SEMESTER – I 4H – 4C

Instruction Hours / week: L: 4 T: 0 P: 0 Marks: Internal: 40 External: 60 Total: 100 End Semester Exam: 3 Hours

Course Objectives

19PHP105A

• Study of materials is always important, for any application, including fabrication of satellites.

MATERIAL CHARACTERIZATION

• To introduce various methods available for characterizing the materials. The characterization of materials specifically addresses that portfolio with which researchers and educators must have working familiarity.

Course Outcomes (COs)

- 1. The students are exposed with X-ray, thermal, microscopic, and electrical methods of characterization.
- 2. Understand and describe the fundamental principles behind the methods of characterization which are included in the curriculum
- 3. Analyze, interpret and present observations from the different methods.
- 4. Evaluate the uncertainty of observations and results from the different methods.

UNIT I- X-RAY TECHNIQUES

Introduction, X-Ray Powder Diffraction - Rotatory crystal method of X ray diffraction -Neutron diffraction – experiment- Particle size - strain determination, Single Crystal X-Ray Structure Determination, GIXRD and its applications, X-Ray Photoelectron Spectroscopy, Surface X-Ray Diffraction.

Neutron Techniques

Introduction, Neutron Powder Diffraction, Single Crystal Neutron Diffraction.

UNIT II- THERMAL ANALYSIS

Introduction - Definitions, Codes of Practice and Nomenclature - thermogravimetric analysis (TGA) - instrumentation - determination of weight loss and decomposition products - differential thermal analysis (DTA) - cooling curves - differential scanning calorimetry (DSC) -

instrumentation - specific heat capacity measurements - determination of thermomechanical parameters.

UNIT III- MAGNETIC ANALYSIS AND OPTICAL MICROSCOPY

Vibrating sample magnetometer - SQUID : Introduction - construction and working principle. Optical microscopy techniques - Bright field - Dark field optical microscopy - phase contrast microscopy - differential interference contrast microscopy - fluorescence microscopy - confocal microscopy - Metallurgical microscope.

UNIT IV - ELECTRON MICROSCOPY AND SCANNING PROBE MICROSCOPY

Electron diffraction technique – High energy electron diffraction – Low energy electron diffraction - Electron microscopy – Scanning electron microscopy (SEM) - FESEM - EDAX - TEM - HRTEM: working principle and Instrumentation - sample preparation - Advantages/disadvantages.

Scanning probe microscopy - STM - AFM - EPMA - working principle and Instrumentation - Advantages/disadvantages.

UNIT V - ELECTRICAL AND OPTICAL METHODS OF CHARACTERIZATION

Two probe and four probe methods - van der Pauw method - Hall probe and measurement - scattering mechanism - C-V, I-V characteristics - Schottky barrier capacitance - impurity concentration - electrochemical C-V profiling - limitations - Introduction to Photoluminescence and Electroluminescence - Applications. Dielectrics - working principle and Instrumentation - Applications.

SUGGESTED READINGS

- Elton N. Kaufmann, Characterization of Materials volumes 1 and 2, John Wiley & Sons, Inc., Hoboken, New Jersey, 2003.
- R.A.Stradling and P.C.Klipstain. Growth and Characterization of semiconductors. Adam Hilger, Bristol, 1990.
- 3. Cullity B D., Stock S R "Elements of X-ray Diffraction", Prentice Hall, Inc 2001.
- J.A.Belk. Electron Microscopy and Microanalysis of Crystalline Materials. Applied Science Publishers, London, 1979.

- 5. Banwell, Fundamentals of Molecular Spectroscopy, McGraw-Hill Education, Pvt. Ltd., 2013.
- D.Kealey & P.J.Haines, Analytical Chemistry, Viva Books Private Limited, New Delhi, 2002.



KARPAGAM ACADEMY OF HIGHER EDUCATION (Deemed to be University Established Under Section 3 of UGC Act 1956) Coimbatore – 641 021.

LECTURE PLAN DEPARTMENT OF PHYSICS

STAFF NAME : Dr. V. SENTHIL KUMAR SUBJECT NAME: MATERIALS CHARACTERIZATIONSUB.CODE:19PHP105A SEMESTER: I CLASS: I M.Sc., (PHYSICS)

| Sl.No. | Lecture Duration Period | Topics to be covered | Support Material/Page Nos. | |
|--------|-------------------------------|---|-------------------------------|--|
| | | UNIT I | | |
| 1. | 1 hr | X-Ray Techniques Introduction, X-Ray Powder Diffraction | T1(835) | |
| 2. | 1 hr | Particle size - strain determination, | J1 | |
| 3. | 1 hr | Single Crystal X-Ray Structure Determination, | T1(850) | |
| 4. | 1 hr | GIXRD and its applications | | |
| 5. | 1 hr | X-Ray Photoelectron Spectroscopy | T1(970) | |
| 6. | 1 hr | Surface X-Ray Diffraction | T1(1007 | |
| 7. | 1 hr | Neutron Techniques Introduction, Neutron Powder Diffraction | T1(1285) | |
| 8. | 1 hr | Single Crystal Neutron Diffraction. | T1(1307) | |
| 9. | 1 hr | Revision | | |
| | | Total Number of Hours Pla | nned For Unit I = 9 | |
| | | | | |
| | | UNIT II | | |
| 10. | 1 hr | Thermal Analysis Introduction - Definitions, Codes of Practice and Nomenclature | T1 (337-344) | |
| 11. | 1 hr | thermogravimetric analysis (TGA) - instrumentation | T1(344-360) | |
| 12. | 1 hr | determination of weight loss and decomposition | T1(352-360) | |

| | | products | | | |
|-----|---|--|----------------------|--|--|
| 13. | 1 hr | differential thermal analysis (DTA) - cooling | T1(362-370) | | |
| | | curves | | | |
| 14. | 1 hr | differential scanning calorimetry (DSC) - | T1(362-370) | | |
| | | instrumentation | | | |
| 15. | 1 hr | specific heat capacity measurements | T1(362-370) | | |
| 16. | 1 hr | determination of thermomechanical parameters | T1(362-370) | | |
| 17. | 1 hr | Revision | | | |
| | | Total Number of Hours Plan | med For Unit II = 8 | | |
| | | | | | |
| | | UNIT III | | | |
| 18. | 1 hr | Magnetic Analysis | W1 | | |
| | | Vibrating sample magnetometer | | | |
| 10 | 1 1 4 | COULD : Introduction construction and | WO | | |
| 19. | 1 nr | SQUID : Introduction - construction and | W Z | | |
| 20 | 1 hr | Ontical Microscony | T1(665-667) | | |
| 20. | 20. I III Optical Microscopy II(003-007) | | | | |
| 21. | 1 hr | Bright field - Dark field optical microscopy - | T1(665-667) | | |
| | | phase contrast microscopy | () | | |
| 22. | 1 hr | differential interference contrast microscopy | T1(665-667) | | |
| 23. | 1 hr | fluorescence microscopy - confocal microscopy | T1(665-667) | | |
| 24. | 1 hr | Metallurgical microscope | T1(665-667) | | |
| 25. | 1 hr | Revision | | | |
| | | Total Number of Hours Plan | ned For Unit III = 8 | | |
| | | | | | |
| | | UNIT IV | | | |
| 26. | 1 hr | Electron Microscopy | T1(1049) | | |
| | | Electron diffraction technique - working | | | |
| | | principle and Instrumentation | | | |
| 27. | 1 hr | High energy electron diffraction – Low energy electron diffraction | T1(1120) | | |
| 28. | 1 hr | Electron microscopy – Scanning electron | T1(1050) | | |
| | | microscopy (SEM) | | | |
| 29. | 1 hr | FESEM – EDAX | W3 | | |
| 30. | 1 hr | TEM - HRTEM - sample preparation - | T1(1063) | | |

| | | Advantages/disadvantages. | | | |
|-----|------|--|----------------------------------|--|--|
| 31. | 1 hr | Scanning Probe Microscopy | W4 | | |
| | | Scanning probe microscopy - working principle | | | |
| | | and Instrumentation | | | |
| 32. | 1 hr | STM | T1(1111) | | |
| 33. | 1 hr | AFM | W5 | | |
| 34. | 1 hr | EPMA - Advantages/disadvantages | W6 | | |
| 35. | 1 hr | Revision | | | |
| | | Total Number of Hours Plann | ed For Unit IV = 10 | | |
| | | | | | |
| | | UNIT V | | | |
| 36. | 1 hr | Electrical and Optical Methods of | W7 | | |
| | | Characterization | | | |
| | | Two probe and four probe methods | | | |
| 37. | 1 hr | Van der Pauw method | W7 | | |
| 38. | 1 hr | Hall probe and measurementT1(411) | | | |
| 39. | 1 hr | scattering mechanism T1(411) | | | |
| 40. | 1 hr | C-V, I-V characteristics | C-V, I-V characteristics T1(456) | | |
| 41. | 1 hr | Schottky barrier capacitance T1(459) | | | |
| 42. | 1 hr | Impurity concentration – electrochemical C-V T1(460) profiling – limitations | | | |
| 43. | 1 hr | Introduction to Photoluminescence and T1(427) Electroluminescence – Applications | | | |
| 44. | 1 hr | Dielectrics - working principle and W5 Instrumentation – Applications W5 | | | |
| 45. | 1 hr | Revision | | | |
| 46. | 1 hr | Old Question Paper Revision | | | |
| 47. | 1 hr | Old Question Paper Revision | | | |
| 48. | 1 hr | Old Question Paper Revision | | | |
| | | Total Number of Hours Plan | ned For Unit V = 13 | | |

Suggested Reading Books

- Elton N. Kaufmann, Characterization of Materials volumes 1 and 2, John Wiley & Sons, Inc., Hoboken, New Jersey, 2003.
- 2. R.A.Stradling and P.C.Klipstain. Growth and Characterization of semiconductors. Adam Hilger, Bristol, 1990.
- 3. Cullity B D., Stock S R "Elements of X-ray Diffraction", Prentice Hall, Inc 2001.
- J.A.Belk. Electron Microscopy and Microanalysis of Crystalline Materials. Applied Science Publishers, London, 1979.
- L. E.Murr. Electron and Ion microscopy and Microanalysis principles and Applications. Marcel Dekker Inc., New York, 1991.
- 6. D.Kealey & P.J.Haines, Analytical Chemistry, Viva Books Private Limited, New Delhi, 2002.
- 7. Banwell, Fundamentals of Molecular Spectroscopy, McGraw-Hill Education, Pvt. Ltd., 2013.
- 8. J1 http://www.jtaphys.com/content/2251-7235/6/1/6
- 9. W1 https://nptel.ac.in/courses/115103030/30
- 10. W2 http://squid.iitd.ernet.in/Basic_Literature.htm
- 11. W3 http://shodhganga.inflibnet.ac.in/bitstream/10603/87209/5/chapter%202.pdf
- 12. W4 https://www.bruker.com/products/surface-and-dimensional-analysis/atomic-forcemicroscopes/campaigns/spm-microscopes-intro-to-scanning-probe-microscopy.html
- 13. W5 http://shodhganga.inflibnet.ac.in/bitstream/10603/70669/11/11_chapter%204.pdf
- 14. W6 https://www.cameca.com/products/epma/technique
- 15. W7 https://nptel.ac.in/courses/115103030/25.

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SYLLABUS

X-Ray Techniques

Introduction, X-Ray Powder Diffraction - Particle size - strain determination, Single Crystal X-Ray Structure Determination, GIXRD and its applications, X-Ray Photoelectron Spectroscopy, Surface X-Ray Diffraction.

Neutron Techniques

Introduction, Neutron Powder Diffraction, Single Crystal Neutron Diffraction.

POWDER X-RAY DIFFRACTION (PXRD)

Powder X-ray diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined.X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined.

Fundamental principles of Powder X-ray diffraction (PXRD)

Max von Laue, in 1912, discovered that crystalline substances act as three-dimensional diffraction gratings for X-ray wavelengths similar to the spacing of planes in a crystal lattice. X-ray diffraction is now a common technique for the study of crystal structures and atomic spacing.

X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample. The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n\lambda=2d \sin \theta$). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted. By scanning the sample through a range of 2 θ angle, all possible diffraction directions of the lattice

should be attained due to the random orientation of the powdered material. Conversion of the diffraction peaks to d-spacing allows identification of the mineral because each mineral has a set of unique d-spacing. Typically, this is achieved by comparison of d-spacing with standard reference patterns.

All diffraction methods are based on generation of X-ray in an X-ray tube. These X-rays are directed at the sample, and the diffracted rays are collected. A key component of all diffraction is the angle between the incident and diffracted rays.

Instrumentation

X-ray diffractometer consist of three basic elements: an X-ray tube, a sample holder, and an X-ray detector. Bruker's X-ray Diffraction D8-Discover instrument. X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. These spectra consist of several components, the most common being K_{α} and K_{β}. K_{α} consists, in part, of K_{α 1} and K_{α 2}. K_{α 1} has a slightly shorter wavelength and twice the intensity as $K_{\alpha 2}$. The specific wavelengths are characteristic of the target material (Cu, Fe, Mo, Cr). Filtering, by foils or crystal monochromator, is required to produce monochromaticX-rays needed for diffraction. $K_{\alpha 1}$ and $K_{\alpha 2}$ are sufficiently close in wavelength such that a weighted average of the two is used. Copper is the most common target material for single-crystal diffraction, with CuK_{α} radiation = 1.5418Å. These X-rays are collimated and directed onto the sample. As the sample and detector are rotated, the intensity of the reflected X-rays is recorded. When the geometry of the incident X-rays impinging the sample satisfies the Bragg Equation, constructive interference occurs and a peak in intensity occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor. The geometry of an X-ray diffractometer is such that the sample rotates in the path of the collimated X-ray beam at an angle θ while the X-ray detector is mounted on an arm to collect the diffracted X-rays and rotates at an angle of 20. The instrument used to maintain the angle and rotate the sample is termed a goniometer. For typical powder patterns, data is collected at 2θ from ~5° to 70°, angles that are preset in theX-ray scan.

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Applications

Powder X-ray diffraction is most widely used for the identification of unknown crystalline materials (e.g. minerals, inorganic compounds). Determination of unknown solids is critical to studies in geology, environmental science, material science, engineering and biology. Other applications include:

- i) Characterization of crystalline materials
- ii) Identification of fine-grained minerals such as clays and mixed layer clays that are difficult to determine optically
- iii) Determination of unit cell dimensions
- iv) Measurement of sample purity

With specialized techniques, XRD can be used to:

- i) Determine crystal structures using Rietveld refinement
- ii) Determine of modal amounts of minerals (quantitative analysis)
- iii) Characterize thin films samples by:
 - a) Determining lattice mismatch between film and substrate and to inferring stress and strain
 - b) Determining dislocation density and quality of the film by rocking curve measurements
 - c) Measuring super lattices in multilayered epitaxial structures
 - d) Determining the thickness, roughness and density of the film using glancing incidence X-ray reflectivity measurements
- iv) Make textural measurements, such as the orientation of grains, in a polycrystalline sample

Strengths and Limitations of Powder X-ray diffraction (XRD) Strengths

- i) Powerful and rapid (< 20 min) technique for identification of an unknown mineral
- ii) In most cases, it provides an unambiguous material determination

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- iii) Minimal sample preparation is required
- iv) XRD units are widely available
- v) Data interpretation is relatively straight forward

Limitations

- i) Homogeneous and single phase material is best for identification of an unknown
- ii) Must have access to a standard reference file of inorganic compounds(d-spacing, hkl)
- iii) Requires tenths of a gram of material which must be ground into a powder
- iv) For mixed materials, detection limit is ~ 2% of sample
- v) For unit cell determinations, indexing of patterns for non-isometric crystal systems is complicated
- vi) Peak overlay may occur and worsens for high angle 'reflections'.

Sample collection and preparation

Determination of an unknown requires: the material, an instrument for grinding, and a sample holder.

Obtain a few tenths of a gram (or more) of the material, as pure as possible

Grind the sample to a fine powder, typically in a fluid to minimize inducing extra strain (surface energy) that can offset peak positions, and to randomize orientation. Powder less than $\sim 10 \ \mu m$ (or 200-mesh) in size is preferred

Data collection, results and presentation

Data collection

The intensity of diffracted X-rays is continuously recorded as the sample and detector rotate through their respective angles. A peak in intensity occurs when the material contains lattice planes with d-spacing appropriate to diffract X-rays at that value of θ . Although each peak consists of two separate reflections (K α_1 and K α_2), at small values of 2 θ the peak locations overlap with K α_2 appearing as a hump on the side of K α_1 . Greater separation occurs at higher values of θ . Typically these combined peaks are treated as one.

Data reduction

Results are commonly presented as peak positions at 2θ and X-ray counts (intensity) in the form of a table or an x-y plot (shown above). Intensity (*I*) is either reported as peak height intensity, that intensity above background, or as integrated intensity, the area under the peak. The relative intensity is recorded as the ratio of the peak intensity to that of the most intense peak.

Determination of an unknown

The d-spacing of each peak is then obtained by solution of the Bragg equation for the appropriate value of λ . Once all d-spacing have been determined, automated search/match routines compare the *d*'s of the unknown to those of known materials. Because each material has a unique set of d-spacing, matching these d-spacing provides an identification of the unknown sample. A systematic procedure is used by ordering the d-spacing in terms of their intensity beginning with the most intense peak. Files of d-spacing for hundreds of thousands of organic and inorganic compounds are available from the International Centre for Diffraction Data as the Powder Diffraction File (PDF). Commonly this information is an integral portion of the software that comes with the instrumentation.

Particle size and strain determination:

Determination of Size and Strain

The previous section explained how size and inhomogeneous strain can broaden the powder diffraction peak. A question that has occupied the minds of many powder diffractionists during the last (20th) century is whether the mean size and strain within a powder can be calculated from the diffraction pattern even when both are present simultaneously. We answer this question in both an approximate and more rigorous fashion.

