



UNIVERSITY OF  
CAMBRIDGE  
Department of Engineering

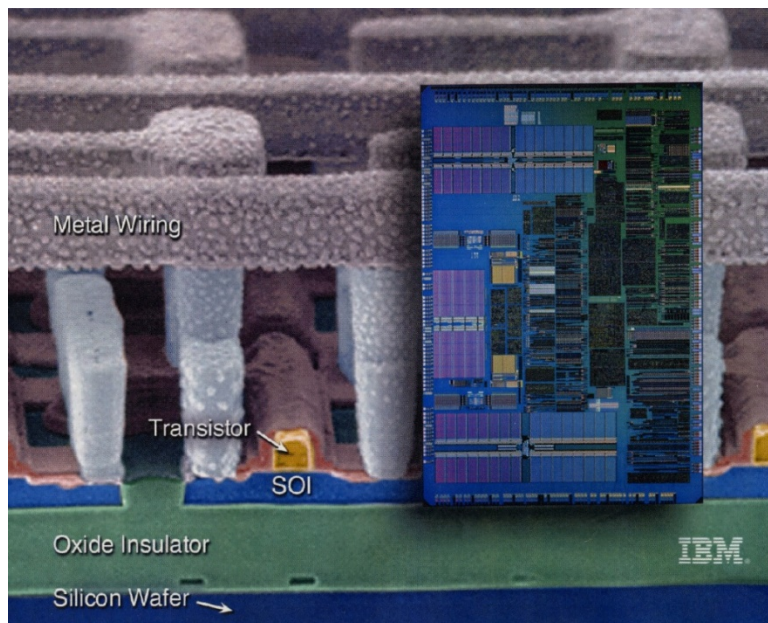
# IB Paper 8 Electrical Engineering

## Lecture 10 Fabricating Devices: Metallisation & Materials Characterisation

<https://www.vle.cam.ac.uk/course/view.php?id=69961>

# Metallisation

- Metals perform vital functions in semiconductor devices
  - Their high conductivity allows them to be used to make electrical contacts in situations where the conductivity of a doped semiconductor is insufficient
  - They are usually used to make conducting tracks and vias to devices
  - In some cases they can also be used to form electrical devices with semiconductors (e.g. metal-semiconductor diodes called **Schottky Diodes**)
  - From a mechanical perspective, metals generally possess high strength and ductility, even at elevated temperatures



- We do not need the same degree of process control as for semiconductors, typically, and so there is a greater emphasis on low cost and high volume manufacture

# Evaporation

- Thermal evaporation is a popular method for producing thin metallic layers due to its simplicity
  - The metal to be deposited is placed on a metal filament (normally W or Mo with a much higher melting point) inside a vacuum chamber which is held at a pressure below  $10^{-4}$  Pa
  - A current is passed through the filament, which heats up causing the metal to evaporate

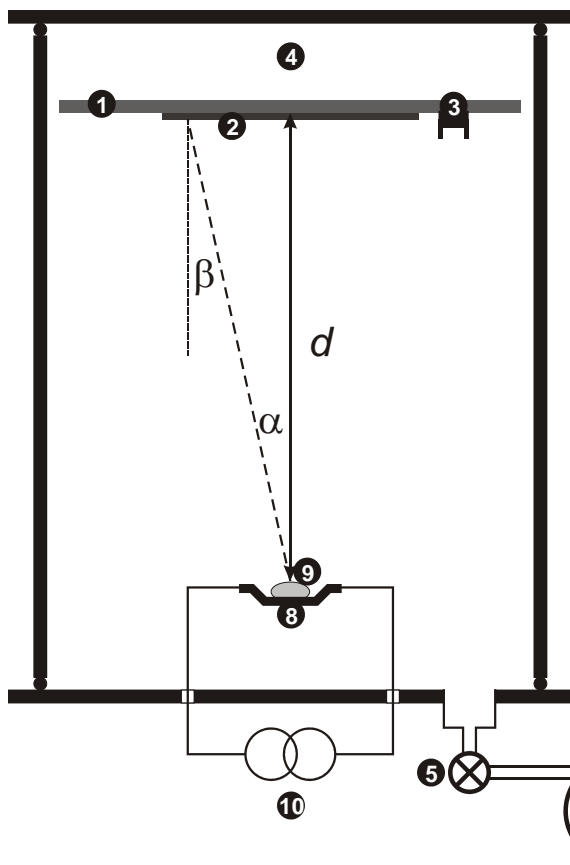
- The flux of atoms evaporating per second,  $F$ , is given by

$$F = N_0 \exp\left(\frac{-\phi_e}{kT}\right) \quad (10.1)$$

- where  $N_0$  is a slowly varying function of temperature and  $\phi_e$  is the activation energy required to evaporate one molecule which is related to the enthalpy of formation of the evaporant,  $H$ , by

$$\phi_e = \frac{H}{N_A} \quad (10.2)$$

- The substrate is placed a short distance away from the evaporating metal, and any molecules which hit the substrate will condense



- ❶ Sample stage
- ❷ Sample for coating
- ❸ Quartz crystal thickness monitor
- ❹ Vacuum chamber
- ❺ Pumping valve
- ❻ Turbo/diffusion and rotary pumps
- ❼ Exhaust
- ❽ Heating filament (W, Mo)
- ❾ Metal to evaporate
- ❿ Current source



