EXAM Advanced Microscopy – SPM and EM

January 27, 2020

- This exam will test your knowledge of scanning probe microscopy and electron microscopy
- This exam covers scanning probe microscopy (40% of the total number of points) and electron microscopy (60% of the total number of points)
- Write your answers to the SPM (question 1) and EM parts (questions 2 to 4) on different sheets of paper.
- Question 4 is a bonus question (10 points). The maximum number of points is 100, though.
- Write your name on each answer sheet.
- Pay attention to your hand writing. If we cannot read your answers, we cannot award points.

Question 1. Scanning Tunneling Microscopy (40 points)

a) Why should you use an atomically sharp tip to obtain STM images? (4 points)

b) What is the difference between the constant-height and the constant-current mode in scanning tunneling microscopy? (4 points)

c) A scanning tunneling microscope can be used to obtain information about the electronic properties of a material using differential conductance spectroscopy and current-distance spectroscopy What information do these two types of spectroscopy give you? (4 points)

d) Figure 1 below shows an STM image of a Si surface. The fast scan direction was from left to right, the slow scan-direction from top to bottom. Note the strong apparent bending of the step edges indicated by the white arrows. Propose an explanation for this bending. (6 points)

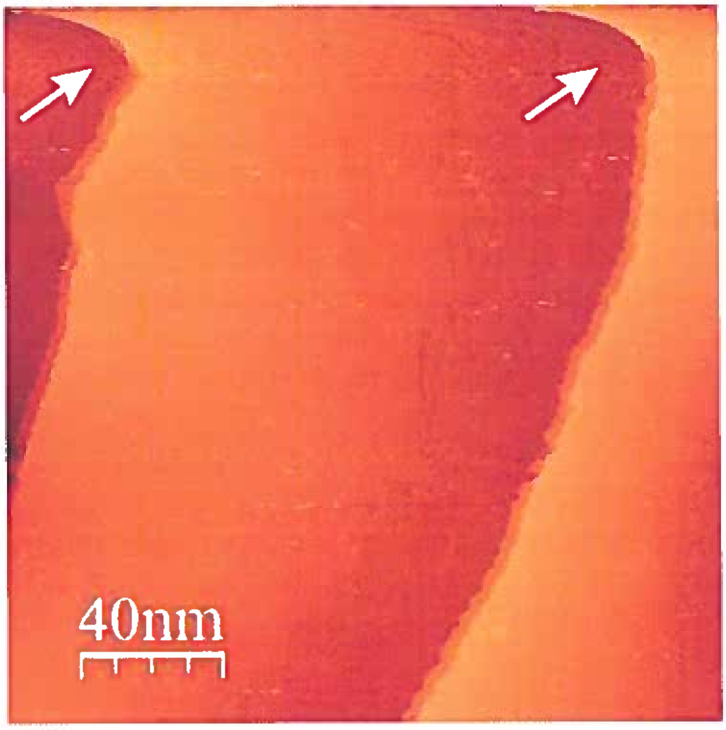

Figure 1. STM image of a Si surface. Taken from Scanning Probe Microscopy, Bert Voigtländer, Springer, 2015.

e) To image atoms, the relative motion between tip and sample should be on the order of 1 pm. Typical building vibrations have amplitudes of 0.1 µm. Explain how the required vertical stability of the tip-sample junction can be achieved. (4 points)

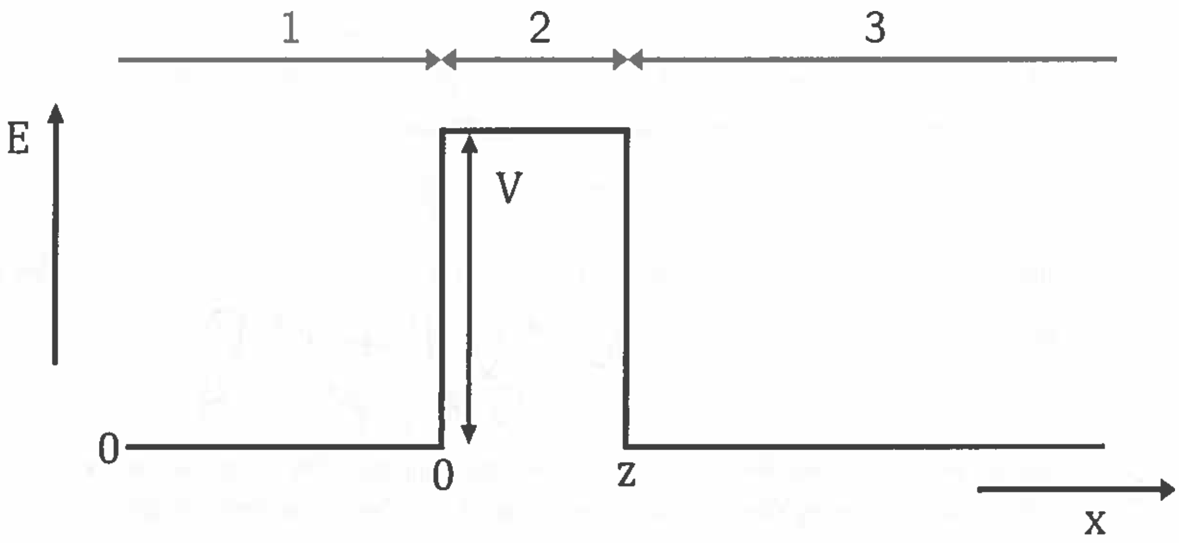

Figure 2. Electron tunneling through a rectangular barrier.

