Loughborough University

23MPB209

Materials Characterisation

Semester 2 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 ALL three questions.

1. (a) You are given a bulk cold rolled aluminium alloy sample of a size about 2cm x 1 cm x 1 cm. Suggest a scanning electron microscopy (SEM) based technique to study the crystallographic orientation of the grains. Justify your choice of technique in terms of the signal generated during sample and electron interaction and how the signals can help you to characterise the requested microstructure of the sample. [6 marks]

   (b) To use the suggested SEM techniques in part a, how would you prepare the Al-alloy sample to obtain the data? [4 marks]

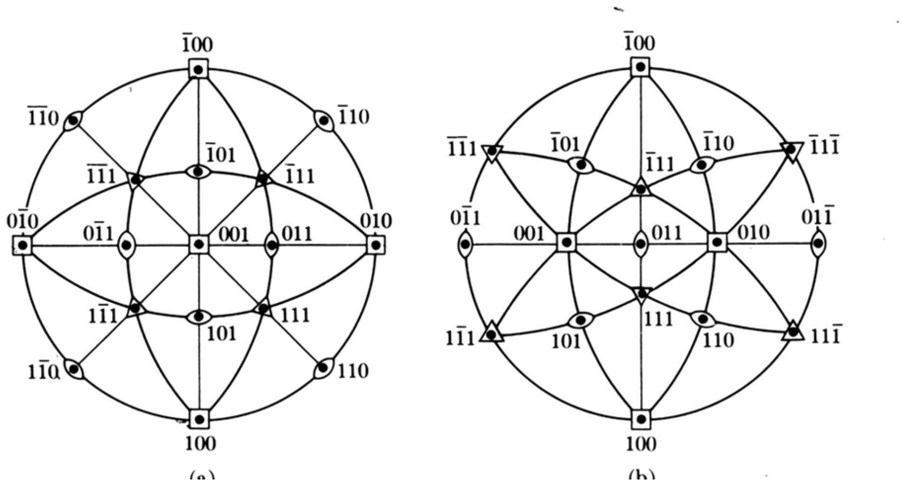
   (c) What is crystallographic texture? [2 marks]

   (d) Name two manufacturing processes that produce a strong texture in metals and alloys, briefly explain in general what texture is formed during the suggested manufacturing processes. [4 marks

   (e) Sketch the following texture components on a 001 pole figure

      (i) Cube texture [2 marks]

      (ii) {100}<110> [2 marks]



001 centred stereogram 011 centred stereogram

Figure Q1 001 and 011 centred stereograms

2. In an energy dispersive X-ray spectroscopy (EDS) experiment, the X-ray counts from a Cu– x wt% Zn specimen and a Cu – 30 wt% Zn standard were as follows:

	Specimen X-ray counts	$Cu_{0.7}Zn_{0.3}$ Standard X-ray counts
Copper	26197	27078
Zinc	12307	11989

(a) What was the composition of the specimen? Give your answers in 2 significant figures. [6 marks]

(b) In an EDS spectrum, a strong peak was detected at 6.403 keV. Using Table Q2b, explain what other peaks you would expect to see. [4 marks]

Table Q2b Energies (eV) of X-ray emission lines

Element	$K\alpha_1$	$K\alpha_2$	$K\beta_1$	$L\alpha_1$	$L\alpha_2$	$L\beta_1$	$L\beta_2$	$L\gamma_1$	$M\alpha_1$
22 Ti	4,510.84	4,504.86	4,931.81	452.2	452.2	458.4			
23 V	4,952.20	4,944.64	5,427.29	511.3	511.3	519.2			
24 Cr	5,414.72	5,405.509	5,946.71	572.8	572.8	582.8			
25 Mn	5,898.75	5,887.65	6,490.45	637.4	637.4	648.8			
26 Fe	6,403.84	6,390.84	7,057.98	705.0	705.0	718.5			
27 Co	6,930.32	6,915.30	7,649.43	776.2	776.2	791.4			
28 Ni	7,478.15	7,460.89	8,264.66	851.5	851.5	868.8			
29 Cu	8,047.78	8,027.83	8,905.29	929.7	929.7	949.8			
30 Zn	8,638.86	8,615.78	9,572.0	1,011.7	1,011.7	1,034.7			
31 Ga	9,251.74	9,224.82	10,264.2	1,097.92	1,097.92	1,124.8			
32 Ge	9,886.42	9,855.32	10,982.1	1,188.00	1,188.00	1,218.5			
33 As	10,543.72	10,507.99	11,726.2	1,282.0	1,282.0	1,317.0			
34 Se	11,222.4	11,181.4	12,495.9	1,379.10	1,379.10	1,419.23			
35 Br	11,924.2	11,877.6	13,291.4	1,480.43	1,480.43	1,525.90			
36 Kr	12,649	12,598	14,112	1,586.0	1,586.0	1,636.6			
37 Rb	13,395.3	13,335.8	14,961.3	1,694.13	1,692.56	1,752.17			
38 Sr	14,165	14,097.9	15,835.7	1,806.56	1,804.74	1,871.72			
39 Y	14,958.4	14,882.9	16,737.8	1,922.56	1,920.47	1,995.84			
40 Zr	15,775.1	15,690.9	17,667.8	2,042.36	2,039.9	2,124.4	2,219.4	2,302.7	