- A high vacuum is required in thermal evaporation to ensure that contamination of the metal layer is minimised
- The source to substrate distance should be small compared with the mean free path in the vacuum
- The time required for a monolayer of the residual vacuum material to hit the substrate surface should be long compared with the time for a monolayer of material to be evaporated

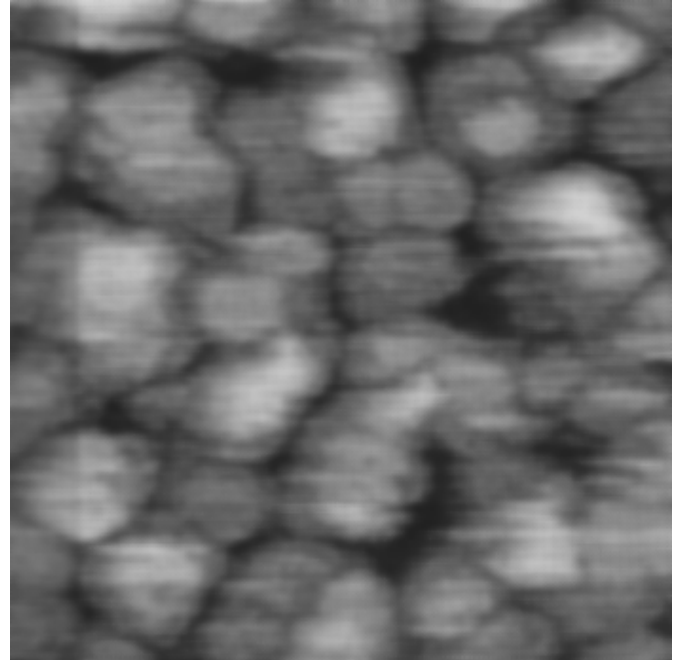
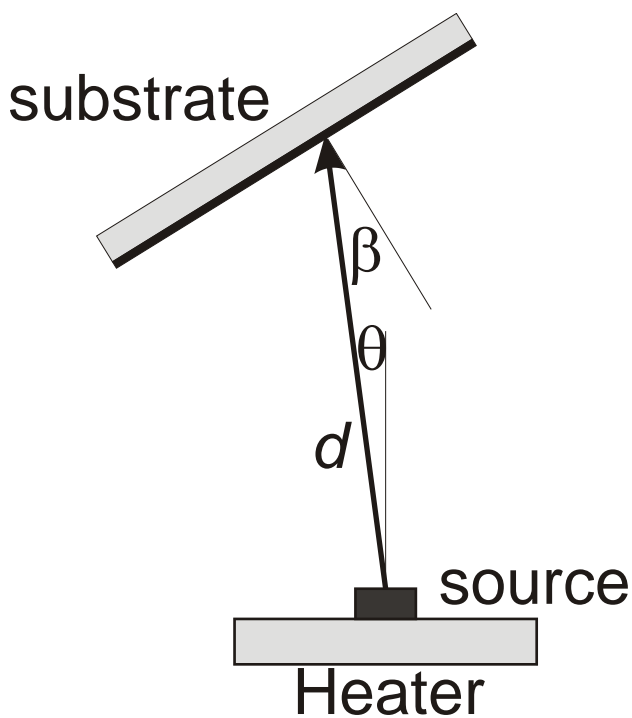
Air Pressure [Pa]	Mean Free Path [m]	Impingement rate [s <sup>-1</sup> m <sup>-2</sup> ]	Time for 1 ML exposure [s]
10 <sup>3</sup>	0.0067	2.9×10 <sup>22</sup>	3.0×10 <sup>-4</sup>
10 <sup>-2</sup>	0.67	2.9×10 <sup>20</sup>	3.0×10 <sup>-2</sup>
10 <sup>-3</sup>	6.7	2.9×10 <sup>19</sup>	3.0×10 <sup>-1</sup>
10 <sup>-5</sup>	6.7×10 <sup>4</sup>	2.9×10 <sup>17</sup>	30
10 <sup>-7</sup>	6.7×10 <sup>6</sup>	2.9×10 <sup>15</sup>	3000

- Typical evaporation rates are ~1 nm s<sup>-1</sup>, which corresponds to a rate of ~2 ML s<sup>-1</sup> (ML is short for monolayer – one layer of atoms)
  - The substrate to sample distance is normally 0.2 – 0.5 m to avoid excessive radiative heating of the sample
  - Therefore, working pressures below ~10<sup>-4</sup> Pa are required
  - Gases (such as O<sub>2</sub>) may however be purposefully added at high pressure to produce metal oxides

- Evaporation results in ‘line of sight’ deposition from what is essentially a point source of evaporating metal
  - Therefore, the deposition rate,  $R$ , at a distance  $d$  from the source is given by

$$R \propto \frac{\cos \beta \cos \theta}{d^2} \quad (10.3)$$

- where angles are as defined in the figure
- Evaporation therefore gives very poor step edge coverage, which is frequently a problem (although can occasionally be put to good use) and films are non-uniform over large areas



