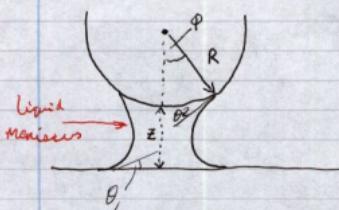
Sphere - Plane interaction

$$F(s) = -\frac{AR}{6s^2}$$

$A \rightarrow$ Hamaker Constant

$R \rightarrow$ Radius of the tip (sphere)

$s \rightarrow$ separation

Capillary Forces

tip - sample form a type of "micro contact"
These are well known to form
nucleation centres for condensation
- humidity & water vapour
can play a dominant rôle

A meniscus can form b/w the tip & the sample
- this produces pressure \Rightarrow force

Capillary force can be estimated from the following equation:

$$F = \frac{4\pi R Y \cos \theta}{1 + \frac{z}{R(1 - \cos \theta)}}$$

This assumes $\theta_1 = \theta_2$

and, $Y =$ surface tension of water = 0.074 N/m ($T = 20^\circ C$)

$R =$ radius of curvature of tip

$\theta =$ contact angle

$\phi =$ angle of the meniscus

$z =$ tip - sample distance

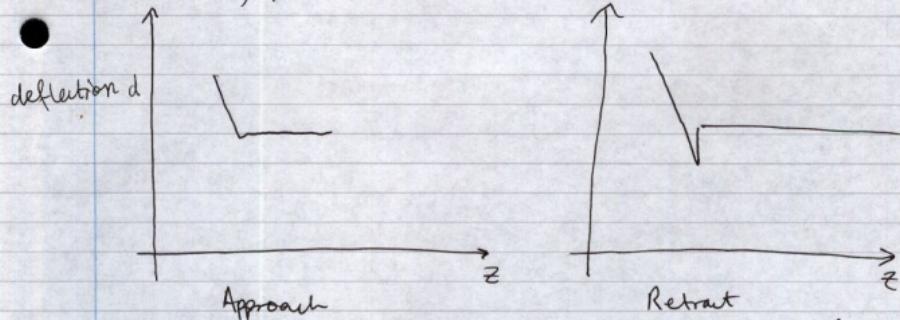
Max. attractive force $F_{max} \approx 4\pi R Y \cos \theta$

$F_{max} \approx 9 \times 10^8 \text{ N}$ which is quite high!
compared to other forces acting in SFM

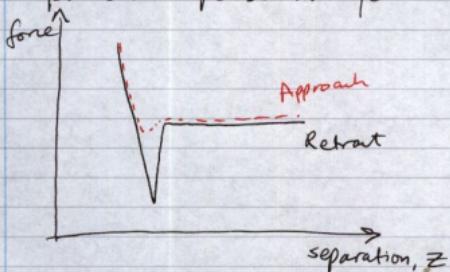
This attractive force can limit the repulsive force in humid conditions - Tip wear evident!

Force-distance curves can tell us all about forces & reactions (e.g. you could functionalise your tip with) specific coatings

for capillary forces;



hysteresis response in force-distance curves



The blunter the tip, the stronger the force.
Interestingly some polymers can act like liquids and show strong capillary/adhesion forces.

Can functionalise tip to look at other types of forces
e.g. 1 Magnetic forces, magnetic force microscopy (MFM)
use a ferromagnetic tip to probe samples magnetic properties.

e.g. 2 potentials, use a charged tip to map attractive/repulsive forces -
force microscopes like Kelvin probe & Scanning Capacitance microscopy.

Cantilever

- vibrating cantilevers are affected by forces
- using optical detection & lock-in-techniques the modulation freq. is chosen to be close to the resonant freq. of the cantilever
- Regulating to a constant amplitude or phase keeps resonance frequency f_r constant

$$f = \frac{1}{2\pi} \sqrt{\frac{k}{m}}$$

$$F = -kx$$

$$f_r \propto \sqrt{k - f'}$$

where f' is the force gradient ($f' = \frac{\partial F}{\partial z}$)

$$f, \propto \sqrt{k - F'}$$

This can be viewed as a modified or corrected spring constant

The probe tip will now measure or "trace" lines of constant force gradient.

sensitivity; minimum detectable force is determined by the sensitivity of the detector/deflection system typically better than 0.01 nm with a ~~1 N/m~~ 1 N/m cantilever ($k = 1 \text{ N/m}$)