f) To understand the exponential dependence of the tunnel current on distance, you studied the transmission of a particle through a rectangular barrier (see Figure 2 above). You solved the Schrödinger equation for each region separately, and required that the wave function is continuous at the boundaries between the regions. The following wave functions were used

$$\psi_1(x) = A_1 e^{ik_1x} + B_1 e^{-ik_1x}$$
$$\psi_2(x) = A_2 e^{k_2x} + B_2 e^{k_2x}$$
$$\psi_3(x) = A_3 e^{ik_3x}$$

The transmission through the barrier, was shown to be

$$T = \frac{|A_3|^2}{|A_1|^2} = \frac{16E(V-E)}{V^2} e^{-2k_2z}$$

with k_2 the wave vector in region 2. In the following, we will set the amplitude of the wave function to unity, i.e. $|A_1|^2 = 1$. We used the ad hoc assumption that T was proportional to the flux density of the particles (the electric current). In the following, we will explore this in more detail. From fluid dynamics, one can derive the following expression that relates a change in particle density to the density of flow of particles:

$$\frac{\partial \rho(x,t)}{\partial t} + \nabla j(x,t) = 0$$

where in quantum mechanics $\rho(x,t) = \psi(x,t)\psi^*(x,t)$ and $j(x,t)$ is the probability density current of one particle (∇ is the derivative with respect to x). $j(x,t)$ can be converted into electric current: multiplying $j(x,t)$ by the number of particles yields the flux of particles through the barrier, and subsequent multiplication by the particle charge yields the electric current density. Finally, if divided by the area of the barrier, an electric current results.

✓ 6

Use the *time-dependent* Schrödinger equation, $i\hbar\frac{\partial}{\partial t}\psi(x,t) = \left(-\frac{\hbar^2}{2m}\nabla^2 + V(x,t)\right)\psi(x,t)$, to show that the following expression holds for the probability current density

$$j(x,t) = \frac{-i\hbar}{2m}\left(\psi^*\underline{\nabla}\psi - \psi\underline{\nabla}\psi^*\right)$$

Hint: evaluate $\frac{\partial\rho(x,t)}{\partial t} = \frac{\partial\psi\psi^*}{\partial t}$ and rewrite the time dependent Schrödinger equation to find an expression for $\frac{\partial\psi}{\partial t}$ and $\frac{\partial\psi^*}{\partial t}$

(10 points)

g) Use the expression determined under f) to calculate the probability current density associated with electron tunneling through a rectangular barrier, i.e. determine an expression for $j(x,t)$. Is it indeed proportional to the transmission to the barrier? (8 points)

Hint: use the explicit form of the wave function in region 3.

Question 2. Physics of electron microscopy (35 points)

Useful constants:

Planck constant: $h=6.626\cdot10^{-34}$ Js
electron rest mass: $m_e=9.109\cdot10^{-31}$ kg
electron charge: $e=1.602\cdot10^{-19}$ C

✗ a) Calculate the acceleration voltage required to obtain electrons with a wavelength of 1.0 picometer. Assume that the problem can be treated non-relativistically. (5 points)

~ b) In reality, the resolution of electron microscopes is always much lower than the wavelength of the electrons. Which factors in the contrast transfer function $H(u)=A(u)E(u)B(u)$ are causing this effect? (5 points)

✓ c) Very thin samples can be considered as a pure phase object. What is meant by a 'pure phase object'? (5 points)

~ d) Mention the type(s) of contrast that can be found in BF-TEM imaging, and the type(s) of contrast that can be found in STEM-HAADF imaging. Why is STEM-HAADF the most popular imaging mode for materials science studies? And why is BF-TEM the most popular imaging mode for life sciences studies? (10 points)

✓ e) For the following four analytical tools, mention whether they mainly rely on *elastic* or on *inelastic* interactions of the primary beam with the specimen. A simple "elastic" or "inelastic" suffices as answer; an explanation does not need to be given. (5 points, all answers need to be correct).

A. EDX
B. EELS
C. Electron diffraction
D. Cathodoluminescence

f) Which of the four methods mentioned under (e) can be used to measure the band gap of semiconductors? No explanation required. (5 points)

Question 3. Interpreting EM images (25 points)

a) Figure 3 shows a BF-TEM image of a crystalline MgO atomic lattice containing CdSe nanocrytals. Both crystals are have a cubic crystal structure (rocksalt) and are in the same orientation. The superposition of the different lattice spacings of MgO and CdSe generates an interference pattern which is called a moiré fringe.

