

**NPTEL Phase – II**  
**Advanced Characterization Techniques**  
**(Instructors: Dr.Krishanu Biswas and Dr.N.P.Gurao, IIT Kanpur)**

**Questionnaires**

**A. Advanced Diffraction Techniques: SAXS, SANS, LEED, RHEED, EXAFS.**

1. What is diffraction? Explain the similarities and differences between scattering and diffraction.
2. Describe the Braggs law in real and reciprocal space?
3. Enumerate three different modes of X-ray diffraction.
4. What are the three different type of diffraction? Which amongst these is valid for conventional X-ray diffraction?
5. Why powder diffraction should be replaced with polycrystalline diffraction?
6. Provide a list of all the additional information provided by X-ray diffractogram along with phase information.
7. Show a schematic explaining the the reason for  $2\theta$ -Intensity rather than  $\theta$ -Intensity plot in polycrystalline XRD.
8. What is X-ray fluorescence?
9. What are the factors contributing to intensity of X-ray peaks?
10. How can you differentiate between the X-ray diffractograms of BCC and SC material?
11. Calculate structure factor for a hexagonal closed packed crystal structure like magnesium. How will the diffractogram of titanium with less than ideal  $c/a$  ratio look compared to that of magnesium?
12. Compare the parallel and para-focusing geometry of diffraction.
13. What are the different optics used in X-ray diffraction? Explain the function of each in brief.
14. Compare and contrast microscopy and scattering as characterization tools.
15. Explain the instrumentation for SAXS.
16. What are the Guiner and Porod regime in SAXS?
17. What is Form factor?
18. What is the size range accessible by SAXS?
19. Explain the geometry of GISAXS.
20. Comment on the origins of Yoneda and Specular peak.
21. Compare SAXS and GISAXS
22. Enlist a few studies using GISAXS in literature and also comment on the complimentary characterization tools used to verify the GISAXS result. Discuss the pros and cons of each in brief.
23. Compare and contrast X-ray and electron diffraction.
24. Explain the similarities and differences in diffraction in 2D and 3D.

25. What is Surface Profile Analysis (SPA) LEED?
26. Draw a schematic showing instrumentation of LEED
27. Compare the electron energy used in LEED compared to conventional SEM.
28. Compare EELS with Electron Back Scatter Diffraction.
29. Explain the origin of streaks in RHEED.
30. Compare LEED and RHEED.
31. Why is RHEED one of the most suited characterization tool for in-situ monitoring of thin film deposition?
32. What is Convergent Beam Electron Diffraction RHEED?
33. Compare RHEED and EBSD.
34. What is the principle of Extended X-ray Absorption Fine Structure (EXAFS) spectroscopy?
35. Explain the instrumentation required for EXAFS.
36. Compare and contrast EXAFS with other techniques that provide chemical composition at atomic level.
37. What is X-ray Absorption Near Edge Structure (XANES) spectroscopy ?
38. Explain different modes of scattering in EXAFS.
39. Explain different type of neutrons and various methods used for their generation ?
40. Explain the concept of scattering cross section. Comment on the scattering cross section of neutrons vis a vis X-rays.
41. Differentiate between neutrons and X-rays and electrons as a diffraction probe.
42. Explain the instrumentation for a typical neutron diffraction experiment.
43. What is time of flight measurement for neutrons?
44. Explain the principle of Small Angle Neutron Scattering (SANS).
45. Compare and contrast SANS and SAXS.
46. What is isotope substitution in SANS?
47. What is Small Angle Polarized Neutron Scattering?
48. Explain different modes of scattering in EXAFS.

## **B. Advanced Surface Characterization Techniques: XPS, AES, SIMS**

1. Explain the difference between XPS and AES along with kinetic energy calculations for each.
2. Which method, AES or XPS, is better for resolving chemical shifts and why? Which direction would a peak in a given spectrum move if an atom loses a valence charge? What if the atom gains a valence charge?
3. Why is the AES uses  $dN/dE$  instead of  $N(E)$  directly?
4. Does the Auger sensitivity depend on the electron beam energy?
5. Is the kinetic energy of the photoelectron dependent on the X-ray beam energy?
6. What is a KLL transition?
7. Can Auger electrons be non-core electrons?
8. Why is AES a more qualitative than quantitative technique?