This corresponds to a minimum detectable force of $F_{\min} = 10^{-7} \text{ N}$

Force gradient detection by interaction with long range V. der Waals forces can be considered in an a.c. mode where the resonant frequency changes and "monitored"

spatially varying force i.e. a force gradient

$$\frac{\partial F_x}{\partial z} = F' \quad \text{gives } k_{\text{eff}} = k - F'$$

$$f = \frac{1}{2\pi} \sqrt{\frac{k_{\text{eff}}}{m}}$$

Change in effective spring constant changes the resonance frequency of the cantilever

$$\omega_0' = \left(\frac{k_{\text{eff}}}{m} \right)^{1/2} = \left(\frac{k - F'}{m} \right)^{1/2}$$

for small F' compared to k , we get by expansion

$$\omega_0' \approx \omega_0 \left(1 - \frac{F'}{2k} \right)$$

where ω_0 is the response with no forces or force gradient acting on cantilever

for attractive forces $F' < 0$ (+ve)

$$\omega_0' < \omega_0$$

PH-863 Probing the Nano Scale

9th March

$$k_{\text{eff}} = k - F'$$

$$\text{where } F' = \frac{dF_n}{dx}$$

\leftarrow normal to the surface/cantilever

$$\therefore F' = \frac{\partial F_n}{\partial z}$$

change in the spring constant, causes a change in the resonant frequency of the cantilever.

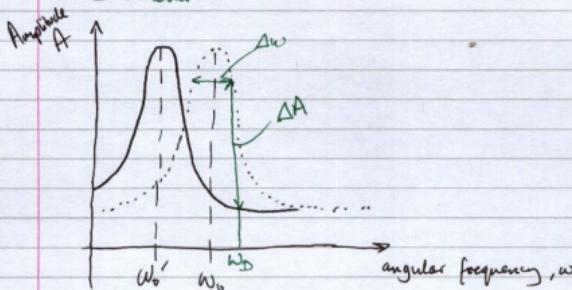
$$\omega_0' = \left(\frac{k_{\text{eff}}}{m} \right)^{\frac{1}{2}} = \left(\frac{k - F'}{m} \right)^{\frac{1}{2}}$$

for small F' compared to k , we get the expression

$$\omega_0' = \omega_0 \left(1 - \frac{F'}{2k} \right)$$

$F' > 0$ for attractive forces,
 $\omega_0' < \omega_0$

response with
no force, free
cantilever



Shift in the resonance curve \Rightarrow when driven at a fixed frequency ω_0 , this results in the change of the oscillation amplitude A , by ΔA . Deflection sensor can measure both change in A , or phase, Δw .

The Amplitude of the cantilever vibration depends on the resonance angular frequency ω_0' according to the normal behavior of a damped harmonic oscillator (Lorenzian lineshape)

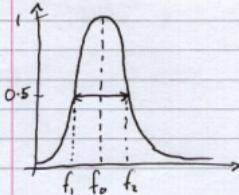
$$A(\omega_0') = A_0 \left(\frac{\omega_0'}{\omega_0} \right)$$

A_0 is the amplitude when $\omega_0' = \omega_0$

$$\left[1 + Q^2 \left(\frac{\omega_0}{\omega_0'} - \frac{\omega_0'}{\omega_0} \right)^2 \right]^{\frac{1}{2}}$$

Q is the quality factor of the cantilever resonance

$Q = \text{quality factor} = \frac{\text{resonance frequency}}{\text{Full width, half max. value}}$
 of the resonant peak (FWHM)



$$Q = \frac{f_0}{f_2 - f_1} \quad \text{high } Q \Rightarrow \text{shaper peak}$$

For maximum sensitivity to changes in ω' the drive frequency is chosen so that $A(\omega')$ has its steepest slope, this occurs at

$$\omega_d \approx \omega' \left(1 \pm \frac{1}{\sqrt{8} Q} \right)$$

Scanning near-field optical microscopy (SNOM), (NSOM)

- this technique utilises optical effects taking place in the near-field.

"near-field optics is the branch of optics that considers configurations that depend on the passage of light to, from, through or near an element with a subwavelength feature, and the coupling of that light to a second element located a sub-distance from the first"

any feature smaller than wavelength

(1928) Edward H Synge proposed a sub-wavelength hole/aperture in a screen as mechanism for imaging at sub-wavelength limits
 his letters to Einstein bore a remarkable similarity to modern SNOM instruments.

(1972) Ash & Nicols used microwaves ($\lambda = 3\text{cm}$) to image metal gratings (0.5mm)

(1984) Pohl's group in Zurich (BM) & Lewis (Imperial)
 published SNOM instrument details - visible light

B.E.E.M: Ballistic Electron emission Microscopy
- used to characterise buried interfaces
sub-surface

- electron transport in structures is very important

- electronic materials/interfaces are normally characterised by tools such as,

I/V , C/V , photoresponse
photoelectron spectroscopy etc...
capacitance/voltage x-rays or UV

All large scale measurements \rightarrow no (little) spatial resolution

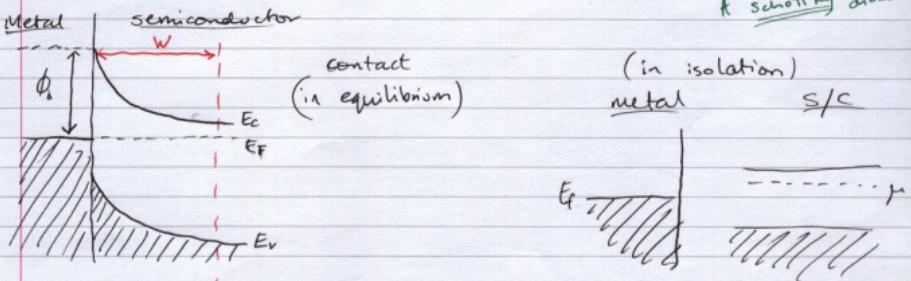
BEEM produces ballistic electrons produced from an STM tip!
a localised probe

↳ when applied to a layered structure it can give v. important information on the "quality" of the interface
i.e. its electronic properties

BEEM was developed in ~1988
how does it work?

