

## Modern Imaging Methods

TIF 030 and FIM 150

Monday January 11<sup>th</sup>, 2010, 14-18

M 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**

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### 1) Basic microscopy (3p)

a) Make schematic drawings of the illumination and imaging paths according to the Köhler design for an inverted optical microscope equipped with an infinity corrected objective. Mark the different conjugate planes and parts of the microscope. (2p)

b) What is approximately the smallest distance between two point-like objects that one can resolve using the highest resolution classical optical microscope available on the market and using visible light? (1p)

### 2) Contrast mechanisms in optical microscopy (4p)

a) Small particles scatter light according to the formula

$$\sigma_{scat}(\omega) \propto \omega^4 |\alpha(\omega)|^2$$

What is the meaning of the quantity  $\alpha$  and how does the formula explain why the sun becomes more red at sunset and sunrise? (2p)

b) Draw a simplified Jablonski diagram for a fluorophore and, using the diagram, describe what is meant by fluorescence quantum efficiency, vibrational relaxation, fluorescence lifetime and non-radiative decay. (2p).

### 3) Modern optical microscopy methods (3p)

a) Briefly describe at least two of the most important requirements for efficient FRET between two different fluorophores (1p)

b) What is the meaning of “confocal” in confocal microscopy and why is it important? (1p)

c) Based on a simplified ray-optics diagram, explain why an object is drawn to the laser focus in an optical tweezers. (1p).

**Question 4: SEM (3p)**

a) Assume that the image in Fig. 1 has been recorded using backscattered electrons. It shows two phases, i.e. ZnO and  $\text{Bi}_2\text{O}_3$ , in a ceramic material. The contrast reveals which phase is which. Which phase is ZnO? Explain your answer. (2p)

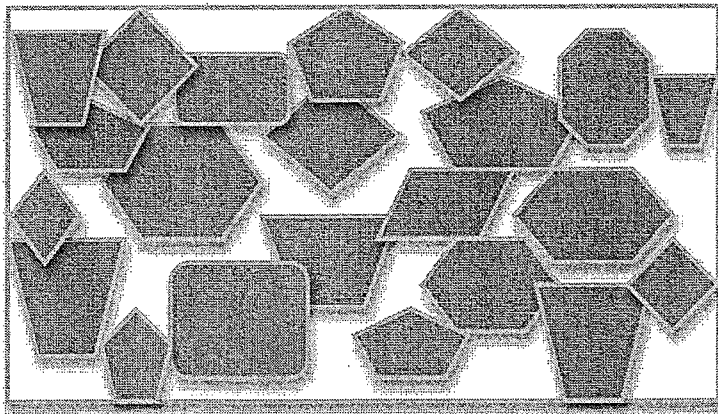


Fig. 1. Schematic illustration of an SEM image, obtained using backscattered electrons, of a planar surface.

b) You are recording one image using the secondary electron and one image using the backscattered electrons. Both images are recorded using identical settings of the SEM. The only difference between the two images is the detector that has collected the signal. Which image has the best spatial resolution? Explain your answer. (1p)

**Question 5: EDX (2p)**

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

b) You are recording a concentration profile across a phase boundary in a bulk specimen using EDS in a SEM. Will the profile be different at a high acceleration voltage compared to a low voltage? If yes, what is the difference between the two profiles? Explain your answer. (1p)

**Question 6: Diffraction (3p)**

a) Draw a schematic diffraction pattern for an amorphous area. Explain the shape of the pattern. (1p)

b) Draw the [110] diffraction pattern for a BCC crystal. Index the pattern. (1p).

c) Draw the Kikuchi lines for the inner 8 diffraction spots for a case when the electron beam is incident along the zone axis. (1p)

**Question 7: TEM (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) Draw the diffraction pattern for the area in the middle of the high TEM resolution image in Fig. 2. Explain how you obtain it. (1p)
- c) How can the spatial resolution be made poorer using the objective aperture? Explain your answer. (1p)

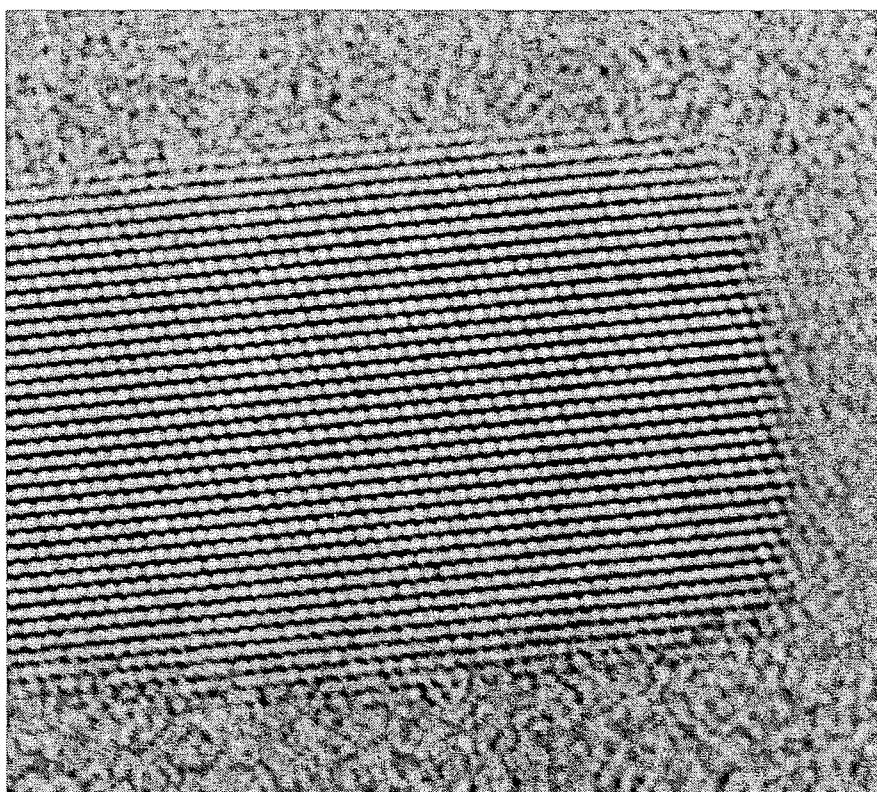


Fig. 2. A high resolution TEM image of an Al thin film. The film is 15 nm in thickness.

