Loughborough University

24MPP567

Advanced Materials Characterisation

Semester 1 2024/25 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 **ALL** questions.

A list of useful equations is given at the end of the exam paper.

1. (a) (i) Index each of the rings in the selected area electron diffraction pattern for a Copper sample (fcc) shown in Figure Q1a. [4 marks]

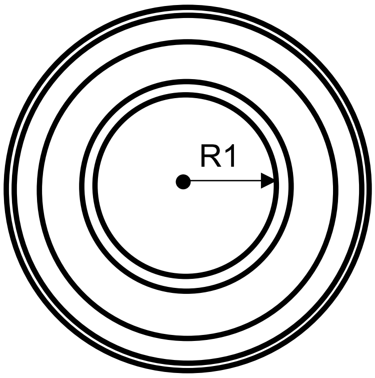

Figure Q1a

Electron diffraction ring pattern of Copper

(ii) Calculate the lattice parameter a of Copper given that $\lambda_{electron} = 0.00251$ nm, camera length $L = 1000$ mm, and $R1 = 12$ mm. Present your answer to four significant figures. [3 marks]

(b) Assuming that any peak broadening of the powder XRD pattern lines is due to the small crystal effect (Scherrer broadening) alone, calculate the following for approximately spherical particles (shape constant = 0.9):

(i) The peak broadening for crystallites with diameters of 50 nm, given that $2\theta = 90°$ and $\lambda = 0.15$ nm, stating your answer in degrees to 2 significant figures. [2 marks]

(ii) The crystallite size associated with a broadened X-ray peak at $2\theta = 160°$ with a full width at half maximum of $1.76°$. Give your answer to two significant figures.

[2 marks]

(c) A Mo powder was ball milled for different times and analysed by powder XRD. The results are shown in Figure Q1c. Explain exactly what causes the observed changes to the shape of the X-Ray diffraction peaks. [5 marks]

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Q1 Continued/…

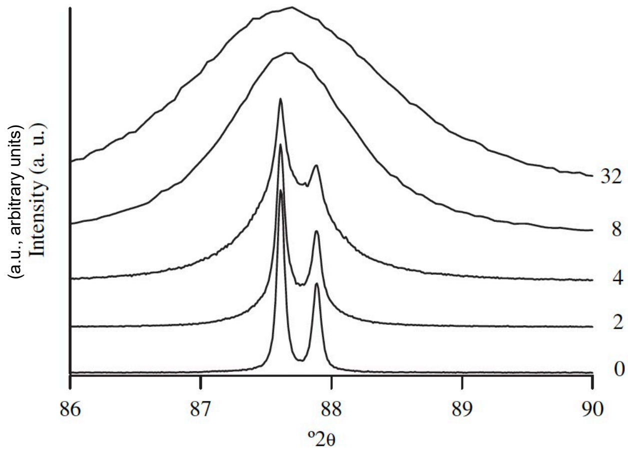

Figure Q1c

XRD line profiles of the Mo {220} reflection starting powder (0 h) also used as reference powder, and ball-milled powder for increasing ball-milling time: 2, 4, 8 and 32 h. [Adapted from DOI: 10.1080/14786430310001605029]

(d) Compare electron diffraction and X-ray diffraction by stating FOUR major differences between the techniques. Focus on their underlying principles and the type of information that they provide. [4 marks]

2. (a) (i) Which scanning electron microscopy (SEM)-based technique can be used to measure the microtexture (i.e., the preferred orientation) of an aluminum sample? [1 mark]

(ii) Explain how the signal used in part (i) is generated. [3 marks]

(iii) Describe how the sample can be prepared for the measurement of microtexture. Identify the most critical preparation step for your suggested technique and explain why such a step is critical to the suggested SEM technique. [4 marks]

(b) Suggest with reasons a technique that can be used to prepare a sample for transmission electron microscopy (TEM) extracted from the area indicated in Figure Q2b. [8 marks]

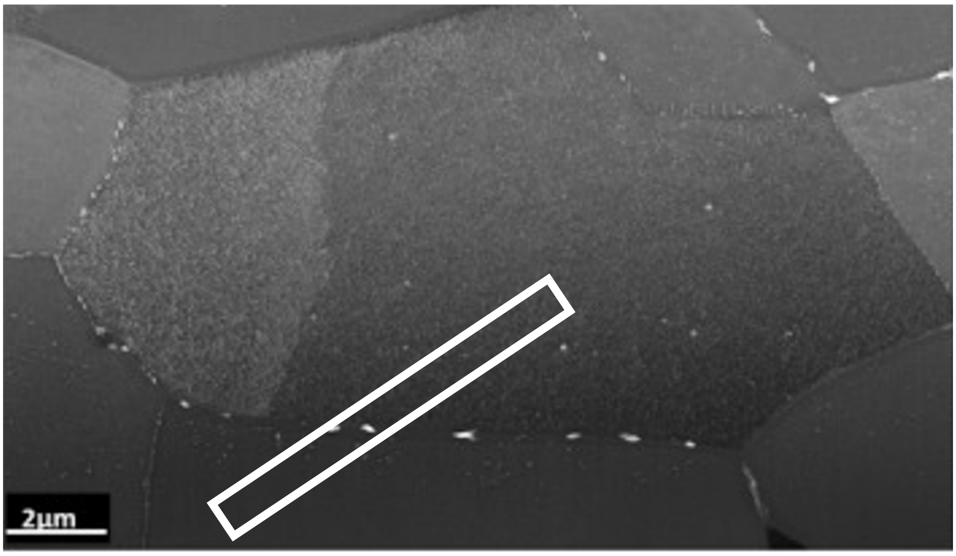

Figure Q2b SEM image of an annealed and polished aluminium sample. The selected area is marked by the rectangle.

(c) In SEM imaging of un-coated polymer particles, you notice that it is difficult to focus the image, and the image appears bright and blurry although these effects can be reduced by changing the acceleration voltage. Suggest the cause of these effects and methods to rectify the problem. [4 marks]

3. (a) Secondary Ion Mass Spectrometry (SIMS) can operate in both static (SSIMS) and dynamic (DSIMS) modes. Compare these two modes by discussing their operational principles, the type of information they provide, and their most suitable applications.

[6 marks]

(b) Explain why SSIMS is very effective in uniquely identifying, for example, a specific polymeric contaminant on a metal surface. [4 marks]

(c) With the aid of a schematic diagram, explain how the output signal is generated in X-ray Photoelectron Spectroscopy (XPS), and describe the applications of XPS resulting from this. [6 marks]

(d) Identify two factors that strongly influence the surface sensitivity of XPS and explain how these factors can increase the surface sensitivity of XPS. [4 marks]

END OF PAPER

Dr Y Tse

List of useful equations

Geometrical relations of diffraction pattern in TEM: $Rd = L\lambda$

Proportional relation for interplanar spacing: $d = \frac{a}{\sqrt{h^2+k^2+l^2}}$

Sherrer's formula: $t = \frac{K\lambda}{B\cos\theta_B}$