- We need an example of a device interface & need to understand how interfaces are important for device physics.

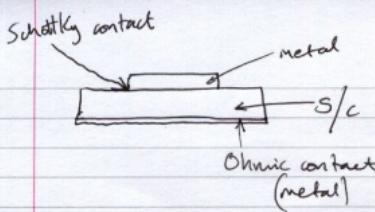
- A metal contact to a semiconductor can form a diode k schottky diode



n-type contact to
a metal and a barrier
forms ϕ_b

Inhibits electron flow from
S/C to metal (forward bias)

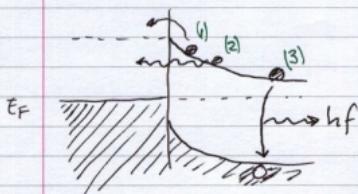
for electrons w/energy less than $e\phi_b$



Tailor ϕ_b properties by understanding the interface physics
e.g. a different crystalline face of a S/C may give different ϕ_b

But can we measure localized charges in ϕ_b ?

Transport over Schottky Barrier



(1) Thermionic emission or diffusion

(2) QM tunnelling - depends on W. depletion width of S/C (narrower better)

(3) Recombination

We are going to consider (1) - thermionic emission-diffusion

for high mobility S/C thermionic emission dominates.

$$\text{The diode eqn} \Rightarrow I = I_0 \left(e^{\frac{V}{nKT}} - 1 \right)$$

I = current

V = applied voltage

n = ideality factor

k = Boltzmann's constant

T = temp (K)

I_0 = saturation current

Moderate forward bias $e^{\frac{V}{nKT}} \gg 1$

$$\therefore I = I_0 e^{\frac{V}{nKT}} \quad [1]$$

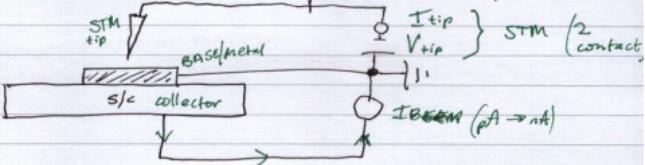
$$I_0 = A^* T^2 e^{-\frac{\phi_b}{kT}}$$

$$A^* = \text{Richardson constant} = \frac{4\pi e M_e k^2}{h^3}$$

reduced mass of electron

By taking \ln of [1] & plotting we get $\ln I$ vs. V and ϕ_b can be determined.
The ideality factor, n should = 1 if it's a perfect diode deviation from $n=1$ indicate problems with diode.

If we form a 3-contact measurement using an STM tip
of tunneling we can observe current flow across buried
interfaces e.g.



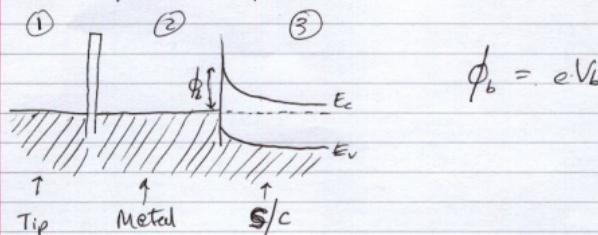
Tunnel Current established b/w tip and "bare" metal contact
 -ve voltage applied to tip - tip becomes electron emitter.
 but some of the tunnel electrons can travel across the barrier b/w the metal and S/C \Rightarrow "collected" in S/C
 \Rightarrow BEEM electrons

Under what conditions?

energy barrier diagrams for 3 contacts

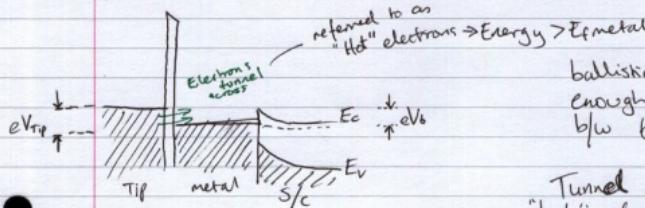
- at equilibrium, $V_{Tip} = 0$

(a) Contants



$$\phi_b = eV_b$$

(b) at $V_{Tip} > V_b$



ballistic electrons are energetic enough to overcome the barrier b/w the metal & S/C

Tunnel electrons after the metal as "hot" electrons.

- Sample must have two layers at least, with potential barriers b/w them
- Schottky is the simplest contact.

Top surface of the metal base is grounded, so the STM measurement is unaffected by the BEEM measurement.

