# UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Session 2009/2010

November 2009

## EBB 245/3 - Characterisation of Engineering Materials
## *[Pencirian Bahan Kejuruteraan]*

Duration : 3 hours
*[Masa : 3 jam]*

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Please ensure that this examination paper contains ELEVEN printed pages before you begin the examination.
*[Sila pastikan bahawa kertas peperiksaan ini mengandungi SEBELAS muka surat yang bercetak sebelum anda memulakan peperiksaan ini.]*

This paper consists of SEVEN questions. THREE questions in PART A and FOUR questions in PART B.
*[Kertas soalan ini mengandungi TUJUH soalan. TIGA soalan di BAHAGIAN A dan EMPAT soalan di BAHAGIAN B.]*

**Instruction:** Answer **FIVE** questions. Answer **ALL** questions from PART A and **TWO** questions from PART B. If candidate answers more than five questions only the first five questions answered in the answer script would be examined.
*[**Arahan**: Jawab **LIMA** soalan. Jawab **SEMUA** soalan dari BAHAGIAN A dan **DUA** soalan dari BAHAGIAN B. Jika calon menjawab lebih daripada lima soalan hanya lima soalan pertama mengikut susunan dalam skrip jawapan akan diberi markah.]*

The answers to all questions must start on a new page.
*[Mulakan jawapan anda untuk semua soalan pada muka surat yang baru.]*

You may answer a question either in Bahasa Malaysia or in English.
*[Anda dibenarkan menjawab soalan sama ada dalam Bahasa Malaysia atau Bahasa Inggeris.]*

In the event of any discrepancies, the English version shall be used.
*[Sekiranya terdapat sebarang percanggahan pada soalan peperiksaan, versi Bahasa Inggeris hendaklah diguna pakai.]*

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**PART A**

***BAHAGIAN A***

1. [a] Give the differences between Optical Microscope and Scanning Electron Microscope, in terms of:

(i) Source of energy

(ii) Magnification

(iii) Type of image revealed

(iv) Sample size

(v) Sample preparation

(vi) Image generation

*Berikan perbezaan antara Mikroskop Optik dan Mikroskop Imbasan Elektron, dari segi:*

*(i) Sumber tenaga*

*(ii) Pembesaran*

*(iii) Jenis imej yang boleh dihasilkan*

*(iv) Saiz sampel*

*(v) Penyediaan sampel*

*(vi) Penghasilan imej*

(60 marks/*markah*)

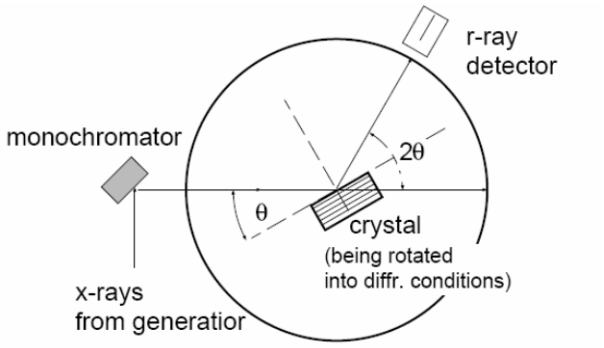
[b] With the help of an appropriate diagram, explain the working principle of Thermogravimetric Analysis (TGA).

*Dengan bantuan gambarajah yang sesuai, huraikan prinsip kerja bagi Analisis Kebezaan Terma (TGA).*

(40 marks/*markah*)

2. [a] Figure 1 shows a set up of an equipment that can be used in analyzing unknown materials. Name this material characterization technique. Explain the principles of this technique.

*Rajah 1 menunjukkan satu susunan peralatan yang boleh digunakan untuk menganalisis bahan yang tidak diketahui. Namakan teknik pencirian bahan ini. Terangkan prinsip utama teknik ini.*



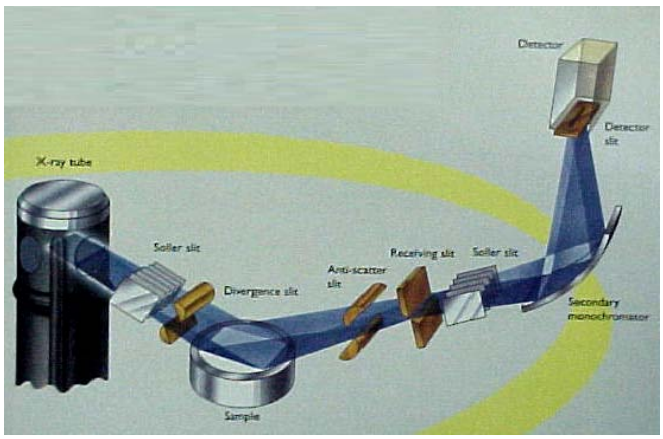




***Figure 1 - Setup for materials analysing equipment***

***Rajah 1 - Satu susunan peralatan bagi menganalisis bahan***

(60 marks/*markah*)

[b] Briefly outline the main steps involve in quantitative analysis of element using an X-ray fluorescence instruments.

