Modern Imaging, Spectroscopy and Diffraction Techniques

TIF 030 and FIM 150
October 20, 2010, 14:00
V Building

Aids: Formula sheets attached to the exam, "Physics Handbook", calculator, and writing tools.

Total marks available from exam: 30
Marks required to pass: 12

Question 1. Basic optical microscopy (3p)
Schematically draw the *imaging* path of an *upright* microscope with an *infinity corrected* objective and equipped with a *transmission* illumination system according to *Köhler*. Track two light rays from the lamp filament to the retina of the eye. Clearly indicate the object plane, the imaging planes and the diffraction planes.

Question 2. Optical microscopy- Contrast mechanisms (3p)
a) Demonstrate, using a simple sketch, that treating an object as a superposition of gratings leads to an estimate of the resolution limit (minimum grating constant) according to d = λ/2NA. (1 p)
b) Write down the function describing the frequency dependence of the dipole polarizability for an electron according to the simplest form of the Lorentz model and explain the different terms. Draw the real an imaginary parts of the function. (1p)
c) Based on the Lorentzian polarizability above, explain why window glass can refract visible light despite being nearly transparent. (1p)

Question 3. Modern optical microscopy (4p)

a) The distance dependence of FRET is described by the formula $E = 1 \big/ 1+(r/R_0)^6$. What does "*E*" mean and what determines "R_0"? (2p)
b) How can stimulated emission in STED microscopy improve the lateral resolution beyond the diffraction limit? (2p)

Question 4. SEM- Imaging. (3p)
(a) Describe spherical and chromatic aberration in a lens. (1p)
(b) What is the most critical parameter that limits the spatial resolution for secondary electron imaging, backscatter electron imaging and EDS-analysis respectively of a bulk specimen in the SEM (1p)
(c) Assume that you are recording an image of a planar specimen in the SEM using the backscattered electrons. The specimen contains Si and Ge and you know from earlier X-ray diffraction experiments that two phases with different composition and lattice parameters are present in the specimen. You observe two intensity

levels in the image, i.e. there are dark and bright domains in the image. What causes the difference in intensity levels? Assume that one of the intensity levels corresponds to pure Si. Is it the lower or the higher level? (1p)

Question 5. EDS- Bulk specimens. (2p)

(a) Draw a typical EDS spectrum including characteristic X-ray peaks and background for oxygen and iron in the interval 0-20 keV. (1p)

(b) You are to carry out EDS analyses of a bulk specimen and you need to optimise the spatial resolution. You need to consider the elements that you are to analyse and the spatial resolution. How do you choose the optimum acceleration voltage? Explain your answer. (1p)

Question 6. TEM- Imaging. (3p)

(a) Draw a schematic ray diagram that shows how a diffraction pattern and an image are formed in the TEM. Include the specimen and the objective lens in the diagram. All other lenses can be omitted. (1p)

(b) Describe the principle of and how Bright Field and Dark Field images can be obtained. Which aperture is used? (1p)

(c) How is energy filtered imaging achieved in a TEM? Explain your answer using a schematic diagram of an EELS spectrum. (1p)

Question 7. TEM- Diffraction. (3p)

The diffraction pattern in Fig. 1 is obtained for a gold crystal with the electron beam incident along the [110] direction in a TEM operated at 200 kV.

(a) Draw the Kikuchi lines corresponding to the 6 diffraction spots closest to 000 in Fig 1. (1p)

(b) Draw the Kikuchi lines for spot A when the crystal is tilted so that the Bragg condition for spot A is fulfilled. (1p)

(c) What happens to the pattern in Fig 1 if the acceleration voltage is reduced to 100 kV? (1p)

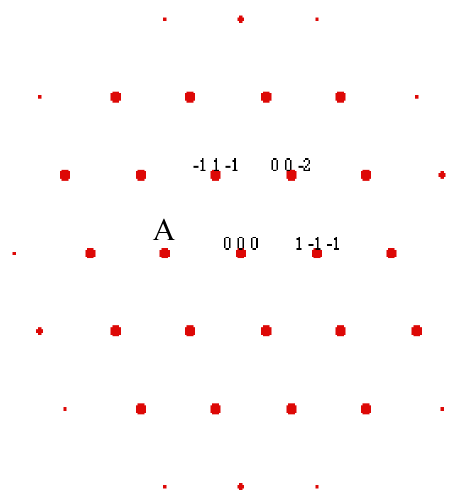


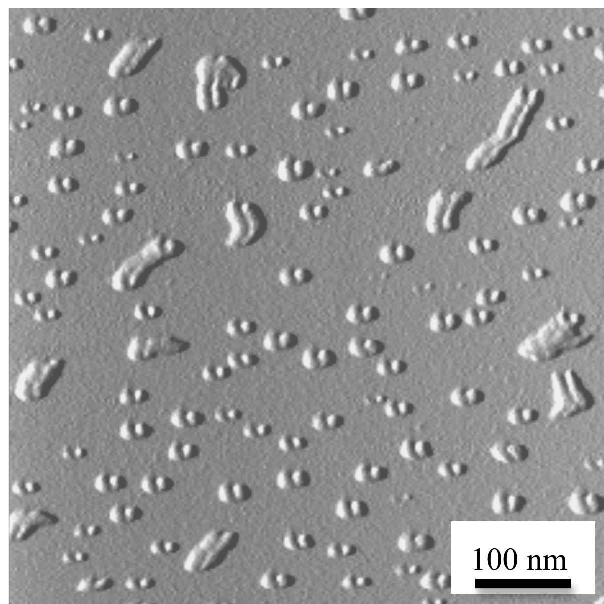
Fig. 1. Diffraction pattern from a gold crystal with the electron beam incident along the [110] direction.