Ballistic current which flows from the metal base to the S/C collector will vary as a fn of two things,
 V_{Tip} & V_b

General Considerations:

- an n-type S/C is used in Schottky measurements for BEEM.

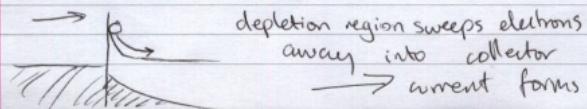
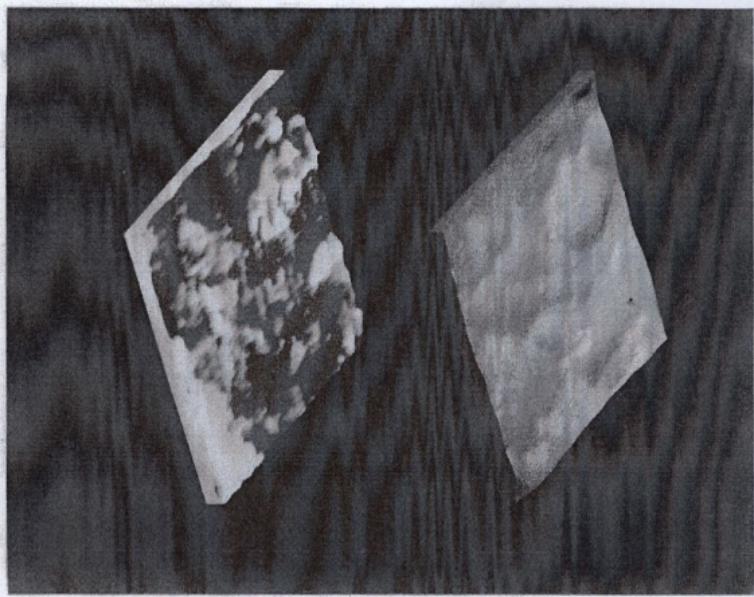


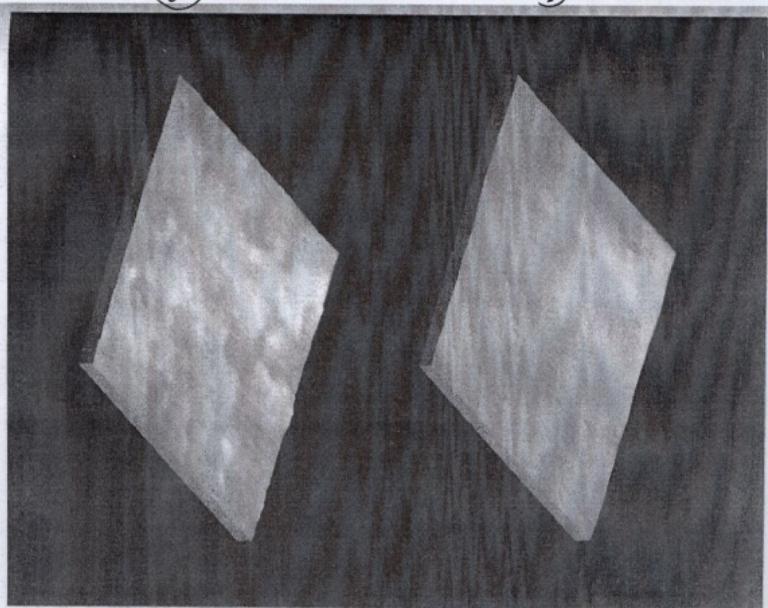
Figure 1



Surface topograph-BEEM image pair for the AuIn/GaAs(100) structure.

An surface. Range of height across this image is 63 Å. (a) Topograph of the obtained at $V = 1.5$ V and $I_t = 1.0$ nA. Collector current ranges from less than 0.1 pA (black) to 14 pA (white).

Figure 2



Topograph-BEEM image pair for Au/AIAs/GaAs(100) fabricated with two monolayers of AIAs. Image area is 510 Å × 390 Å. (a) Topograph of the Au surface. Range of height in this image is 24 Å. (b) BEEM in interface image, obtained at $V = 1.5$ V and $I_t = 1.0$ nA. Average collector current across the image is 2.0 pA; RMS variation is 0.7 pA.

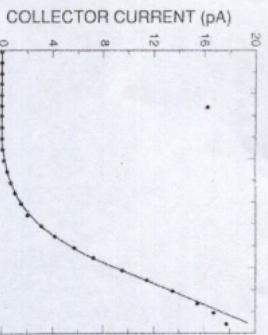
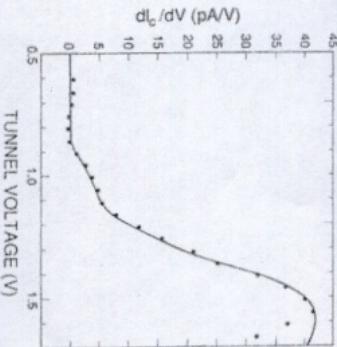


Figure 3

BEMM I_c - V spectrum (circles) for Au/n-GaAs(100). Plotted tunnel voltage is equivalent to $V_{\text{sample}} - V_{\text{tp}}$. Also shown is a fit of a three-threshold model to the data. Three different V_b parameters and three separate R values are allowed to vary during the fit. Fixed values of 3 eV and 15 Å were used for Φ and s . The thresholds derived from the fit are at 0.89 V, 1.18 V, and 1.86 V.