9. Can the Auger electron emission process be initiated with x-rays?
10. Besides the beam electrons, how many electrons are involved in AES?
11. Can x-rays eject non-core electrons?
12. What is the maximum depth of XPS?
13. Why is XPS a more qualitative than quantitative technique?
14. How many electrons are involved in XPS?
15. Is the kinetic energy of the photoelectron dependent on the X-ray beam energy?
16. Determine the Auger transitions that produced electrons with the provided information.  
The binding energy information for Mg is:  
Subshell 1s(K) 2s (L1) 2p<sup>1/2</sup> (L23)  
BE (eV) 1407 98 45
17. What are the three type of observed peaks originating in photo emission in XPS?
18. List the similarities and differences of XPS and AES.
19. What is the difference between Auger electrons and x-ray electrons?
20. What is the sampling depth of XPS?
21. What are the most common notation designated in Auger electrons and what each letter corresponds to in terms of location?
22. How charging can be rescued almost completely?
23. How is the excitation energy affects the KE of Auger and how in XPS?
24. How it is called the separation between the two peaks in XPS and how different the values are of spin orbital splitting of a core level of an element in different compounds?
25. What is Mass Spectrometry? What is secondary ion mass spectroscopy (SIMS)?
26. What are different parts of a typical mass spectrometer?
27. What is the typical resolution of SIMS?
28. What are different types of SIMS available?
29. What are important precautions one needs to take into consideration while preparing samples for SIMS?
30. Compare three techniques, XPS, Auger and SIMS in terms of applicability and resolution.

### **C. Advanced Microscopic Techniques: TEM: HR,HAADF,STEM, In-situ; EBSD,AFM, STM, Laser Confocal Microscopy**

1. What is the difference between TEM and HRTEM?
2. Explain the concept of spherical and chromatic aberration in electromagnetic lens.
3. Using the concept of spherical aberration, obtain the condition for optimum resolution.
4. What is Scherzer focus? Why it is important for atomic resolution imaging.
5. What is called 'aberration corrected' high resolution electron microscopy?
6. What are different types of "aberration corrected" high resolution electron microscopy?
7. What is information limit? Using "aberration corrected" high resolution electron microscope, what is the level of information limit one can obtain?
8. What is HAADF? How a HAADF image collected from a typical foil?
9. What kind of information one can obtain from A HAADF image? Is there any similarity between the Back Scattered Electron imaging in SEM and HAADF in TEM? If so, elaborate in details
10. Can HAADF be used to obtain atomic structure of a solid?
11. What is STEM? How does it work?
12. What kinds of information one can obtain from STEM bright field and dark images
13. What is the meaning of in situ microscopy? What are different types of in situ microscopic techniques? Elaborate each one of these techniques?
14. What precautions one needs to take into consideration while carrying out in situ heating experiments in an advanced TEM?
15. What kinds of phase transformation are studied using cold stage in situ TEM experiments
16. What is micro-texture?
17. Explain the principle of EBSD?
18. Why the sample is tilted by 70 degree for routine EBSD measurements ?
19. What is Hough Transform?
20. Explain the strategy to carry out EBSD analysis on two phase structures like alpha-beta titanium alloy? Explain the strategy in terms of sample preparation and EBSD parameter selection to obtain correct results.
21. Enumerate the information obtained from routine EBSD scan for a single phase material.
22. Explain the utility of EBSD as the most important tool for Grain Boundary Engineering.
23. Explain the instrumentation for EBSD.
24. Briefly explain the basic principle behind atomic force microscopy (AFM)?
25. What information one can obtain by using AFM?
26. What is the difference between AFM and STM (scanning tunneling microscopy)? Why AFM is so popular in the research community?
27. Can STM be used for non-conducting samples? If yes, how?
28. What is confocal microscopy? Why it is important in the advanced research?
29. Normally, confocal microscopes are utilized to image biological samples. Provide examples where confocal microscope can used for materials science applications
30. Explain the near field optical microscopy in brief.

