23MPP567

Advanced Materials Characterisation

Semester 1 2023/24 In-Person Exam paper

This examination is to take place in-person at a central University venue under exam conditions. The standard length of time for this paper is **2 hours**.

You will not be able to leave the exam hall for the first 30 or final 15 minutes of your exam. Your invigilator will collect your exam paper when you have finished.

Help during the exam

Invigilators are not able to answer queries about the content of your exam paper. Instead, please make a note of your query in your answer script to be considered during the marking process.

If you feel unwell, please raise your hand so that an invigilator can assist you.

You may use a calculator for this exam. It must comply with the University's Calculator Policy for In-Person exams, in particular that it must not be able to transmit or receive information (e.g. mobile devices and smart watches are **not** allowed).

Answer **THREE** questions. Each question carries 20 marks.

Useful equations are included at the end of the exam paper.

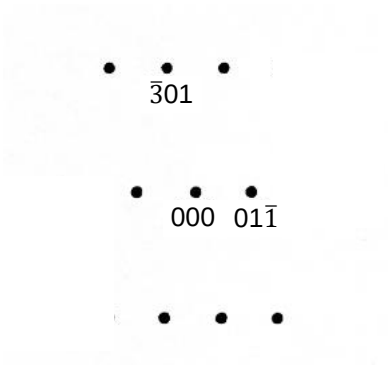
1. (a) (i) A cold rolled iron sample is analysed by X-ray diffraction (XRD). The diffracted beam emerges at the following angles (2θ) to the incident beam:

44.67°
65.02°
82.33°

If the X-ray wavelength (λ) is 0.154 nm and iron has a bcc crystal structure, calculate the lattice parameter (*a*) of the unit cell, showing your working. [6 marks]

(ii) The cold-rolled iron sample is then annealed at 700°C. Recrystallization occurs after annealing. Describe the changes that you might expect in the XRD pattern of the annealed sample compared with XRD pattern of the original sample. [4 marks]

(b) (i) Sketch and index the following diffraction pattern. [3 marks]



(ii) What is the beam direction of the above diffraction pattern? [2 marks]

(c) Calculate the Bragg angle for the (111) plane of copper (*a* = 0.362 nm), using electron diffraction with λ = 0.00251 nm. [2 marks]

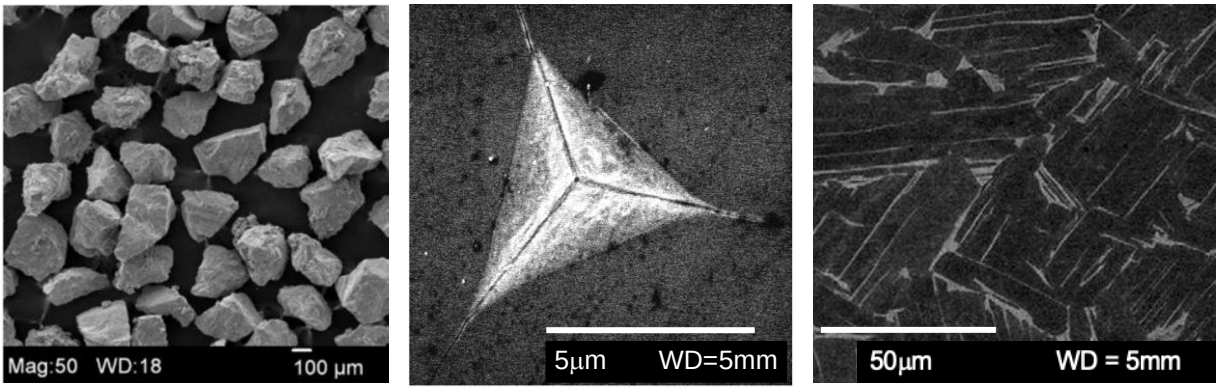
(d) Describe three differences between X-ray and electron diffraction. [3 marks]

2. (a) You are given three micrographs of different materials as shown in Fig Q2. You are asked to choose the following detectors (listed i to iii below) within an SEM or dualbeam FIB to reproduce the images of the samples. Justify your choice of detector for each sample, including the reasons why you would use it and what information the choice of detector provides about the sample in question.

(i) Everhart-Thornley detector (ETD); [4 marks]

(ii) In-lens detector; [4 marks]

(iii) Solid state detector. [4 marks]



(a) (b) (c)

Figure Q2 SEM images of (a) unpolished titanium alloy powder obtained with accelerating voltage of 10 kV, (b) hardness indent on silicon obtained with accelerating voltage of 5 kV (c) polished titanium aluminide sample obtained with accelerating voltage of 10 kV. (WD=working distance, Mag=Magnification).

(b) (i) You are asked to characterise the deformation microstructure underneath the indented area on the silicon sample using a transmission electron microscope (TEM). Suggest a sample preparation method to achieve this and provide a justification for your answer. [4 marks]

(ii) State four damages / changes in the materials crystal lattice after the sample preparation you suggested in part (b)(i). [4 marks]

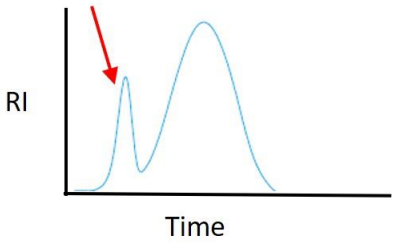
3. You work for a materials characterisation company and a customer approaches you requesting help to characterise their new paint formulation. The only information you have is that this is a dispersion of polymer particles in a liquid. The customer states that they need to check the reproducibility and stability of their formulation.