Williamson-Hall Plot

This method is attributed to G.K.Williamson and his student, W.H.Hall (Acta Metall. 1, 22-31 (1953)). It relies on the principle that the approximate formulae for size broadening, β_L , and strain broadening, β_e , vary quite differently with respect to Bragg angle, θ :

$$\beta_{\rm L} = \mathbf{K} \lambda$$

 $L\cos\theta$

 $\beta_e = \mathbf{C}\varepsilon \tan\theta$

One contribution varies as $1/\cos\theta$ and the other as $\tan\theta$. If both contributions are present then their combined effect should be determined by convolution. The simplification of Williamson and Hall is to assume the convolution is either a simple sum or sum of squares (see previous discussion on Sources of Peak Broadening within this section). Using the former of these then we get:

Κλ

$$\beta_{tot} = \beta_e + \beta_L = \mathbf{C}\varepsilon \tan\theta +$$

 $L\cos\theta$

If we multiply this equation by $\cos\theta$ we get:

Κλ

L

 $\beta_{\text{tot}} \cos\theta = \mathbf{C}\varepsilon \sin\theta +$ ____

and comparing this to the standard equation for a straight line (m = slope; c = intercept)

y = mx + c

we see that by plotting $\beta_{tot}\cos\theta$ versus $\sin\theta$ we obtain the strain component from the slope (C ϵ) and the size component from the intercept (**K** λ /*L*). Such a plot is known as a Williamson-Hall plot and is illustrated schematically below (note that this plot could alternatively be expressed in reciprocal space parameters, β^* versus d^*):

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sinθ

It can be a good idea to label each data point on the Williamson-Hall plot according to the index of its reflection to see whether any pattern emerges: for example, in the illustration several h00 reflections seem to lie slightly above the mean straight line suggesting that the powder crystallites are thinnest in the <100> crystal direction. However the Williamson-Hall method has many assumptions: its absolute values should not be taken too seriously but it can be a useful method if used in the relative sense; for example a study of many powder patterns of the same chemical compound, but synthesised under different conditions, might reveal trends in the crystallite size/strain which in turn can be related to the properties of the product.

Warren-Averbach method

This method is attributed to B.E.Warren and B.L.Averbach (J.Appl.Phys. 21, 595(1950) and ibid 23, 497 (1952)) and has become accepted as one of the more rigorous and widely used methods for separating the effects of size and strain. The treatment behind the method is quite

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complex and involves expressing the peak intensity in terms of a Fourier sum, which has distinct mathematical advantages (half of the terms - the sine components - turn out to be negligible, and (de-)convolution is conveniently handled in Fourier space). A further trick used is to arrange the powder diffraction peaks of a given pattern into h,k,l-indexed groups possessing a common crystallographic direction (e.g. $\langle 00n \rangle = \langle 001 \rangle$, $\langle 002 \rangle$, $\langle 003 \rangle$ where *n* is referred to as the order). This has two particular advantages: it overcomes the problem of asymmetrically sized/strained crystallites as each group analysis then becomes specific for a given direction (i.e. <001> in the above example), and the size/strain separation is simplified by the fact that the size effects are independent of order, n, whereas the strain effects vary with order. The method is exact if the strain distribution in the crystallites are Gaussian, and is still a good approximation otherwise provided the crystallite lattice distortions are small. The kind of final values provided by the method are an average (area-weighted) crystallite size, a parameter that is related to the crystallite size distribution and an average strain (for a characteristic size), all specified for a particular crystallographic direction. Even though the method is clearly not straightforward most commercial powder diffractometer systems provide a Warren-Averbach analysis routine on their software with easy-to-follow instructions.

GIXRD and its Applications

X-ray powder diffraction is most widely used for the identification of unknown crystalline materials (e.g. minerals, inorganic compounds). Determination of unknown solids is critical to studies in geology, environmental science, material science, engineering and biology.

Other applications include:

- characterization of crystalline materials
- identification of fine-grained minerals such as clays and mixed layer clays that are difficult to determine optically
- determination of unit cell dimensions
- measurement of sample purity
- ▶ With specialized techniques, XRD can be used to:

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- determine crystal structures using Riveted refinement
- determine of modal amounts of minerals (quantitative analysis)
- characterize thin films samples by:
 - determining lattice mismatch between film and substrate and to inferring stress and strain
 - determining dislocation density and quality of the film by rocking curve measurements
 - measuring super lattices in multilayered epitaxial structures
 - determining the thickness, roughness and density of the film using glancing incidence X-ray reflectivity measurements
- > make textural measurements, such as the orientation of grains, in a polycrystalline sample
- Strengths and Limitations of X-ray Powder Diffraction (XRD)?

Strengths

- > Powerful and rapid (< 20 min) technique for identification of an unknown mineral
- > In most cases, it provides an unambiguous mineral determination
- Minimal sample preparation is required
- > XRD units are widely available
- Data interpretation is relatively straight forward

Limitations

- > Homogeneous and single phase material is best for identification of an unknown
- Must have access to a standard reference file of inorganic compounds (d-spacing's, *hulls*)
- Requires tenths of a gram of material which must be ground into a powder
- > For mixed materials, detection limit is $\sim 2\%$ of sample
- For unit cell determinations, indexing of patterns for non-isometric crystal systems is complicated
- > Peak overlay may occur and worsens for high angle 'reflections'

X-ray Photoelectron Spectroscopy (XPS)

Photoelectron spectroscopy utilizes photo-ionization and analysis of the kinetic energy distribution of the emitted photoelectrons to study the composition and electronic state of the surface region of a sample.

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Traditionally, when the technique has been used for surface studies it has been subdivided according to the source of exciting radiation into :

| X-ray Photoelectron Spectroscopy (XPS) | - using soft x-rays (with a photon energy |
|--|---|
| | of 200-2000 eV) to examine core-levels. |
| Ultraviolet Photoelectron Spectroscopy (UPS) | - using vacuum UV radiation (with a |
| | photon energy of 10-45 eV) to examine |
| | valence levels. |

The development of synchrotron radiation sources has enabled high resolution studies to be carried out with radiation spanning a much wider and more complete energy range (5 - 5000 + eV) but such work remains a small minority of all photoelectron studies due to the expense, complexity and limited availability of such sources.

Physical Principles

Photoelectron spectroscopy is based upon a single photon in/electron out process and from many viewpoints this underlying process is a much simpler phenomenon than the Auger process. The energy of a photon of all types of electromagnetic radiation is given by the Einstein relation :

E = h v

Where

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h - Planck constant (6.62 \times 10^{-34} \text{ J s})
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v - frequency (Hz) of the radiation

Photoelectron spectroscopy uses monochromatic sources of radiation (i.e. photons of fixed energy).

In XPS the photon is absorbed by an atom in a molecule or solid, leading to ionization and the emission of a core (inner shell) electron. By contrast, in UPS the photon interacts with valence levels of the molecule or solid, leading to ionisation by removal of one of these valence electrons.

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The kinetic energy distribution of the emitted photoelectrons (i.e. the number of emitted photoelectrons as a function of their kinetic energy) can be measured using any appropriate electron energy analyser and a photoelectron spectrum can thus be recorded.

The process of photoionization can be considered in several ways : one way is to look at the overall process as follows :

$$A + hv \rightarrow A^+ + e^-$$

Conservation of energy then requires that :

$$\mathbf{E}(\mathbf{A}) + hv = \mathbf{E}(\mathbf{A}^+) + \mathbf{E}(\mathbf{e}_-)$$

Since the electron's energy is present solely as kinetic energy (KE) this can be rearranged to give the following expression for the KE of the photoelectron:

$$\mathbf{KE} = h\mathbf{v} \cdot (\mathbf{E}(\mathbf{A}^+) \cdot \mathbf{E}(\mathbf{A}))$$

The final term in brackets, representing the difference in energy between the ionized and neutral atoms, is generally called the *binding energy* (BE) of the electron - this then leads to the following commonly quoted equation :

KE = hv - BE

An alternative approach is to consider a one-electron model along the lines of the following pictorial representation ; this model of the process has the benefit of simplicity but it can be rather misleading.

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The BE is now taken to be a direct measure of the energy required to just remove the electron concerned from its initial level to the vacuum level and the KE of the photoelectron is again given by :

KE = hv - BE

NOTE - the binding energies (BE) of energy levels in solids are conventionally measured with respect to the Fermi-level of the solid, rather than the vacuum level. This involves a small correction to the equation given above in order to account for the *work function* (ϕ) of the solid, but for the purposes of the discussion below this correction will be neglected.

Experimental Details

The basic requirements for a photoemission experiment (XPS or UPS) are:

- 1. a source of fixed-energy radiation (an x-ray source for XPS or, typically, a He discharge lamp for UPS)
- 2. an electron energy analyser (which can disperse the emitted electrons according to their kinetic energy, and thereby measure the flux of emitted electrons of a particular energy)

3. a high vacuum environment (to enable the emitted photoelectrons to be analysed without interference from gas phase collisions)

Such a system is illustrated schematically below:



There are many different designs of electron energy analyser but the preferred option for photoemission experiments is a concentric hemispherical analyser (CHA) which uses an electric field between two hemispherical surfaces to disperse the electrons according to their kinetic energy.

X-ray Photoelectron Spectroscopy (XPS)

For each and every element, there will be a characteristic binding energy associated with each core atomic orbital i.e. each element will give rise to a characteristic set of peaks in the hotoelectron spectrum at kinetic energies determined by the photon energy and the respective binding energies.

The presence of peaks at particular energies therefore indicates the presence of a specific element in the sample under study - furthermore, the intensity of the peaks is related to the concentration of the element within the sampled region. Thus, the technique provides a *quantitative analysis of the surface composition* and is sometimes known by the alternative acronym, ESCA (Electron Spectroscopy for Chemical Analysis).

The most commonly employed x-ray sources are those giving rise to :

Mg K_{α} radiation : hv = 1253.6 eV Al K_{α} radiation : hv = 1486.6 eV

The emitted photoelectrons will therefore have kinetic energies in the range of ca. 0 - 1250 eV or

0 - 1480 eV . Since such electrons have very short IMFPs in solids, the technique is necessarily surface sensitive.

Example 1 - the XPS spectrum of Pd metal

The diagram below shows a real XPS spectrum obtained from a Pd metal sample using

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Mg K_{\alpha} radiation
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- the main peaks occur at kinetic energies of ca. 330, 690, 720, 910 and 920 eV.

Since the photon energy of the radiation is always known it is a trivial matter to transform the spectrum so that it is plotted against BE as opposed to KE.



The most intense peak is now seen to occur at a binding energy of ca. 335 eV

Working downwards from the highest energy levels

- the valence band (4*d*, 5*s*) emission occurs at a binding energy of *ca*. 0 8 eV (measured with respect to the Fermi level, or alternatively at ca. 4 12 eV if measured with respect to the vacuum level).
- 2. the emission from the 4*p* and 4*s* levels gives rise to very weak peaks at 54 eV and 88 eV respectively
- 3. the most intense peak at ca. 335 eV is due to emission from the 3*d* levels of the Pd atoms, whilst the 3*p* and 3*s* levels give rise to the peaks at ca. 534/561 eV and 673 eV respectively.
- 4. the remaining peak is not an XPS peak at all ! it is an Auger peak arising from x-ray induced Auger emission. It occurs at a kinetic energy of ca. 330 eV (in this case it is really meaningless to refer to an associated binding energy).

These assignments are summarised below



It may be further noted that

- there are significant differences in the natural widths of the various photoemission peaks
- the peak intensities are not simply related to the electron occupancy of the orbitals

Exercise 1 - the XPS spectrum of NaCl

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Spin-Orbit Splitting

Closer inspection of the spectrum shows that emission from some levels (most obviously 3p and 3d) does not give rise to a single photoemission peak, but a closely spaced doublet.

We can see this more clearly if, for example, we expand the spectrum in the region of the 3d emission ...



The 3*d* photoemission is in fact split between two peaks, one at 334.9 eV BE and the other at 340.2 eV BE, with an intensity ratio of 3:2. This arises from spin-orbit coupling effects in the final state. The inner core electronic configuration of the initial state of the Pd is :

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 $(1s)^{2} (2s)^{2} (2p)^{6} (3s)^{2} (3p)^{6} (3d)^{10} \dots$

with all sub-shells completely full.

The removal of an electron from the 3*d* sub-shell by photo-ionization leads to a $(3d)^9$ configuration for the final state - since the *d*-orbitals (l = 2) have non-zero orbital angular momentum, there will be coupling between the unpaired spin and orbital angular momenta.

Spin-orbit coupling is generally treated using one of two models which correspond to the two limiting ways in which the coupling can occur - these being the LS (or Russell-Saunders) coupling approximation and the *j*-*j* coupling approximation.

If we consider the final ionised state of Pd within the Russell-Saunders coupling approximation, the $(3d)^9$ configuration gives rise to two states (ignoring any coupling with valence levels) which differ slightly in energy and in their degeneracy ...

^{2D}_{5/2}
^{2D}_{5/2}

$$g_J = 2x\{5/2\}+1 = 6$$

^{2D}_{3/2}
 $g_J = 2x\{3/2\}+1 = 4$

These two states arise from the coupling of the L = 2 and S = 1/2 vectors to give permitted J values of 3/2 and 5/2. The lowest energy final state is the one with maximum J (since the shell is more than half-full), i.e. J = 5/2, hence this gives rise to the "lower binding energy" peak. The relative intensities of the two peaks reflects the degeneracies of the final states ($g_J = 2J + 1$), which in turn determines the probability of transition to such a state during photoionization.

The Russell-Saunders coupling approximation is best applied only to light atoms and this splitting can alternatively be described using individual electron *l*-*s* coupling. In this case the resultant angular momenta arise from the single hole in the *d*-shell; a *d*-shell electron (or hole) has l = 2 and s = 1/2, which again gives permitted *j*-values of 3/2 and 5/2 with the latter being lower in energy.

The peaks themselves are conventionally annotated as indicated - note the use of lower case lettering

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This spin-orbit splitting is of course not evident with *s*-levels (l = 0), but is seen with *p*,*d* & *f* core-levels which all show characteristic spin-orbit doublets.

Chemical Shifts

The exact binding energy of an electron depends not only upon the level from which photoemission is occurring, but also upon :

- 1. the formal oxidation state of the atom
- 2. the local chemical and physical environment

Changes in either (1) or (2) give rise to small shifts in the peak positions in the spectrum - so-called *chemical shifts*.

Such shifts are readily observable and interpretable in XP spectra (unlike in Auger spectra) because the technique :

- is of high intrinsic resolution (as core levels are discrete and generally of a well-defined energy)
- is a one electron process (thus simplifying the interpretation)

Atoms of a higher positive oxidation state exhibit a higher binding energy due to the extra coulombic interaction between the photo-emitted electron and the ion core. *This ability to discriminate between different oxidation states and chemical environments is one of the major strengths of the XPS technique*.

In practice, the ability to resolve between atoms exhibiting slightly different chemical shifts is limited by the peak widths which are governed by a combination of factors ; especially

- the intrinsic width of the initial level and the lifetime of the final state
- the line-width of the incident radiation which for traditional x-ray sources can only be improved by using x-ray monochromators
- the resolving power of the electron-energy analyser

In most cases, the second factor is the major contribution to the overall line width.

Angle Dependent Studies

the degree of surface sensitivity of an electron-based technique such as XPS may be varied by collecting photoelectrons emitted at different emission angles to the surface plane. This approach may be used to perform non-destructive analysis of the variation of surface composition with depth (with chemical state specificity).

Surface X-Ray Diffraction

A similar idea allows the direct recovery of surface electron density from measured intensities of reciprocal lattice rods in surface X-ray diffraction. Fig. 5 is a schematic of a diffraction pattern from a (2x2) reconstructed InSb(111) surface. The reciprocal lattice rods run through the Bragg spots on this diagram, and perpendicular to the plane of the figure.





It will be noted that the diffraction amplitudes of the integer-order rods are a sum of those from the bulk and surface regions of the sample, while the fractional-order rods arise solely from diffraction from the reconstructed surface region.

The integer-order rods may be treated in a manner entirely analogous to the structure completion problem described in the previous section. The bulk is the known part of the structure and the surface the unknown part. However, inclusion of just the integer-order (crystal truncation) rods alone in the reconstruction algorithm gives only the *average* structure (Fig. 6a) within the surface unit cell (i.e. one which has the bulk 2D periodicity). Nevertheless, this gives an excellent starting electron density for further refinement by inclusion in the algorithm of the fractional-order rod intensities, which finally recovers the accepted vacancy-buckling model [12] in which

the Sb atoms in the top double layer closest to the vacancies have moved towards those holes. Such a tendency is apparent even after just 10 iterations of the algorithm following the inclusion of the fractional-order intensities (Fig. 6b). The final reconstructed image (not shown) is even capable of resolving bucklings normal to the surface of as little as 0.2 Å.



NEURON DIFFRACTION:

Neutron diffraction or elastic neutron scattering is the application of neutron scattering to the determination of the atomic and/or magnetic structure of a material. A sample to be examined is placed in a beam of thermal or cold neutrons to obtain a diffraction pattern that provides information of the structure of the material. The technique is similar to X-ray diffraction but due to their different scattering properties, neutrons and X-rays provide complementary information: X-Rays are suited for superficial analysis, strong x-rays from synchrotron radiation are suited for shallow depths or thin specimens, while neutrons having high penetration depth are suited for bulk samples.

Construction and working of Single Crystal Neutron Diffraction:

The technique requires a source of neutrons. Neutrons are usually produced in a nuclear reactor or spallation source. At a research reactor, other components are needed, including a crystal monochromators, as well as filters to select the desired neutron wavelength. Some parts of the setup may also be movable. At a spallation source, the time of flight technique is used to sort the energies of the incident neutrons (higher energy neutrons are faster), so no monochromatic is needed, but rather a series of aperture elements synchronized to filter neutron pulses with the desired wavelength.

The technique is most commonly performed as powder diffraction, which only requires a polycrystalline powder. Single crystal work is also possible, but the crystals must be much larger than those that are used in single-crystal X-ray crystallography. It is common to use crystals that are about 1 mm³.

Summarizing, the main disadvantage to neutron diffraction is the requirement for a nuclear reactor. For single crystal work, the technique requires relatively large crystals, which are usually challenging to grow. The advantages to the technique are many - sensitivity to light atoms, ability to distinguish isotopes, absence of radiation damage, as well as a penetration depth of several cm.

Nuclear Scattering:

Like all quantum particles, neutrons can exhibit wave phenomena typically associated with light or sound. Diffraction is one of these phenomena; it occurs when waves encounter obstacles whose size is comparable with the wavelength. If the wavelength of a quantum particle is short enough, atoms or their nuclei can serve as diffraction obstacles. When a beam of neutrons emanating from a reactor is slowed down and selected properly by their speed, their wavelength lies near one angstrom (0.1 nanometer), and the typical separation between atoms in a solid material. Such a beam can then be used to perform a diffraction experiment. Impinging on a crystalline sample, it will scatter under a limited number of well-defined angles, according to the same Bragg's law that describes X-ray diffraction.