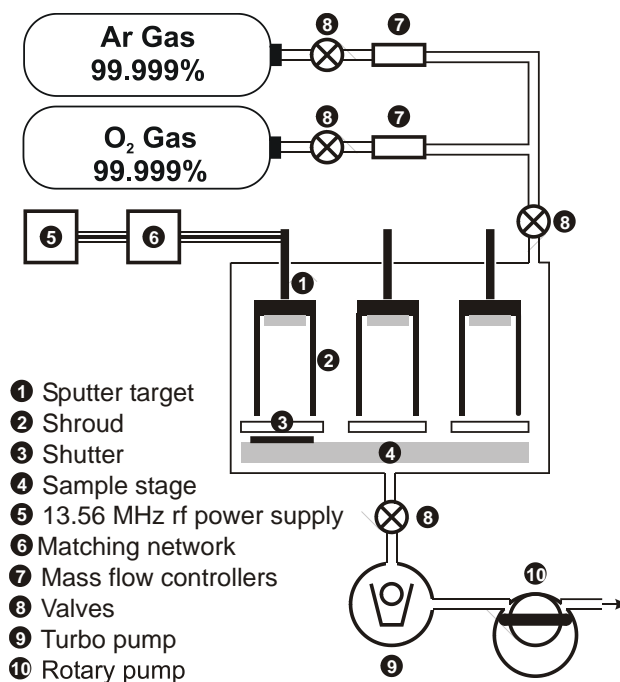
AFM Image of a  $2\mu\text{m} \times 2\mu\text{m} \times 40\text{nm}$  thermally evaporated gold layer on mica

- Evaporation produces generally amorphous metallic layers, frequently with a high surface roughness
  - The kinetic energy associated with depositing atoms is very low ( $\sim 0.1$  eV) so there is little possibility of damage to underlying layers, although thermal damage can occur
- In addition to resistive heating of a filament (which can be a source of contamination) other methods of evaporating metals are employed
  - ***Electron beam (e-beam)***
    - An e-beam locally melts a metal in a crucible
    - The crucible is not exposed to the melt, and contamination is reduced
    - X-rays can be emitted by the impacting electrons, resulting in radiation damage of the sample (and operator?!)
  - ***Laser heating***
    - A very expensive, but low contamination option
  - ***rf induction heating***



# Sputtering

- Sputtering has become probably the most popular method for producing metallic layers
  - The basic principle behind sputter coating is that ions of an inert gas are fired at a block of the desired source material (the **target**) under vacuum
  - An incident ion passes its kinetic energy to atoms at or near the target surface, allowing them to escape into the vacuum
  - The substrate to be coated is placed a short distance away from the target
  - Any released atoms hitting the substrate will condense and form the desired coating





- There are several means by which the incident ions may be generated
  - Application of a high, negative dc voltage to the target with respect to a nearby anode creates a plasma discharge, although this can only be employed for conducting targets
  - Application of an rf electric field to the target also produces a plasma discharge, but the rf electric field will not be screened by any insulating surface to the target, allowing the sputtering of insulators such as  $\text{SiO}_2$ 
    - Furthermore, a magnetic field may be used to guide ions onto the target to improve the sputter rate
    - This technique is known as rf magnetron sputtering and is particularly versatile
- The addition of oxygen or nitrogen gas to the argon will cause the formation of a metal oxide or nitride insulator, such as  $\text{TiN}$  or  $\text{Al}_2\text{O}_3$ , and this is called reactive sputtering, whilst alloys can be formed by sputtering an alloy target or sputtering from several targets simultaneously

- In order to impart a significant kinetic energy to the target, a relatively massive inert gas atom is required, and Ar is most frequently employed
  - The **sputter yield** is defined as the number of atoms removed per incident ion
  - Most metals have a sputter yield of  $\sim 1$  for Ar ions in the energy range 500 to 3000 eV
- The sputter yield,  $S$ , is dependent on several factors

### 1. Ion type

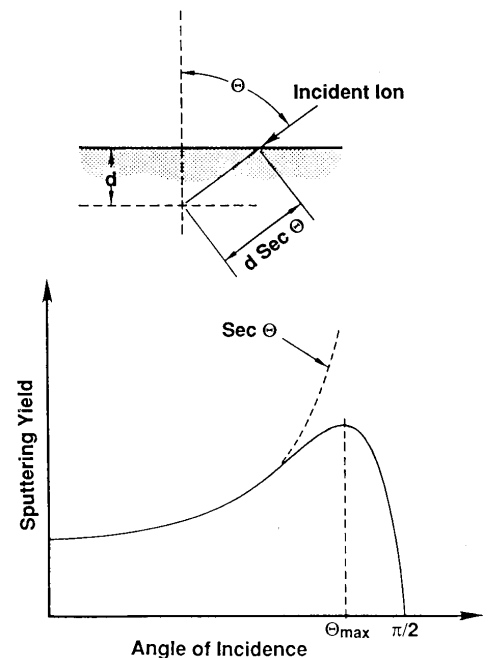
### 2. Target material

### 3. Ion energy

- A minimum ion energy in the range of 10 to 30 eV (the sputter threshold) is required for any release of target material
- The yield then increases linearly with energy up to  $\sim 1$  keV