**Question 8: Analytical EM (3p)**

An EDS-analysis is carried out in a TEM at 100 kV. The spectrum shows K-lines from Ca, Ni and Mo. The number of counts summed over the energy ranges corresponding to the Ca, Ni and Mo lines are 22 300, 6 700 and 17 200 respectively. The background intensities are 150, 200 and 570 counts. The electron probe diameter is 0.5 nm.

- a) Which background intensity belongs to Ca, Ni and Mo? 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. You see from the contrast that the specimen thickness is varying along the boundary. How would you choose the position for the concentration profile to improve the spatial resolution of the measured concentration profile across the boundary? Explain your answer. (0.5p)

**Question 9 SPM- drift and noise issues in SPM (3 points)**

Drift and noise are serious issues, which need to be carefully considered whenever acquiring and interpreting SPM images.

a) What is the most common source of drift in SPMs? Describe two strategies to get around (or live) with this problem! (0.75 points)

b) Assume that you have a vertical drift in your scanning tunneling microscope (STM), which drives the tip away from the sample at a constant speed of 0.1nm/sec; assume that there is no lateral drift. Will you be able to run the STM in constant-height and/or constant-current mode despite this drift? Decide for each of the two modes if this is possible. If yes, describe how this drift will affect your image(s); if no, motivate why you think it is not possible. (1.0 points)

c) You observe periodic features in one of your SPM images, which you suspect to be related to electronic noise rather than true surface features. In order to check whether you are indeed looking at electronic noise, you reduce the scan size by 50% while keeping the scan speed constant. How will this change of scan size affect your image if the periodic features are due to noise? (0.25 points)

d) The schematic below illustrates a simple setup, which helps to reduce noise in SPM images. Will this setup effectively remove acoustic noise? If yes, why? If no, how could you reduce acoustic noise instead? (0.5 points)

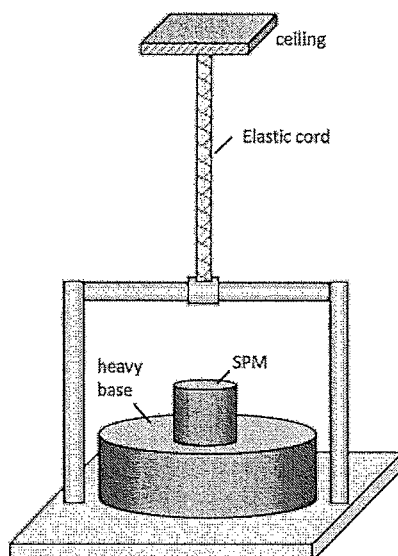
e) You would like to build a setup (according to the schematic below), which removes mechanical vibrations of frequencies larger than 0.3 Hz. Calculate the minimum height of a room, which is able to house your setup! (0.5 points) (Hints: the resonance frequency of the setup below is

$$f_0 = (1/2\pi) (k/m)^{1/2}$$

where  $k$  is the spring constant of the elastic cord and  $m$  is the total mass of base and SPM. In equilibrium the following relationship holds:

$$k\Delta l$$

where  $g = 9.8\text{ms}^{-2}$  and  $\Delta l$  is the elongation of the cord.)



**Question 10- Atomic Force Microscopy – AFM (3 points)**

a) Describe three main aspects of your choice concerning the principle of an atomic force microscope operated in contact mode. (0.75 points)

b) What are the two major characteristics, which distinguish contact-mode from tapping-mode AFM? (0.5 points)

c) What is the major distinguishing characteristic when comparing scanning tunneling microscopy (STM) to AFM? (0.25 points)

d) In constant-force contact mode, one sometimes records significant height variations when imaging samples, which are known to actually exhibit a completely flat topography. What could be the origin of these “artificial” height variations? List two possible origins. (0.5 points)

e) “Creep” is a scanner artifact, which is very prominent in AFM images acquired with older, so-called “open-loop” AFMs. What is creep, and by imaging which kind of sample can you easily detect creep? (0.5 points)

Decide whether the following tasks can be tackled with AFM, and briefly motivate your answer!

f) You would like to measure the exact spacing between (identical) metallic nanoparticles, which are arranged in a regular, orthogonal lattice on a glass substrate. The diameter of the dots is roughly 2-3nm and their spacing is expected to be in the 70-80nm range. (0.25 points)

g) You would like to investigate the (comparably fast) diffusion of oxygen atoms on a Ru(0001) single crystal surface (very flat) at slightly elevated temperature. The size of an oxygen atom is on the order of 6Å. (0.25 points)

## Formula sheet

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

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

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

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

$$n > (5/C)^2$$

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

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

$$I \propto U \rho_s(E, r) e^{-2 \frac{\sqrt{2m_e(\phi - E_F)}}{\hbar} d}$$

Thermal expansion coefficient of piezo materials:  $4.7 \cdot 10^{-6} \text{ K}^{-1}$