Figure 4



Derivatives dI_c/dV of the experimental and theoretical spectra multiple-threshold nature of the data can clearly be seen.

in a p-type



accumulation region

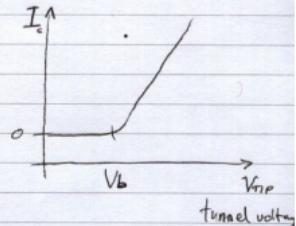
electrons remain
at junction.

Consider Conditions for BEEM

By varying V_{TIP} the energy of the "hot electrons" can be varied (i.e. their energy distribution)
 $\therefore V_{\text{TIP}}$ allows a spectroscopy measurement of interface transport to be performed.

V_{TIP} vs. BEEM current $I_{\text{beam}} (I_c)$

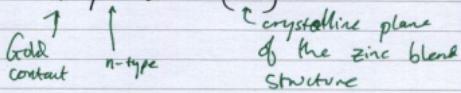
for $V_{\text{TIP}} < V_b \rightarrow$ No BEEM current!
thus on-set of I_c indicates V_b .



n.b. Scattering of electrons is an issue!
metal layer must be thin ($< 10^{\text{s}} \text{ nm}$)
so that scattering does not dominate
and inhibit localised BEEM measurement.

BEEM EXAMPLE

Interface between Au / n-GaAs (100)



On Handout:

Figure 1 :

- (a) STM topography
 - (b) BEEM interface image
- regions of high current, regions of low current
 $I_c \uparrow \rightarrow V_b \uparrow$ High V_{TIP}

VARIATION IN V_b !!!

\rightarrow not related to defects or cleanliness of interface
but Ga preferentially bonds to Au, As remains at the interface. As islands at interface.

Figure 2 : uses an interfacial layer to stop Ga reaction
AlAs used, Al stays bonded to As.

*Following from Handout *

Let's look at the conservation laws in the "classical" non-relativistic case for the photon \leftrightarrow particle interaction

$$(i) \boxed{\text{Momentum}} \quad p_i = m v_i$$

$$\lambda = \frac{h}{P}$$

$$R = \frac{2\pi}{\lambda} \quad P_{ph} = h \cdot k$$

$$|k| = \frac{2\pi}{\lambda} = \frac{\omega}{c}$$

i=initial
f=final

wave vector

$$\text{Conservation: } \underline{p_i} + \underline{t k} = \underline{p_f}$$

(ii) **Energy** Internal energy affects particle

photon
tw

$$\dots E_i + KE_{\text{external}}$$

$$\text{Conservation: } E_i + t_{\text{tw}} = E_f$$

often written as; $t_{hw} = t_{hw_f} = E_f - E_i$ (*)

↑
photon energy to create
a transition b/w energy
levels

However, we have stated that the photon moves with relativistic velocity (speed of light) and hence E has to be formulated in relativistic fashion.

Thus we find for the energy conservation;

$$f(w_{fi}) = \sqrt{p_f^2 c^2 + (M_0 c^2 + E_f)^2} - \sqrt{p_i^2 c^2 + (M_0 c^2 + E_i)^2}$$

This becomes rather complex, we don't like $\sqrt{\cdot}$

We can do an expansion

(e.g. $\sqrt{1+x}$ with small x , $= 1 + x - \frac{x^2}{2} + \dots$)

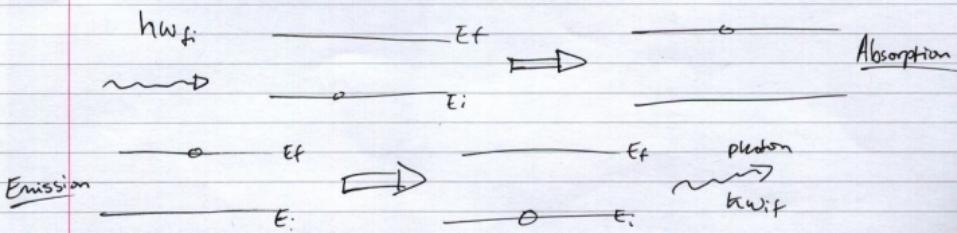
For the specific case, the parameter x can be $\frac{v}{c}$, this is small
(without the details);

$$\hbar\omega_f = \hbar\omega_i + \dots \text{ a few terms of correction}$$

photon energy for the particles at rest, $v=0$

one of these is important to the laser cooling, related to photon recoil.

Thus far we have a photon being absorbed by the particle. But once the "atom" is excited it tends to live for only a certain period in the higher internal energy state, before decaying in energy to the lower state.