Neutrons and X-rays interact with matter differently. X-rays interact primarily with the electron cloud surrounding each atom. The contribution to the diffracted x-ray intensity is therefore larger for atoms with larger atomic number (Z). On the other hand, neutrons interact directly with the *nucleus* of the atom, and the contribution to the diffracted intensity depends on each isotope; for example, regular hydrogen and deuterium contribute differently. It is also often the case that light (low Z) atoms contribute strongly to the diffracted intensity, even in the presence of large Z atoms. The scattering length varies from isotope to isotope rather than linearly with the atomic number. An element like vanadium is a strong scattered of X-rays, but

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its nuclei hardly scatter neutrons, which is why it is often used as a container material. Nonmagnetic neutron diffraction is directly sensitive to the positions of the nuclei of the atoms.

Unlike X-rays, neutrons scatter mostly from the nuclei of the atoms, which are tiny. Furthermore, there is no need for an atomic form factor to describe the shape of the electron cloud of the atom and the scattering power of an atom does not fall off with the scattering angle as it does for X-rays. Diffract grams therefore can show strong well defined diffraction peaks even at high angles, particularly if the experiment is done at low temperatures. Many neutron sources are equipped with liquid helium cooling systems that allow data collection at temperatures down to 4.2 K. The superb high angle (i.e. high *resolution*) information means that the atomic positions in the structure can be determined with high precision. On the other hand, Fourier maps (and to a lesser extent difference Fourier maps) derived from neutron data suffer from series termination errors, sometimes so much that the results are meaningless.



KARPAGAM ACADEMY OF HIGHER EDUCATION, COIMBATORE-21 DEPARTMENT OF PHYSICS CLASS : 1 M.SC PHYSICS BATCH: 2019-2021 PART A : MULTIPLE CHOICE QUESTIONS (ONLINE EXAMINATIONS) SUBJECT : MATERIAL CHARACTERIZATION SUBJECT CODE : 19PHP105A UNIT-1

| SL.NO. | QUESTIONS | opt 1 | opt2 | opt3 | opt4 | Answer |
|--------|--|------------------------------|---------------------------------|--|-----------------------|------------------------------|
| 1 | Thin film may be arbitrarily defined as a solid layer having a thickness varying from a few \hat{A} to about | 10mm | 10cm | 10µm | 10^-3 nm | 10µm |
| 2 | Thickness of ultra thin film is | >1000Å | 50-100Å | 100-1000Å | none of these | 50-100Å |
| | An ideal film extended infinite in directions but excited along | ture and a dimension | and a dimension | three and more | and the second | turn and a dimension |
| 2 | An idea rum extended infinity in | two and z direction | one and z direction | three and none | none of these | two and z direction |
| 4 | behaving as | semiconductors | conductors | insulators | dielectrics | semiconductors |
| 5 | A freshly formed film surface becomes highly reactive due to | high volume to surface ratio | high surface to volume ratio | high thickness | none of these | high surface to volume ratio |
| 6 | Characteristic of a surface are more often observed in | amorphous | single crystal | polycrystalline | thin films | thin films |
| 7 | Thin films can be prepared from a variety of materials such as | metals | semiconductors | insulators | all the above | all the above |
| 8 | The primary requirement for the methods (a) Thermal deposition and Sputtering is a | high vacuum | higher than ambient pressure | ambient pressure | none of these | high vacuum |
| q | involves the evanoration or sublimation of the material in vacuo by thermal energy | snuttering | CVD | Thermal denosition | chemical deposition | Thermal deposition |
| | | 100 | | | 1000 | |
| 10 | when an me molecules are condensed, sucking coefficient has a max, value of | 100 | 1 | 10 | 1000 | 1 |
| 11 | When none is deposited, sticking coefficient has a min. value of | 1 | 10 | 0 | -1 | 0 |
| 12 | velocity distribution of evaporant molecules will depend on the | nature of the source | nature of the substrate | nature of the target | none of these | nature of the source |
| 13 | The emitted vapour stream will have the same velocity distribution in all directions thus resembling the emission from | surface source | point source | cylindrical source | prism source | point source |
| 14 | Forthe emission velocity of the vapour stream will decrease with the increase of the angle of inclination | point source | surface source | cylindrical source | none of these | surface source |
| 15 | If =0, then a surface source resembles a | cylindrical source | point source | cylindrical as well as point source | none of these | point source |
| 16 | Heating sources are made from | refractory metals | semiconductors | insulators | none of these | refractory metals |
| | The amount of deposits received by a substrate will depend not only on the nature of the source but also | | | source as well as substrate | | |
| 1/ | on the inclination of the vapour stream with the Positioning of the substrate with respect to the source as well as the nature of the source considerably | substrate-normal direction | source-normal direction | normal | none of these | substrate-normal direction |
| 18 | affects the | film growth | film thickness | film purity | none of these | film thickness |
| 19 | A highgenerally improves the physical characteristics of the deposited films | source temperature | substrate temperature | nature of the substrate | substrate thickness | substrate temperature |
| 20 | Higher vapour pressure constituent will tend to vaporise at a | lower temperature | higher temperature | temperature | none of these | lower temperature |
| 21 | There is no accumulation of the charge on the heated boat in | Thermal evaporation | Electron beam method | sputtering | Flash evaporation | Flash evaporation |
| 22 | Which method is generally adopted when a material has a tendency to decompose or dissociate during evaporation | Flash evaporation | Electron beam method | sputtering | CVD | Flash evaporation |
| 23 | A constant composition film can be produced by | Electron beam method | sputtering | Flash evaporation | CVD | Flash evaporation |
| 24 | In which method several sources are simultaneously used for the evaporation of the constituent species | multi evaporation process | Electron beam method | sputtering | evaporation process | multi evaporation process |
| 25 | The simulation of the side of the second form different second is because as | | | | Electron been method | |
| 23 | The simultaneous deposition process from different sources is known as | co-evaporation | muiti evaporation process | evaporation process | Electron beam method | co-evaporation |
| 26 | In electron beam method the electron beam is accelerated withvoltage | 2V | 4kV | 10V | 2-10kV | 2-10kV |
| 27 | Which method is now routinely used for the production of metal film resistors and others | Electron beam method | multi evaporation process | sputtering | Flash evaporation | Electron beam method |
| 28 | There aretypes of electron beam guns | three | two | one | five | three |
| 29 | In, electrons are emitted from hair pin type of filament | work accelerated gun | self-accelerated gun | bent beam electron gun | none of these | self-accelerated gun |
| 30 | In Electron beam is bent by an appropriate magnetic field | work accelerated gun | self-accelerated gun | bent beam electron gun | none of these | bent beam electron gun |
| 31 | The deposition of metal films by sputtering from a cathode by the glow discharge method was first observed by | Wehner | buckel and hilsch | grove | pease | grove |
| 32 | Sputtering yield increases with the of the bombarding ions | mass | energy | mass and energy | none of these | mass and energy |
| 33 | Auser transition takes place along with emission of | -electrons | -electrons | -electrons | radiation less | -electrons |
| | If the process does not involve any chemical reaction between bombarding gas ions and the cathode is | and a second state | dealed and a | 11.4 million (1997) | | had have he |
| 34 | known as | reactive sputtering | physical sputtering | high pressure sputtering | both b and c | both b and c |
| 35 | If the process involve any chemical reaction between bombarding gas ions and the cathode is known as | reactive sputtering | physical sputtering | high pressure sputtering | both a and c | both a and c |
| 36 | Glow discharge is used to clean | dirt | plastics | polymers | none of these | dirt |
| 37 | The simplest method of sputtering is by the | glow discharge technique | Electron beam method | Flash evaporation | evaporation process | glow discharge technique |
| 38 | The minimum voltage at which breakdown takes place is called | transition region | abnormal glow discharge | break down voltage | normal glow discharge | break down voltage |
| 39 | In glow discharge sputtering, the region where there is a large increase in voltage as well as current is called the | abnormal glow region | townsend region | normal glow discharge | transition region | abnormal glow region |
| 40 | In glow discharge sputtering, the region where there is a large increase in current at a constant voltage is called the | abnormal glow region | townsend region | normal glow discharge | transition region | townsend region |

| 41 | A continuous sputtering can take place only in | transition region | townsend region | normal glow discharge | abnormal glow region | abnormal glow region |
|----|--|----------------------------|----------------------------------|------------------------------------|------------------------------|------------------------------------|
| | | | | | | |
| 42 | in glow discharge sputtering, luminosity of the is the most intense | cathode glow | negative glow | anode glow | faraday dark space | negative glow |
| 43 | Which technique is particularly suitable for refractory materials which caannot easily be deposited by other methods | glow discharge sputtering | Electron beam method | Flash evaporation | evaporation process | glow discharge sputtering |
| 44 | In method, gases like oxygen or nitrogen are used as media | glow discharge sputtering | Electron beam method | Flash evaporation | reactive sputtering | reactive sputtering |
| 45 | Different types of films of mixed compositions can be prepared comparatively easily by | Low pressure sputtering | reactive sputtering | Electron beam method | glow discharge sputtering | reactive sputtering |
| 46 | Enhanced ionisation can be achieved in the | r.f.field | d.c.voltage | reactive sputtering | low pressure sputtering | r.f.field |
| 47 | Sputtering of cathode materials in presence of inert or active gases either at | low pressure | medium pressure | high pressure | all the above | high pressue |
| | | | | | | |
| 48 | In low pressure sputtering, ionisation of gas molecules takes place at low vacuum say | 10^-1 torr | 10^-15 torr | 10^-10 torr | 10^-6 torr | 10^-1 |
| 49 | In RF sputtering method an impedance matching between is necessary | Power supply and electrode | Discharge tube and DC voltage | Power supply and discharge tube | voltage | Power supply and discharge tube |
| 50 | RF sputtering technique is particularly useful for the deposition of | Insulator | Dielectrics | Semiconductor | Conductor | Dielectrics |
| | | | | | | |
| 51 | The deposition of films from gaseous phases by thermal decomposition is known as | chemical vapour deposition | sputtering | Thermal deposition | evaporation process | chemical vapour deposition |
| 52 | Which technique is used for the preparation of various inorganic as well as organic compounds | chemical vapour deposition | sputtering | Thermal deposition | evaporation process | chemical vapour deposition |
| 53 | When tetra ethoxy silane is heated to a temperature of about 700-900°C, a dielectric film ofis formed | SiO2 | Si | SiH4 | с | SiO2 |
| | | | | | | |
| 54 | Gunther method has been used for the preparation of wafers of CdS,CdSe etc. | Single crystal | Polycrystal | Amorphous | both a and c | Single crystal |
| 55 | Which method has three temperature zones | Vapour phase reaction | vapour transportation method | Disproportionation method | pyrolysis | vapour transportation method |
| | The SiO2 films can be produced by the reaction of SiCl4 vapours with CO2 in the presence of H2 gas is | | | | | |
| 56 | known as | Gunther method | Philips process | Disproportionation method | pyrolysis | Philips process |
| 57 | Which technique is often used for the preparation of very thick films and wafers and even flat shaped crystals of several mm sizes | Vapour phase reaction | vapour transportation method | Disproportionation method | pyrolysis | vapour transportation method |
| 58 | Which method depends on the difference in the stability of polyvalent metal compounds at two temperatures | Vapour phase reaction | vapour transportation method | Disproportionation method | pyrolysis | Disproportionation method |
| 59 | Which method depends on the deposition of films from aqueous solutions either by passing a current or by chemical reactions | chemical deposition | chemical vapour deposition | evaporation process | sputtering | chemical deposition |
| | | | | | - | - |
| 60 | In process metal films can be deposited without passing any current | electrodeposition | anodic oxidation | electroless plating | chemical reaction | electroless plating |
| 61 | In mass method balance methods is made of | silica | glass | quartz | magnesium | quartz |
| | | a | | | a | |
| 62 | in crystal oscillator thickness measurement depends on the oscillation of a | Quartz fibre | Quartz crystal | Quartz amorphous | Quartz film | Quartz crystal |
| 63 | Which method uses interference fringes for the measurement of film thickness? | crystal oscillator | photometric | ellipsometry | interferometry | interferometry |
| 64 | Which of the following technique comes under mass methods | crystal oscillator | photometric | ellipsometry | interferometry | crystal oscillator |
| 65 | Which is a very accurate method which is eminently suitable for measuring the thickness of surface layers or films as well as their optical constants | ellipsometry | interferometry | photometric | microbalance | ellipsometry |
| | Degreasing and cleaning are carried out in a closed chamber by suspending the substrates in vapours of | | | | | |
| | organic solvents such as | carbon tetrachloride | alcohol | trichloro ethylene | both a and c | both a and c |

CLASS: IMSC PHYSICS

COURSE NAME: MATERIAL CHARACTERIZATION

COURSE CODE: 19PHP105A UNIT II (Thermal Analysis)

BATCH-2019-2021

SYLLABUS

Thermal Analysis

Introduction - Definitions, Codes of Practice and Nomenclature - thermogravimetric analysis (TGA) - instrumentation - determination of weight loss and decomposition products - differential thermal analysis (DTA) - cooling curves - differential scanning calorimetry (DSC) - instrumentation - specific heat capacity measurements - determination of thermomechanical parameters.

THERMAL ANALYSIS

Thermal analysis is a branch of materials science where the properties of materials are studied as they change with temperature. Several methods are commonly used – these are distinguished from one another by the property which is measured:

Dielectric thermal analysis (DEA): dielectric permittivity and loss factor

Differential thermal analysis (DTA): temperature difference versus temperature or time Differential scanning calorimetric (DSC): heat flow changes versus temperature or time

Dilatometer (DIL): volume changes with temperature change

Dynamic mechanical analysis (DMA or DMTA) : measures storage modulus (stiffness) and loss modulus (damping) versus temperature, time and frequency

Evolved gas analysis (EGA): analysis of gases evolved during heating of a material, usually decomposition products

Laser flash analysis (LFA): thermal diffusivity and thermal conductivity

Thermo gravimetric analysis (TGA): mass change versus temperature or time

Thermo mechanical analysis (TMA): dimensional changes versus temperature or time

Thermo-optical analysis (TOA): optical properties

Derivatography: A complex method in thermal analysis simultaneous thermal analysis (STA) generally refers to the simultaneous application of thermogravimetry (TGA) and differential scanning calorimetric (DSC) to one and the same sample in a single instrument. The test conditions are perfectly identical for the TGA and DSCsignals (same atmosphere, gas flow rate, vapor pressure of the sample, heating rate, thermal contact to the sample crucible and

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sensor, radiation effect, etc.). The information gathered can even be enhanced by coupling the STA instrument to an Evolved Gas Analyzer (EGA) like Fourier transform infrared spectroscopy (FTIR) or mass spectrometry (MS).

Other, less common, methods measure the sound or light emission from a sample, or the electrical discharge from a dielectric material, or the mechanical relaxation in a stressed specimen. The essence of all these techniques is that the sample's response is recorded as a function of temperature (and time).

It is usual to control the temperature in a predetermined way - either by a continuous increase or decrease in temperature at a constant rate (linear heating/cooling) or by carrying out a series of determinations at different temperatures (stepwise isothermal measurements). More advanced temperature profiles have been developed which use an oscillating (usually sine or square wave) heating rate (Modulated Temperature Thermal Analysis) or modify the heating rate in response to changes in the system's properties (Sample Controlled Thermal Analysis).

In addition to controlling the temperature of the sample, it is also important to control its environment (e.g. atmosphere). Measurements may be carried out in air or under an inert gas (e.g. nitrogen or helium). Reducing or reactive atmospheres have also been used and measurements are even carried out with the sample surrounded by water or other liquids. Inverse gas chromatography is a technique which studies the interaction of gases and vapors with a surface - measurements are often made at different temperatures so that these experiments can be considered to come under the auspices of Thermal Analysis.

Atomic force microscopy uses a fine stylus to map the topography and mechanical properties of surfaces to high spatial resolution. By controlling the temperature of the heated tip and/or the sample a form of spatially resolved thermal analysis can be carried out.

Thermal analysis is also often used as a term for the study of transfer through structures. Many of the basic engineering data for modeling such systems comes from measurements of heat capacity and thermal conductivity.

The thermal analysis is one of the important and oldest methods to analyze the thermal behavior of the materials. Sophisticated instruments have been developed to get useful quantitative and qualitative information on the desired materials with respect to temperature change. The advanced thermal analysis instruments are used to measure various properties such as weight loss, phase transformation, glass transition, specific heat capacity, the energy of enthalpy, thermo mechanical behavior, viscoelastic properties and reaction kinetics (Michael 2004).

1.7.5.1 Thermogravimetric analysis

Thermogravimetric analysis (TGA) measures the rate of change in mass of the sample as the function of temperature or time under controlled atmosphere. TGA is widely used to identify the oxidation, thermal stability and volatility properties of samples. Thermobalance consists of a programmable temperature controller with a computer and an electronic microbalance with the furnace which controls the temperature and simultaneously measures the heating or cooling temperature and weight of the sample. The three important thermogram signals are collected when analyzing the samples, which are, temperature, weight and rate of weight change (i.e., differential thermogravimetric). The loss of mass is recorded with respect to temperature or time. Owing to the nature of the bond between molecules, the mass loss percentage is varied when increasing the temperature. The schematic diagram of TG analyzer is shown in Figure 1.7.



The schematic diagram of TG analyzer

Thermogravimetry provides a quantitative measurement of any weight changes associated with thermally induced transitions. For example TG can record directly the loss in weight as a function of temperature or time for transitions that involves dehydration or decomposition. The rates of these thermally induced processes are often a function of the molecular structure charges in weight result from physical and chemical bonds forming and breaking at elevated temperature. TG data are useful in characterizing materials as well as in investigating the thermodynamics and kinetics of the reaction and transitions that result from the application of heat to these materials. The usual temperature range for TG is from ambient to 1200 °c in either inert or reaction atmosphere.

Instrumentation

In TG the weight of the sample is continuously recorded as the temperature is increased. Samples are placed in a crucible or a shallow disc that is positioned in a furnace on a quartz beam attached to an automatic recording balance. TG instrument that contains a taut- ban suspension electromechanical transducer. The horizontal quartz beam is maintained in the null position by the current flowing through the transducer coil of an electromagnetic balance. A pair of photo sensitive diodes acts as a position sensor to determine the movement of the beam. Any change in the weight of the sample causes a deflection of the beam, which is sensed by one of the photo diodes. The beam is then restored to the original null position by a feedback current sent from the photo diodes to the coil of the balance.

TG curve

The weight change TG curve can be understood by using the curve for a single component and binary mixtures. The crucible is removed from the thermo balance then it is cleaned and dried and again put in position in the thermo balance. Then, thermo balance is operated to get a base line which is indicated by dash line. The substance to be determined is precipitated by a suitable precipitant and then transferred to the crucible. Now the heating in the thermo balance is started and the weight loss curve is recorded as usual to get the thermo gram.

Application

TG is useful to determine the correct drying temperatures of precipitates.

