MLL203: Characterization of Materials – II **Minor 1**

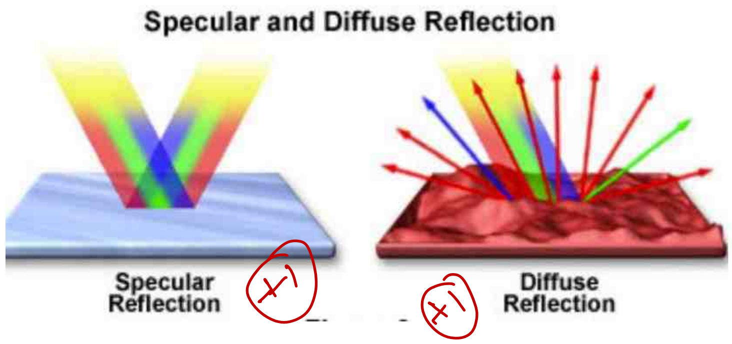
Date: 12-09-2023 Duration: **2 hrs** Total Marks: 80

Instructions: Sketch/diagram not required unless specified. There is no negative marking, however, step marking is applicable.

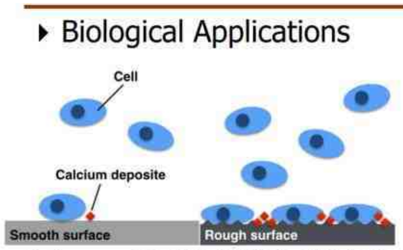
1. What are the conditions to achieve super-hydrophobicity as observed in case of lotus leaf? **(4 marks)**

Surface with multiscale surface roughness (combination of rough features at different lengthscales) in combination with hybrophobic nature (low surface energy) gives rise to superhydrophobicity.

2. How does surface play a role in optical properties and biological compatibility of materials? **(4 marks)**



figures are not mandatory



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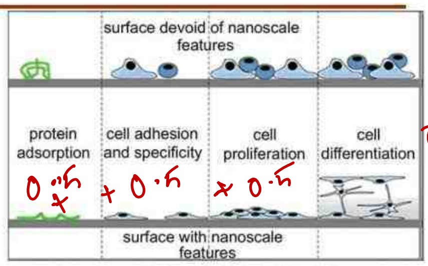

https://www.mdpi.com/1996-1944/13/5/1188/htm

3. What is the purpose of sample polishing for optical microscopy? What is the role of the etching process? Calculate and discuss the highest resolution that can be achieved using optical microscopy? **(8 marks)**

Sample polishing is done to make surface flat and reflective else it will appear completely dark due to very high roughness. Etching is performed to selectively dissolve/roughen regions such as grain boundaries, phase boundaries and other defects etc. which provides contrast corresponding to these features due to variation in light scattering. If etching is not performed, then entire surface would reflect light equally and microscopic features will not be observed.

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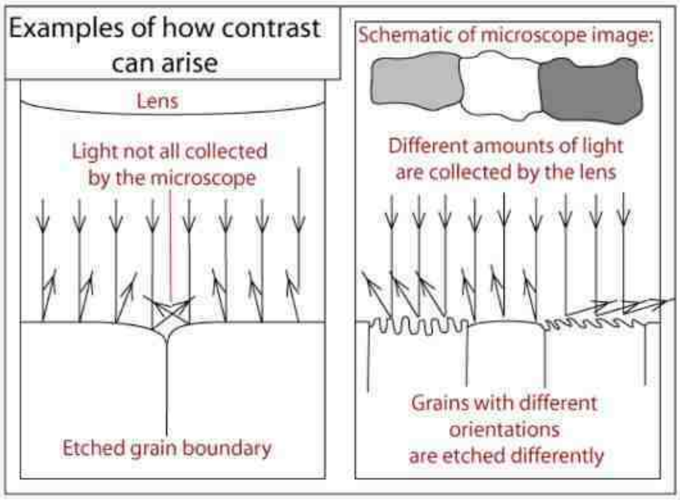

$d = \frac{0.61\lambda}{n \sin\beta}$ (+5 marks)

β should be large for better resolution (smaller d value)

assume n = 1 (in air)

& sin β = 1 (max possible value)