## D. Advanced Spectroscopic Techniques: Vis, UV, FTIR, Raman, STEM-EELS

1. What is the principle and instrumentation of FTIR
2. Write a note on sample handling in IR spectroscopy and give its applications.
3. Discuss the choice of solvents effects in UV spectroscopy. Factors
4. Give the theoretical principle of mass spectroscopy with the aid of neat diagram of double focusing mass spectrophotometer. Write a brief note on hyphenation of GC & LC with MS.
5. Fourier transformation
6. Applications UV visible spectroscopy.
7. Application of atomic absorption spectroscopy.
8. Application of Raman and IR spectroscopy
9. Vibrational energy level of a molecule corresponds to region of electromagnetic spectrum.
  - a) X ray
  - b) UV
  - c) IR
  - d) Radio wave
10. A change in electric dipole moment during molecular motion is associated with
  - a) X ray
  - b) UV
  - c) IR
  - d) Radio wave
11. In Raman scattering the frequency of the scattered radiation is that the incident radiation
  - a) Equal to
  - b) Lower than
  - c) Higher than
  - d) Either lower or higher than
12. A 3p orbital has .....radial nodes
13. Which among the following molecule is IR inactive but Raman active?
  - a) HCL;
  - b) N<sub>2</sub>
  - c) SO<sub>2</sub>
  - d) CH<sub>4</sub>
14. Name the spectroscopic technique used in molecular mass determination
15. Non-polar molecules are microwave active
16. The number of bands in the IR Spectrum of a molecule is the same as the number of normal modes of vibration.
17. Calculate the frequency of light emitted when an electron jumps from the first excited state of butadiene to its ground state using particle in a box approximation. Given that length of the carbon chain in butadiene is 578 PM.

18. Compare and contrast  $\sigma$ ,  $\sigma^*$ ,  $\pi$ , and  $\pi^*$  molecular orbitals
19. The microwave spectrum of HCL consists of a series of equally spaced lines separated by  $20.86 \text{ cm}^{-1}$ . Calculate the bond length of HCL
20. State and explain Einstein law of photochemical equivalence. What is meant by quantum yield of a photochemical reaction?
21. Briefly explain the quantum theory of Raman effect
22. Why must the excitation source for fluorescence spectrometry be more powerful than for absorption spectroscopy?
- Because the sample won't fluoresce if the incident radiation is of low power
  - Because the magnitude of the output signal is proportional to the power of the incident radiation
  - To allow for scattering by the sample
  - To suppress Raman scattering
23. Spatial coherence refers to
- Prediction of amplitude and phase at any position at a given time
  - Prediction of amplitude and phase at any time at a given position
  - High monochromaticity
  - Diffraction limited collimation
24. Problem(s) with short (in time) laser pulse is(are)
- Non-linearity may induce complications in the dynamics of interest
  - Lack of spectral sensitivity due to broad spectrum of the laser
  - Difficult to measure proper time zero
  - Instrument response function may be too large
25. Which of the following statement(s) is(are) correct for population inversion
- For a 2-level system, a steady state population inversion may be achieved by using direct optical pumping between the two levels at 0K.
  - For a 2-level system, a steady state population inversion may be achieved by using direct optical pumping between the two levels at very high temperature.
  - For a 3-level system, a steady state population inversion may be achieved by using direct optical pumping between first two levels at 0K.
  - For a 3-level system, a steady state population inversion may be achieved by using direct optical pumping between the first and third levels at any temperature.

26. [X] Associate each of the following spectra with the following media; DCM, DMF, Gas phase, Cyclohexane

- a) (i) --> Gas phase; (ii) --> DCM; (iii) --> DMF; (iv) --> Cyclohexane
- b) (i) --> Cyclohexane; (ii) --> DCM; (iii) --> DMF; (iv) --> Gas phase
- c) (i) --> Cyclohexane; (ii) --> Gas phase; (iii) --> DMF; (iv) --> DCM
- d) (i) --> Gas phase; (ii) --> Cyclohexane; (iii) --> DCM; (iv) --> DMF

27. During the measurement of fluorescence lifetime of a molecule, the emission is collected at the magic angle polarization

- a) To remove the polarity effect
- b) To remove the quenching effect by dissolved oxygen
- c) To remove the orientational relaxation time component
- d) None is correct

28. For a molecule, with absorption maxima at 400 nm ( $20000 \text{ cm}^{-1}$ ) with a width of  $2000 \text{ cm}^{-1}$  the maximum possible value of molar extinction coefficient is

- a)  $115500 \text{ l mol}^{-1} \text{ cm}^{-1}$
- b)  $100500 \text{ l mol}^{-1} \text{ cm}^{-1}$
- c)  $85500 \text{ l mol}^{-1} \text{ cm}^{-1}$  Page 1 of 3
- d)  $70500 \text{ l mol}^{-1} \text{ cm}^{-1}$

29. Among the following, which is(are) true

- a) Stimulated emission is a process of transition of atom or molecule from a higher state to a lower one without any outer effect.
- b) The laser source may emits a wide-band of frequency so that there may be many colours in the spectrum.
- c) The number of spontaneous emission depends on the population of the state.
- d) In acousto-optic Q-switch, when an acoustic signal is applied to the transducer, light is diffracted out of the intracavity beam.