### 4. Angle of incidence

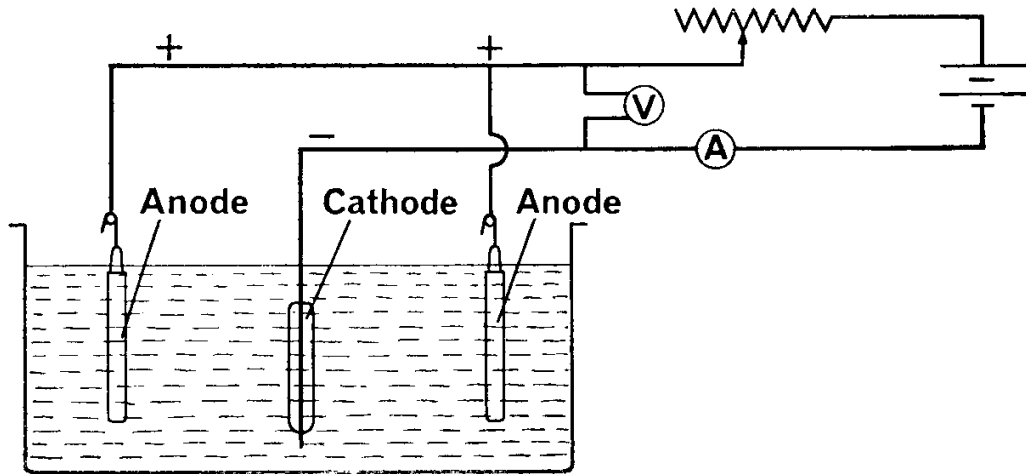
- At glancing angles, there is negligible sputtering yield
- Yield then increases rapidly up to a maximum at an angle of  $\Theta_{max}$  before slowly decreasing towards normal incidence



- The large area of the target (typically similar to the substrate) leads to improved step coverage and uniformity relative to evaporation
  - However, sputtering is a more costly process to implement, whilst secondary electron bombardment of the sample can lead to excessive sample heating as well as defect creation
  - The latter of these means that semiconductors produced by this technique are usually of poor quality
  - Argon may also be incorporated into the substrate, leading to a porous sample
  - Finally, the deposition rate,  $R$ , by sputtering can be very slow (normally well below  $1 \text{ nm s}^{-1}$ )

# Electroplating

- Electroplating is the application of metallic coatings to conductive surfaces by electrochemical processes



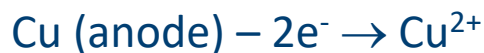
- The sample to be coated, which must have a conductive surface, is placed in a solution of the metal salt to be deposited together with another conducting plate and these are connected as the cathode and anode respectively to a low voltage, dc power supply
- In aqueous solution, the metal salt will dissociate, as will the water to a small extent
  - For example, for the plating of copper using copper sulphate,
$$\text{CuSO}_4 \rightarrow \text{Cu}^{2+} + \text{SO}_4^{2-}$$
$$\text{H}_2\text{O} \leftrightarrow \text{H}^+ + \text{OH}^-$$
  - Under the application of a bias to the electrodes, the positive ions will migrate to the cathode

- The metal ions will be deposited onto the electrode surface while the  $H^+$  ions will mostly react to form gaseous hydrogen, although a small quantity may be incorporated into the metal

- The reaction at the anode will depend on its nature
  - For an ***inert anode***, the hydroxyl ions will be discharged,



- As the anode is inert, the salt ions cannot react and so are not discharged
- This has the disadvantage that the metal ion is depleted from the solution with time
- Alternatively, the anode may be made from the depositing metal, in which case it will be dissolved into the solution in preference to any other electrochemical reactions at low voltages
- For example, the reaction at a copper anode will be



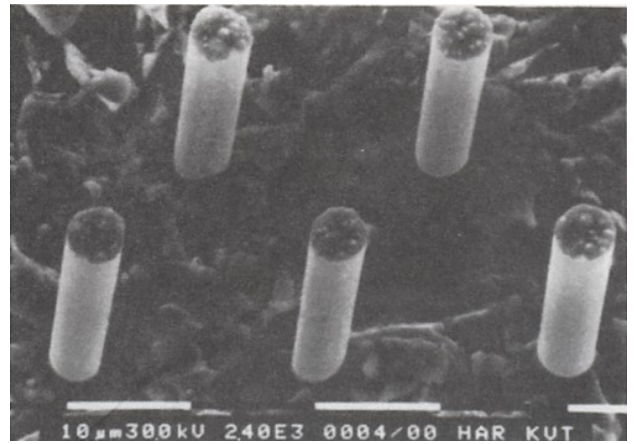
- From the Faraday Law of electrolysis, the mass of metal deposited per unit area per unit time,  $M$ , is given by

$$M = \frac{JA}{ZF} \quad (10.4)$$

- where, assuming 100% current efficiency,  $J$  is the current density *due to metal ions*,  $A$  and  $z$  are the atomic weight and valency of the metal respectively and  $F$  is the Faraday constant, which is 96500 C
- If the density of the layer is  $\rho$ , then a thickness  $R$  of material will be deposited in one second (deposition rate) where

$$R = M/\rho \quad (10.5)$$

- Electroplating has been applied to the production of high aspect ratio structures