λ = 380 - 700 nm (visible light)

hence $d = \frac{0.61 \times 380}{1}$ to $\frac{0.61 \times 700}{1}$

= 250 nm to 462 nm

4. What is the fundamental difference between the working principle of scanning tunnelling microscopy and atomic force microscopy techniques? Which one of these techniques is more versatile for study of different classes of materials and why? **(4 marks)**

Electron tunnelling between a conductive tip and sample to detect and image the surface vs. tip-sample force detection via bending/deflection of a flexible cantilever which is tracked using a laser beam. (+1) (+1)
STM can work only for conductive samples as it is based on electron tunnelling whereas AFM can be used for all types of materials. (+1) (+1)

5. For a TEM, given the collection angle of 10 mrad and electron beam acceleration voltage of 100 V, calculate fo find out whether it is sufficient to achieve atomic resolution (typical value of 0.2 nm)? Assume that aberrations can be neglected. **(8 marks)**

$$eV = \frac{1}{2}mv^2 \qquad v = \sqrt{\frac{2eV}{m}} \qquad \lambda = \frac{h}{mv}$$

assume sin β = β

$$\lambda = \frac{h}{\sqrt{2meV}} \qquad d = \frac{0.612 * \lambda}{n \sin\beta}, \quad d = \frac{0.753}{\beta\sqrt{V}}$$ (+4)

(+2) upon solving d = 7.5 nm >> 0.2 nm

(+2) ∴ it is not possible to achieve atomic resolution

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6. Fill Y/N in the columns below to indicate possibility of the given TEM sample preparation technique for different types of materials: **(10 marks)**

	Grids	Ion Milling	Electropolishing	FIB	Microtomy
Polymers	N	Y	N	Y	Y
Ceramics	N	Y	N	Y	N
Metals	N	Y	Y	Y	N
Particles in solution	Y	N	N	N	N

+0.5 for each option

7. What type of samples require chemical fixation and cryo fixation, and which technique you would prefer? What is the purpose of staining the sample? Is cooling rate critical for cryo-fixation? **(5 marks)**

+1

These techniques are used for biological samples. Cryo fixation is preferred as we completely freeze the biological sample, preventing any metabolic activity or degradation. +1

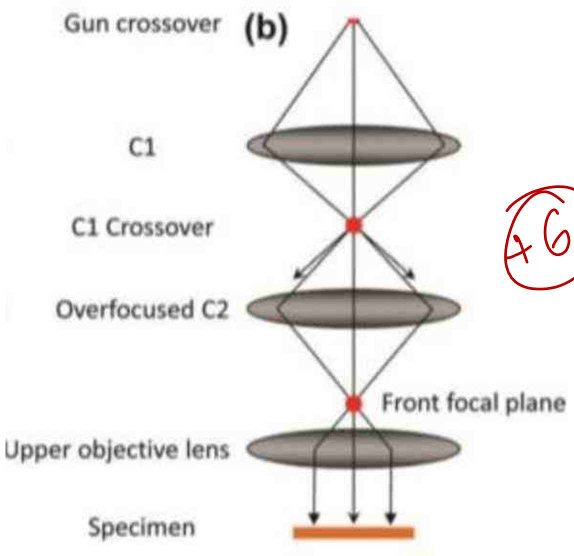
Staining is performed to increasing imaging contrast in TEM as biological samples have very low mass/density and are nearly electron transparent. +1

Cooling rate is very critical else water will convert to crystalline ice and this would result in volumetric expansion, generating stresses and will damage soft materials. +2

8. Briefly discuss the role of two condenser lenses in TEM. Draw schematic showing lenses producing ideal/preferred beam ray diagram before it reaches the specimen. **(10 marks)**