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Q2 Continued/…

(c) In a dynamic mechanical analysis (DMA) experiment, the complex elastic modulus E^* of a composite material, composed of a polymer and a filler, $E^*$is measured.

The storage modulus (E') of the material as a function of temperature for a range of filler concentrations is shown in the graph in Figure Q2c, with the different filled symbols representing different filler concentrations.

(i) Why is there a drop in the value of *E'* at higher temperatures? [2 marks]

(ii) Why is this drop dependent on the concentration of filler? [2 marks]

(iii) Which of the curves corresponds to the polymer without any filler additions? [2 marks]

Justify all your answers.

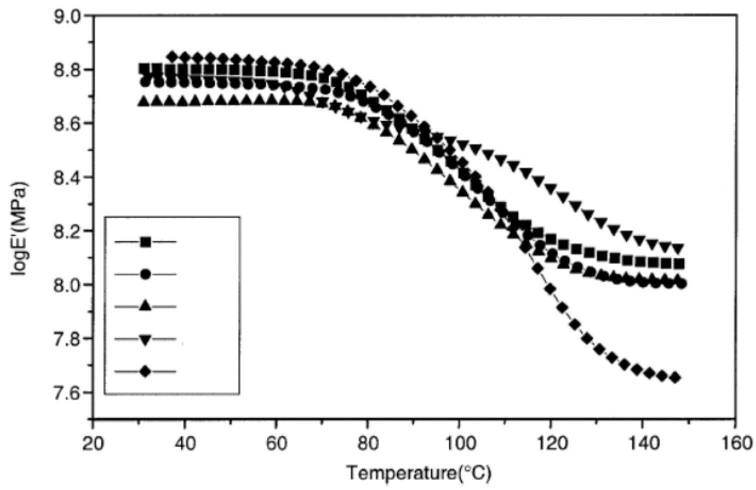

Figure Q2c. The storage modulus (E') of the material as a function of temperature for a range of filler concentrations.

(iv) For one of the composites, at 120°C a value for tanδ is measured as 0.25. Assuming a storage modulus E' at this temperature of 8.1 MPa, what is the loss modulus, E'' of the composite? [2 marks]

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Q2 Continued/…

(d) Bentonite is a type of layered silicate, often used as filler in polymers to enhance water absorption. Bentonite layers can pack forming a structure where the interlayer distance is 1.497 nm.

(i) If you analyse this structure using Wide Angle X-Ray Scattering with a source of $\lambda = 1.54$ Å, at what value of 2θ would you expect to observe a peak? [2 marks]

(ii) If you produce an intercalated composite of Bentonite with an amorphous polymer, having an interlayer distance of 25.0 Å, at what value of 2θ would you expect the peak to shift? [2 marks]

3. (a) In a differential scanning calorimetry (DSC) experiment, samples of polypropylene with and without dibenzylidene sorbitol (DBS), a nucleating agent, are characterised. For the sample with DBS, an enthalpy of fusion of $\Delta H_m = 65.2\, J/g$ is obtained, whereas for the sample without DBS the value obtained is $\Delta H_m = 45.2\, J/g$.

(i) What is the degree of crystallinity of each of the two samples? Assume a heat of fusion for polypropylene of $\Delta H_m^0 = 207\, J/g$. [4 marks]

(ii) Why does the crystallinity vary when DBS is added? [2 marks]

(iii) It is observed that in the sample with DBS, the crystallisation peak is shifted to higher temperatures with respect to the sample without DBS. Why would this shift be useful from an application point of view? Justify your answer. [2 marks]

(b) Poly(paraphenylenevinylene) (PPV) is a conductive polymer with great potential for application in solar cells and light emitting diodes (LEDs). The graph in Figure Q3b shows the variation in absorbance seen in infrared spectra of polymer samples after being exposed to a xenon lamp for varying times. The direction of the arrows indicates increasing exposure time.