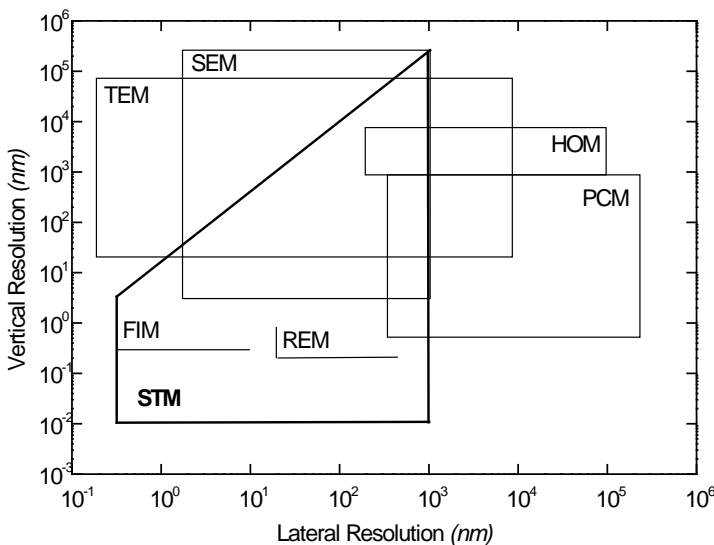
## Transparent Conducting Oxides

- Transparent conducting oxides (TCOs) are a class of optically transparent materials which nevertheless do conduct electricity
  - The most common TCO is tin doped indium oxide (more usually, but ambiguously called **indium tin oxide**, ITO)
  - It has a band gap of 3.75 eV, allowing visible radiation to pass
  - It has a low electron mobility ( $\sim 1 \times 10^{-3} \text{ m}^2 \text{ V}^{-1} \text{ s}^{-1}$ ) and so must be heavily doped to yield a reasonable conductivity
  - Provides a means of providing optically transparent contacts and is most frequently employed in solar cells
  - TCO may be deposited by reactively sputtering indium and tin in an oxygen environment
  - More simply, the alloy of the correct composition may be purchased as a target and used in an rf sputtering system with little process development

# Characterisation Techniques

- The fabrication of a typical electronic device involves many process steps and several materials
  - A failure at any stage in the fabrication process can lead to complete device failure
  - It is therefore imperative to have the ability to characterise individual materials and process steps so that the cause of device failure can be rapidly determined
- In general, this material characterisation can be classified into one of three groups

## 1. Microscopy



STM – scanning tunnelling microscopy  
HOM – high resolution optical microscopy  
PCM – phase contrast microscopy  
TEM – transmission electron microscopy  
SEM – scanning electron microscopy  
REM – reflection electron microscopy  
FIM – field ion microscopy

## 2. Physical Characterisation

- E.g. x-ray diffraction

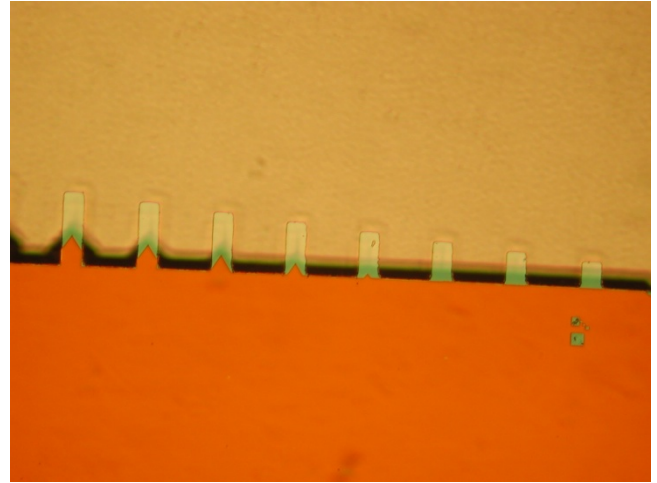
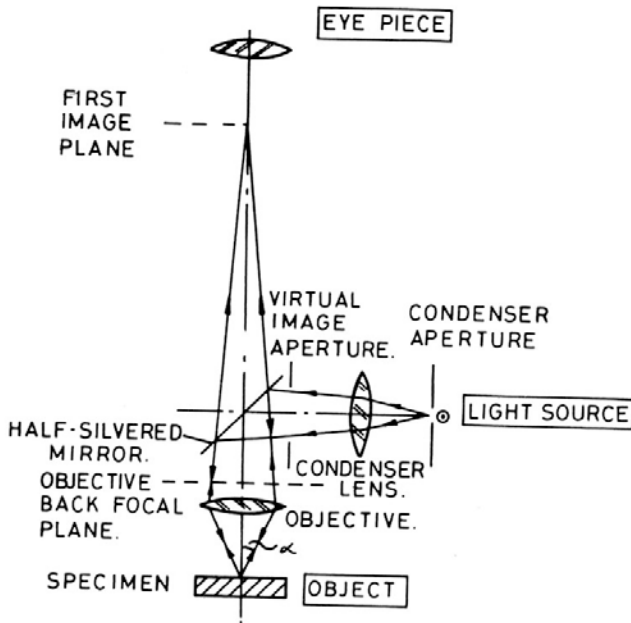
## 3. Electrical Characterisation

- Not considered here



# Optical Microscopy

- The ability to visually see a microstructure is a critical part of material characterisation
  - Probably the most critical parameter in microscopy is resolution – the ability to resolve two distinct points



- For a simple optical system comprising an objective and condenser, it can be shown that the resolving power is given by

$$\delta = \frac{C\lambda}{n \sin \alpha} \quad (10.6)$$

- where  $\lambda$  is the wavelength of the illumination,  $n$  is the refractive index of the medium between the sample and the objective,  $\alpha$  is the semi-angle subtended by the objective at the sample and  $C$  is a coherence constant, which is normally 0.61
- Optical microscopy is quick, non-destructive and can even be used to measure vertical distances by adjusting focus