*Terangkan secara ringkas langkah-langkah utama yang terlibat dalam analisis kuantitatif unsur menggunakan peralatan pendaflor sinar-X.*

(40 marks/*markah*)

3. [a] Describe briefly using electromagnetic concepts, how resonance radiation and resonance fluorescence are generated. In your explanation, use a sodium atom as an example.

*Terangkan secara ringkas berdasarkan konsep electromagnetik, bagaimana radiasi resonan dan radiasi florosen dihasilkan. Gunakan atom sodium sebagai contoh di dalam penerangan anda.*

(30 marks/*markah*)

[b] Most spectroscopic instruments in the UV/visible and IR regions are made up of five main components. Name and describe each of the components and its functions. Show schematically a typical set-up of a single-beam spectrophotometer with a complete labeling.

*Kebanyakan peralatan spektroskopik UV/visible and IR terdiri dari lima komponen utama. Nama dan terangkan setiap satu dari lima komponen tersebut. Seterusnya, lakarkan secara skematik dengan penglabelan yang lengkap.*

(30 marks/*markah*)

[c] A method of analysis yields weights for gold that are low by 0.4 mg. Calculate the percent relative error caused by this uncertainty. Consequently, the method described above is to be used for analysis of ores that assay about 1.2% gold. What minimum sample weight should be taken if the relative error resulting from 0.4 mg loss is not to exceed:

(i) - 0.2%
(ii) - 0.5%
(iii) - 0.8%
(iv) - 1.2%

*Kaedah analisis menghasilkan emas seberat 0.4 mg lebih rendah. Kirakan peratusan ralat yang terhasil daripada ketidaktentuan ini. Seterusnya, kaedah diatas digunakan untuk menganalisa bijih yang mengandungi 1.2% emas. Berapakah berat minima sampel yang patut diambil sekiranya ralat relatif yang terhasil daripada kekurangan 0.4 mg tidak melebihi:*

(i) - 0.2%
(ii) - 0.5%
(iii) - 0.8%
(iv) - 1.2%

(40 marks/*markah*)

**PART B**

***BAHAGIAN B***

4. [a] One of the steps in sample preparation for microstructure observation using Optical Microscope is etching. Describe it.

*Satu dari langkah penyediaan sampel bagi pemerhatian mikrostruktur menggunakan Mikroskop Optik ialah punaran. Perihalkan.*

(20 marks/*markah*)

[b] Scanning Probe Microscopy (SPM) is a group of microscopic techniques that measure morphology and properties of surfaces on atomic scale. List three (3) advantages and disadvantages of this technique.

*Mikroskopi Imbasan 'Probe' (SPM) merupakan satu kumpulam teknik mikroskopik yang mengukur sifat-sifat dan morfologi permukaan dalam skala atom. Senaraikan tiga (3) kelebihan dan kekurangan teknik ini.*

(30 marks/*markah*)

[c] With the help of appropriate diagram and flow chart, explain the working principle of Transmission Electron Microscopy (TEM).

*Dengan bantuan gambarajah dan cartalir yang sesuai, huraikan prinsip kerja bagi Mikroskop Transmisi Elektron (TEM).*

(50 marks/*markah*)

5. [a] Briefly describe on the working principle of Differential Scanning Calorimetry (DSC).

*Huraikan secara ringkas prinsip kerja Kalorimetri Imbasan Kebezaan (DSC).*

(40 marks/*markah*)

[b] Sketch a TG – DTA curve for montmorillonite clay which involves reactions as follows:

(i) Loss of interlayer absorbed water at 114°C.

(ii) Dehydroxylation reaction of the mineral at 704°C.

(iii) Endothermic event at 895°C and an exothermic event at 940°C due to structural changes.

*Lakaran keluk TG – DTA bagi tanah liat monmorilonit yang melibatkan tindakbalas-tindakbalas seperti berikut:*

*(i) Kehilangan air terserap antaralapisan pada 114°C.*

*(ii) Tindakbalas penyahhidroksilan mineral padat 704°C.*

*(iii) Tindakbalas Endotermik pada 895°C dan eksotermik pada 940°C akibat dari perubahan struktur.*

(40 marks/*markah*)

[c] List four (4) application of Thermodilatometry (TD).

*Senaraikan empat (4) kegunaan termodilatometri (TD).*

(20 marks/*markah*)

6. [a] By referring to suitable examples, describe the purposes of atomic absorption spectroscopy. What would be the expected results obtained from this technique?

*Dengan merujuk contoh-contoh yang sesuai, terangkan tujuan bagi spektroskopi nyalaan atom. Apakah bentuk keputusan yang dijangka akan diperolehi dari teknik ini?*

(30 marks/*markah*)

[b] Explain the advantages of graphite furnace atomic absorption compared to flame atomic absorption spectroscopy?