TG is useful to find suitable standards in analytical chemistry.

Thermo balance study can be used in determining the purity of various substances.

TG can also be used to study the thermal effects on induced magnetism and evaluating the effects of heat on ferromagnetic materials.

TG has been used to study the reaction between certain oxides.

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DIFFERENTIAL THERMAL ANALYSIS

In differential thermal analysis (DTA), the temperature difference between the inert reference and a test sample is measured and recorded. The furnace is heated under defined atmospheric condition. The variation of heat absorption or desorption of the sample is compared with an inert reference sample. From DTA instruments the thermal properties and phase variation of the sample can be identified but this method cannot quantify the enthalpy change. Low heating rate and sample weight lead sharper peak of high resolution which also influences the properties of decomposition phenomenon and kinetics. In the case of single-componentsystems, only simple phase transformation occurs. For multiple components, many phases such as formation and deformation of materials can be noticed.

Differential thermal analysis (DTA) technique is simple as it involves the technique of recording the difference in temperature between a substance and a reference material against either time or temperature as the two specimens are subjected to identical temperature in an environment heated or cooled at a controlled rate. Thus a differential thermo gram consists of a record of the difference in sample and reference temperature (differential temperature ΔT) plotted as a function of time 't', sample temperature (T_s), reference temperature (T_r) or furnace temperature (T_f).

Instrumentation

A large number of different types of instruments are available for DTA studies. However a typical DTA apparatus is shown. The various components of a DTA apparatus are as follows

Furnace

This is a device for heating the sample in the apparatus, one always prefers a tabular furnace. This is constructed with an appropriate material (wire or ribbon) wound on a refractory tube. Such furnace possess the desired characteristics for good temperature regulation and programming these are fairly inexpensive. Generally the choice of the resistance material as well as refractory is decided from the intended maximum temperature of operation and gaseous environments.
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Sample holder

This is used to contain the sample as well as the reference material. Generally sample holder made up of platinum is employed. In the most of the DTA instruments, specimen holder assemblies are identical isolated cups supported on thermocouple as well as metallic or nonmetallic blocks with wells. In these assemblies, the sample or the reference material can either be packed in the respective wells, which fit tightly into the wells resulting effectively heat transfer. In these assemblies, the reference temperature may be measured either at the center of reference or sample using the respective arm of the differential thermocouple.

Temperature controller/ sensors

In order to control temperature, the three basic elements are required, they are sensor, control element and heater. The control element governs the rate of heat input required to match the heat loss from the system. The location of the sensor with respect to the heater and made of heat transfer measure the time elapsed between sensing and variation in heat input. It there occurs adjustment between these three components, successful temperature control is possible.

Recorder

This is to record the DTA curve in thermo analytical studies the signals obtained from the sensors can be recorded in which the signal trace is produced on paper or film, by ink, heating stylus, electric writing or optical beam.

DC Amplifier and furnace programmer

In DTA apparatus, generally a low level Dc amplifier is employed and the main function of furnace temperature programmer is to increase the temperature of the furnace at a steady rate.

DTA curve

In the ideal DTA curve there is an exothermic peak and an endothermic peak. Both the shape and size of the peak may furnished good information about the nature of test sample. Generally, sharp endothermic peaks give ideas of change in crystallinity or fusion processes whereas broad end. In most of the cases, physical changes give rise to endothermic curves whereas chemical reactions give rise to exothermic peaks. The origin of the temperature

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difference in the sample (ΔT) lies in the energy difference between the product and the reactants or between the product and the reactants or between the two phases of a substance. This energy differences is manifested as enthalpic changes either exothermic or endothermic.

Application

Fingerprinting by the pattern of thermo grams, qualitative and quantitative analyses by peak area or height for a wide range of materials.

Study of thermal characteristics, stability, degradation and reaction kinetics on small samples, over a temperature range of -175 °C to 1000 °C and above.

DIFFERENTIAL SCANNING CALORIMETRY

Differential scanning calorimetry (DSC) is one of the most important analytical tools in the thermal analysis that measures the flow of heat in the sample and gives the information about the change in physical properties with the function of temperature. The DSC instrument is also similar to DTA apparatus but the electrical energy is used to restore the cooler of the both sample and reference materials. Figure 1.8 illustrate the schematic diagram of DSC apparatus. Depending on nature of sample endothermic or exothermic process occurs during heat flow through the sample. The DSC instrument can be operated in two ways, heating and cooling with constant heating or cooling rate. The schematic (Figure 1.9) representation reveals the possible transitions that are produced from DSC curve but all the parameters could not occur in a single compound or sample.



The schematic diagram of DSC apparatus

KARPAGAM ACADEMY OF HIGHER EDUCATION CLASS: IMSC PHYSICS COURSE NAME: MATERIAL CHARACTERIZATION COURSE CODE: 19PHP105A UNIT II (Thermal Analysis) BATCH-2019-2021 Orystallization Orystallization

Melting

Temperature

Possible thermal behavior of sample with respect to temperature and heat flow

Thermal kinetics

The thermal parameters of material play an important role in nucleation formation (Cahoon 2012). Thermal kinetics is one of the most important analysis to understand the nucleation mechanism in every crystal growth process. The knowledge about thermal kinetics is useful to grow better quality crystals from melt growth techniques. By using Arrhenius equation, thermal kinetic parameters were calculated for many inorganic glasses and compounds such as hydrated iron sulphate (Straszko et al 1997), dolomite (Olszak-Humienik&Możejko 1999), molybdate (IV) and tungstate (IV) with chromium (III) (Ali &Kowsar 1998) and cobalt sulphate hexahydrate (Straszko et al 2000). Even though there are many characterization and interpretations on organic crystals grown by solution and melt growth methods, only limited investigation reports are available on thermal kinetic parameters of organic materials. The researchers have performed non-isothermal and isothermal studies for various compounds, in which non - isothermal condition is implemented for a wide range of temperature in lesser time duration (Sidel et al 2011). Arrhenius equation is used generally to evaluate the thermal kinetic parameters. However, different types of equations were derived from Arrhenius equation by other researchers like Kissingers (Kissinger & Homer 1957), Ozawa (Ozawa 1970), Augis and Bennet (Augis & Bennett 1978), Kissinger - Akahira - Sunose (KAS) and Ozawa - Flynn - Wall (OFW) (Benavidez et al 2011) to estimate the values of thermal kinetic parameter.

The data conversion from thermogram given by the thermal analysis instrument into kinetic parameters such as activation energy, the order of reaction, rate constant and frequency

$$\frac{\mathrm{d}\alpha}{\mathrm{d}t} = \mathrm{k.}\,\mathrm{f}(\alpha) \tag{1.2}$$

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where, k is the rate constant and α can be evaluated from the thermogravimetric curve of the material.

The classic Kissinger equation is used for the calculation of thermal kinetic parameters from DTA curves. Kinetic parameters and reaction mechanism can be determined simultaneously by generalized Kissinger equation. The Kissingers equation gives an analytical solution, which correlate the kinetic behavior of melting or decomposition peak temperatures. The better results were achieved from Kissinger equation which is due to the consideration of many assumptions in the equation. Hence, the application of Kissinger equation is extended widely. The Kissinger equation is represented as,

$$\ln\left(\frac{\beta}{T_{m}^{2}}\right) = \frac{-E_{a}}{RT_{m}} + \ln\left(\frac{K_{0}R}{E_{a}}\right)$$
(1.3)

While applying this equation to non - first - order kinetics, similar results are obtained. The activation energy is calculated using isoconversional methods which are alogarithmic form of the kinetic equation. Owing to the repeatability of this calculation method, it is recommended for calculating the activation energy of various materials.

The degree of conversion (α) is the most useful parameter for the calculation of kinetic parameters at single heating rate and it is defined by the relation,

$$\alpha = \frac{M_s - M(t,T)}{M_s - M_f} \tag{1.4}$$

where, M_s - initial mass, M_f - final mass and M(t,T) - instantaneous mass which depends on temperature and time. The calculated values of α can be substituted in such kinetic equations derived by Coats and Redfern or Friedman methods, the thermal kinetic parameter can be evaluated.

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factor or pre-exponential factor is mainly based on utilization of classical kinetic laws. The rate

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SL.NO. QUESTIONS opt 1 opt2 opt3 opt4 Answe In electron diffraction technique maximum voltage upto 60-63keV 60-65eV 60-65keV 60-63eV 60-65keV SEM LEED HEED XPS .Is used to analyse the surface layers of metals, semiconductor and insula XPS The surface layers of Ni single crystal was first made by oth a and b oth a and In Finch type camera, a maximum voltage is limited upto 100keV 200keV 5keV 40keV 65keV In HEED method the accelerating potential of electrons is about 40 to 100keV 60 to 200keV to 500eV oth a and b both a and b In LEED method the accelerating potential of electrons is abo to 500eV 50 to 200keV 40 to 100keV one of these 5 to 500eV 500Å For transmission method the material thickness should be lower that 1000Å 10000Å 3000Å 500Å 10^-9 to 10^-10 8 The vacuum conditions in LEED is in an order of . 10^-9 to 10^-10 torr 10^-1 to 10^-2 torr < 10^-1 torr 10^-4 to 10^-6 torr is used as a standard material in HEED graphite nagnesiun graphite pper Which method provide information of both surface layers and different layers constituting the material can .EED HEED SEM ГЕМ HEED cannot pas elastically scattered electrons annot passes nelastically scattered lectrons annot passe nelastically cattered elec es inelastically s elastically 11 The function of repeller in LEED attered electron attered electi ormation of oxide ayers 12 A LEED concerns primarily about the nature of the ill the abov ill the abo rface laye 13 Slow electrons because of their low energy interact.... ...with matter eakly trongly nediumly both a and b strongly 14 The resolution in an electron microscope is sbout. 5-10Å <5Å >5Å 100Å 5-10Å ... is one of the most useful and versatile instruments for the investigation of surface topography, anning electron canning electron LEED HEED TEM 15 microstructural feature, etc icroscope microscope <100eV >100eV 16 The secondary electrons have very low energy say ... <50eV 60eV <50eV eld emission uger electron 17 SEM technique along with..... Is widely used for surface studies ooth a and b spectroscopy lectron microprobe nicroscopy both a and b positions of the peaks in the sitions of the peaks shifts of the ESCA he shape of the ESCA peaks 18 The composition of the surface material is readily determined from the in the spectra all the abo spectrais a very useful tool for the analysis of organic compounds and polymer materials of thickness 19 about 40 to 100Å SEM LEED HEED XPS XPS 20 In SIMS, the sputtered particles come out as. all the abov all the abo egative i utral 21 The condensation of vapour atoms on a neutral solid takes place from a Condition of vapours supersaturated under saturated either a or b supersaturated aturated nucleation involves film formation over a foreign substrate surface. homogeneo either a or b both a and b heterogeneou heterog If the substrate is of the same material as the vapour atoms then the nucleation process is called either a or b both a and b homoge eteroger 24 Formation of critical nucleus involves the of heat of condensation of vapour atoms release absorption adsorption transfer releas 25 Impinging atoms can be reflected back to vapour state without... ondensation condensation vaporation absorption desorption 26 Evaporation can take place at stages ... 2,3 and 4 ,2,3 and 5 ,2 and 3 ,4 and 5 ,2,3 and 5 27 A higher Ts generally favours a larger grain size due to the increasingof the surface ad-atoms obility nobility absorptic dsorption onder 28 A ...size of area can be examined with mass spectroscopy nicro 29 If the ions have the same energy, the radii of curvatures will be olum

| 30 | is a very sensitive method to identify the elemental composition of a film | mass spectroscopy | SIMS | SEM | XPS | mass spectroscopy |
|----|--|---|-----------------------------|--|--------------------|--------------------|
| | | | | | | |
| 31 | Adsorbed species after migration collide to form | subcritical nuclei | critical nuclei | cluster | either a or b or c | either a or b or c |
| | To a thermally available and an entry of impiration atoms, the marketability of condensed atoms stability to the | | | abaamtian | | |
| 32 | in a mermany equilibrated system of impinging atoms, the probability of condensed atoms sucking to the substrate surface is known as | thermal accomodation | sticking coefficient | coefficient | either a or b | either a or b |
| | | | | | | |
| 33 | is a microscopic and short-lived phase(metastable) | surface-solid phase | surface-liquid phase | surface-gas phase | bulk-gas phase | surface-gas phase |
| | | | | | | |
| 34 | Ad-atoms also called | monomers | dimers | trimers | polymers | monomer |
| | | | | | | |
| 35 | When thermal equilibrium is established the flux of impinging atoms on the substrate will be equal to theflux of atoms | re-evaporations | desorbed | adsorbed | either a or b | either a or b |
| | | | | | | |
| 36 | The surface diffusion generally takes place byof an ad-atom from one possible site to a next one | jumping | bonning | moving | either a or b | either a or b |
| 50 | next one | Jumping | nopping | lioving | | chaler a or b |
| 27 | The root mean square distance travelled by an ad-atom beforeis an important factor in | | decompliant | | and the shore | decomption |
| 57 | nucleanon process | evaporation | desorption | condensation | none of the above | uesorption |
| | | | atomistic or statistical | | | |
| 38 | In order to explain the nucleation and cluster formationmodels have been found useful | capillarity | models | BCF model | both a and b | both a and b |
| | Capillarity Model is an extension of the classical theory of Volmer-Weber-Becker-Doring forof | | | | | |
| 39 | a supersaturated vapour phase to the liquid state | phase transition | adsorption | condensation | none of the above | condensation |
| 40 | The surface energy will of course increase withr | increasing | decreasing | constant | either a or b | increasing |
| 41 | If a molecular volume of the film material is condensed from a supersaturated vapour pressure to equilibrium pressure of bulk liquid G _c is given by | -(KT/V)ln(p/p,) | (KT/V)ln(p/p _c) | (KT)ln(p/p_) | (KT)ln(R/R,) | -(KT/V)ln(p/pe) |
| 43 | | | | | | |
| 42 | For high supersaturations the critical cluster may consist ofatom | one | two | three | many | one |
| | From critical nuclei of 2 and 3 atoms stable clusters of 3 and 4 atoms in And | | | square and | triangular and | triangular and |
| 43 | arrangements respectively are obtained | single and square | triangular and square | hexagonal | hexagonal | square |
| | | direct addition of atoms from the vapour | from other | by the diffusion controlled process | | |
| 44 | The formation of these islands and their growth take place either by | phase | environment | of ad-atoms | any of the above | any of the above |
| | | | | | | |
| 45 | Islands consist of comparatively larger nuclei say greater than | 10Å | 1Å | 10nm | 1mm | 10Å |
| | | | | | | |
| 46 | Thecontrolled process is more commonly observed except at low substrate temperature | evaporation | diffusion | absorption | desorption | diffusion |
| | | | | | | |
| 47 | The smallest stable nuclei are of radii of about | 10Å | 5Å | 10cm | 1mm | 5Å |
| | | | | | | |
| 48 | Most embryos prior to the formation of island structure are of sizes about | 15-30Å | greater than 30Å | less than 15Å | none of the above | 15-30Å |
| | | | | | | |
| 49 | An island will be made up aboutatoms or more | 50-100 | 100-200 | 300-600 | 10-200 | 50-100 |
| | · · · · · · · · · · · · · · · · · · · | | | | | |
| 50 | The surface migration distance of ad-atoms of silver deposited from the vapour phase has been estimated to be about | 500nm | 500µm | 500Å | 300nm | 500Å |
| 51 | The coalescence involves considerable transfer of Between island by diffusion | atoms | molecules | mass | volume | mass |
| 57 | The time of coalescence is very short say about | 0.6second | 3second | 30second | 40second | 0.6second |
| 52 | with film thickness holes or gans will decrease in size | increasing | decreasing | constant | none of the above | increasing |
| | waitthin unceases, notes or gaps will decrease in size | mercasing | occreasing | deposition | none of the above | mercasing |
| 54 | The minimum film thickness for the continuous stage is also dependent on the | nature of the deposits | modes of deposition | parameters | all the above | all the above |
| 55 | The number of can be reduced by the thermal annealing process | foreign species | impurities | grain boundaries | all the above | grain boundaries |
| 56 | Defects such ascan also develop during nucleations and grain growths | twinning | stacking fault | dislocation | all the above | all the above |
| 57 | films often develop surface asperities and roughness | thicker | thin | very thick | none of the above | thicker |
| 58 | The formation of larger masses of nuclei resulting in the Of the nucleation density | decrease | increase | either a or b | no change | decrease |
| 59 | In an electron diffraction camera monochromatic electrons pass through an aperture of a | Diaphragm | Specimen | Focusing coil | Screen | Diaphragm |
| 60 | In electron diffraction camera works as the anode normally at the earth potential | Mono chromatic electron beam source | Specimen | Diaphragm | Focusing coil | Diaphragm |
| 61 | Electron beam should be | Coherent | Monochromatic | High intense | Both a& b | Both a& b |

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SYLLABUS

Magnetic Analysis

Vibrating sample magnetometer - SQUID : Introduction - construction and working principle. **Optical Microscopy**

Optical microscopy techniques - Bright field - Dark field optical microscopy - phase contrast microscopy - differential interference contrast microscopy - fluorescence microscopy - confocal microscopy - Metallurgical microscope.

VIBRATING-SAMPLE MAGNETOMETER (VSM)

A vibrating-sample magnetometer (VSM) is a scientific instrument that measures magnetic properties. Simon Finer at MIT Lincoln Laboratory invented VSM in 1955 and reported it in 1959. A sample is first magnetized in a uniform magnetic field. It is then sinusoidal vibrated, typically through the use of a piezoelectric material. Commercial systems use linear actuators of some form. Historically, these systems were developed using modified audio speakers, though this approach was dropped due to the interference through the produced in-phase magnetic noise, as the magnetic flux through a nearby pickup coil varies sinusoidal. The induced voltage in the pickup coil is proportional to the sample's magnetic moment, but does not depend on the strength of the applied magnetic field. In a typical setup, the induced voltage is measured with an amplifier using the piezoelectric signal as a frequency reference. It is also possible to record the hysteresis curve of a material by sweeping the magnetic field. The idea of vibrating sample came from D. O. Smith's vibrating-coil magnetometer.

A schematic drawing of the VSM sample holder and detection mechanism of VSM is shown in Figure 30.01. The set up was invented by Simon Foner at MIT in 1955.

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Figure 30.01: Schematic of sample holder and VSM mechanism.

• A sample to be studied is placed inside the uniform magnetic field and then vibrated sinusoidally using vibrator made of linear actuators or modified audio speakers.

• The induced voltage in the pickup coil is proportional to the sample's magnetic moment, but does not depend on the strength of the applied magnetic field.

• In a typical setup, the induced voltage is measured through the lock-in amplifier using the piezoelectric signal as its reference signal. Lock-in principle of measurement allows the measurement of signal weaker than the noise. By measuring in the field of an external electromagnet, it is possible to obtain the hysteresis curve of a material.

• VSM measures the magnetization of a small sample of magnetic material placed in an external magnetizing field by converting the dipole field of the sample into an AC electrical signal.

SQUID

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A **SQUID** (for **superconducting quantum interference device**) is a very sensitive magnetometer used to measure extremely subtle magnetic fields, based on superconducting loops containing Josephson junctions.

SQUIDs are sensitive enough to measure fields as low as 5 at $(5 \times 10^{-18} \text{ T})$ with a few days of averaged measurements.^[1] Their noise levels are as low as 3 fT·Hz^{-1/2}.^[2] For comparison, a typical refrigerator magnet produces 0.01 Tesla (10^{-2} T) , and some processes in animals produce very small magnetic fields between 10^{-9} T and 10^{-6} T . Recently invented SERF atomic magnetometers are potentially more sensitive and do not require cryogenic refrigeration but are orders of magnitude larger in size (~1 cm³) and must be operated in a near-zero magnetic field.

OPTICAL MICROSCOPE

The **optical microscope**, often referred to as the **light microscope**, is a type of microscope that uses visible light and a system of lenses to magnify images of small subjects. Optical microscopes are the oldest design of microscope and were possibly invented in their present compound form in the 17th century. Basic optical microscopes can be very simple, although many complex designs aim to improve resolution and sample contrast.

The image from an optical microscope can be captured by normal, photosensitive cameras to generate a micrograph. Originally images were captured by photographic film, but modern developments in CMOS and charge-coupled device (CCD) cameras allow the capture of digital images. Purely digital microscopes are now available which use a CCD camera to examine a sample, showing the resulting image directly on a computer screen without the need for eyepieces.

Alternatives to optical microscopy which do not use visible light include scanning electron microscopy and transmission electron microscopy and scanning probe microscopy.

On 8 October 2014, the Nobel Prize in Chemistry was awarded to Eric Bet zig, William Mourner and Stefan Hell for "the development of super-resolved fluorescence microscopy," which brings "optical microscopy into the nan dimension

Types:

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There are two basic types of optical microscopes: simple microscopes and compound microscopes. A simple microscope is one which uses a single lens for magnification, such as a magnifying glass. A compound microscope uses several lenses to enhance the magnification of an object. The vast majority of modern research microscopes are compound microscopes while some cheaper commercial digital microscopes are simple single lens microscopes. Compound microscopes can be further divided into a variety of other types of microscopes which differ in their optical configurations, cost, and intended purposes

Simple microscope

A simple microscope uses a lens or set of lenses to enlarge an object through angular magnification alone, giving the viewer an erect enlarged virtual image.^{[3][4]} The use of a single convex lens or groups of lenses are found in simple magnification devices such as the magnifying glass, loupes, and eyepieces for telescopes and microscopes.

Compound microscope

A compound microscope uses a lens close to the object being viewed to collect light (called the objective lens) which focuses an image of the object inside the microscope (image 1). That image is then magnified by a second lens or group of lenses (called the eyepiece) that gives the viewer an enlarged inverted virtual image of the object (image 2). The use of a compound objective/eyepiece combination allows for much higher magnification. Common compound microscopes often feature exchangeable objective lenses, allowing the user to quickly adjust the magnification. A compound microscope also enables more advanced illumination setups, such as phase contrast.

Other microscope variants

- There are many variants of the compound optical microscope design for specialized purposes. Some of these are physical design differences allowing specialization for certain purposes:
- Stereo microscope, a low-powered microscope which provides a stereoscopic view of the sample, commonly used for dissection.

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- Comparison microscope, which has two separate light paths allowing direct comparison of two samples via one image in each eye.
- Inverted microscope, for studying samples from below; useful for cell cultures in liquid, or for metallography.
- Fiber optic connector inspection microscope, designed for connector end-face inspection
- Traveling microscope, for studying samples of high optical resolution.

Other microscope variants are designed for different illumination techniques:

- Petrographic microscope, whose design usually includes a polarizing filter, rotating stage and gypsum plate to facilitate the study of minerals or other crystalline materials whose optical properties can vary with orientation.
- Polarizing microscope, similar to the petrographic microscope.
- Phase contrast microscope, which applies the phase contrast illumination method.
- Epifluorescence microscope, designed for analysis of samples which include fluorophores.
- Confocal microscope, a widely used variant of epifluorescent illumination which uses a scanning laser to illuminate a sample for fluorescence.
- Two-photon microscope, used to image fluorescence deeper in scattering media and reduce photobleaching, especially in living samples.
- Student microscope an often low-power microscope with simplified controls and sometimes low quality optics designed for school use or as a starter instrument for children.
- Ultra microscope, an adapted light microscope that uses scattering to allow viewing of tiny particles whose diameter is below or near the wavelength of visible light (around 500 nanometers); mostly obsolete since the advent of electron microscopes

Digital microscope

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A digital microscope is a microscope equipped with a camera allowing observation of a sample via a computer. Microscopes can also be partly or wholly computer-controlled with various levels of automation. Digital microscopy allows greater analysis of a microscope image, for example measurements of distances and areas and quantitation of a fluorescent or histological stain.

Low-powered digital microscopes, USB microscopes, are also commercially available. These are essentially webcams with a high-powered lens and generally do not use Trans illumination. The camera attached directly to the USB port of a computer, so that the images are shown directly on the monitor. They offer modest magnifications (up to about 200×) without the need to use eyepieces, and at very low cost. High power illumination is usually provided by an LED source or sources adjacent to the camera lens.

Digital microscopy with very low light levels to avoid damage to vulnerable biological samples is available using sensitive photon-counting digital cameras. It has been demonstrated that a light source providing pairs of entangled photons may minimize the risk of damage to the most lightsensitive samples. In this application of ghost imaging to photon-sparse microscopy, the sample is illuminated with infrared photons, each of which is spatially correlated with an entangled partner in the visible band for efficient imaging by a photon-counting camera

Lighting techniques

While basic microscope technology and optics have been available for over 400 years it is much more recently that techniques in sample illumination were developed to generate the high quality images seen today.

In August 1893 August Koehler developed Koehler illumination. This method of sample illumination gives rise to extremely even lighting and overcomes many limitations of older techniques of sample illumination. Before development of Koehler illumination the image of the light source, for example a light bulb filament, was always visible in the image of the sample.

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The Nobel Prize in physics was awarded to Dutch physicist Frits Zernike in 1953 for his development of phase contrast illumination which allows imaging of transparent samples. By using interference rather than absorption of light, extremely transparent samples, such as live mammalian cells, can be imaged without having to use staining techniques. Just two years later, in 1955, Georges Nomarski published the theory for differential interference contrast microscopy, another interference-based imaging technique

Fluorescence microscopy

Modern biological microscopy depends heavily on the development of fluorescent probes for specific structures within a cell. In contrast to normal Trans illuminated light microscopy, in fluorescence microscopy the sample is illuminated through the objective lens with a narrow set of wavelengths of light. This light interacts with fluorophores in the sample which then emit light of a longer wavelength. It is this emitted light which makes up the image.

Since the mid-20th century chemical fluorescent stains, such as DAPI which binds to DNA, have been used to label specific structures within the cell. More recent developments include immunofluorescence, which uses fluorescently labelled antibodies to recognize specific proteins within a sample, and fluorescent proteins like GFP which a live cell can express making it fluorescent.

Components:

All modern optical microscopes designed for viewing samples by transmitted light share the same basic components of the light path. In addition, the vast majority of microscopes have the same 'structural' components ^[28] (numbered below according to the image on the right):

- **L**yepiece (ocular lens)
- **4** Objective turret, revolver, or revolving nose piece (to hold multiple objective lenses)
- 4 Objective lenses
- **4** Focus knobs (to move the stage)
- Coarse adjustment
- Fine adjustment

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- **4** Stage (to hold the specimen)
- Light source (a light or a mirror)
- Diaphragm and condenser
- **4** Mechanical stage

Eyepiece (ocular lens)

The eyepiece, or ocular lens, is a cylinder containing two or more lenses; its function is to bring the image into focus for the eye. The eyepiece is inserted into the top end of the body tube. Eyepieces are interchangeable and many different eyepieces can be inserted with different degrees of magnification. Typical magnification values for eyepieces include $5\times$, $10\times$ (the most common), $15\times$ and $20\times$. In some high performance microscopes, the optical configuration of the objective lens and eyepiece are matched to give the best possible optical performance. This occurs most commonly with apochromatic objectives.

Objective turret (revolver or revolving nose piece)

Objective turret, revolver, or revolving nose piece is the part that holds the set of objective lenses. It allows the user to switch between objective lenses.

Objective

At the lower end of a typical compound optical microscope, there are one or more objective lenses that collect light from the sample. The objective is usually in a cylinder housing containing a glass single or multi-element compound lens. Typically there will be around three objective lenses screwed into a circular nose piece which may be rotated to select the required objective lens. These arrangements are designed to be par focal, which means that when one changes from one lens to another on a microscope, the sample stays in focus. Microscope objectives are characterized by two parameters, namely, magnification and numerical aperture. The former typically ranges from $5 \times$ to $100 \times$ while the latter ranges from 0.14 to 0.7, corresponding to focal lengths of about 40 to 2 mm, respectively. Objective lenses with higher

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magnifications normally have a higher numerical aperture and a shorter depth of field in the resulting image. Some high performance objective lenses may require matched eyepieces to deliver the best optical performance

Oil immersion objective

Some microscopes make use of objectives or water-immersion objectives for greater resolution at high magnification. These are used with material such as immersion oil or water and a matched cover slip between the objective lens and the sample. The refractive index of the index-matching material is higher than air allowing the objective lens to have a larger numerical aperture (greater than 1) so that the light is transmitted from the specimen to the outer face of the objective lens with minimal refraction. Numerical apertures as high as 1.6 can be achieved. The larger numerical aperture allows collection of more light making detailed observation of smaller details possible. An oil immersion lens usually has a magnification of 40 to $100 \times$

Focus knobs

Adjustment knobs move the stage up and down with separate adjustment for coarse and fine focusing. The same controls enable the microscope to adjust to specimens of different thickness. In older designs of microscopes, the focus adjustment wheels move the microscope tube up or down relative to the stand and had a fixed stage.

Frame

The whole of the optical assembly is traditionally attached to a rigid arm, which in turn is attached to a robust U-shaped foot to provide the necessary rigidity. The arm angle may be adjustable to allow the viewing angle to be adjusted.

The frame provides a mounting point for various microscope controls. Normally this will include controls for focusing, typically a large knurled wheel to adjust coarse focus, together with a

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smaller knurled wheel to control fine focus. Other features may be lamp controls and/or controls for adjusting the condenser.

Stage

The stage is a platform below the objective which supports the specimen being viewed. In the center of the stage is a hole through which light passes to illuminate the specimen. The stage usually has arms to hold slides (rectangular glass plates with typical dimensions of 25×75 mm, on which the specimen is mounted).

At magnifications higher than $100 \times \text{moving}$ a slide by hand is not practical. A mechanical stage, typical of medium and higher priced microscopes, allows tiny movements of the slide via control knobs that reposition the sample/slide as desired. If a microscope did not originally have a mechanical stage it may be possible to add one.

All stages move up and down for focus. With a mechanical stage slides move on two horizontal axes for positioning the specimen to examine specimen details.

Focusing starts at lower magnification in order to center the specimen by the user on the stage. Moving to a higher magnification requires the stage to be moved higher vertically for re-focus at the higher magnification and may also require slight horizontal specimen position adjustment. Horizontal specimen position adjustments are the reason for having a mechanical stage.

Due to the difficulty in preparing specimens and mounting them on slides, for children it's best to begin with prepared slides that are centered and focus easily regardless of the focus level used.

Light source

Many sources of light can be used. At its simplest, daylight is directed via a mirror. Most microscopes, however, have their own adjustable and controllable light source – often a halogen lamp, although illumination using LEDs and lasers are becoming a more common provision. Koehler illumination is often provided on more expensive instruments.

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Condenser

The condenser is a lens designed to focus light from the illumination source onto the sample. The condenser may also include other features, such as a diaphragm and/or filters, to manage the quality and intensity of the illumination. For illumination techniques like dark field, phase contrast and differential interference contrast microscopy additional optical components must be precisely aligned in the light path.

Magnification

The actual power or magnification of a compound optical microscope is the product of the powers of the ocular (eyepiece) and the objective lens. The maximum normal magnifications of the ocular and objective are $10\times$ and $100\times$ respectively, giving a final magnification of 1,000.

Magnification and micrographs

When using a camera to capture a micrograph the effective magnification of the image must take into account the size of the image. This is independent of whether it is on a print from a film negative or displayed digitally on a computer screen.

In the case of photographic film cameras the calculation is simple; the final magnification is the product of: the objective lens magnification, the camera optics magnification and the enlargement factor of the film print relative to the negative. A typical value of the enlargement factor is around $5\times$ (for the case of 35 mm film and a 15×10 cm (6×4 inch) print).

In the case of digital cameras the size of the pixels in the CMOS or CCDdetector and the size of the pixels on the screen have to be known. The enlargement factor from the detector to the pixels on screen can then be calculated. As with a film camera the final magnification is the product of: the objective lens magnification, the camera optics magnification and the enlargement factor.

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Other techniques

Modern microscopes allow more than just observation of transmitted light image of a sample; there are many techniques which can be used to extract other kinds of data. Most of these require additional equipment in addition to a basic compound microscope.

- Reflected light, or incident, illumination (for analysis of surface structures)
- Fluorescence microscopy, both:
 - Epifluorescence microscopy
 - Confocal microscopy
- Micro spectroscopy (where a UV-visible spectrophotometer is integrated with an optical microscope)
- Ultraviolet microscopy
- Near-Infrared microscopy
- Multiple transmission microscopy for contrast enhancement and aberration reduction.
- Automation (for automatic scanning of a large sample or image capture)

Applications

Optical microscopy is used extensively in microelectronics, nanophysics, biotechnology, pharmaceutics research, mineralogy and microbiology.

Optical microscopy is used for medical diagnosis, the field being termed histopathology when dealing with tissues, or in smear tests on free cells or tissue fragments.

In industrial use, binocular microscopes are common. Aside from applications needing true depth perception, the use of dual eyepieces reduces strain associated with long workdays at a microscopy station. In certain applications, long-working-distance or long-focus microscopes are beneficial. An item may need to be examined behind a window, or industrial subjects may be a hazard to the objective. Such optics resemble telescopes with close-focus capabilities

Measuring microscopes are used for precision measurement. There are two basic types. One has a reticle graduated to allow measuring distances in the focal plane. The other (and older) type has

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simple crosshairs and a micrometer mechanism for moving the subject relative to the microscope.



KARPAGAM ACADEMY OF HIGHER EDUCATION, COIMBATORE-21 DEPARTMENT OF PHYSICS CLASS : I M.SC PHYSICS BATCH: 2019-2021 PART A : MULTIPLE CHOICE QUESTIONS SUBJECT : MATERIAL CHARACTERIZATION SUBJECT : MATERIAL CHARACTERIZATION SUBJECT CODE : 19PHP105A UNIT-III

| SL.NO. | QUESTIONS | opt 1 | opt2 | opt3 | opt4 | Answer |
|--------|---|-------------------------------|---|---|-------------------------------|-------------------------------|
| 1 | In the crystal growth process, supersaturation will be attained | when temperature increases | when temperature decreases | when salt dissolves completely | none of these | when temperature increases |
| 2 | Heterogeneous nucleation is typically much faster than homogeneous nucleation because nucleation barrier isat a surface | higher | much lower | much higher | lower | much lower |
| 3 | The solubility diagram is between temperature and | concentration | time | pressure | none of these | concentration |
| 4 | Bridgman crystal growth method is based on | gel growth | solution growth | melt growth | vapour growth | melt growth |
| 5 | The spontaneous formation of crystalline nuclei in the interior of the parent phase is called \ldots . | Homogeneous nucleation | heterogeneous nucleation | instability | seed | Homogeneous nucleation |
| 6 | A crucible used in the bridgman crystal growth method is | quartz | glass | plastics | metals | quartz |
| 7 | The gibbs free energy changes from | 0 to infinity | -1 to 0 | 0 to 1 | none of these | 0 to infinity |
| 8 | The solubility of most substances | does not relate | increases | decreases | none of these | increases |
| 9 | According to BCF theory, the important factor for growth parameter is | surface diffusion | volume diffusion | ternary growth | pressure | surface diffusion |
| 10 | The vapour transport is carried out in | crucibles | Al rod | quartz | silicon | Al rod |
| 11 | An example for physical gel is | silica | polyacrylamide | tetramethoxy silane | agar-agar | silica |
| 12 | A decrease of crystalis demanded simultaneously with the development of greater crystal dimensions | defects | quality | inhomogeneities | both a and c | both a and c |
| 13 | The photonic device evolution is due to the advent ofCrystals | compound semiconductor | metal-organic | organic | inorganic | compound semiconductor |
| 14 | The major use of GaAs at present is in the area of | microwave devices | high speed digital integrated circuits | substrates for epitaxial layer growth | all the above | all the above |
| 15 | InP is well established as the base material for | optical components | electronic component | mechanical component | opto-electronic components | opto-electronic components |
| 16 | The are among the most widespread type of scintillation materials | oxide crystals | organic crystals | halogenide scintillators | quartz | halogenide scintillators |
| 17 | Alkali halide scintillators like | NaI:TI | KMgF3 | CsCaBr3 | RbMgF3 | NaI:TI |
| 18 | Fluorperovskites such as | CsSrBr3 | LiF:Eu | CsI | LiF:Eu | CsSrBr3 |
| 19 | The applications of alkali halide scintillators are | UV applications | ultrasound applications | IR optics | all the above | all the above |
| 20 | Pick one of the requirements for the scintillators | slow scintillation decay | fast scintillation rise | low light yield | slow scintillation rise | fast scintillation rise |
| 21 | Attractive oxide scintillation crystals are | BGO | KCaF3 | NaI:TI | KDP | BGO |
| 22 | Nonlinear optical crystals are very important for | Jewelry | watch | scintillation | laser frequency conversion | laser frequency conversion |
| 23 | KDP is a suitable crystal for Of huge laser systems for fusion experiment | high harmonic generation | low harmonic generation | Acousto optic crystals | semiconductors | high harmonic generation |
| 24 | KTP is | hygroscopic | non hygroscopic | mechanically hard | both b and c | both b and c |
| 25 | Borate crystals are used in | IR optics | NLO | coherent UV sources | all the above | coherent UV sources |
| 26 | The success of the borate crystals can be attributed to the unique structural characteristics ofGroups | boron-oxygen | boron | oxygen | halide | boron-oxygen |
| 27 | Pick one of the following which is promising for UV generation | beta-barium borate | LiF:Eu | KDP | КТР | beta-barium borate |
| 28 | is more suitable for 4HG and 5HG of Nd:YAG laser | yittrium calcium borate | cesium lithium borate | KDP | КТР | cesium lithium borate |
| 29 | are suitable for 2nd and 3rd harmonic generation of Nd:YAG laser radiation | YCa4O(BO3)3 | GdxY1- xCa4O(BO3)3 | cesium lithium borate | both a and b | both a and b |
| 30 | Choose organic NLO materials from the following | L-Arginine phophate | urea | 4-dimethylamino-N- methyl-4-stilbazolium tosylate | all the above | all the above |
| 31 | crystals due to the piezoelectric property and ability to rotate a plane of polarization and high transparency are used in electronics and optical instrumentation | Quartz | KDP | urea | all the above | Quartz |
| 32 | KTP is a useful nonlinear optical crystal to get efficient green light by the frequency doubling of | Nd:YAG laser | He-Ne laser | Ti:Sapphire laser | all the above | Nd:YAG |
| 33 | Crystal growth is aprocess | non-equilibrium | equilibrium | stable | none of these | non-equilibrium |
| 34 | Many Properties of Are obscured by the effects of grain boundaries | physical and liquids | magnetic and solid | physical and solids | physical and gas | physical and solids |

| | | | | - | | _ |
|----------------------|---|--|--|---|--|--|
| 35 | Growth methods are classified intotypes | three | two | four | none of these | three |
| 36 | The third step in the growth of crystals is | successive growth of crystals to yield distinct faces | achievement of supersaturation | formation of crystal nuclei | supercooling | successive growth of crystals to yield distinct faces |
| 27 | The cluster consisting of few stores or molecules is called | nuclei | seed | crustal | polycrystal | nuclei |
| 57 | The cluster consisting of rew atoms of molecules is caned | nuciei | seeu | crystai | porycrystar | nuclei |
| 38 | The kinetics of phase change take place in Different stages | five | three | four | two | four |
| 39 | The development of the supersaturated state may arise due to change in | temperature | pressure | chemical or physical condition | all the above | all the above |
| 40 | Once embryos achieve the Size there is a high probability that they will grow | nano | micro | bulk | critical | critical |
| 41 | critical nuclei have more than bulk phases of the same mass | pressure | concentration | energy | energy and concentration | energy |
| 42 | William Thomson showed that the vapour pressure of a liquid in the form of a drop or radius r is greater than | vapour pressure over a plane liquid surface | saturation vapour pressure over a plane liquid surface | vapour pressure inside a liquid | none of these | saturation vapour pressure over a plane liquid surface |
| 43 | J.J.Thomson modified Thomsons equation to show that the melting point of a small crystal isof a crystal | less than that | more than that | equal | a or b | less than that |
| 44 | Since the two phases(i.e. crystal and supercooled melt) are in equilibrium, their chemical potentials must be | equal | not equal | high | low | equal |
| | A | | | | | |
| 45 | The solubility of a crystal increases as the size of the crystal | decreases | increases | not changes | a or b | decreases |
| 46 | If C_i and C are the concentrations of solution with a small crystal of radius r and wit a large crystal of radius r = respectively then | KTln(C ₁ /Ci)=2 V _{II} /r | KTln(CI /C1)=2 V _{II} / | KTln(C ₁ /CI)=2 V _{II} | $KTln(C_1/CI) = V_{II}$ | KTln(C ₁ /Ci)=2 V _{II} /r |
| 47 | Any crystal smaller than r will dissolve in the supersaturated solution since its solubility is than Q | lower | greater | equal | a or b | greater |
| 48 | The size corresponding to the maximum free change is called | nucleus | small nucleus | critical nucleus | large nucleus | critical nucleus |
| 49 | The surface energy term with \vec{r} and the volume energy term With \tilde{r} | decreases, increases | increases,decreases | constant, increases | decreases,constant | increases,decreases |
| 50 | The free energy change associated with the critical nucleus is | G*=16 ³ /3 G _v ² | $G^*=16$ ² /3 G_v^2 | $G^{*}=$ ³ /3 G_{v}^{2} | $G^*=16^{-3/}G_v^2$ | $G^*=16^{-3/3}$ G_v^{-2} |
| 51 | The expression for G_{i} for the nucleation from solution is given by | G _v =-(kT/v)lnS | G _v =(kT/v)lnS | G _v =-(kT)lnS | G _v =-lnS | G _v =-(kT/v)lnS |
| 52 | The number of critical nuclei formed per unit time per unit volume is known as the | rate of formation | nucleation | rate of nucleation | seed growth | rate of nucleation |
| | The puplication on the surface of a puplicant is known as | hataraganaous nualti | homogeneous | nomo or heterogeneous | none of these | heterogeneous |
| 53 | The nucleation on the sufface of a nucleant is known as | neterogeneous nucleation | C(i)=C(1)exp[G(i*)/ | C(i*)=exp[G(i*)/kT | C(i*)=C(1)exp[- | C(i*)=C(1)exp[- |
| 54 | The concentration of the critical nuclei is given as | $C(i^*)=C(1)exp[-G(i^*)]$ | kT] | 1 | G(i*)/kT] | G(i*)/kT] |
| 55 | | | | | | |
| | _{sv} is the interfacial energies between | liquid-substrate | vapour-substrate | vapour-solid | substrate-vapour | vapour-substrate |
| 56 | w is the interfacial energies between The frequency of the continuous X-rays emitted from the tube | liquid-substrate do not depend on the target | vapour-substrate depends only on the potential difference | vapour-solid directly proportional to the potential difference | substrate-vapour all of the above | vapour-substrate all of the above |
| 56 | w is the interfacial energies between The frequency of the continuous X-rays emitted from the tube The frequencies of the characteristic X-rays emitted from an X-ray tube | liquid-substrate do not depend on the target do not depend on potential difference | vapour-substrate depends only on the potential difference depends only on the atomic number of the target | vapour-solid directly proportional to the potential difference can be increased by increasing atomic number of target | substrate-vapour all of the above all of the above | vapour-substrate all of the above all of the above |
| 56 57 58 | ₂₀ is the interfacial energies between The frequency of the continuous X-rays emitted from the tube The frequencies of the characteristic X-rays emitted from an X-ray tube The penetrating power of X-rays emitted from an X-ray tube depends on | liquid-substrate do not depend on the target do not depend on potential difference filament current | vapour-substrate depends only on the potential difference depends only on the atomic number of the target applied potential difference | vapour-solid directly proportional to the potential difference can be increased by increasing atomic number of target atomic number of target | substrate-vapour all of the above all of the above both b and c | vapour-substrate all of the above all of the above both b and c |
| 56 57 58 59 | " is the interfacial energies between The frequency of the continuous X-rays emitted from the tube The frequencies of the characteristic X-rays emitted from an X-ray tube The penetrating power of X-rays emitted from an X-ray tube depends on which of the following can be deflected by an electric field | liquid-substrate do not depend on the target do not depend on potential difference filament current X-rays | vapour-substrate depends only on the potential difference depends only on the atomic number of the target applied potential difference UV-rays | vapour-solid directly proportional to the potential difference can be increased by increasing atomic number of target atomic number of target cathode rays | substrate-vapour all of the above all of the above both b and c microwaves | vapour-substrate all of the above all of the above both b and c cathode rays |
| 56 57 58 59 | w is the interfacial energies between The frequency of the continuous X-rays emitted from the tube The frequencies of the characteristic X-rays emitted from an X-ray tube The penetrating power of X-rays emitted from an X-ray tube depends on which of the following can be deflected by an electric field | liquid-substrate do not depend on the target do not depend on potential difference filament current X-rays | vapour-substrate depends only on the potential difference depends only on the atomic number of the target applied potential difference UV-rays | vapour-solid directly proportional to the potential difference can be increased by increasing atomic number of target atomic number of target cathode rays | substrate-vapour all of the above all of the above both b and c microwaves | vapour-substrate all of the above all of the above both b and c cathode rays |

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SYLLABUS

Electron Microscopy

Electron diffraction technique – High energy electron diffraction – Low energy electron diffraction - Electron microscopy – Scanning electron microscopy (SEM) - FESEM - EDAX - TEM - HRTEM: working principle and Instrumentation - sample preparation - Advantages/disadvantages.

Scanning Probe Microscopy

Scanning probe microscopy - STM - AFM - EPMA - working principle and Instrumentation - Advantages/disadvantages.

LOW-ENERGY ELECTRON DIFFRACTION

Low-energy electron diffraction (LEED) is a powerful method for determining the geometric structure of solid surfaces. The phenomenon was first observed by Davisson and Germer in 1927 and provided the earliest direct experimental proof of the wavelike properties of electrons.

LEED has since evolved into one of the most powerful and widespread tools of the surface scientist. It is similar to X-ray diffraction (XRD) in the type of information that it provides,

and XRD will be frequently referred to for analogy throughout this unit. The most obvious difference is that a beam of electrons, rather than x rays, is used. Since electrons

have a mean free path (or attenuation length) measured in angstroms (A°) as opposed to microns (m) for x rays, LEED is particularly sensitive to surface geometry.

LEED is a structural technique that can provide essentially two levels of information. In both cases, it is most commonly used to determine the structure of a solid surface when the bulk structure of the material is alreadyknown by other means (e.g., XRD). It is possible, and indeed straightforwardly simple, to use LEED to determine both the absolute dimensions of the surface unitcell and the unit cell symmetry. One can easily deduce that, for example, a surface has a twodimensional (2D)unit cell twice as large as that of the bulk; this often allows reasonable guess at the true structure. Sometimes it issufficient to know that a particular phase is present without knowing the details of the structure.

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For example, the (2_4) reconstruction of GaAs (nomenclature explained below) has been found to be optimal for device growthFarrow, 1995). Determination of the exact atomic positionsis more difficult, but quantitative experiments canelucidate this second level of information. Sophisticated calculations, generally run on a workstation, can provide atomic coordinates with a typical precision of _0.05 Awhich is generally more than adequate to determine the adsorption site of a molecule or the atomic positions in areconstructed surface.

Many different surfaces can be examined by LEED, andtheir terminology must be understood. We assume familiarity with the basic crystal structures [face-centered cubic(fcc), zincblende, etc.]. A particular crystal face is specifiedby its Miller indices. These are of the form (hkl), and theideal bulk termination can be determined in the followingway: place one corner of the unit cell at the origin of a Cartesiancoordinate system and construct a plane that intersects at the reciprocal of the respective Millerindex (see SYMMETRY IN CRYSTALLOGRAPHY). In the generalcase, this plane would pass through the points (1/h, 0, 0),(0, 1/k, 0), and (0, 0, 1/l). A Miller index of zero indicates that the plane is parallel to that axis. Further examplescan be found in any solid-state textbook (e.g., Ashcroftand Mermin, 1976; Kittel, 1986). It is conceptually usefulto regard a surface as being composed of stacked planes ofatoms, each parallel to the topmost surface plane (see SYMMETRYIN CRYSTALLOGRAPHY).

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The structure of an actual surface is usually very different from an ideal termination of the bulk. The low-Miller- index surfaces—(100), (110), (111)—are relatively flat, buthigher-index surfaces generally have a large number of steps and often kinks as well. Adatoms and adsorbed molecules also appear in many systems. Figure 1 illustrates some of these features. LEED is sensitive to any suchstructures that recur at regular intervals, that is, withmeasurable periodicity.LEED is a mature area of surface science and it is usefulto consider the state of the art in this field. A great dealof work has been done on clean metal and semiconductorsurfaces, and much of this is considered "done"; currentwork in this area consists mostly of refinements. Mostlow-index clean metal surfaces are found to merely relaxthe spacing between the topmost layers. Higher-indexmetal surfaces tend to be less closely packed and moreprone to reconstruction. Clean semiconductor surfacesalmost always reconstruct. Adsorbed atoms and moleculesare sometimes found to lift reconstructions, and they often modify the dimensions and orientation of the surface unitcell themselves. Several atoms and simple molecules (H, O,S, CO, C2H) have been investigated on low-Miller-indexsurfaces of semiconductors (Si, GaAs) and most transitionmetals. An important direction of current research is thegrowth of ordered crystalline epitaxial films for a variety of purposes. They serve as models for catalytic systems(Boffa et al., 1995), model magnetic structures, or avenues for studying otherwise intractable systems (Roberts et al;

1998), to name a few examples. Other recent advancesseek to extend the power of the technique to systems4with disorder in geometry (Do"1 et al., 1997; Starke et al., 1996) or composition authier et al., 1985; Crampin et al., 1991). Because of the time-consuming trial-and-error computationsinvolved in a structural determination, there is also interest in developing a LEED-ased holographictechnique for rapid adsorption site determination (Saldin, 1997).

SCANNING ELECTRON MICROSCOPY (SEM)

In scanning electron microscope, electron beam scans across the sample surface and collects scattered electrons for imaging. Because the image is formed using backscattered signals instead of forward-transmitted signals, the electron beam energy does not need to be high (~40

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keV) and the sample also does not require electronic transparency; it has only a conductivity requirement to prevent charging.

Scanning electron microscopy (SEM) is one of the most widely used techniques for the characterization. SEM is providing not only topographical information like optical microscopes but also information of chemical composition near the surface. SEM creates magnified images by using electrons instead of light. It gives detailed 3-D images at higher magnification than an optical microscope. The various processes of interaction of primary electron beam with specimen in an electron microscope are shown in Figure 2.7. In the scanning electron microscope, back scattered electrons and secondary electrons are captured by a detector to form the image.



Interaction between primary electron beam and the sample in anelectron microscope

Secondary electronsarise due to inelastic collision between primary electrons (the beam) and loosely bound electrons of the conduction band or tightly bound valence electrons. The energy transferred is sufficient to overcome the work function which binds them to the solid and hence they are ejected. The ejected electrons have 5-10 eV energy and they are detected by scintillator / photomultiplier tube. Back scattered electrons arise due to elastic collisions between the incoming electron and the nucleus of the target atom (i.e. Rutherford scattering). Higher the

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atomic number, higher is the number of back scattered electrons. They are detected by semiconductor detectors.

Most of the samples used in the study are low conducting specimens and hence the samples were coated with gold by sputtering method. The morphology of the materials were recorded using a scanning electron microscope (HITACHI COM-S-4200) operated at an accelerating voltage of 16 kV. The samples were suspended in methanol and the specimen stub was dipped into the liquid and removed. The sample powder deposited onto the surface of the stub evenly when methanol was evaporated. This specimen was coated with gold for two minutes. The beam is scanned over the specimen surface in synchronism with the beam of a cathode ray tube (CRT) display screen. Materials can be studied properly only when they are electrically conducting, otherwise they give rise to charging phenomena resulting blurred images.

Energy Dispersive X-ray Spectroscopy

Energy dispersive X-ray (EDX) spectroscopy is an analytical technique used for the elemental analysis of the sample. A high energy beam of electrons is focused on the sample. The incident beam of electrons excites an electron in the inner shell of an atom thus creating an electron-hole. An electron from an outer higher energy shell then fills the hole. The difference in energy between higher energy and lower energy shell may be released in the form of an X-ray. The number and energy of X-rays emitted from a specimen can be measured by an energy dispersive spectrometer. As the energy of X-rays is characteristic of the difference in energy between the two shells and atomic structure of the element from which they are emitted, this allows the elemental identification and hence the composition of the specimen to be measured. The detector converts X-ray energy into voltage signals and the information is sent to the pulse processor, which measures the signals and passes them onto an analyzer for data display and analysis.

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A silicon semiconductor is used with high impedance zone which is radiation sensitive. Each absorbed X-ray quantum is converted into a cloud which consists of pairs of charge carriers i.e. electrons and holes. Hence, the energy of the X-ray photons can be calculated. Due to high voltage, which is applied between both ends of the sensitive area of the detector, the charge carriers are moved and collected at the ends of the sensitive zone. With a charge sensitive preamplifier, the charge quantity is finally converted into a voltage pulse. The pulse is amplified and shaped in a spectroscopic amplifier.

TRANSMISSION ELECTRON MICROSCOPY (TEM)

Transmission Electron Microscopy (TEM) has, in three decades time, become amainstay in the repertoire of characterization techniques for materials scientists.TEM's strong cards are its high lateral spatial resolution (better than0.2 nrn"point-to-point" on some instruments) and its capability to provide both imageand diffraction information from a single sample. In addition, the highly energetic beam of electrons used in TEM interacts with sample matter to produce character-

istic radiation and particles; these signals often are measured to provide materialscharacterization using EDS, EELS, EXELFS, backscattered and secondary electronimaging, to name a few possible techniques.

Basic Principles

In TEM, a focused electron beam is incident on a thin (less than 200 nm) samplr. The signal in TEM is obtained from both undeflected and deflected electrons that penetrate the sample thickness.

A series of magnetic lenses at and below the sampleposition are responsible for delivering the signal to a detector, usually a fluorescentscreen, a film plate, **or** a video camera. Accompanying this signal transmission is amagnification of the spatial information in the signal by as little as 50 times to as much as a factor of 10'. This remarkable magnification range is facilitated by thesmall wavelengthof the incident electrons, and is the key to the unique capabilities associated withTEM analysis. A schematic of a TEM instrument, showing thelocation of a thin sample and

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the principal lenses within a TEM column, is illustrated in Figure 1a.Figure Ib shows a schematic for the ray paths of both unscattered and scattered electrons beneath the sample.



Schematic diagram a TEM instrument

Resolution

The high lateral spatial resolution in a TEM instrument is a consequence of several features of the technique. First, in the crudest sense, TEM has high spatial resolution because it uses a highly focused electron beam**as** a probe. This probe is focused at the specimen to a small spot, often ap or less in diameter. More importantly, the probe's source is an electrongun designed to emit a

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highly coherent beam of monoenergetic electrons of exceedingly small wavelength. The wavelength, **h**, of **IO0**keV electrons is only 0.0037 nm, much smaller than that of light, X rays, or neutrons used in other analytical techniques. Having such small wavelengths, sinceelectrons in a TEM probe are in phaseas they enter the specimen, their phase relationships upon exiting are correlated with spatial associations between scatteringcenters (atoms) within the material. Finally, high lateral spatial resolution is maintained via the useof extremely thin samples. In most TEM experiments, samples thinned usually to less than200 nm. For most materials this insures relatively **few** scattering events as each electron traverses the sample. Not only does this limitspreading of the probe, but muchof the coherency of the incident source is **also** retained.

ATOMIC FORCE MICROSCOPY (AFM)

The ability of AFM to create three-dimensional micrographs with resolution down to nanometer scale renders it as an essential tool for imaging the surfaces. It provides nanoscale to atomic-scale microscopy by measuring the tip-surface interactions from forces and translating these to a sensor to provide information on the topographic features of the surface. The forces involved include van der Waals, electrostatic, frictional and magnetic. Typically, forces measured are in the range 10^{-3} to 10^{-6} N. The small probe-sample separation makes it possible to take measurements over a small area. AFM operates by measuring attractive or repulsive forces between a tip and the sample.

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Schematic diagram of Atomic Force Microscope (AFM)

In a typical instrument (Figure 2.8), an atomically sharp tip is made to scan over a surface with feedback mechanisms that enable the piezoelectric scanners to maintain the tip at a constant force (to obtain height information), or height (to obtain force information) above the sample surface. Tips are typically made from Si_3N_4 or Si, and extended down from the end of a cantilever. The nanoscope AFM head employs an optical detection system in which the tip is attached to the underside of a reflective cantilever. A diode laser is focused onto the back of a reflective cantilever. As the tip scans the surface of the sample, moving up and down with the contour of the surface, the laser beam is deflected off the attached cantilever into a dual element photodiode. The photodetector measures the difference in light intensities between the upper and lower photodetectors, and then converts to voltage. Feedback from the photodiode difference signal, through software control from the computer, enables the tip to maintain either a constant

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force or constant height above the sample. In the constant force mode the piezoelectric transducer monitors real time height deviation. In the constant height mode the deflection force on the sample is recorded. The latter mode of operation requires calibration parameters of the scanning tip to be inserted in the sensitivity of the AFM head during force calibration of the microscope. The primary purpose of this instrument is to quantitatively measure surface roughness with a nominal 5 nm lateral and 0.01 nm vertical resolutions on all types of samples. Three dimensional topographical maps of the surface are then constructed by plotting the local sample height versus horizontal probe tip position. Image contrast can be achieved in many ways. The three main classes of interactions are contact mode, tapping mode and non-contact mode.

Contact mode is the most common method of operation of the AFM. As thename suggests, the tip and sample remain in close contact as the scanning proceeds. As the tip is moved for raster-scan across the surface, it gets deflected as it moves over the surface corrugation. In constant force mode, the tip is constantly adjusted to maintain a constant deflection, and therefore constant height above the surface. It is this adjustment that is displaced as data. However, the ability to track the surface in this manner is limited by the feedback circuit. Sometimes the tip is allowed to scan without this adjustment, and one measures only the deflection. This is useful for small, high speed atomic resolution scans, and is known as variable-deflection mode. Because the tip is in hard contact with the surface, the stiffness of the lever needs to be less that the effective spring constant holding atoms together, which is in the order of 1-10 nN / nm. Most contact mode levers have a spring constant of < 1 N/m.

Tapping mode is also frequently used in AFM. When operated in air or other gases, the cantilever is oscillated at its resonant frequency (often hundreds of kilohertz) and positioned above the surface so that it only taps the surface for a very small fraction of its oscillation period. This is still contact with the sample, but the very short time over which this contact occurs means that lateral forces are dramatically reduced as the tip scans over the surface. When imaging

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poorly immobilized or soft samples, tapping mode may be a far better choice than contact mode for imaging. The advantage of tapping the surface is improved lateral resolution on soft samples. In a non-contact mode, a stiff cantilever is oscillated in the attractive regime, meaning that the tip is quite close to the sample, but not touching it. The forces between the tip and sample are quite low, on the order of pico Newton (10^{-12} N) . The detection scheme is based on measuring changes to the resonant frequency or amplitude of the cantilever. This is a very difficult mode to operate in ambient conditions with the AFM.



KARPAGAM ACADEMY OF HIGHER EDUCATION, COIMBATORE-21 DEPARTMENT OF PHYSICS CLASS : I M.SC PHYSICS BATCH: 2019-2021 PART A : MULTIPLE CHOICE QUESTIONS (ONLINE EXAMINATIONS) SUBJECT : MATERIAL CHARACTERIZATION SUBJECT CODE : 19PHP105A UNIT-IV

| SL.N O. | QUESTIONS | opt 1 | opt2 | opt3 | opt4 | Answer |
|------------|---|--|---|---|---|---------------------------------------|
| | | | | | | |
| 1 | In Bridgman technique, the rates of movement are mostly in the range | 1-30mm/hr | 0.1-200mm/hr suitable for low | 200-300mm/hr adhesion of the solid | above 300mm/hr | 1-30mm/hr |
| 2 | The advantages of the Bridgman technique are | low-cost | melting point materials | materials to the ampoule wall | both a and b | both a and b |
| | In Bridgman technique, the melt temperature | | | | | |
| 3 | interface | decreases | increases | increases or decreases adhesion of the solid | constant | increases |
| 4 | A disadvantage in Bridgman technique is | nucleate dislocations in the material | low-cost | materials to thee ampoule wall | both a and c | both a and c |
| | | | | | single or | |
| 5 | The container wall acts as a preferential, spurious nucleation site, resulting in | polycrystalline growth | single crystal growth | amorphous growth | polycrystal growth | polycrystalline growth |
| 6 | The requirement that the freezing isotherm should move systematically through the molten charge can be satisfied by moving | the crucible | the furnace | changing the furnace temperature | any one of the above | any one of the above |
| 7 | The Bridgman technique cannot be used for materials which | decompose before melting | undergo solid state phase transformation | decompose after melting | both a and b | both a and b |
| 8 | Bridgman technique is best suited for | high melting point materials | high vapour pressure materials | low melting point materials | both high and low melting point materials | low melting point materials |
| 9 | The characteristic of a container | not react with the melt | smaller coefficient of thermal expansion than the crystal | smaller thermal conductivity than the crystal | all the above | all the above |
| 10 | The materials with covalently bound crystals are often compatible with containers made of | alassas | anld | oilsean | alotinum | alaasaa |
| 10 | | glasses | gold | silver | platinum | glasses |
| 11 | Graphite crucible is a useful container material for | ionic materials | some covalently bound materials | some metallic materials | all the above | all the above |
| 12 | which one of the following comes under crystal pulling technique | Bridgman | zone refining | skull melting | Czochralski | Czochralski |
| 13 | Which method can produce crystals, weighing from several grams to many kilograms | crystal pulling | zone melting | slow evaporation | chemical vapour deposition | crystal pulling |
| 14 | crystal growth is a complex process, which usually takes place by a | phase change | solid phase | liquid phase | vapour phase | phase change |
| 15 | Irregular convective flow in crystal pulling can be produced due to | higher temperature gradients | concentration gradients | high vapour pressure | both a and b | higher temperature gradients |
| 16 | The seed is withdrawn from the melt at such a rate that the crystal Is gradually increased to its desired value | length | weight | diameter | both length and diameter | diameter |
| 17 | Growth at constant diameter is maintained by adjustments of the | rate of pulling | rate of liquid level drop | heat fluxes into and out of the system | all the above | all the above |
| 18 | The seed can be pulled from the melt at a rate which matches the rate at which liquid crystallizes on the | seed | container | crystal | puller | seed |
| | | | | | | |
| 19 | With angle of contact <0 the crystal will | narrow | parallel | grow out | either b or c | narrow |
| 20 | With angle of contact =0 the crystal will | narrow | parallel | grow out | either a or c | parallel |
| 21 | With angle of contact >0 the crystal will | parallel | narrow | grow out | either a or b | grow out |
| 22 | The terminology necking means reducing the dia gradually to grow as a In diameter | 2-3mm | 10mm | 40mm | 0.5mm | 2-3mm |
| 23 | The pull rod provides rotation and facility to lift and lower the crystal | Bridgman | czochralski | zone melting | both a and b | czochralski |
| | The average growth rate can be obtained by equating the mass of the | | | | | |
| 24 | formed | solid the thermal | liquid | liquid or solid | solid and liquid | solid |
| 25 | The important parameter governing the onset and nature of convection pattern in melts | characteristics of the material | the aspect ratio of the container | the imposed temperature gradients | all the above | all the above |
| 26 | The Prandtl number is dependent only on the | contained geometry | applied temperature gradient | characteristics of the liquid | the aspect ratio of the container | the aspect ratio of the container |
| 27 | In high Prandtl number systems such as oxides, strong fluid flow is largely | distributed throughout the bulk | confined to boundary layer regions | confined to central regions | all the above | confined to boundary layer regions |
| 28 | In low Prandtl number materials such as metals and semiconductors the streamlines are mor generally | confined to boundary layer regions | distributed throughout the bulk | confined to central regions | none of the above | distributed throughout the bulk |
| 29 | In horizontal systems the temperature gradient is characterised by the | Rayleigh number | Prandtl number | Grashof number | none of the above | Ravleigh number |

| 30 | In vertically driven convection systems, the relevant dimensionless number is the | Rayleigh number | Prandtl number | Grashof number | none of the above | Grashof number |
|----|--|--|---|---|--|---|
| 31 | The Prandtl number is defined as the ratio | / | QI/ | VI/ | /D | / |
| 32 | Ravleigh number Ra is defined by | gd ⁴ T _v / | gI ⁴ T _H / ² | 1 | VI/ | gd ⁴ T./ |
| | | | | | | |
| 33 | Grashor number Gr is defined by As the Rayleigh(or Grashof number) increases with increase in, the fluid flow | / | gd I√ | gi i _H / - | /D | g1 1 _H / - |
| 34 | begins to move by convective flow | Т | | gI ⁴ T _H / ² | both a and b | Т |
| 35 | Typical Bridgman growth ampoules haveaspect ratio | large d/a | small d/a | large or small d/a | none of the above | large d/a |
| 36 | Forced convection flow patterns in Czochralski melts are due to various combinations of | crystal rotation | seed rotation | crucible rotation | seed rotation and crucible rotation | seed rotation and crucible rotation |
| 37 | The shape of the growth surface is dependent upon its | crystallographic orientation | local temperature gradient | both a and b | none of the above | both a and b |
| 38 | The simplest and the best case from the point of view of good quality growth is the | concave interface | convex interface | planar interface | both a and c | planar interface |
| 39 | Which model calculates, for a given instant of growth, the global heat transfer, the shape of the solid-liquid interface, natural and forced convection | Time dependent model with imposed crystal shape | Time dependent model with unknown crystal shape | Quasi-steady state model | both a and c | Quasi-steady state model |
| 40 | The model calculates, as functions of time | Time dependent model with imposed crystal shape | Time dependent model with unknown crystal shape | Quasi-steady state model | both a and b | both a and b |
| 41 | In Model, the variation of the tri-junction radius as a function of time is calculated | Quasi-steady state model | Time dependent model with imposed crystal shape | Time dependent model with unknown crystal shape | both a and b | Time dependent model with unknown crystal shape |
| 42 | single crystals are used in the laser systems | Nd:YAG | Ti:Al ₂ O ₃ | YAP | all the above | all the above |
| 43 | The Czochralski system for the growth of oxides incorporates an induction heating system operating at | 455kHz | 455Hz | 1kHz | 100Hz | 455kHz |
| 44 | and, are two ferroelectric oxides | Lithium niobate and Ti:Al ₂ O ₃ | Lithium niobate and Lithium tantalate | BGO and Lithium niobate | Aluminium and iron oxide | Lithium niobate and Lithium tantalate |
| 45 | Lithium niobate and Lithium tantalate have been widely investigated due to an | piezoelectricity | pyroelectricity | electro-optic property | all the above | all the above |
| 46 | Equilibrium referes to a growth speed of | 0mm/min | 10mm/min | 2mm/min | none of these | 0mm/min |
| 47 | Lithium niobate and Lithium tantalate crystals are | biaxial crystals | uniaxial crystals | symmetric crystals | none of the above | uniaxial crystals |
| 48 | Lithium tantalate and Lithium niobate crystals are used for | laser systems | surface acoustic wave | magnetic bubble devices | optical components | surface acoustic wave |
| 49 | A good ratio between the crucible diameter and the crystal diameter is | 1 | 2 | 10 | 3 | 2 |
| 50 | The size of the crystal is limited by the | size of the crucible | thermal gradients in the crystallization area | pull rate | both a and b | size of the crucible |
| 51 | crystal has been studied extensively for ecourtic wave devices | Lithium niobate | NdYAG | YAP | langasite | langasite |
| 52 | Isomorphic crystals have | zero temperature coefficient | large electromechanical coupling factor | congruent melting point | all the above | all the above |
| 53 | Dissociable compounds can be grown by | CZ method | sealed tube methods | vertical pulling methods | both b and c | both b and c |
| 54 | Pick out the suitable property of encapsulant | melt after the compound melts | more dense than the melt of the compound | transparent | not wet the crucible | transparent |
| 55 | Boron tri oxide has proved to be one of the best encapsulants fortyp | metals | semiconductor | insulator | both a and b | both a and b |
| | The technological importance of GaAs and InP comes about because of their use in | alestenia destant | abotonia d | monutin d | hoth o or ³ | hoth a and t |
| 56 | | electronic devices | photome devices | magneuc devices | bour a and b | |
| 57 | 11ne paper published by Pfann(1952) was related to Technique | Zone melting | Zone refining | float zone | all the above | all the above |
| 58 | In zone refining, if freezing is rapid enough, k may actually | approach unity | less than unity | more than unity | 0 to 1 | approach unity |
| 59 | process is used for the growth of zirconium oxide | Zone melting | Skull melting | cz | both a abd c | Skull melting |
| 60 | is a process, which dynamically controls both the heat input and the heat extraction in a crystal growth furnace | Skull melting | Zone melting | Bridgman | Heat exchanger | Heat exchanger |

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UNIT V (Electrical and Optical Methods of Characterization)

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SYLLABUS

Electrical and Optical Methods of Characterisation

Two probe and four probe methods - van der Pauw method - Hall probe and measurement - scattering mechanism - C-V, I-V characteristics - Schottky barrier capacitance - impurity concentration - electrochemical C-V profiling - limitations - Introduction to Photoluminescence and Electroluminescence - Applications. Dielectrics - working principle and Instrumentation - Applications.

ELECTRICAL PROPERTIES

Electrical properties such as resistivity, carrier concentration and Hall mobility were estimated from the mobility were estimated from the four probe method and Hall measurements in van der Pauw configuration at room temperature.

FOUR-POINT COLLINEAR PROBE METHOD

The most common way of measuring the resistivity of a semiconductor material is using a four-point collinear probe (Hartnagell et al 1995). This technique involves bringing four equally spaced probes in contact with a material of unknown resistance. The probe array is placed in the centre of the material, as shown in Figure 2.9.



Schematic diagram of four point collinear setup

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The two outer probes are used for applying the current source and the two inner probes are used for measuring the resulting voltage difference across the surface of the sample. The sheet resistance is calculated as follows,

$$R_{sh} = \frac{\pi}{In_2} \times \frac{V}{I} \times K \tag{2.2}$$

where, R_{sh} is the sheet resistance (Ω /cm), V is the measured voltage (volts), I is the source current (amperes), K is a correction factor based on the ratio of the probe to wafer diameter and on the ratio of wafer thickness to probe separation.

Van der Pauw configuration

The van der Pauw method involves applying a current and measuring the voltage using four small contacts on the circumference of a flat, arbitrarily shaped sample of uniform thickness (van der Pauw 1983). This method is particularly useful for measuring very small samples because geometric spacing of the contacts is unimportant. Effect due to the size of a sample, which is the approximate probe spacing, is irrelevant. Using this method, the resistivity can be derived from a total of eight measurements that are made around the periphery of the sample with the configurations shown in Figure 2.10.
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Fig. 2.10 Symmetric contacts in van der Pauw configuration

The samples used were $1 \times 1 \text{ cm}^2$ in size and the electrical contact was established using indium metal or silver paste. The gold spring probes were placed at the corners of the sample symmetrically as shown in Figure 2.10. A constant current of 1mA was supplied to the sample. The sample was subjected to uniform magnetic field of 0.57 Tesla. A known current was passed through the electrodes of the samples and voltage across the other two electrodes was measured. The polarity was reversed for each measurement and the voltage was measured. Once all the voltage measurements are taken, two values of resistivity, ρ_A and ρ_B are derived as follows,

$$\rho_A = \frac{\pi}{In2} f_A t_s \frac{(V_1 - V_2 + V_3 - V_4)}{4I}$$
(2.3)

$$\rho_{B} = \frac{\pi}{In2} f_{B} t_{s} \frac{(V_{5} - V_{6} + V_{7} - V_{8})}{4I}$$
(2.4)

where, ρ_A and ρ_B are resistivities in $\Omega\text{-}cm,\,t_s$ is the sample thickness in nm, $V_1\!\!-\!\!V_8$

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represents the voltages measured by the voltmeter, I is the current passed through the sample in amperes, f_A and f_B are geometrical factors based on sample symmetry. Once ρ_A and ρ_B are known, the average resistivity (ρ_{AVG}) can be determined as follows:

$$\rho_{AVG} = \frac{\rho_A + \rho_B}{2} \tag{2.5}$$

The Hall signal is measured between two ends while the current is passing through the other two ends. Hall mobility (μ) is given by the ratio

$$\mu = \Delta R \times \frac{10^8}{BR_{sh}} \tag{2.6}$$

where, ΔR is the change in resistance due to magnetic field (B) which was applied to measure Hall voltages and R_{sh} is sheet resistance of the material. Carrier concentration is determined using the relation

$$\rho = \frac{1}{en\mu} \tag{2.7}$$

The Hall coefficient R_H is given by,

$$R_{\rm H} = \rho \times \mu \tag{2.8}$$

The type of carriers can be understood from the sign of the R_H and thus the type of conductivity can be identified. The negative value of R_H corresponds to the flow of electrons (n-type) and positive value corresponds to the flow of holes (p- type) (Schroder Dieter 1998).

THICKNESS MEASUREMENT

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Thickness plays an important role in the properties of thin films. The results obtained can be reproduced only when the film thickness and the deposition parameters are kept constant. In the present work, the film thickness was determined by employing weight gain method, optical interference technique and stylus profilometry.

The initial estimation of thickness was done using the conventional gravimetric method. In this method, the weight of the substrates before (W_1) and after deposition (W_2) was used to find out the weight of the film deposit (W_3) by the given relation $W_3 = (W_2 - W_1)$. Thus knowing the weight of the material coated on the area and the film density (ρ_d) , the thickness (t) of the film can be obtained by the relation (Elangovan and Ramamurthi 2003).

$$t = \frac{W_3}{A\rho_d} \tag{2.9}$$

The thickness of the layers deposited onto the glass plates was calculated from the optical interference maxima (λ_1 , λ_2) appearing in the transmission spectra of the films and the refractive index (n) of the film material according to the formula (Swanepol 1983)

$$t = \frac{\lambda_1 \lambda_2}{2n(\lambda_1 - \lambda_2)} \tag{2.10}$$

The optically and gravimetrically measured thickness was consequently verified by the stylus profilometry technique. The Hall effect measurement setup used in this work is shown in the Figure 2.11.

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Hall effect measurement system used for present work

PHOTOLUMINESCENCE

Luminescence refers to the emission of light by a material through any processotherthan blackbody radiation. The term Photoluminescence (PL) narrows this down to any emissionof light that results from optical stimulation. Photoluminescence is apparent in everyday life,for example, in the brightness of white paper or shirts (often treated with fluorescent whiteners to make them literally glow) or in the light from the coating on a fluorescent lamp. The detection and analysis**of** this emission is widely used as an analytical tool due to its sensitivity, simplicity, andlow cost. Sensitivity is one of the strengths of the PL technique, allowing very smallquantities (nanograms)

or low concentrations (parts-per-trillion) of material to beanalyzed. Precise quantitative concentration determinations are difficult unless conditions*can* be carefully controlled, and many applications of PL are primarilyqualitative.

PL is often referred to **as** fluorescence spectrometry **or** fluorometry, especially when applied to molecular systems. Uses forPL are found in many fields, including environmental research, pharmaceutical and food analysis, forensics, pesticidestudies, medicine, biochemistry, and semiconductors and materials research. PLcan be used**as** a tool for quantification, particularly for organic materials, whereinthe compoundof interest can be dissolved in an

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appropriate solvent and examinedeither as a liquid in a cuvetteor deposited onto a solid surface like silica gel, alumina,

or filter paper. Qualitative analysis of emission spectra is used to detect the presence of trace contaminants or to monitor the progress of reactions. Molecular applications include thin-layer chromatography (TLC) spot analysis, the detection of aromatic compounds, and studies of protein structure and membranes. Polymers are studied with regard to intramolecular energy transfer processes, conformation, configuration, stabilization, and radiation damage.

Many inorganic solids lend themselves to study by**PLY** to probe their intrinsicproperties and to look at impurities and defects. Such materials include alkali halides, semiconductors, crystalline ceramics, and glasses. In opaque materials PL isparticularly surface sensitive, being restricted by the optical penetration depth and carrier diffusion length to a region of 0.05 to several pm beneath the surface.

Emission spectraof impurity levels are used to monitor dopants in 111-V, 11-VI, and group IV compounds, as well as in dilute magnetic and other chalcogenidesemiconductors.PL efficiency can be used to provide a measure of surfice damagedue to sputtering, polishing, or ion bombardment, and it is strongly affected bystructural imperfections arising during the growth of films like Sicand diamond.Coupled with models of crystalline band structure, PL is a powerful tool for monitoring the dimensions and other properties of semiconductor superlattices and quantum wells (man-made layered structures with angstrom-scale dimensions).The ability to work with low light levels makes it well suited to measurements onthin epitaxial layers.

Basic Principles

In **PLY** a material gains energy by absorbing light at some wavelength by promotingan electron from a low to a higher energy level. This may be described**as** making a transition from the ground state to an excited state of an atomor molecule, or from the valence band to the conduction band of a semiconductor crystal (electron-holepair creation). The system then undergoes a nonradiative internal relaxation involveing interaction with crystallineor molecular

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vibrational and rotational modes, and the excited electron moves to a more stable excited level, suchas the bottom of the

conduction bandor the lowest vibrational molecular state.

If the cross-coupling is strong enough this may include a transition to a lowerelectronic level, such**as an** excited triplet state, a lower energy indirect conduction band,or a localized impurity level. A common occurrence in insulators and semiconductors is the formation of a bound state between an electron and a hole (called an exciton) or involving a defect or impurity (electron bound to acceptor, excitons bound to vacancy, etc.).

After a system-dependent characteristic lifetime in the excited state, which maylast from picoseconds to many seconds, the electronic system will return to theground state. In luminescent materials some orall of the energy released during thisfinal transition is in the form of light, in which case the relaxation is said to be radiative. The wavelength of this emission is longerthan that of the incident light. Thisemittedlight is detected **as** photoluminescence, and the spectral dependence of its analyzed to provide information about the properties of the material.

The time dependence of the emission can also be measured to provide informationabout energy level coupling and lifetimes. In molecular systems, we use different terminologyto distinguish between certain PL processes that tend to be fast (submicrosecond), whose emission we**call** fluorescence, and other, slower ones (*lo4* sto 10 s) which are said to generate phosphorescence. The light involved inPL excitation and emission usually Us in the range 0.6- 6 eV (roughly 200-2000 nm). Many electronic transitions of interest lie in this range, and efficient sources and detectors for these wavelengths are available. Toprobe higher energy transitions, UPS, *XPS*, and Auger techniques become useful.X-ray fluorescence is technically a high-energy form of PL involvingX rays and coreelectrons insteadof visible photons and valence electrons. Although lower energy intraband, vibrational, and molecular rotational processes may participate in PL, they are studied more effectively by Raman scattering IR absorption.

Since the excited electronic distribution approaches thermal equilibrium with the lattice before recombining, only features within an energy range of -kT of the lowest excited level (the

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band edge in semiconductors) are seen in a typical PL *emission* spectrum. It is possible, however, to monitor the intensity of the PL **as** ahnction of the wavelength of the *incident* light. In this way the emission is used **as** a probe of the absorption, showing additional energy levels above the band gap.

DIELECTRIC ANALYSIS

The dielectric property is an important analysis in the field of research for the materials like crystals, alloys, glasses and electrically non-conducting samples. Each materialhas its own electrical characteristics which depend on the insulation or dielectric properties. The dielectric property is also related to the parameters like substrate impedance, loss and resonator frequency. Precise measurements of dielectric behavior for the materials can lead to reliable information to propose applications.

A material which has the capability to store charges or energy when applying an electric field is called dielectric. The complex dielectric constant contains two parts, which are real part and an imaginary part. They represent the capacity of storage and dielectric loss of the sample respectively. The dielectric constant or relative permittivity is defined as, the ratio of the capacitancewith dielectric to the capacitance without dielectric. The value of dielectric constant (ε_r) is given by the formula,

$$\varepsilon_{\rm r} = \frac{Ct}{A\varepsilon_0} \tag{1.5}$$

where, A - cross-sectional area of the area, ε_0 - permittivity of free space, t - crystal thickness and C - capacitance.

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The parallel plate capacitance method is used to analyze the capacitance and dielectric loss with respect to frequency. An alternating voltage is applied across the parallel plates which have a dielectric material in between. The schematic diagram of parallel plate capacitor is shown in Figure.



The schematic diagram of parallel plate capacitor

Dissipation factor or dielectric loss (D) is an energy loss in a dielectric sample due to applied alternating electric field. In other words, while the dipoles are rotating, a kind of internal friction arises to respond to the alternative electric field, which in turn gets converted as heat by the material. From the dielectric loss values, the quality, purity, deterioration or contamination of the material can be identified. The addition of external impurities or moisture can change the values of D. The conductivity of materials also depends on the value of dielectric loss factor.

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There are four types of polarizations responsible for dielectric behavior, which are, space charge or interfacial, orientation, ionic and atomic or electronic polarization. The ionic and electronic polarizations are strongly independent of temperature and orientational and space charge polarizations are temperature dependent. Their response to external frequency depends on their inertia. At lower frequencies, having enough time to accumulate the charges, materials exhibit a higher dielectric constant values than at higher frequencies. The Figure 1.11 represents the variation of frequency versus polarization mechanism.





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Т

Т

| SL.NO. | QUESTIONS | opt 1 | opt2 | opt3 | opt4 | Answer |
|----------------|--|---|--|---|--|---|
| 1 | Who coined the word 'nanotechnology'? | Eric Drexler | Richard Feynmann | Sumio Tijima | Richard Smalley | Eric Drexler |
| | The size of nanonarticles is between nm | 100 to 1000 | 0.1 to 10 | 1 to 100 | 0.01 to 1 | 1 to 100 |
| | The size of nanoparticles is between nn. | 100101000 | 0.110 10 | 110100 | 0.01 10 1 | 10100 |
| 3 | "There is plenty of room at the bottom." This was stated by | Eric Drexler | Richard Feynmann | Harold Croto | Richard Smalley | Richard Feynmann |
| 4 | Who prepared and explained nanotubes for the first time? | Sumio Tijima | Richard Smalley | Eric Drexler | Richard Feynmann | Richard Smalley |
| 5 | Carbon atoms make type of bond with other carbon atoms. | covalent | ionic | metallic | hydrogen | covalent |
| 6 | Which of the following is an allotrope of carbon? | diamond | graphite | carbon nanotube | all of the above. | diamond |
| 7 | Fullerene or bucky ball is made up of carbon atoms. | 100 | 20 | 75 | 60 | 60 |
| 8 | There are types of CNT | 2 | 3 | 4 | 1 | 2 |
| 9 | Which of the following statement/s is are true? | ratio is very large for nanomaterials. | The cut-off limit of human eye is 10-5 m. | Hardness of a SWNT is about 63 x 109 Pa. | Carbon nanotubes are cylindrical fullerenes. | Carbon nanotubes are cylindrical fullerenes. |
| 10 | The thermal stability of a nanotube is seen up toK in air. | 100 | 1000 | 2000 | 3100 | 1000 |
| 11 | The width of a carbon nanotube is nm | 1 | 1.3 | 2.5 | 10 | 1.3 |
| 12 | In radial direction, the thermal conductivity of a nonotube iswatt/(m K). | 3500 | 385 | 350 | 0 | 0 |
| | | | 1000 | | | |
| 13 | The thermal stability of a nanotube is seen up to K in vacuum. | 100 | 1000 | 2200 | 3100 | 3100 |
| 14 | The thermal conductivity of an SWNT along length is <u>watt/(m.K)</u> . | 35 | 350 | 385 | 3500 | 3500 |
| 15 | The tensile strength of an MWNT is Pa. | 63 x 10 ⁶ | 63 x 10 ⁷ | 63 x 10 ⁸ | 63 x 10 ⁹ | 63 x 10 ⁹ |
| 16 | The compressive strength of a nanotube its tensile strength. | is less than | is greater than | is equal to | may be greater than | is less than |
| 17 | The hardness of a standard SWNT is Pa. | 63 x 10 ⁶ | 25 x 10 ⁶ | 25 x 10 ⁹ | 25 x 10 ⁻⁹ | 25 x 10 ⁹ |
| 18 | The bulk modulus of a standard SWNT is that of diamond. | less than | greater than | equal to | less than or equal to | greater than |
| 19 | How much current can be passed through 1 cm ² cross-section of a metal nanotube? | 10 ⁻⁹ A | 10 ⁹ A | 1000 A | 0.001 A | 10 ⁹ A |
| 20 | The electrical conductivity of a nanotube is times that of copper. | 10 | 100 | 1000 | 1/100 | 1000 |
| 21 | An MWNT possesses electrical superconductivity up to temperature of | 12 K | 12°C | 100 K | 100° | 12 K |
| 22 | The thermal conductivity of a standard SWNT along its length is watt//m K) | 3500 | 385 | 35000 | 35 | 3500 |
| | | | | | | |
| 23 | Nanoscience can be studied with the help of | quantum mechanics | macro-dynamics | Newtonian mechanics | geophysics | quantum mechanics |
| 24 | Carbon nanotubes offer the advantage over metals of being: | good electrical conductors The tubes are connected together by covalent C C | light and flexible The tubes are randomly organized with the ayer of | good absorbers of heat The tubes are aligned, axes parallel, with yan dar Waale | strong. The bundles are of discrete sizes, and dipole dipole | good electrical conductors The tubes are connected together by covalent C C |
| 25 | Nanotubes usually form in bundles. Which is the best description of such a bundle? | bonds | the tubes lying in random | forces operating between | forces hold the tubes | bonds |
| 26 | The structure of SWCNT can be conceptualized by wrapping athick layer of graphite into a seamless cylinder | ten atom | two atom | one atom | none of the above | one atom |
| 27 | A single-walled nanotube (SWNT) can have a diameter of and a length of | 2nm,100µm | 10nm,200µm | 10mm,200µm | 18nm,200mm | 2nm,100µm |
| 28 | A single-walled nanotube (SWNT) can make a one dimensional structure called a | nanodot | nanoclusters | nanoparticles | nanowire | nanowire |
| 29 | Carbon nanotubes can be made by | laser evaporation | carbon arc methods | chemical vapor deposition | all the above | all the above |
| 30 | In laser evaporation method, a quartz tube containing argon gas and a graphite target are heated to | 100°C | 1200°C | 1000°C | 500°C | 1200°C |
| 31 | In laser evaporation, thesweeps the carbon atoms from the high-temperature zone to the colder copper collector | oxygen | CO ₂ | argon | hydrogen | argon |
| 22 | | | 1 | CUD | N/D | |
| 32 | The Method can produce single-walled nanotubes of diameters 1-5nm with a length of 1µm. Themethod allows continuous fabrication and the most favorable method for scaleup and | carbon arc method | laser evaporation | CVD | PVD | carbon arc method |
| 33 | production | chemical vapor deposition | laser evaporation | carbon arc | PVD | chemical vapor deposition |
| 34 | Most MWCNTs have a diameter of close to | 100nm | 10nm | 1nm | 1000nm | 10nm |
| | | | | | | |
| 35 | Inmethod a laser is aimed at a block of graphite | Laser ablation | Pulsed laser vaporization | CVD | both a and b | both a and b |
| 35 36 | Inmethod a laser is aimed at a block of graphite In CVD, the synthesis of CNT is performed at | Laser ablation 1100°C | Pulsed laser vaporization 500°C | CVD 100°C | both a and b 3000°C | both a and b 1100°C |
| 35 36 37 | In method a laser is aimed at a block of graphite In CVD, the synthesis of CNT is performed at At atm. Pressure | Laser ablation 1100°C carbon arc method | Pulsed laser vaporization 500°C laser evaporation | CVD 100°C chemical vapor deposition | both a and b 3000°C High-pressure CO conversion | both a and b 1100°C High-pressure CO conversion |

| 39 | Magnetoresistance is a phenomenon whereby the resistance of a material is changed by the application of a | AC magnetic field | DC magnetic field | DC electric field | none of the above | DC magnetic field |
|----|--|-------------------------|-----------------------------|---------------------------|-------------------------------|----------------------------|
| 10 | | negative | | negative or positive | | |
| 40 | The decrease in resistance with increasing DC magnetic field is | magnetoresistance | positive magnetoresistance | magnetoresistance | none of the these | negative magnetoresistance |
| | | | | | | |
| 41 | Carbon nanotubes have young's moduli ranging from | 2.8-3.8TPa | 1.28-1.8TPa | 1.28-1.8Pa | 1.28-1.8MPa | 1.28-1.8TPa |
| | | | | | | |
| | | | | | | |
| 42 | Young's modulus of carbon nanotubes is almosttimes that of steel | 10 | 20 | 100 | 2 | 10 |
| | | | | | | |
| 43 | Carbon nanotubes have so in their structure | many defects | few defects | moderate defects | all the above | few defects |
| | | | | | | |
| | | | | | | |
| 44 | The carbon-carbon bonds are | sp ² hybrids | sp ³ hybrids | sp hybrids | sp ² or sp hybrids | sp ² hybrids |
| | | | | | | |
| 45 | carbon nanotubes are about times stronger than steel | 30 | 10 | 20 | 2 | 20 |
| | carbon nanorabes are aboutthirds stronger than seen | 50 | 10 | 20 | ~ | 20 |
| | | | | | | |
| 46 | Single electron tunneling occurs when the capacitance of the nanotube is | so small | high | moderate to low | very high | so small |
| | | | | | | |
| 47 | In the metallic state the combestivity of the constance is | soons binb | hi sh | | di | |
| 47 | In the metanic state the conductivity of the hanotubes is | very nign | ingn | low | meanam | very nigh |
| | | | | | | |
| 48 | Carbon naotubes can carry aampheres per square centimeter | trillion | million | billion | thousand | billion |
| | | | | | | |
| | | - | | | | |
| 49 | nanotubes have a very high thermal conductivity, almost a factor of more than that of | 2, copper | 4,diamond | 2, silver | 2, diamond | 2, diamond |
| | | small diamater and low | low mechanical strength and | High accept ratio and | low conductivity and | High acpact ratio and |
| 50 | Advantages of carbon nanotubes on memory devices are | aspect ratio | aspect ratio | conductive along the axis | mechanical strength | conductive along the axis |
| | | | | | | |
| | | | | | | |
| 51 | Nano electronic devices using CNT can be manufactured using | Bottom up approach | top down approach | both a and b | none of the above | Bottom up approach |
| | | 11-1 | | | | |
| 52 | Advantages of carbon nanotubes based biosensors | sensitivity | fast response | slow response | both a and b | both a and b |
| 52 | Advantages of earborn nanotabes based biosensors | sensurity | nust response | sion response | bour a und b | bour a and b |
| | is a powerful technique to calculate how tubes of different dia. Are distorted when placed | | | | | |
| 53 | on a substrate | molecular mechanics | biosensors | memory devices | none of the above | molecular mechanics |
| | | | | | | |
| 54 | The diameter of constaling and to form the sizes is | 2 | 1.4 | £ | 100 | 1.4 |
| J4 | The diameter of hanotubes used to form the migs is | Juni | 1.44000 | Jun | TOOIIII | 1.4000 |
| | | | | | | |
| 55 | The high Of CNTs means that they will be poor transmitters of electromagnetic energy | mechanical stability | thermal conductivity | electrical conductivity | none of these | electrical conductivity |
| | | | | | | |
| | | | | | | |
| 56 | UNTs are metallic or semiconducting, depending on the | diameter of the tube | chirality of the tube | temperature | both a and b | both a and b |
| | There are a variety of structures of carbon nanotubes and these various structures have | | | | | |
| 57 | | same | different | both a and b | none of the above | different |
| | · · · | | | | | |
| | In carbon arc, carbon atoms are ejected from theelectrode and form nanotubes on | | | | | |
| 58 | theelectrode | positive, negative | negative,positive | either a or b | none of these | positive, negative |
| | | | | | | |
| 50 | If no catalysts are used, the tubes are | pested | multiwalled | single walled | either a or b | either a or b |
| 37 | and a second | promotion of | | | | |
| | | | | | | |
| 60 | The carbon arc method can produce | MWNT | nested | SWNT | all the above | SWNT |