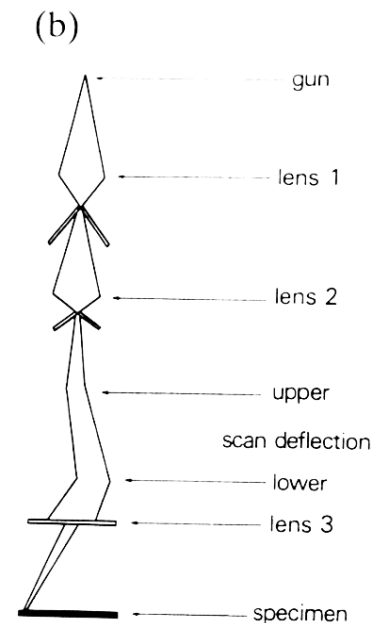
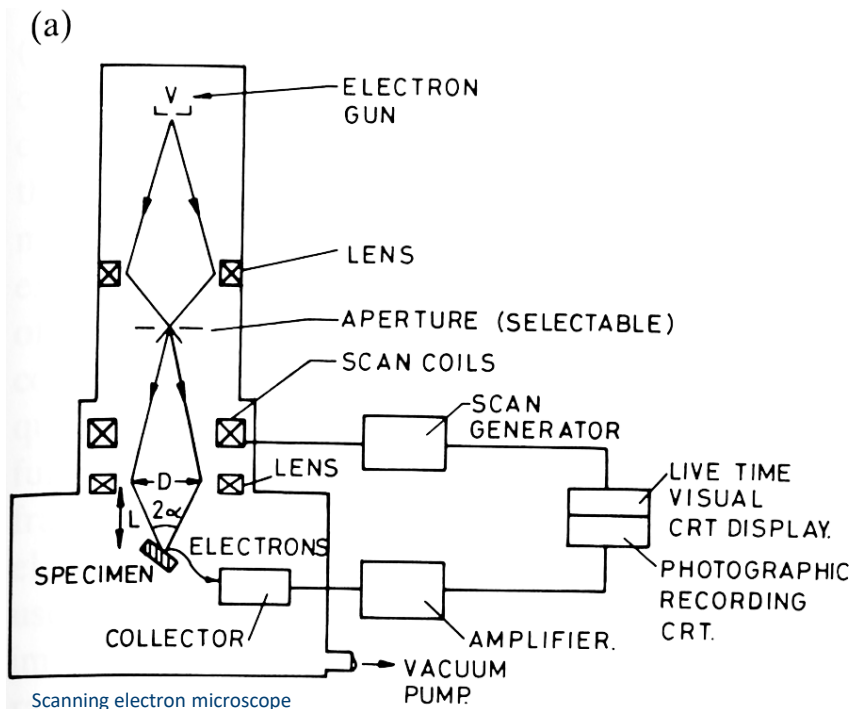
# Electron Microscopy

- The major limitation of optical microscopy is the relatively large wavelength of visible light (~300 nm in the blue) which limits resolving power

- The quantum mechanical wavelength of an electron is much smaller than that of light, given by the de Broglie equation,

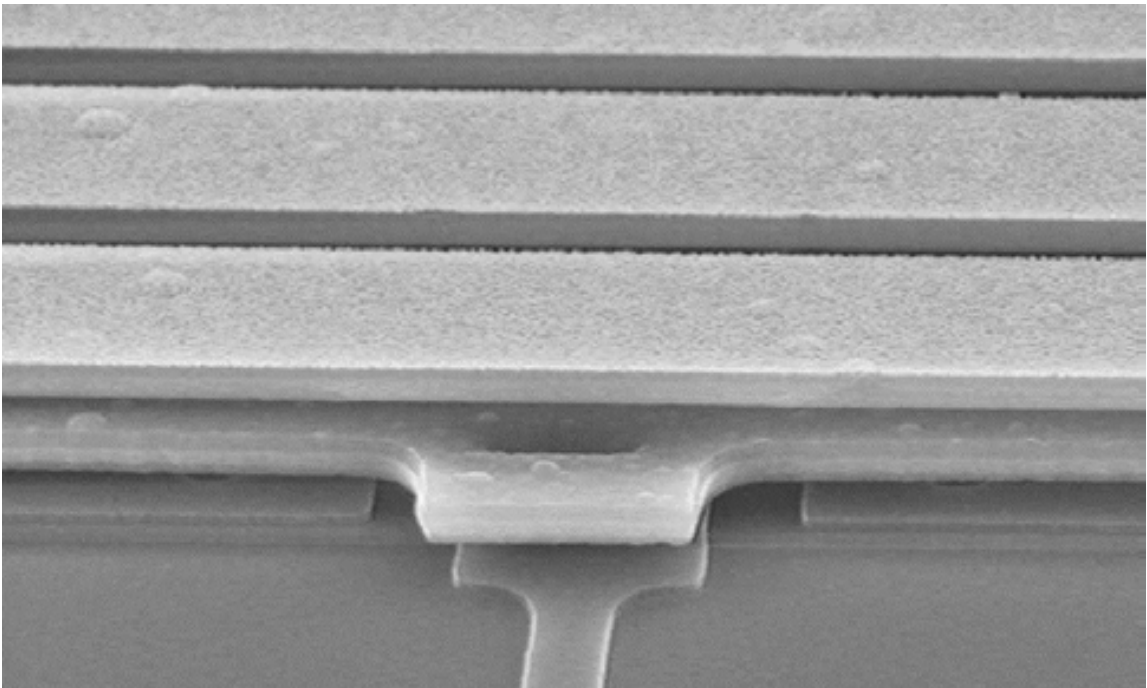
$$\lambda = h/p \quad (10.7)$$

- where  $h$  is the Planck constant and  $p$  is the momentum of the electron
- and therefore far smaller features can be imaged