*Perihalkan kelebihan serapan atom relau grafit berbanding spektroskopi serapan atom nyalaan?*

(30 marks/*markah*)

[c] Consider the quantitative analysis result of a dry limestone sample which is obtained from XRF analysis:

| ELEMENT | SPECTRUM | WT% |
|---|---|---|
| Mg | Mg Kα | 1.2 |
| Ca | Ca Kα | 90.0 |
| Fe | Fe Kα | 2.3 |
| Si | Si Kα | 6.5 |

Mg and Ca are found as carbonates, while Fe and Si as oxides. Convert the data to mol % of the actual substances ($CaCO_3$, $MgCO_3$, $Fe_2O_3$ and $SiO_2$) present in the limestone. Mol weight: Ca = 40.1; Mg = 24.3; C = 12.0; O = 16.0 and Fe = 55.8, Si = 28.1. Spectra of oxygen and carbon are not considered.

*Pertimbangkan keputusan analisis kuantitatif sampel batu kapur kering yang diperolehi dari keputusan XRF:*

| ***ELEMENT*** | ***SPECTRUM*** | ***WT%*** |
|---|---|---|
| *Mg* | *Mg Kα* | *1.2* |
| *Ca* | *Ca Kα* | *90.0* |
| *Fe* | *Fe Kα* | *2.3* |
| *Si* | *Si Kα* | *6.5* |

*Mg dan Ca ditemui sebagai karbonat, manakala Fe dan Si sebagai oksida. Tukarkan data tersebut kepada mol % bagi bahan sebenar($CaCO_3$, $MgCO_3$, $Fe_2O_3$ dan $SiO_2$) yang hadir dalam batu kapur tersebut. Berat mol: Ca = 40.1; Mg = 24.3; C = 12.0; O = 16.0 dan Fe = 55.8, Si = 28.1. Spektrum oksigen dan karbon tidak dipertimbangkan.*

(40 marks/*markah*)

7. [a] How do systematic errors arise? Explain the difference between constant and proportional error, random and systematic error, mean and median, absolute and relative errors.

*Bagaimana ralat sistematik terhasil? Terangkan perbezaan di antara ralat tetap dan ralat langsung, ralat rawak dan ralat sistematik, purata dan median, ralat mutlak dan ralat relatif.*

(20 marks/*markah*)

[b] A portable photometer with linear response to radiation registered 73.6 μA with a blank solution in the light path. Replacement of the blank with an absorbing solution yielded a response of 24.9 μA. Calculate:

(i) the percent transmittance of the sample solution
(ii) the absorbance of the sample solution
(iii) the transmittance to be expected for a solution in which the concentration of the absorber is one-third that of the original sample
(iv) the transmittance to be expected for a solution that has twice the concentration of the sample

*Potometer mudah alih dengan tindakbalas terus terhadap radiasi merekodkan 73.6 μA dengan larutan kosong di bawah alur sinar. Penggantian kosong kepada larutan penyerap menghasilkan tindakbalas 24.9 μA. Kirakan:*

*(i) peratusan pembalikan larutan sampel*
*(ii) penyerapan larutan sampel*
*(iii) pembalikan yang dijangkakan untuk larutan yang berkepekatan 1/3 daripada sampel asal*
*(iv) pembalikan yang dijangkakan untuk larutan yang berkepekatan 2 kali ganda sampel asal*

(40 marks/*markah*)

[c] The following data were taken from a diode array spectrophotometer in an experiment to measure spectrum of the Co(II)-EDTA complex. The column labelled $\mathbf{P_{solution}}$ is the relative signal obtained with sample solution in the cell after substraction of the dark signal. The column labelled $\mathbf{P_{solvent}}$ is the reference signal obtained with only solvent in the cell after substraction of dark signal. Find the transmittance at each wavelength, and the absorbance at each wavelength. Plot the spectrum of the compound.

*Data berikut diambil daripada spektrophotometer diod tata susunan di dalam ujikaji mengukur spektrum Co(II)-EDTA. Turus berlabel $P_{solution}$ merupakan isyarat relatif yang diperolehi dari larutan sampel selepas pengasingan isyarat gelap. Turus berlabel $P_{solvent}$ diperolehi sebagai isyarat rujukan pelarut di dalam sel selepas pengasingan isyarat gelap. Dapatkan penghantaran dan penyerapan pada setiap panjang gelombang. Plotkan spektrum kompoun terlibat.*

(40 marks/*markah*)

| **Wavelength (nm)** | $\mathbf{P_{solvent}}$ | $\mathbf{P_{solution}}$ |
|---|---|---|
| 350 | 0.002689 | 0.002560 |
| 375 | 0.006326 | 0.005995 |
| 400 | 0.016975 | 0.015143 |
| 425 | 0.035517 | 0.031648 |
| 450 | 0.062425 | 0.024978 |
| 475 | 0.095374 | 0.019073 |
| 500 | 0.140567 | 0.023275 |
| 525 | 0.188984 | 0.037448 |
| 550 | 0.263103 | 0.088537 |
| 575 | 0.318361 | 0.200872 |
| 600 | 0.394600 | 0.278072 |
| 625 | 0.477018 | 0.363525 |
| 650 | 0.564295 | 0.468281 |
| 675 | 0.655066 | 0.611062 |
| 700 | 0.738180 | 0.704126 |
| 725 | 0.813694 | 0.777466 |
| 750 | 0.885979 | 0.863224 |
| 775 | 0.945083 | 0.921446 |
| 800 | 1.000000 | 0.977237 |

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