Question 8. TEM- EDS. (3p)
An EDS-analysis is carried out in a TEM at 100 kV. The spectrum shows K-lines from Al, Ti and Mo. The number of counts summed over the energy ranges corresponding to the Mg, Ti and Zn lines are 33 700, 15 200 and 16 400 respectively. The background intensities are 190, 310 and 480 counts. The specimen thickness is 50 nm and the probe diameter is 0.5 nm.

(a) Which background intensity belongs to Mg, Ti and Zn? Explain your answer. (0.5p)
(b) Calculate the composition in weight per cent. Neglect the absorption. (2p)
(c) Assume that you would like to investigate if there is segregation of an element to a grain boundary. Explain the effect of specimen thickness and acceleration voltage on the spatial resolution of the analysis. (0.5p)

Question 9 SPM- Image/tip artifacts (3p)
SPM images might be affected by unwanted artifacts. This question deals with the sources of image artifacts and how they can be minimized or avoided.

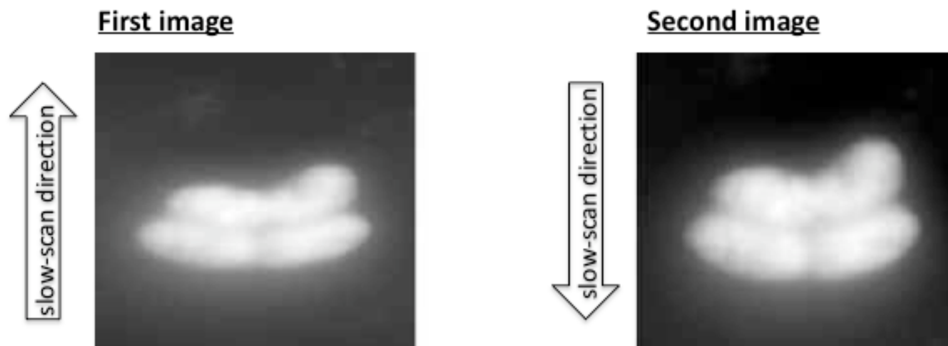
a) You are trying to image single-stranded DNA, which you have immobilized on a solid support, in constant-force contact-mode. From literature you know that the DNA strands in question are expected to be several hundred nanometers long, while their diameter is only a few nanometers. The image below is the result of one of your scans – unfortunately, it contains several tip-related artifacts. List three such artifacts and describe their origin! (1.5 points)



b) For each of the artifacts you identified in a) above describe how you could circumvent or minimize the respective artifact! (0.75 points).

c) You have acquired two sequential images of a cluster of bacteria (see below). The first image was scanned with the slow-scan direction from bottom to top, while the second

scan had the slow-scan direction from top to bottom. Although you have not changed any of the acquisition parameters, the two images are different. What could be the origin of this difference? (0.25 points)



d) Which measures can you take to minimize the artifact in c) above? List two strategies! (0.5 points)

Question 10. SPM- Force spectroscopy (3p)
Force spectroscopy is a powerful tool to directly measure intermolecular forces. This question discusses several aspects related to the acquisition and interpretation of force curves.

a) Describe the main steps required to acquire the raw data, which are necessary to construct a force-distance curve using an Atomic Force Microscope (AFM). (1 point)

b) Which quantities need to be calibrated in order to transform the raw data you get in a) to a force-distance plot? (0.25 points)

c) Which of the following three scanner artifacts might affect/disturb the acquisition of force-distance curves? 1) Creep – 2) Hysteresis – 3) Bow. Briefly motivate your answer. (0.75 points)

d) Could you use force spectroscopy to detect a vertical drift in your AFM? If yes, how would you go about? If no, why not? (Hint: "vertical drift" here shall imply that the tip is moving towards the sample surface in an uncontrolled way, i.e. without the piezo scanner being actuated). (0.5 points)

e) The cantilever spring constant is among the most critical parameters, which one needs to adjust carefully when acquiring force-distance curves. Depending on the application, one may face an upper and/or a lower limit on the "acceptable" force constant. Explain this statement by presenting two examples where the selection of force constant might be critical! (0.5 points)

Formula sheet

Element (A)	k_{ASi} (1) 100 kV
Na	5.77
Mg	2.07 ± 0.1
Al	1.42 ± 0.1
Si	1.0
P	
S	
Cl	
K	
Ca	1.0 ± 0.07
Ti	1.08 ± 0.07
V	1.13 ± 0.07
Cr	1.17 ± 0.07
Mn	1.22 ± 0.07
Fe	1.27 ± 0.07
Co	
Ni	1.47 ± 0.07
Cu	1.58 ± 0.07
Zn	1.68 ± 0.07
Ge	1.92
Zr	
Nb	
Mo	4.3
Ag	8.49
Cd	10.6
In	
Sn	10.6
Ba	

$\lambda = h / [2m_0eV(1 + eV/2m_0c^2)]^{1/2}$

$d_p = (d_g^2 + d_s^2 + d_d^2 + d_c^2)^{1/2}$

$r_{Sch} = 0.66\ C_s^{1/4}\ \lambda^{3/4}$

$n > (5/C)^2$

$2\ d_{hkl} \sin\Theta = n\lambda$

$b = 7.21 \times 10^5\ (\rho/A)^{1/2} t^{3/2}\ (Z/E_0)$

$$I \propto U\rho_s(E,r)e^{-2\frac{\sqrt{2m_e\phi}}{\hbar}d} \text{ with } \phi = \tfrac{1}{2}\left(\phi_{sample} + \phi_{tip}\right)$$