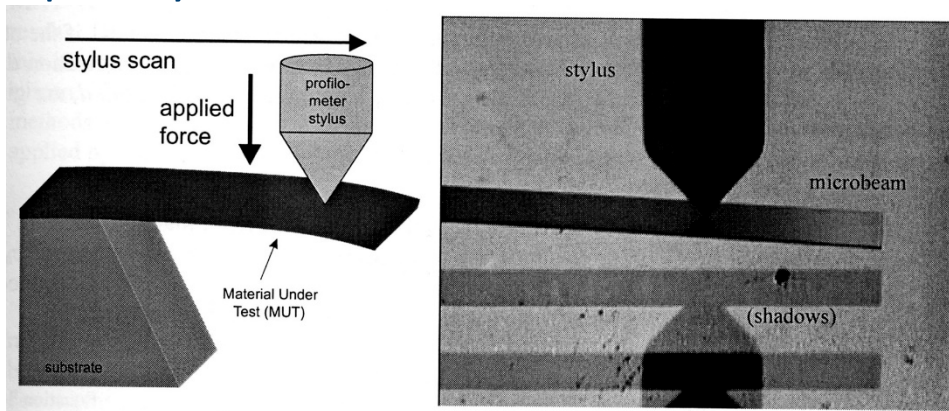
- In the scanning electron microscope (SEM) an electron beam is emitted from a hot filament and accelerated across a potential of 0 to 30 kV
  - An aperture limits the beam width and divergence
  - The beam is focussed from the aperture onto the sample, across which it is scanned
  - Electrons emitted from the sample surface are collected and amplified to produce an image
  - As the focussing distance between the aperture and the sample is large, the SEM has an excellent depth of focus (up to several mm) within which the image is sharp
  - The sample must be earthed to avoid charging up, and therefore the imaging of insulators is problematic

Polychromix Polychromator



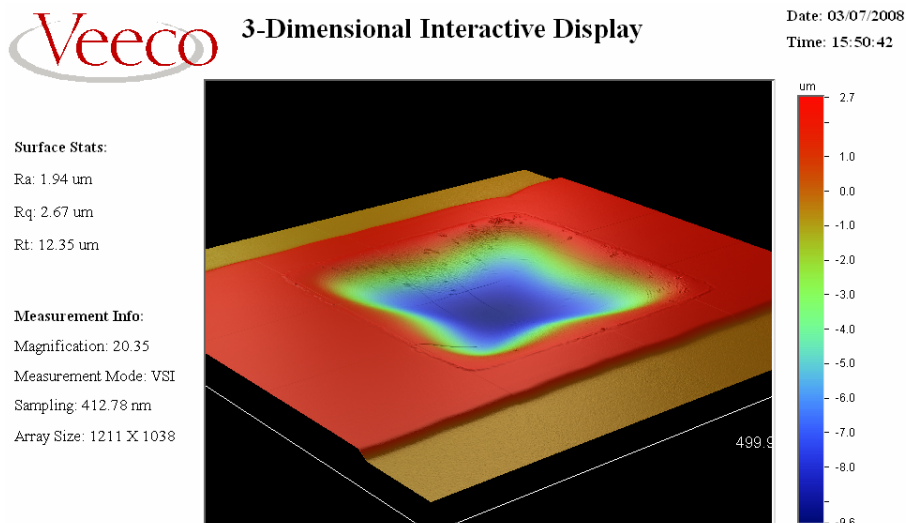
# Profilometry

- The surface topography of a sample may also be determined using ***stylus profilometry***
  - A sharp tip (usually  $\sim 10\ \mu\text{m}$  radius) is pressed onto the sample surface with a known force ( $\sim 1\ \text{mg}$ ), scanned over the surface and the vertical deflection of the tip is measured to give a plot of the topography
  - It is frequently used to determine the thickness of layers



M. A. Hopcroft, M. Phil Thesis (2002)

- ***Optical profilometry*** uses a white light source, and interferometer to determine the height of structures without contact

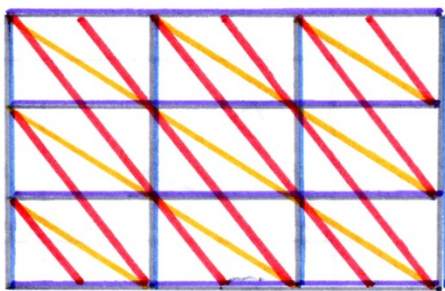


# X-Ray Diffraction

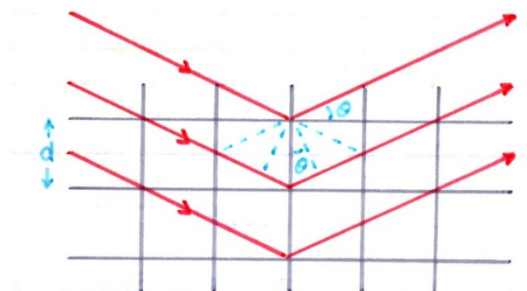
- X-rays (high energy photons) have a wavelength that is of the same order as the interatomic spacing ( $\sim 0.1 - 1 \text{ nm}$ )
- When a coherent source of x-rays is incident on a crystal, reflection will take place from each of the atomic layers, leading to the formation of a diffraction pattern
- The Bragg equation for diffraction states that constructive interference will only occur when

$$n\lambda = 2d \sin \theta \quad (10.8)$$

- where  $n$  is an integer,  $d$  is the lattice spacing and  $\theta$  is the angle of incidence
- Diffraction can occur from any set of planes
- A plane is defined by its **Miller indices** ( $h \ k \ l$ ): the number of times the plane cuts each side of the unit cell



(100)  
(010)  
(110)  
(210)



- Therefore, for a given set of planes ( $h \ k \ l$ ) in a cubic unit cell with side lengths  $a$ ,  $b$  and  $c$ , the plane separation in equation 10.8 will be

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \quad (10.9)$$



- We would therefore expect to see a set of diffraction peaks associated with each combination of  $h$ ,  $k$  and  $l$
- However, crystals are rarely simple and additional atoms within each unit cell will affect the diffraction pattern
- We need to introduce a structure factor,  $F_{hkl}$ , the modulus of which gives the amplitude of the wave diffracted by a particular set of planes, and is given by

$$|F_{hkl}| = \sum_{n=1}^N f_n \exp[2\pi j(hu_n + kv_n + lw_n)] \quad (10.10)$$

- where  $u_n$ ,  $v_n$  and  $w_n$  are the fractional positions of the atoms in the unit cell and  $f$  is the scattering factor for the particular atom
- Hence for a face centred cubic atomic structure with identical atoms at positions  $(0\ 0\ 0)$ ,  $(\frac{1}{2}\ \frac{1}{2}\ 0)$ ,  $(\frac{1}{2}\ 0\ \frac{1}{2})$  and  $(0\ \frac{1}{2}\ \frac{1}{2})$  we find a structure factor of

$$|F_{hkl}| = f \exp[2\pi j(0)] + f \exp\left[2\pi j\left(\frac{h}{2} + \frac{k}{2}\right)\right] + f \exp\left[2\pi j\left(\frac{h}{2} + \frac{l}{2}\right)\right] + f \exp\left[2\pi j\left(\frac{k}{2} + \frac{l}{2}\right)\right]$$

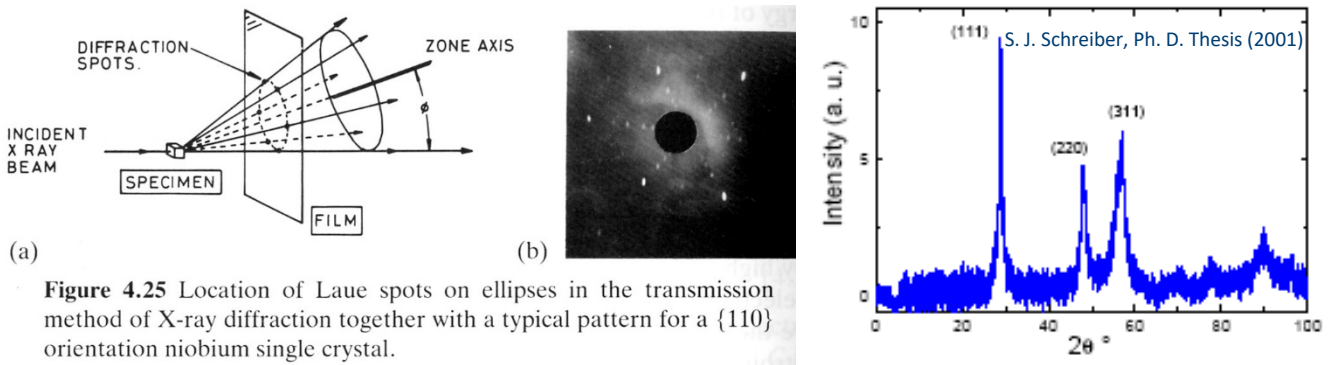
$$|F_{hkl}| = f\{1 + \exp[\pi j(h + k)] + \exp[\pi j(h + l)] + \exp[\pi j(k + l)]\} \quad (10.11)$$

- If  $h$ ,  $k$  and  $l$  are all odd or all even, then  $F = 4f$  and there will be a diffraction peak associated with this plane
- However, if they are mixed odd and even, then  $F = 0$  and there will be no diffraction peak
- Hence peaks associated with  $(1\ 1\ 1)$ ,  $(2\ 0\ 0)$  and  $(2\ 2\ 0)$  planes will be present, whilst those associated with  $(1\ 0\ 0)$ ,  $(1\ 1\ 0)$  and  $(1\ 1\ 2)$  will not

- In this way, XRD allows a determination of the lattice parameters and structure

Bravais lattice	Diffraction present	Diffraction absent
Simple	All	None
Base centred	$h$ and $k$ not mixed	$h$ and $k$ mixed
Body centred	$(h + k + l)$ even	$(h + k + l)$ odd
Face centred	$h$ , $k$ and $l$ not mixed	$h$ , $k$ and $l$ mixed

- A single crystal will produce a pattern of spots



- In polycrystalline materials, as all crystal orientations are generally present, diffraction rings are produced, and so X-ray intensity may simply be plotted as a function of angle, as for the microcrystalline silicon sample shown above
- In amorphous materials, broad, diffuse rings are produced
- If the material possesses an intrinsic stress, then the crystal lattice will be distorted, and this results in a well-defined broadening of the diffraction peaks as a function of  $(h\ k\ l)$ , allowing the stress to be measured
- A number of different X-ray diffraction techniques exist to permit a range of microstructural properties to be investigated