Loughborough University

22MPB209

Materials Characterisation

Semester 2 2022/23 | 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.

<u>Help during the exam</u>

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.

1. (a) Calculate the lattice constant **"a"** of a fcc material from the X-ray diffraction pattern (Figure Q1a) obtained using Cu radiation with wavelength λ = 0.1541 nm. [6 marks]

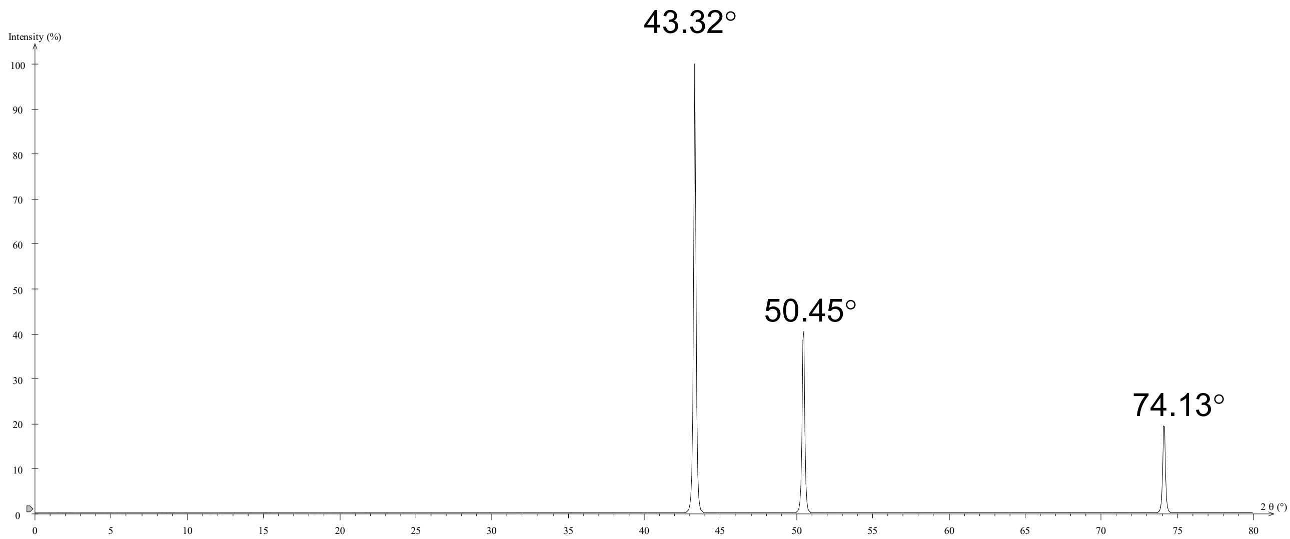

Figure Q1a: XRD pattern of a fcc material.

(b) Crystallographic texture can affect the properties of metals and alloys. In the case of electrical steel Fe-3%Si for manufacturing transformers, please answer the following questions:

(i) Name and state the orientation component of the favourable texture in Fe-3%Si steel. [2 marks]

(ii) With the aid of Figure Q1b, plot the favourable texture orientation component of Fe-3%Si steel in a 001 pole figure. [3 marks]

(iii) Explain the importance of the favourable texture of Fe-3%Si steel. [4 marks]

(iv) Suggest a processing route to obtain the favourable texture in Fe-3% Si steel. [2 marks]