C1 controls the beam size +2

C2 controls the beam brightness +2



+6

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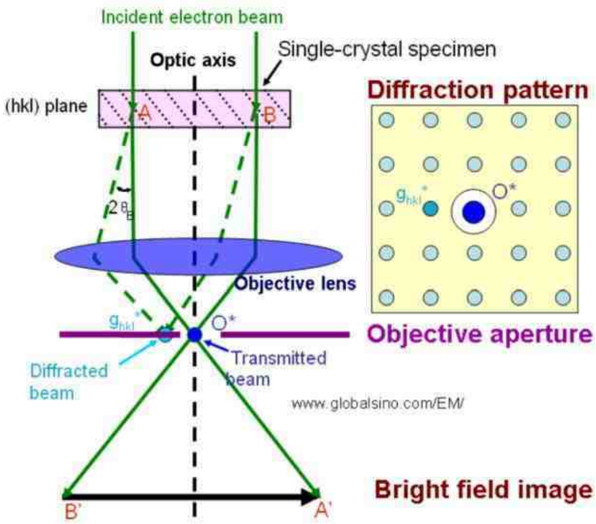
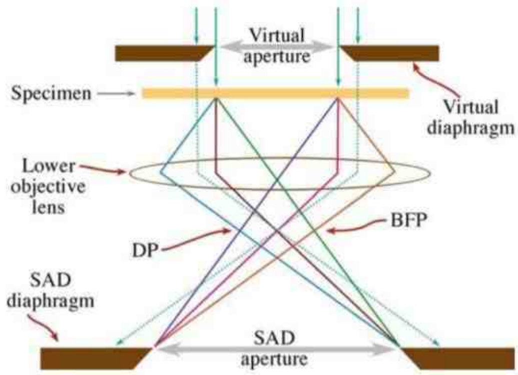
9. What specific sample information can be achieved by objective aperture and selected area diffraction aperture in TEM? Discuss where these apertures are placed in TEM. **(5 marks)**

Objective aperture is utilized for choosing between direct beam and diffracted beam to form bright field or dark field images. It is placed in back focal plane of the TEM objective lens.

SAD aperture is to select a small region of interest to collect diffraction pattern from that specific region. It is placed in the image plane of the TEM objective lens.





10. Derive the expression for practical resolution (a function of wavelength) of a TEM that can be achieved in presence of spherical aberration. How do we eliminate spherical aberration? **(10 marks)**

$$r = (r_{th}^2 + r_{sph}^2)^{1/2}$$

$$r(\beta) \approx \left[\left(\frac{\lambda}{\beta}\right)^2 + (C_s\beta^3)^2\right]^{1/2}$$

$$\frac{\lambda^2}{\beta^3} \approx C_s^2\beta^5$$

→ solving for r_{min} → $\frac{dr}{d\beta} = 0$

$$\beta_{opt} = A \frac{\lambda^{1/4}}{C_s^{1/4}}$$

$$r_{min} \approx B\ (C_s\lambda^3)^{1/4}$$

smaller β values & using

Spherical aberration can be eliminated using Cs corrector, which is a sequence of hexapole and transfer lenses to produce negative Cs values to negate the aberration.

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11. Discuss how EELS can detect specific elements present in the specimen, thickness of specimen and its conduction/valence electron density. **(6 marks)**

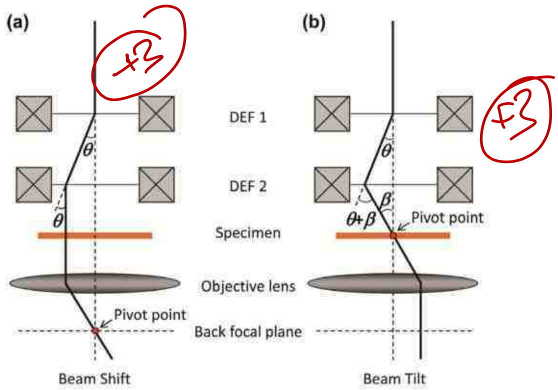
EELS can detect specific elements by analyzing the ionization edge peaks.

Sample thickness can be inferred by looking at the nature of the plasmons peak (as thickness of sample increases, the intensity of plasmon peak inreases and multiple peaks can be observed).

Electron density can be measured using the energy values of the plasmon peaks.

12. With simple schematics, discuss why beam shift and beam tilt is require and how it can be achieved in TEM. **(6 marks)**

- Beam shift and beam tilt
- The beam shift and beam tilt are controlled by deflector coils.
- Beam shift and tilt could be mixed, that is, the operation of beam shift may produce beam tilt, and the beam tilt may produce beam shift.



.16 *Beam shift (a) and beam tilt (b) by deflector coils.*