(i) What process is taking place in the polymer? [2 marks]

(ii) Why does one of the highlighted peaks increase in intensity and the other decrease as exposure time increases? [4 marks]

Give reasons for your answers. Note that the peak at 960 cm^{-1} corresponds to C=C bonds.

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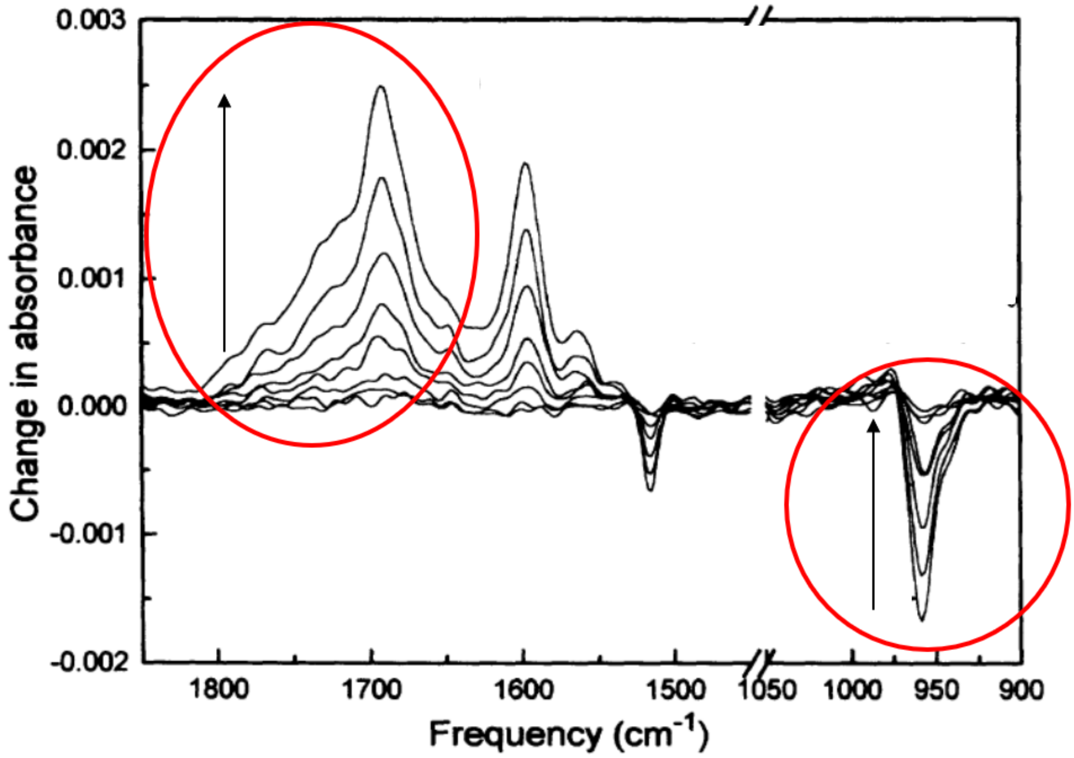

Figure Q3b. The variation in absorbance seen in infrared spectra of polymer samples after being exposed to a xenon lamp for varying times.

(c) For the following samples, choose the most appropriate characterisation technique or techniques to determine its composition if you have access to Raman spectroscopy, attenuated total reflection infrared spectroscopy, and transmission infrared spectroscopy. Explain the reasons for your choice.

(i) Polluted air from a contaminated area; [2 marks]

(ii) A photovoltaic panel; [2 marks]

(iii) A dispersion of impurities in water. [2 marks]

END OF PAPER

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List of equations and constants

Interplanar spacing $d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$

Bragg's equation 2dsinθ =nλ

Damping ratio: $tan\delta = E''/E'$

Density of crystalline unit cell: $\rho_c = \frac{nM}{N_a V}$

Cristallinity: $\%\chi_c = 100\ x\ \frac{\rho - \rho_a}{\rho_c - \rho_a}$

Crystallinity and melting enthalpy: $\chi_c = \frac{\Delta H_m}{\Delta H_m^0}$

Beer-Lambert law: $A = \epsilon bc$

Avogadro's number $N_A = 6.022\ x\ 10^{23} mol^{-1}$

Main functional groups on IR spectra: