

RAJARATA UNIVERSITY OF SRI LANKA
FACULTY OF APPLIED SCIENCES

Bachelor of Science Honours in Applied Sciences
B.Sc. (Joint Major) in Chemistry and Physics
Fourth Year-Semester I Examination –July/August 2023

PHY 4308 - CHARACTERIZATION TECHNIQUES

Time: Three (03) hours

Answer ALL questions.

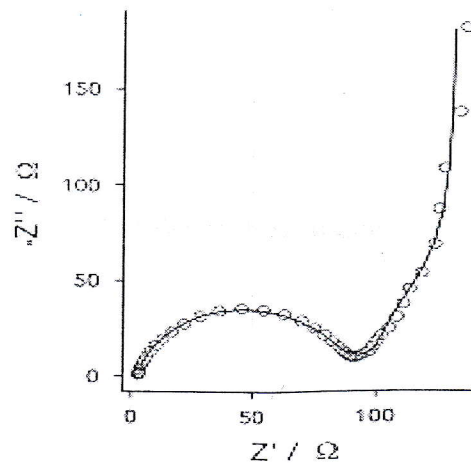
1. a) Distinguish between Resolution and Magnification of a microscope. (05 marks)
- b) i. State the *Rayleigh Criterion* for the resolution of two point objects. (05 marks)
- ii. Two stars emitting light of wavelength 550 nm are 3.7×10^{11} m apart and equally distant from the Earth. A telescope which has an objective lens of diameter 1.02 m, just resolves these stars as separate objects. Calculate the approximate distance between these stars and the Earth (assume that the distance between the stars and the Earth is very much greater the distance between the stars). (10 marks)
- iii. Explain why the resolution of Electron Microscopy is better than that of the Optical Microscopy. (10 marks)
- c) What are the image contrast mechanisms employed in Scanning Electron Microscopy (SEM)? Discuss each of them in detail. (10 marks)
- d) Fractography is the study of the fracture surfaces of materials. In many cases fractography requires examination at a finer scale which is usually carried out using SEM. Explain how the fracture surface of the following materials are analyzed using SEM (pay much attention on the sample preparation and SEM mode used).
 - i. Brittle fracture of a ceramic material (electrically non-conducting). (05 marks)
 - ii. Ductile fracture of a metal (electrically conducting). (05 marks)

2. a) Describe principle, instrumentation and applications of Thermogravimetry (TG) and Differential Thermal Analysis (DTA) (20 marks)
- b) What kind of DTG and TG curves would you expect to obtain on heating samples of the following?
- $Ca_2C_2O_4 \cdot H_2O$ with w% 12.3, 19.2 and 30.5 weight loss. Write the decomposition steps
 - NH_4NO_3
 - $CaCO_3$ contaminated with SiO_2 (20 marks)
- c) Outline basic differences between DTA and Differential Scanning Calorimetry (DSC) (10 marks)
- 3) a) Spectroscopy is an area of study that measures and interprets the electromagnetic spectra that result from the interaction between electromagnetic radiation and matter.
- Write three types of motions which can be considered as kinetic energy of a molecule.
 - Briefly explain four types of techniques which are used in optical spectroscopy measurements with aid of an appropriate diagram.
 - What are the basic components of a spectroscopy instrument?
 - Write three methods used for wavelength selection. What are the advantages and disadvantages of each of them?
 - How many modes of vibrations occur in the linear molecules which can be detected by the Fourier Transform Infrared (FTIR) spectroscopy. Obtain the number of modes of vibrations for CO_2 molecule. (25 marks)
- b) An electrolyte is sandwiched between perfectly non-blocking electrodes.
- Draw an equivalent circuit to represent the electrical behavior of the electrolyte sample.
 - Show that the frequency response of the above circuit has the following expressions for the real and imaginary parts of the total impedance Z .

$$Z' = \frac{R}{1 + (\omega RC)^2} \text{ and } Z'' = \frac{-\omega R^2 C}{1 + (\omega RC)^2}$$

Contd.

- iii. The figure below shows a Nyquist plot for an electrolyte sandwiched between perfectly blocking electrodes.

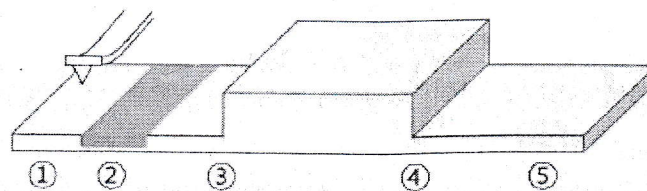


Explain why the given Nyquist plot is different from the ideal behavior?

- iv. Briefly explain the variation of impedance value with frequency for pure resistor and pure capacitor. Draw Nyquist plot for each of them.
- v. What is the difference between blocking electrode and non-blocking electrode in DC polarization test (Use appropriate graph/figure to explain the answer).

(25 marks)

- a) Atomic Force Microscopy (AFM) is a high-resolution type of Scanning Probe Microscopy (SPM) in nano range.
- Briefly explain the working principle of AFM using an appropriate diagram.
 - Following figure shows the topography of a sample. Areas (2) has a higher friction than those of other areas.

