UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Session 2012/2013

January 2013

EBB 511/3 – Materials Characterisation Techniques

Duration : 3 hours

Please ensure that this examination paper contains SIX printed pages before you begin the examination.

This paper consists of SEVEN questions.

Instruction: Answer **FIVE** questions. If a candidate answers more than five questions only the first five questions answered in the answer script would be examined.

The answers to all questions must start on a new page.

All questions must be answered in English.

1. [a] Draw a schematic diagram of a diffractometer, and label each component and briefly describe them.

(30 marks)

[b] Phase identification is one of the main applications of XRD and Hanawalt method is one of the techniques used to sort out the XRD patterns. Explain this technique and use appropriate diagram to illustrate your answer.

(50 marks)

[c] The limit of phase detectability by manual XRD is usually about 1 - 2% concentration range. With automated XRD this can be improved further. Discuss how can this be achieved.

(20 marks)

2. [a] The electron gun is one of the important components in a Scanning Electron Microscopy (SEM). Briefly describe the types of electron gun used in SEM and what are the advantages and disadvantages.

(50 marks)

[b] Secondary electrons are the most widely used interaction with which to form image in Scanning Electron Microscopy (SEM). Why?

(30 marks)

[c] What are the factors affecting aberration of lenses? How do you minimize lens aberration in Scanning Electron Microscopy (SEM)?

(20 marks)

3. [a] Electromagnetic lens systems employ very small angular apertures, primarily because of their large spherical aberration. Explain why the spherical aberration and other lens defects limit the resolving power of a Transmission Electron Microscope (TEM).

(40 marks)

[b] What are the three primary contrast mechanisms in Transmission Electron Microscope (TEM) and describe any TWO (2) of them.

(40 marks)

[c] Describe the difference between bright field and dark field imaging modes.

(20 marks)

4. [a] Describe using a schematic diagram the working of a Scanning Tunelling Microscope (STM). The answer should include the underlying principle and the imaging process.

(60 marks)

[b] In an Atomic Force Microscopy (AFM) there are 3 modes of operation i.e. Contact mode, Noncontact mode and Tapping Mode. Describe these modes and what are their advantages and disadvantages.

(40 marks)

5. [a] Fourier Transform Infrared (FTIR) is one of the techniques used to identify materials. What is the underlying principle of this technique? What do you understand by the term IR active and IR inactive?

(30 marks)

[b] What are the advantages of Fourier Transform Infrared (FTIR) compared to IR?

(20 marks)

[c] Briefly describe the Attenuated Total Reflectance (ATR) technique.

(20 marks)

[d] By using appropriate diagram explain the working of a Michelson interferometer.

(30 marks)

6. [a] What is thermal analysis? Why temperature and environment (atmosphere) in thermal analysis need to be controlled?

(20 marks)

[b] You were given a polymer composite sample that contains carbon fibre and glass fibre with the composition shown below:

Material	Weight (%)
Polymer	60
Carbon fibre	Unknown
Glass fibre	Unknown

(i) Design a thermal gravimetry analysis (TGA) test on how to determine the percentage of carbon and glass fibre in the sample.

(ii) If the composition of carbon fibre is 15%, plot thermal gravimetry analysis (TGA) curve with the test parameters shown below:

Test temperature: 50 – 600°C

Atmosphere: Oxygen

(60 marks)

[c] Calculate the percentage of crystallinity for polyethylene (PE) phase in a PE/EPDM blend (55/45 by weight). It was given that:

ΔH_f = 40.07 J/g

ΔH°_f (PE) = 209 J/g

(20 marks)

7. [a] Describe how viscoelastic property of a material can be determined by using dynamic mechanical thermal analysis (DMTA) and describe also Argand chart.

(20 marks)

[b] For a Thermal Gravimetry Analysis (TGA) curve, a carbon black-filled rubber shows a 5% loss of mass at 92 – 252ºC and 75% mass loss at 402 – 502ºC. The test stop at 600ºC and residue of 20% remains.

(i) Plot the curve and interpret the result

(ii) If isothermal test was continued at 600ºC for 30 minutes, plot the expected curve and interpret the result if no residue remains at the end.

(60 marks)

[c] Why test temperature for thermal gravimetry analysis (TGA) is higher than test temperature for differential scanning calorimetry (DSC)?

(20 marks)

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