30. In a particular He-Ne laser, the distance between two end mirror is 60 cm. The natural band width of the laser is 1.0 GHz. The numbers of longitudinal modes present in the output of the laser is

- a) 2
- b) 3
- c) 4
- d) 5

31. In TCSPC system, the function of CFD is
- Reduce amplitude jitter of PMT
  - Reduce the time jitter induced by the amplitude jitter of PMT
  - Reduce the time jitter of laser
  - Reduce the time jitter induced by transit time spread of PMT
32. Because of the excimer formation, one get a
- New red shifted structured emission compared to the monomer fluorescence
  - New red shifted broad emission compared to the monomer fluorescence
  - New blue shifted structured emission compared to the monomer fluorescence
  - New blue shifted broad emission compared to the monomer fluorescence
33. The steady state fluorescence anisotropy of fluorescein in water is 0.03 and of fluorescein-labeled protein in water is 0.25. The fluorescence anisotropy is near zero for the free fluorescein however the fluorescence anisotropy is high for the fluorescein-labeled protein. This is because
- Excited state lifetime of the free fluorescein is very small compared to the labeled fluorescein
  - Solvation time inside the protein is 100 times slower compared to bulk
  - Excited state proton transfer takes place in fluorescein, and the rate is slow in protein compared to bulk.
  - The rotation of the free fluorescein in water is faster compared to the fluorescein labeled in the protein.
34. The specific intermolecular interaction that leads to the sixth power dependence in the efficiency of energy transfer with the intermolecular distance is
- Exchange interaction
  - Coulombic interaction
  - Van der Waals interaction
  - Nuclear interaction
35. Molar extinction coefficient of coumarin is 40000 lit mole<sup>-1</sup> cm<sup>-1</sup> at 400 nm. Molecular cross-section of coumarin is  $20 \times 10^{-16}$  cm<sup>2</sup>. The percentage of absorbed 400 nm photons by coumarin is about
- 5.0 %
  - 7.5 %
  - 10.0 %
  - 12.5 %

36. The rate of photo-induced electron transfer reaction depends on

- a) Reaction free energy
- b) Solvent reorganization energy
- c) Temperature
- d) None of the above

37. Oxygen as a triplet molecule is able to quench efficiently the fluorescence of certain fluorophores. For a particular fluorophore it has been observed that the degree of quenching increase on increasing the concentration, pressure and temperature of the oxygen-containing media. Such quenching is

- a) Static quenching
- b) Dynamics quenching
- c) Both static and dynamic quenching
- d) Insufficient information to comment

38. A protein has two tryptophan. Four X-ray diffractionists are studying the protein. The different understanding about the position of the tryptophan by these four people is given below. First person: One internal tryptophan and a surface exposed tryptophan surrounded by Glu (Negatively charged) and Asp (Negatively charged) Second person: One internal tryptophan and a surface exposed tryptophan surrounded by Gln (Neutral) and two Asn (Neutral) Third person: Both tryptophan are on the surface and surrounded by Glu (Negatively charged) and Asp (Negatively charged) Fourth person: Both tryptophan are on the surface and surrounded by Gln (Neutral) and two Asn (Neutral) You have perform a fluorescence measurement and discovered that

- a) Addition of I<sup>-</sup> or Cs<sup>+</sup> (both are quencher) decreases the tryptophan fluorescence with similar magnitude and the quenching data for I<sup>-</sup> is given below.
- b) Addition of quencher causes a shift of emission maximum to shorter wavelength.

According to your experiment, the correct structure is given by the

- a) First person
- b) Second person
- c) Third person
- d) Fourth person

39. Melittin (a protein) contains one tryptophan. The emission spectrum of melittin and its dansyl derivative are shown below. Given that the R0 value for tryptophan dansyl pair is 23.6 Å.... The distance between tryptophan and dansyl group in melittin is about

- a) 8 Å ...
- b) 16 Å ...
- c) 24 Å ...
- d) 32 Å ...