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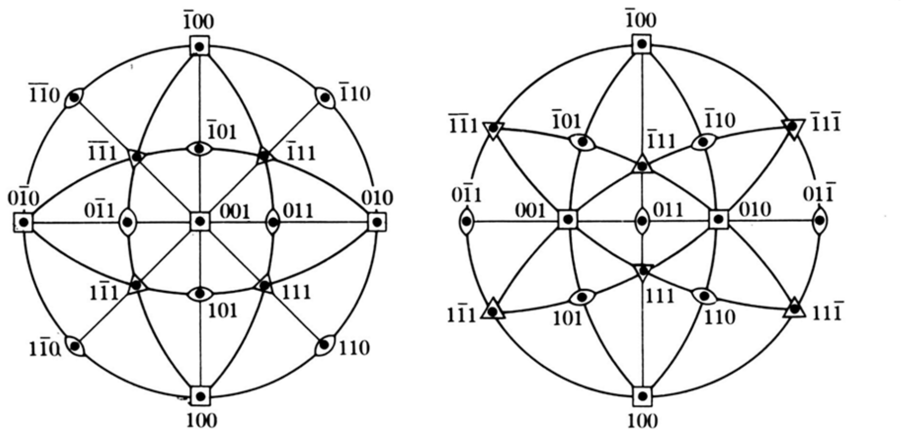
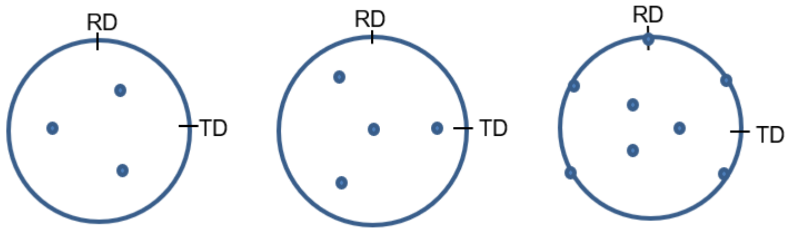
Q1 Continued/…



001 centred stereogram 011 centred stereogram

Figure Q1b: 001 and 011 centred stereograms

(c) (i) Name the following texture component. [1 mark]



100 pole figure 111 pole figure 110 pole figure

(ii) Sketch the {100}<110> texture component in a 001 pole figure. [2 marks]

2. (a) Ti-6Al-4V alloy contains two phases at room temperature namely α and β phases. α phase is Al-rich phase and β phase is V-rich phase. The microstructure of the polished Ti-6Al-4V alloy is shown in Figure Q2a.

(i) What scanning electron microscopy (SEM) imaging mode was used to obtain the image? [1 mark]

(ii) State why such image mode is used. [1 mark]

(iii) State what microstructural information of the sample you can get from those images. [2 marks]

(iv) Explain how the signals are generated. [4 marks]

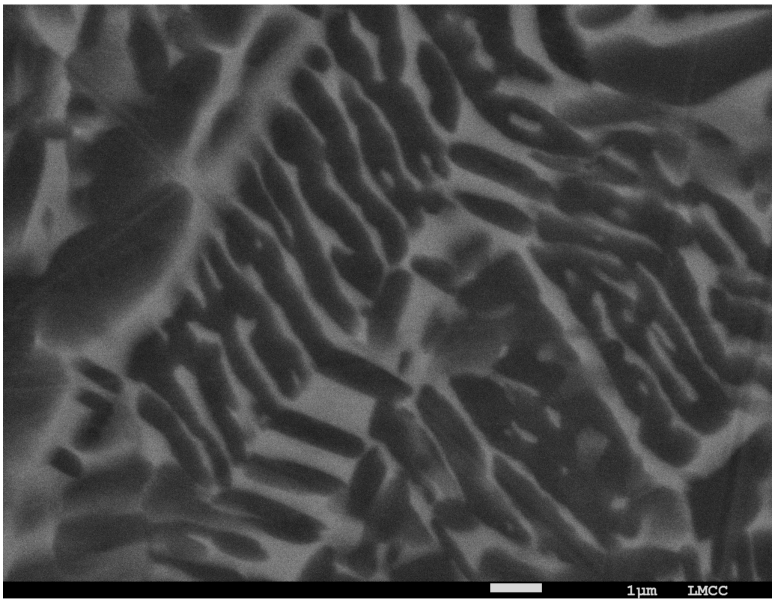


Figure Q2a: Microstructure of Ti-6Al-4V.

(b) In a SEM energy dispersive X-ray spectroscopy (EDS) experiment, the X-ray intensities from the specimen of unknown composition and from a standard containing 50% of Nickel and 50% Aluminium were shown in Table Q2b. What was the composition of the specimen? [6 marks]

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Q2 Continued/…

Table Q2b: Measured X-ray intensity for a known and unknown specimen.

	Specimen	Al Kα	Ni Kα
Known	Ni-50%Al	7943	14989
Unknown	Ni-x%Al	3124	19988

(c) In an EDX experiment, a strong peak was detected at 4.51 keV. Using Table Q2c below, explain what other peaks you would expect to see. [4 marks]

Table Q2c: Energies (eV) of X-ray emission lines

Element	Kα_1	Kα_2	Kβ_1	Lα_1	Lα_2	Lβ_1	Lβ_2	Lγ_1	Mα_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	

(d) EDS can be performed in transmission electron microscope (TEM). What are the advantages of using TEM-EDS over SEM-EDS and why? [2 marks]

3. (a) An X-ray scattering experiment on a sample semicrystalline poly (vinyl chloride) (PVC) reveals an orthorhombic unit cell ($\alpha = \beta = \gamma = 90°$) with a = 1.02 nm, b = 0.53 nm, and c = 0.50 nm. The density of the purely crystalline phase of this sample has been measured using buoyancy experiments as $\rho_c = 1.54\ g/cm^3$.

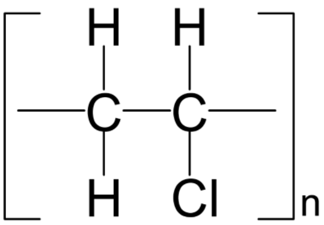

Figure Q3: Chemical formula of poly(vinyl chloride) (PVC)

(i) Calculate the number of vinyl chloride molecules in each unit cell by rounding to the nearest integer. [6 marks]

(ii) Do other PVC samples exist where there is a different number of vinyl chloride molecules in the unit cell? Explain why that would happen. [2 marks]

(iii) The density of fully amorphous PVC is $1.37\ g/cm^3$ and of fully crystalline is $\rho_c = 1.54\ g/cm^3$. What is the % crystallinity of a sample which has a density of $1.40\ g/cm^3$? [2 marks]

(b) For the following samples, choose what would be the best characterisation technique to determine its composition amongst, Raman spectroscopy, attenuated total reflection infrared spectroscopy, or transmission infrared spectroscopy. Explain the reasons for your choice.

(i) A blood sample; [2 marks]

(ii) An anticorrosion coating on a piece of pipe; [2 marks]

(iii) A chemical gas. [2 marks]

(c) Sketch a Storage modulus (E') versus Temperature graph that you would observe when performing a Dynamic Mechanical Analysis on a semicrystalline polymer, starting from below its glass transition and reaching above its melting temperature. [4 marks]

4. (a) Tocopheryl acetate is a compound of natural origin, used as additive for its antioxidant properties. It can absorb infrared (IR) radiation with a strong peak at 1759 cm^{-1}. A solution of this compound, of concentration 0.5 x 10^{-4} mol L^{-1}, is placed in a cuvette of 1 cm path length, and it is irradiated by IR light, with intensity I_0 = 100. The intensity read by the detector at 1759 cm^{-1} is I = 9.00. Calculate:

(i) The absorbance A at 1759 cm^{-1} [1 mark]

(ii) The extinction coefficient of tocopheryl acetate at this wavelength, ε_{1759} [2 marks]

(iii) The concentration of a solution of tocopheryl acetate showing A = 1.5 when placed in a cuvette of 2 cm path length; [2 marks]

(iv) Which functional group could be responsible for this specific absorption? [1 mark]

(b) 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 λ = 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, what value of 2θ would you expect the peak to shift to? [2 marks]

(iii) Bentonite shows a strong IR absorption at 3600 cm^{-1}: which functional group can you relate to this peak? [1 mark]

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Q4 Continued/…

(c) (i) Explain the main differences in the operation of Fourier Transform spectrometers and traditional spectrometers. [4 marks]

(ii) Why is it an advantage to not have slits in the Fourier Transform spectrometer? [1 mark]

(iii) Explain what the fingerprinting region of an IR spectrum is. [2 marks]

(iv) Why does Raman detect signals from homonuclear diatomic molecules whereas FTIR does not? [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 $2d \sin\theta = n\lambda$

Volume of unit cell: $V = abc \times \sin\alpha \times \sin\beta \times \sin\gamma$

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

Crystallinity: $\%\chi_c = 100 \times \frac{\rho - \rho_a}{\rho_c - \rho_a}$

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

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

Main functional groups on IR spectra: