# UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Session 2007/2008

October/November 2007

## 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 any **FIVE** questions. If a candidate answers more than five questions, only the first five answers 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] Briefly explain the working principle and applications of a Scanning Tunneling Microscope (STM). Compare this equipment with the other microscopy (Electron and Optical microscopy, and Atomic Force Microscopy).

(40 marks)

[b] Explain the various interactions between electromagnetic radiation and matter in spectroscopic analytical methods. How can we use these types of interactions to identify and determine chemical analysis, chemical structure and many other applications of the spectroscopic methods. Give appropriate examples.

(40 marks)

[c] Why do optical microscope and electron microscope have different resolutions? Explain what is meant by resolution and what are the factors that determine the resolution of a microscope.

(20 marks)

2. [a] Discuss for each case; the importance of the issue, the method of analysis and how the result is interpreted to solve the issue or problem.

(i) The analysis of engine oil for the early detection of machine component failure.

(ii) Identification of narcotic drugs (in liquid, tablets and powders form).

(iii) Analysis of steel sample during scrap melting in the electric arc furnace.

(iv) Characterize the transmission loss of silicon photocells (solar cells application).

(v) Characterize effect of sol-gel concentration, pH value, calcination time and temperature on grain size of nano-$TiO_2$ powders.

(100 marks)

3. [a] Explain the working principle and applications of a UV-Vis spectroscopy. Use appropriate examples.

(40 marks)

[b] Discuss the following:

(i) Electron interactions in electron microscopy.

(ii) Bragg's Law in X-ray diffraction for identifying crystalline materials.

(iii) Energy-dispersive XRF compared to wavelength-dispersive XRF.

(iv) Atomic and molecular transitions in spectrometry techniques.

(60 marks)

4. [a] Explain the working principle and applications of a Nuclear Magnetic Resonance (NMR) spectrometry.

(40 marks)

[b] A sample of stainless steel (0.320 g) was weighed out and dissolved in nitric acid. The resulting solution was made up to 1 $dm^3$ with water. Five standards and the sample solution were analysed for nickel consecutively on a flame atomic absorption spectrophotometer with the following results:

| SOLUTION | NI CONCENTRATION/PPM | ABSORBANCE |
|----------|----------------------|------------|
| 1 | 2 | 0.126 |
| 2 | 4 | 0.250 |
| 3 | 6 | 0.374 |
| 4 | 8 | 0.500 |
| 5 | 10 | 0.626 |
| sample | - | 0.220 |

Calculate the amount of nickel in the steel.

(60 marks)

5. [a] Discuss the following cases and suggest the characterization method (or methods) that each case might require:

    (i) A student wants to produce an in-situ $FeAl/(TiB_2 + Al_2O_3)_p$ composites at various reinforcement percentage. The system that he is using is $Fe + Al + TiO_2 + B = FeAl + TiB_2 + Al_2O_3$.

    (ii) A researcher wants to study the effect of using various organic templates in producing vanadium oxide nano-tube.

    (iii) A researcher is involved in the study of nano-particles with a core-shell structure. The analysis should include the determination of the particles size and how to confirm the existence of core-shell structure.

The discussion should include the characteristic required and how each of the method that you suggest can be used to characterize them.

(25 marks each)

[b] Describe the basic function of an X-ray tube and how does it provide a monochromatic radiation.

(25 marks)

6. [a] What is the principle of Thermodilatometry and how the coefficient of expansion of a solid can be determined by this technique?

(30 marks)

[b] Outline the principles of a LASER dilatometer and how is it superior over a conventional dilatometer.

(30 marks)

[c] Describe the procedure, indicating the necessary formulations, to determine the purity of a substance using the differential scanning calometry technique.

(40 marks)

7. [a] Consider a chemical reaction involving nucleation and growth and obeying the Johnson – Mehl equation, $[- \ln (1 - \alpha)]^{1/n} = kt$. Outline the methodology to calculate the activation energy of the reaction through differential and integral approaches.

(50 marks)

[b] Discuss the importance of temperature measurement and calibration in a thermogravimetric experiment and how the calibration can be done?

(25 marks)

[c] Discuss in brief how the atmosphere prevailing during a thermogravimetric experiment may affect the TG/DTA results?

(25 marks)

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