UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Session 2008/2009

November 2008

EBB 511/3 - Materials Characterization Techniques

Duration : 3 hours

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

This paper contains SEVEN questions.

Instructions: Answer **FIVE** questions. If a candidate answers more than five questions only the first five questions in the answer sheet will be graded.

Answer to any question must start on a new page.

All questions must be answered in English.

1. [a] In X-ray Diffraction (XRD) "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_α radiation in the 2θ angular range of 30 to 70°. 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		λ = 0.154056 nm	
Peak #	2θ (°)	hkl	FWHM (°)	FWHM (rad) = B_i	B_o (°)
1	38.52	111	0.103	1.8×10^{-3}	0.187
2	44.76	200	0.066	1.2×10^{-3}	0.206
3	65.13	220	0.089	1.6×10^{-3}	0.271

B_o = observed broadening

B_i = instrumental broadening

(60 marks)

2. [a] Explain the basic principle of FTIR. Name the three techniques available in IR spectroscopy for surface analysis. Briefly describe one technique that can analyse surface layer in the range ~0.5 to 3 μm.

(50 marks)

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

(30 marks)

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[c] Briefly discuss the application of FTIR in surface analysis of nanomaterials.

(20 marks)

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

(50 marks)

[b] Secondary electrons are the most widely used interaction to form image in SEM. Why?

(30 marks)

[c] Briefly explain how chemical analysis is carried out in SEM.

(20 marks)

4. [a] Draw a schematic diagram of a Transmission Electron Microscope (TEM). Label each component clearly.

(30 marks)

[b] What is/are the most important differences between TEM and SEM.

(20 marks)

[c] Explain the basic principle of UV-Vis spectrometry. Briefly describe how the band gap determination of a semiconductor material can be carried out.

(50 marks)

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

(50 marks)

6. Consider the following decomposition reaction that is being studied for DTA-TG analysis : $CaCO_3 = CaO + CO_2$ under the following experimental conditions : max temperature = 1100°C, heating rate = 10 K/min, sample weight = 50 mg, physical state of sample = powder, reference material = Al_2O_3

(a) If the experiment is conducted under a flowing stream of CO_2, N_2, and vacuum of 0.1 atm separately, what are the changes do you expect in the corresponding TG plots?

(40 marks)

(b) If the sample is compacted to form a disc, how does it affect the DTA-TG plots?

(30 marks)

(c) What are the effects of heating rate and sample mass on the results obtained from the above TG-DTA experiment?

(30 marks)

7. [a] Consider the reduction reaction of pure liquid FeO by solid powdered carbon at three different constant temperatures. Assume that the reaction follows a nucleation and growth (Johnson - Mehl type) reaction rate model. Also assume that the change in weight of the sample is only through the evolution of carbon monoxide (CO) according to the reaction : $FeO + C \rightarrow Fe + CO$. Illustrate the steps involved to calculate the reaction rate and the activation energy by employing the integral method of kinetic analysis.

(50 marks)

[b] Outline the principles of thermomechanical analysis (TMA) of materials showing the various types of loading configurations and typical TMA plots. How is it different from thermodilatometry (TD)?

(50 marks)

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