## UNIVERSITI SAINS MALAYSIA

Supplementary Semester Examination Academic Session 2004/2005

May 2005

## EBB 511/3 - Materials Characterisation Techniques

Duration: 3 hours

Please check that this examination paper consists of FIVE (5) pages of printed material before you begin the examination.

This paper contains SEVEN questions.

<u>Instructions:</u> Answer <u>FIVE</u> questions. If a candidate answer more than five questions, only the first five answered will be examined and awarded marks.

Answer to any question must start on a new page.

All questions must be answered in English.

1. (a) What is thermal analysis? Why do we need to control temperature and environment (atmosphere) in thermal analysis?

(30 marks)

(b) A carbon fiber-reinforced polymer shows (in nitrogen) a 3% loss of mass at 90 – 250°C and 75% mass loss at 400 – 500°C. The test stop at 600°C and residue of 22% remains. Plot the curve and describe every weight loss in that curve. If nitrogen was replaced by air at 500°C, plot the expected curve.

(50 marks)

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

$$\Delta H_f = 40.07 \text{ J/g}$$
  
 $\Delta H^{\circ}_f (PE) = 209 \text{ J/g}$ 

(20 marks)

- 2. (a) Write short note on the topics listed below:
  - i. Differential scanning calorimetry (DSC)
  - ii. Thermal mechanical analysis (TMA)
  - iii. Differential thermal analysis (DTA)
  - iv. Thermogravimetry analysis (TGA)
  - v. Scanning electron microscope energy dispersive analysis (SEM-EDA)

(50 marks)

(b) Describe the principle of scanning electron microscope (SEM) with the help of a diagram. What happens when an electron beam was focused on a sample?

(50 marks)

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 (a) With the help of a diagram, describe how viscoelastic property of a material can be determined using dynamic mechanical thermal analysis (DMTA).

(50 marks)

(b) Describe the formation and importance of secondary and back scatted electrons in SEM imaging.

(50 marks)

4. (a) "The structure factor is independent of the shape and size of the unit cell'.

Explain this statement. (You can cite appropriate examples to support your discussion).

(40 marks)

(b) Aluminium metal powder was obtained by filing a bulk specimen; i.e. it is effectively a cold-worked specimen. An X-ray diffraction of this specimen was recorded with Cu  $K_{\alpha}$  radiation in the  $2\theta$  angular range of 30 to  $70^{\circ}$ . Three peaks were indexed in this range. For comparison purposes and to calculate the instrumental broadening, the sample was annealed and its XRD was also recorded. With the information given in the table below determine the lattice strain and the crystallite size.

Table 1: Full-Width at Half-Maxima of Annealed Aluminum Specimen

Material: Annealed aluminum		Radiation: Cu Kα		$\lambda = 0.154056 \text{ nm}$	
Peak #	2θ (°)	hkl	FWHM (°)	FWHM (rad) = B <sub>i</sub>	B <sub>0</sub> (°)
1	38.52	111	0.103	1.8 x 10 <sup>-3</sup>	0.187
2	44.76	200	0.066	1.2 x 10 <sup>-3</sup>	0.206
3	65.13	220	0.089	1.6 x 10 <sup>-3</sup>	0.271

 $B_0$  = observed broadening

B<sub>i</sub> = instrumental broadening

(60 marks)

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5. (a) Explain the basic principle of FTIR. Name the three techniques available in IR spectroscopy for surface analysis. Briefly describe one that can analyse surface layer in the range  $\sim 0.5$  to 3  $\mu m$ .

(50 marks)

(b) Give a schematic diagram of an atomic absorption spectrometer. Briefly describe the important components.

(50 marks)

6. (a) Atomic Force Microscopy (AFM) is a very versatile technique for measuring surface topography. Describe the principle and operation of an AFM. What is the advantage(s) and disadvantage(s) of non-contact mode.

(50 marks)

- (b). Write short notes on any (2) of the following:
  - i. Scanning Tunneling Microscope (SPM)
  - ii. Lateral Force Microscopy
  - iii. Magnetic Force Microscopy
  - iv. Scanning Thermal Microscopy
  - v. Scanning Capacitance Microscopy

(50 marks)