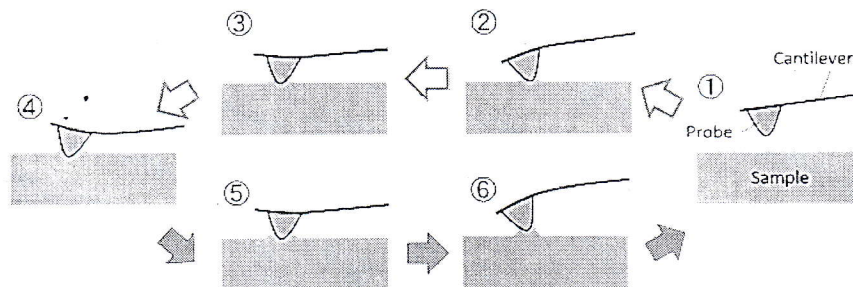


Draw AFM image, Lateral force microscopy (LFM) image from left to right and LFM image from right to left for the given sample.

- iii. What are the advantages and disadvantages of contact mode, non-contact mode and tapping mode of AFM?

Contd.

- iv. Explain the force spectroscopy technique of the AFM and draw the graph using the following diagram.



(25 marks)

- b) Nuclear Magnetic Resonance (NMR) spectroscopy is a technique used for determining the content and purity of a sample as well as its molecular structure.

- i. What type of compounds can be determined using NMR? Elaborate your answer using atomic mass and atomic number.
- ii. Explain briefly why the protons that are closer to the electronegative atom have higher chemical shift in ^1H -NMR spectrum.
- iii. Draw the NMR spectrum for the following chemicals considering peak split under spin-spin coupling (J coupling) effect.
 - a. $\text{CH}_3\text{CH}_2\text{COOH}$
 - b. $\text{CH}_3\text{COOCH}_2\text{CH}_3$
 - c. CH_3CHCl_2
- iv. Explain why Tetramethylsilane (TMS) is used as the baseline material in NMR?
- v. Briefly explain the splitting pattern of NMR peaks with equivalent and non-equivalent J coupling.

(25 marks)

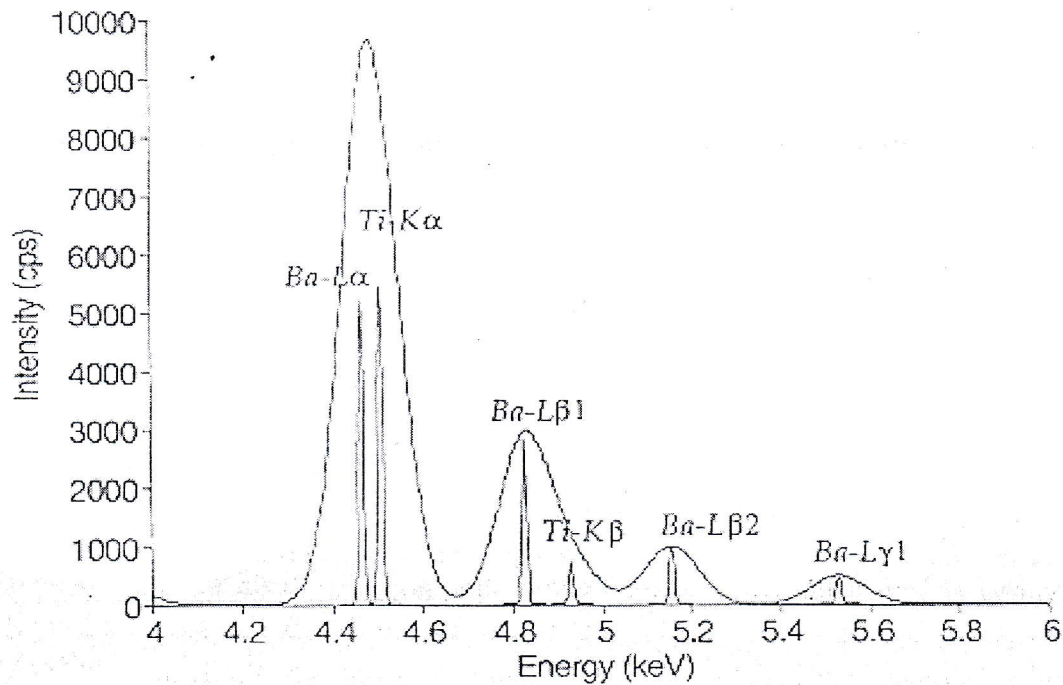
- 4) a) Discuss the following techniques used for elemental analysis associated with Scanning Electron Microscopy (SEM).

- i. Wavelength Dispersive X-Ray microanalysis (WD)
- ii. Energy Dispersive X-Ray microanalysis (ED)

(20 marks)

Contd.

b)



Superposed WD and ED spectra of a BaTiO₃ sample are shown in the above diagram. Compare and contrast the above two spectra. (10 marks)

- c) "X-Ray microanalysis cannot distinguish between isotopes of the same element" Substantiate the above statement. (10 marks)
- d) Discuss in detail the "X-Ray mapping" used for imaging the special distribution of chemical elements within a given sample. (10 marks)

End.