One finds in general that $\hbar\omega_f - \hbar\omega_i = \hbar\Delta\omega (\neq 0)$

absorbed emitted

For the relativistic case of absorption/emission cycle, we find (after some maths)

$$\hbar\Delta\omega \equiv \frac{1}{2} \frac{\hbar^2 \omega_0^2}{mc^2} \left(\equiv \frac{\text{square of the photon energy}}{\text{rest mass energy}} \right) \quad (1)$$

This change in energy must be related to a change in velocity - because we can't change the quantum character of the atom or photon.

$$\hbar\Delta\omega = \frac{1}{2} m \Delta v^2 \quad (2)$$

We can find Δv value in the following way;

$$\Delta v = \frac{\Delta p}{m} = \frac{h/|e|}{M} = \frac{h \omega_0}{Mc}$$

~~from eqns (1) & (2)~~

For Na atom (sodium)

$$\Delta v = \frac{\Delta p}{m} = \frac{\hbar / k}{m} = \frac{\hbar \omega_0}{mc}$$

Numerical example: Sodium atom (Na)

$$m_{\text{Na}} = 23 \times 1.66 \times 10^{-27} \text{ kg}$$

$$\hbar = 1.05 \times 10^{-34} \text{ J.s}$$

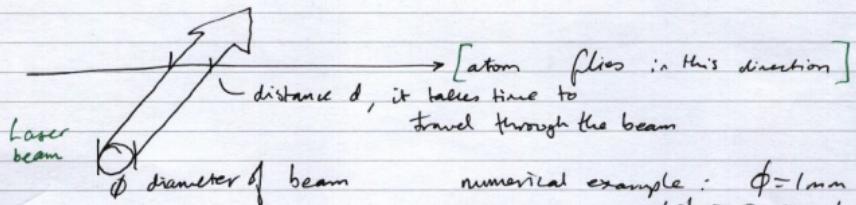
$$\omega_0 = \text{Na D-line} = 3.2 \times 10^{15} \text{ s}^{-1}$$

$$c = 3 \times 10^8 \text{ m/s}$$

$$\Delta v \approx 0.03 \text{ m/s}$$

This value is very small! But it is for a single event!
So a single event is insufficient to achieve the goal of stopping/cooling the (Na) atom.

Multiple events can do the trick!



$$\text{numerical example: } \phi = 1 \text{ mm} \\ |v| = 1000 \text{ m/s} \\ \Rightarrow T = 1 \mu\text{s}$$

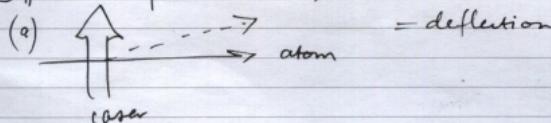
This is a long time in comparison to radiative lifetime

→ for atoms is typically $10 - 100 \text{ ns}$

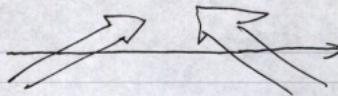
⇒ multiple emission & absorption events will occur
∴ multiple energy/momentum transfers

NOTE: momentum transfer needs to be in opposite direction of atom's ~~path~~ travel
↳ i.e. is along direction of the incoming photon

Different manipulation cases;



(b) heating / cooling
accelerating decelerating



Effects of (b) are exploited in laser cooling (and subsequently trapping)

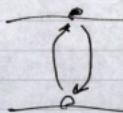
Let's see how this works for the Na atom from our previous example.

Laser beam diameter $\phi = 1\text{mm}$
most probable velocity of Na at $300\text{K} \approx 400\text{m/s}$

\Rightarrow transit time of atom through photon beam.
 $T = \frac{0.001}{400} = 2.5 \times 10^{-6} = 2.5\mu\text{s}$

transit time for absorption or emission for D-line
 $T_D \approx 16\text{ns}$

$$T_{\text{up-down}} = 2 \times 16\text{ns} = 32\text{ns}$$



transition cycles during passage
of atom through the beam $n = \frac{T}{T_D} = \frac{2.5\mu\text{s}}{16\text{ns}} \approx 78$

remember that ΔV
for single event $= 0.03\text{m/s}$

$$\Delta V_{\text{TOTAL}} = n \cdot \Delta V \approx 2.3\text{m/s}$$

If we relate this to the most probable velocity
 $v_p \approx 400\text{m/s}$

we need to repeat this process a number of times to cool down

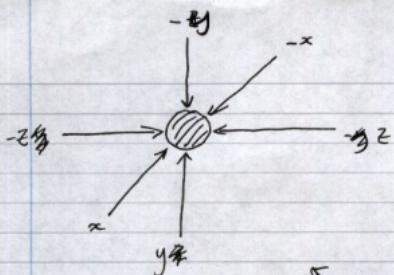
$$\frac{v_p}{\Delta V_{\text{TOTAL}}} = \frac{400}{2.3} \approx 173$$

The interaction ϕ needs to be $173 \times 1\text{mm} \approx 173\text{mm}$

this is possible
in the lab

So far this is all in 1-D

To cool atoms in 3-D we need to expand our treatment
 Total of 6 laser beams, $\pm x, \pm y, \pm z$ (counter propagating beam)



With this method of cooling one can actually get very low temperatures
 → helping to set up a optical based trap

→ Magneto-optical trap (MOT)

This is often ~~termed~~ termed as an "OPTICAL MOLASSES"

without magnetic field, particles can drift out of the "molasses" region

becomes a trap with a mag. field

But the temperature in the overlap region can get very "cold"

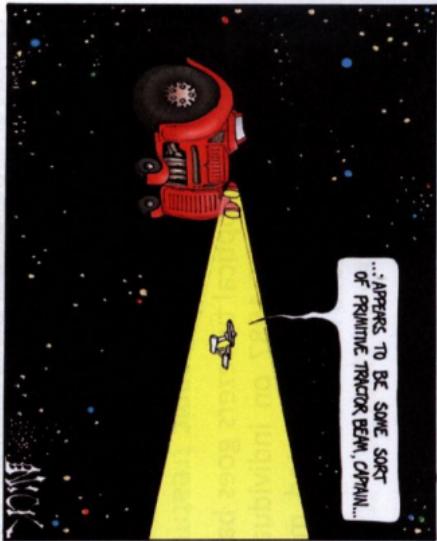
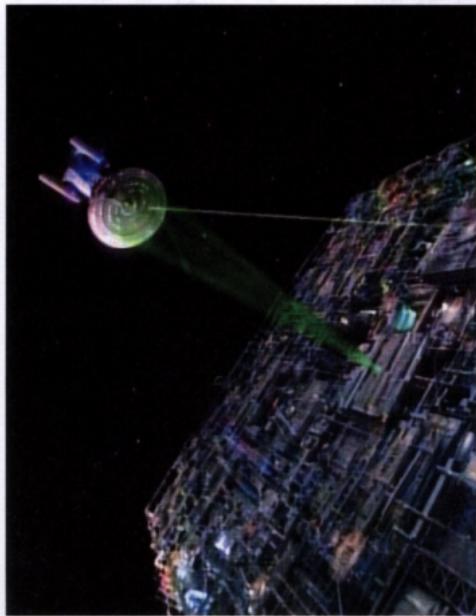
$$\text{Temp}_{\text{laser cooling}} \approx 1 \mu\text{K}$$

recall $v \approx \sqrt{T}$

$$v = v_{\text{thermal}} \sqrt{\frac{10^{-6} \text{ K}}{300 \text{ K}}} = 400 \times 6.06 \times 10^{-3} \\ = \underline{\underline{0.023 \text{ m/s}}}$$

24th April

Obiects



Optical Tweezers

We've seen how we can cool atoms in a small volume (and discussed how, with a magnetic field, a magneto-optical trap can be generated). BUT what about larger particles.

In Sci-Fi we have the Borg ship holding the Starship Enterprise in a Tractor beam....
definitely sci-fi!

However smaller particles can be trapped and manoeuvred in an optical beam. Particle sizes range from a few microns, up to ~ 100 microns. These particles are made up of a large number of atoms - as long as the particles are 'transparent' to the laser light being used, then in principle the same effect as that discussed for single atoms applies - namely photo recoil - or transfer of momentum.

The origins of optical tweezers goes back to the mid-1980's when in experiments in the period of 1985-1987 on individual biological specimens, such as viruses and bacteria, successfully demonstrated manipulation in 3D. The first experiments were performed in Bell labs in the USA.

So how do optical tweezers work?

Schematic diagram
complex optical tweezers setup
involves lots of mirrors

Optical Tweezers

Since the particles in question consist of a large number of closely packed atoms, the individual atom treatment of photon momentum transfer can not be applied any longer.

Rather a global, macroscopic treatment is required, although the particles are still only of micron dimensions overall.

We know from classical mechanics that change in momentum is nothing else than a force exerted on a particle.

i.e.

$$\underline{F} = m \cdot \underline{a} = \frac{d}{dt}(m \underline{v}) = \dot{\underline{p}}$$

When light is absorbed, reflected or diffracted by the particle, minute forces are generated acting on the particle. The forces can be of the order of a few pN when using a few mW of laser power.

Optical Tweezers

One can crudely calculate the force from a formula which gives the photon recoil force for a sphere under the influence of a laser beam of power P.

i.e.

$$|F| = Q \frac{n_1 P}{c}$$

n_1 = refractive index of the particle's environment

P = laser power

Q = scaling factor, taking into account the particle's shape and refractive index.

c = speed of light

(due to absorption or transfer of momentum)

Typically in optical tweezers:

$Q \approx 0.1 - 0.5$

$n_1 = 1$ (air)/1.33 (water)/ higher for other liquids

P \approx 10 mW (HeNe lasers widely used)

$$\Rightarrow |F| = 1.1 \times 10^{-11} N \cong 10 pN$$

sounds v. small
but can manipulate
a tiny polystyrene bead

Optical Tweezers

So how do we exploit this force for manipulation of a particles position?