(a) (i) Suggest TWO techniques that may be used to characterise this sample, stating what information you would gain from each and whether it could help you to monitor reproducibility, stability or both. [4 marks]

(ii) Explaining your reasoning, describe what questions would you need to ask the customer to help you to determine the best sample preparation methods for the samples. [4 marks]

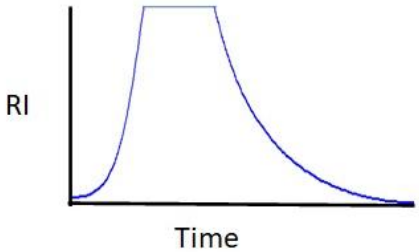
(b) Size exclusion chromatography (SEC) is a technique used to measure molecular weight of polymers in solution. The traces below show some non-ideal time versus refractive index (RI) plots that have been obtained for another customer's samples. State what may cause the issues in each case, why it would be a problem when trying to analyse samples and suggest how these could be rectified in practice.

(i)



[3 marks]

(ii)



[3 marks]

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(c) The samples are usually run at 1 ml/min on 2 mixed bed columns (plus a guard column), and one sample takes 30 minutes to complete. The customer wants to speed up the analysis. Discuss one way that this could be achieved, stating how this will speed up the analysis, and explaining the negative impact that this may have on the results. [2 marks]

(d) (i) Describe one other detector that can be used in SEC and explain what additional information can be gained from this. [2 marks]

(ii) In general SEC instruments tend to have 1-3 detectors, when many more are available. Discuss the factors that limit the use of multiple detectors on one system. [2 marks]

4. (a) (i) In light scattering analysis particle size is determined using the Stokes-Einstein equation.

$$Dh = kT/3\pi\eta_s D$$

You have a sample of crosslinked polymer particles dispersed in an unknown solvent. Given that they have a radius of 90 nm, identify the solvent used given the following information. [5 marks]

$k = 1.38 \times 10^{-23}$ m^2 kg s^{-2} K^{-1}

η_s= solvent viscosity

D = 8.1 x 10^{-12} m^2/s

Solvent	**Dynamic Viscosity at 30°C in mPa.s**
Acetone	0.30
Water	0.80
Ethanol	0.98

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(ii) Light scattering can also be used to analyse the size of linear polymers in solution. Explain why the concentration of these polymers is important when referring to the Stokes-Einstein equation above. [3 marks]

(b) In a small-angle X-ray scattering (SAXS) what does the scattering vector q represent? Describe what information is obtained from a polymer system at (a) low q, (b) intermediate q, and (c) high q. [4 marks]

(c) The ^{1}H NMR spectrum of C_3H_7Cl is shown below in Figure Q4c.

(i) Draw the 2 possible molecular structures of the compound and explain which one would result in the spectra below based on the number of peaks. [2 marks]

(ii) Correctly attribute each peak to their corresponding protons. Explain your reasoning based on peak position and splitting. [6 marks]

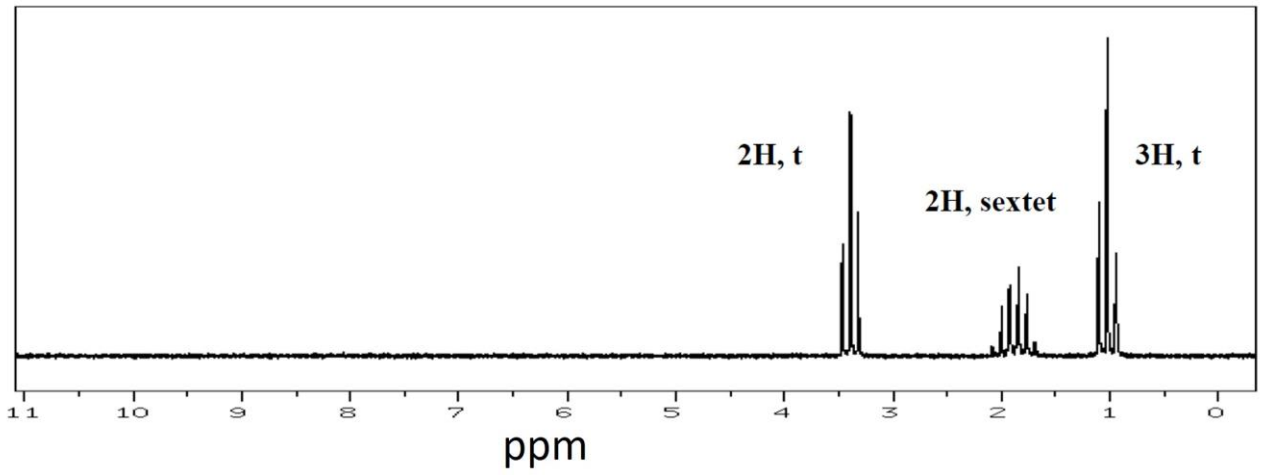

Figure Q4c – NMR spectrum of C_3H_7Cl (where t represents triplet).

END OF PAPER

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List of equations

Bragg's law	$n\lambda = 2d\sin\theta$
Interplanar spacing	$d = \frac{a}{\sqrt{h^2+k^2+l^2}}$