Figure 3. High-resolution TEM image of MgO containing CdSe nanocrystals. [M.A. van Huis *et al.*, *Acta Materialia* **53** (2005) 1305-1311].

The (longer) spacing of the moiré fringes is related to the atomic lattice spacing of MgO and CdSe as follows:

$$\frac{1}{d_{\text{fringes}}} = \left|\frac{1}{d_{\text{MgO}}} - \frac{1}{d_{\text{CdSe}}}\right|$$

From the figure, it is clear that exactly 4 MgO lattice fringes fit into one moiré fringe. The lattice spacing of MgO is d_{MgO}=2.11 Å. Derive the lattice spacing of CdSe (d_{CdSe}). (10 points)

Hint: Mind the absolute sign "|..|". Lattice spacings are typically between 2 and 3 Å.

b) As the BF-TEM image of Figure 3 is a projection, one does not know whether the CdSe nanocrystals are embedded, or on top, or at the bottom, of the MgO film. What would be a good way to obtain also depth-resolved information? (5 points)

c) Consider Figure 4. It shows a so-called supraball consisting of silica-coated Au nanorods. Using a focused ion beam (FIB), thin planar layers can be milled away, and after removal of every layer a SEM image is taken. From this stack of depth-resolved SEM images, a 3D reconstruction can then be obtained.

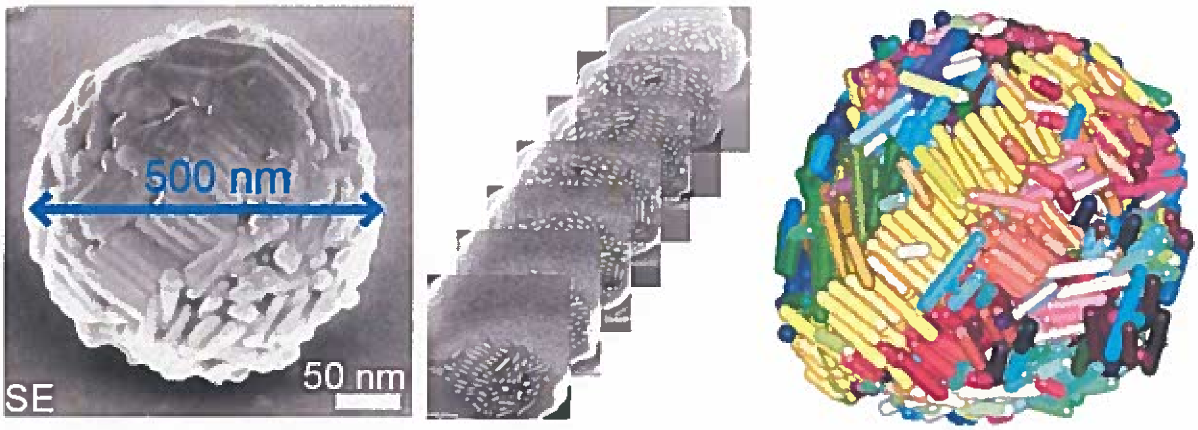

Figure 4. Left: SEM image of a supraball, consisting of silica-coated Au nanorods. Middle: sequence of images obtained by milling away thin slabs with the FIB and subsequent imaging by SEM. Right: 3D reconstruction of the object. [J.E.S. van der Hoeven, *et al.*, *Nanoscale* **11** (2019) 5304-5316].

Give a general definition of tomography. Does the 'slice-and-view' method shown in Figure 4 qualify as tomography? And would the Crowther criterion apply here? (10 points)

Question 4 (BONUS: 10 points. The maximum number of points is 100, though).

Consider a cubic PbSe nanocrystal with a size of 10 nm. The nanocrystal is covered with oleic acid molecules. These are linear molecules (long carbon backbone), with chemical formula $C_{18}H_{33}O_2$. They have their polar head attached to the surface and their non-polar head pointing outward.

The nanoparticles are imaged using STEM-HAADF. Compare the Z-contrast intensity from a volume of 1 cubic nanometer of PbSe to the intensity of a volume of 1 cubic nanometer of molecules. Give the ratio of the two. Will the layer of surfactant molecules be visible in the STEM-HAADF image?

Data:

Atomic numbers of the elements: $Z_{Pb}=82$, $Z_{Se}=34$, $Z_C=6$, $Z_H=1$, $Z_O=8$.
Atomic density of Pb and Se: $n_{Pb}=n_{Se}=18\ \text{nm}^{-3}$
The molecules are 2 nm long and their surface coverage (surface density) is 5.0 molecules nm^{-2}.

Hint: as an intermediate step, first calculate the intensity from a single oleic acid molecule.