One specific change from the atom cooling is that we generate an inhomogeneous radiation field in the area of the particle – we focus the beam.

For the sake of simplicity, let us assume the particle is a sphere. Depending on where the rays within the beam enter the sphere, they are refracted and bent, and the particle behaves like a small lens.

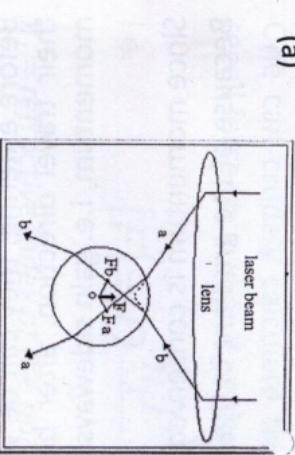
Before entering, they can be viewed as having no momentum perpendicular to their travel direction, after being deflected/refracted they appear to change momentum, i.e. gain sideways momentum component.

Since momentum is conserved, there is a transfer of momentum to the particle. Because of the gradient in the beam, a gradient in force is generated.

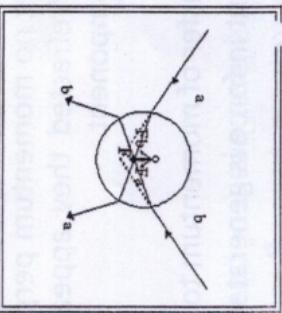
The particle is actually pulled towards the high-force region, i.e. towards the light focus.

Optical Tweezers

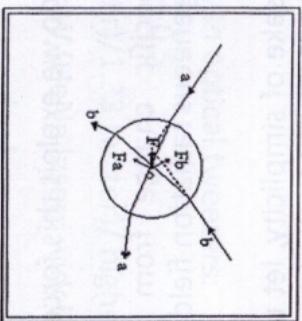
(a)



If the object is placed below the centre of the focus, the resulting force in the tweezers acts in the upward direction



If the object is placed above the centre of the focus, the resulting force in the tweezers acts in the downward direction



(c)

If the object is placed to the right of the focus, the resulting force in the tweezers acts towards the centre, i.e. to the left

Optical Tweezers

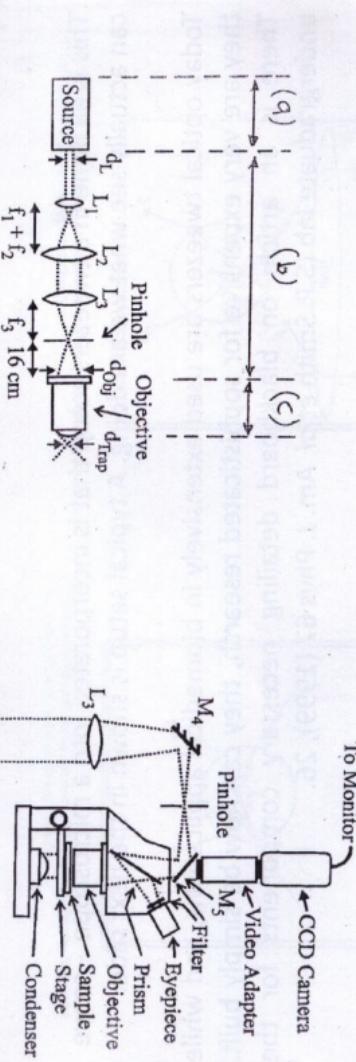
- (a) If the particle is below the centre of focus, the resulting force on the particle is upwards
- (b) If the particle is above the centre of focus, the resulting force on the particle is downwards
- (c) If the particle is to the right of the focus, the resulting force on the particle is to the left, i.e. To the centre of the tweezers.

This is all done in a tweezers setup that is incorporated into a microscope - so we can actually see what we are doing. A typical setup is shown in the next slide.

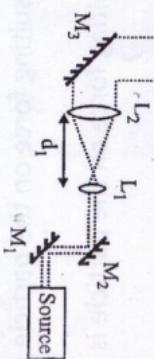
Today optical tweezers are used extensively in biomedical applications, and while they are very expensive for sophisticated research, they can now be simply built. There is an article on blackboard detailing necessary components for the undergraduate lab (S.P. Smith *et al*, Am. J. Phys **67** (1999), 26).

See also the Nature paper from 1992 by S.M. Block.

Optical Tweezers



Principle set-up with optical elements:



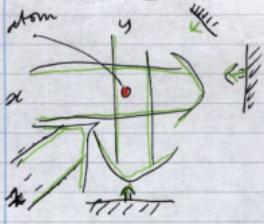
Simplified schematic diagram of a complete optical tweezers set-up, incorporated into a microscope

- (a) Laser source
(e.g. HeNe laser)
- (b) Beam shaper
(beam expander plus pin hole)
- (c) Short focal length lens
(microscope objective)

PH-361 Probing Nano Scale

Handout Notes

24th April



atom cooling
 $\propto \frac{1}{t}$

Counter propagating beams

atom cooled in overlap region