EXAM COVER PAGE

COURSE: **Advanced Superstructures: Scattering and Microscopy**
COURSE CODE: **SK-BASSM**
EXAM: FINAL
TIME: 31-01-2022 from 9.00h to 12.00h
LENGTH:2h30m (plus 25m extra time=2h55m)
PLACE: Educatorium Megaron

<u>IMPORTANT</u>
- PLACE YOUR ID CARD (WITH PHOTO) ON THE TABLE
- DO EACH PART (SCATTERING, EM, OM) ON A SEPARATE SHEET
- WRITE YOUR NAME AND STUDENT NUMBER ON EVERY ANSWER SHEET

EXAM SPECIFICS
- Each part of this exam (scattering, OM) counts for 25% of the final grade.
- The distribution of points is given for each question.
- When answering the questions, please take the following into account: answers in English or Dutch are allowed for scattering. EM must be answered in English. **Please ensure that your handwriting is readable!**

PERMITTED EQUIPMENT
calculator

GOOD LUCK!

Andrei Petukhov, Friedrich Förster

GENERAL EXAMINATION RULES
- You are not allowed to leave the exam room in the first 30 minutes. Latecomers are allowed in up to 30 minutes after the start time.
- All electronic equipment needs to be switched off (including mobile phones), with the exception of electronic equipment allowed by the examiner.
- Your coat and closed bag are placed on the ground.
- Raise your hand when you need to go to the bathroom. 1 person at a time. Place your mobile phone visibly on your table just before you go.
- Raise your hand if you have a question about the exam, or need extra paper, etc.
- Not following the instructions of the surveillant (examiner) can lead to exclusion from the exam.
- When fraud is suspected the surveillant (examiner) will collect evidence of the fraud, will file a report, and will allow you to finish your exam. The examiner will send the evidence, report and the exam to the Exam Committee within one working day and will inform the Education Manager of the suspicion of fraud. See also EER article 5.14.
- Upon receiving your result you can request the examiner for access to your graded exam. Possibly, a collective meeting will be organized.

SCATTERING [100 points total]

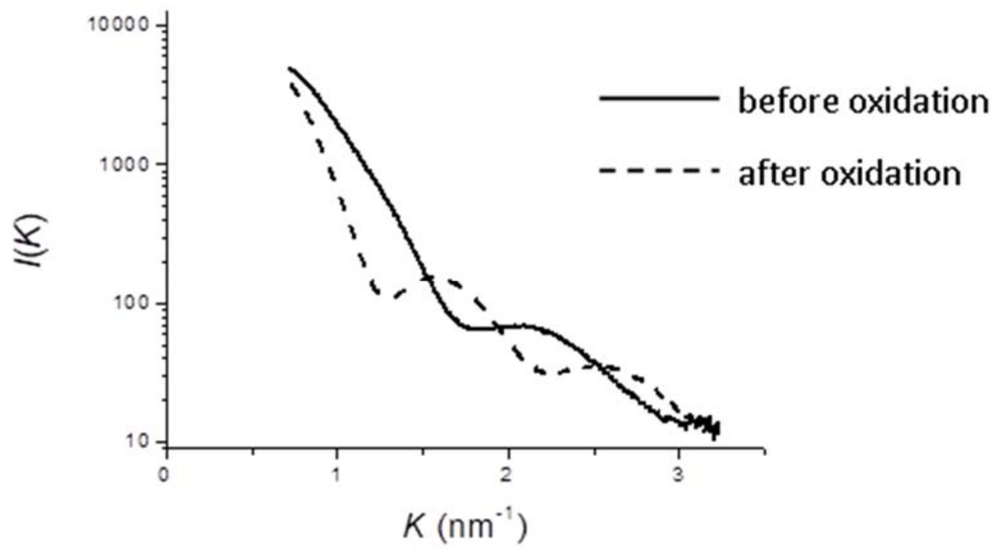
Please use separate pieces of paper for EM and Scattering.
Don't forget to write down your name on every piece of paper!
For scattering you can write your answers either in English or in Dutch.

1. Explain the idea of contrast variation using the H/D substitution. Is it used in light, x-ray or neutron scattering? Why is it useful? [10 points]

2. What is the difference between the Guinier range and the Porod range? What type of information can be obtained from the measurements in these two cases? [10 points]

3. What is the radius of gyration? How can it be determined from the scattering data? [10 points]

4. The figure below displays the intensity of the small-angle x-ray scattering $I(K)$ as a function of the scattering wavevector K for iron nanoparticles before (solid line) and after (dashed line) oxidation.



a). The data is taken between K_{min} = 0.8 nm^{-1} and K_{max} = 3.2 nm^{-1}. What range of scattering angles θ was used in this experiment? The x-ray wavelength was λ = 0.1 nm. [15 points]

b) Do the particles become larger or smaller after the oxidation? Clearly motivate your answer! [10 points]

c) Give a rough estimate of the particle diameter *after* oxidation. [15 points]

d) Explain why the minima in the curve are not very deep. Give two possible reasons [10 points]

5. Imagine that one studies the same system (as above) using light scattering.
   a) What range of K can be reached with light having the wavelength of $\lambda = 628$ nm? Assume that the average refractive index in the sample is n = 1.5 and that the scattering angle can be varied between 5 and 180 degrees. [10 points]

   b) Can light scattering supplement the x-ray data shown above? Are there advantages/disadvantages in using light? Describe at least 3 advantages or disadvantages of using light in this case. [10 points]

<u>Possibly useful relations</u>

$$2d \sin\frac{\theta}{2} = n\lambda$$

$$K = \frac{4\pi}{\lambda} n \sin\frac{\theta}{2}$$

$$P(K) = \left(3\,\frac{\sin Ka - Ka\cos Ka}{(Ka)^3}\right)^2$$, minima at Ka = 4.49, 7.72, 10.90, …

$$R_g{}^2 = \frac{\sum_i b_i(\mathbf{r}_i - \mathbf{R}_b)^2}{\sum_i b_i}$$

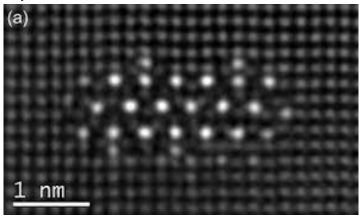
$$I_{\mathrm{sc}}(K) \cong I_{\mathrm{sc}}(K \to 0)\exp\left(-\frac{1}{3}K^2 R_g{}^2\right)$$

$$I_{\mathrm{sc}}(K) \cong \frac{A}{K^4}$$

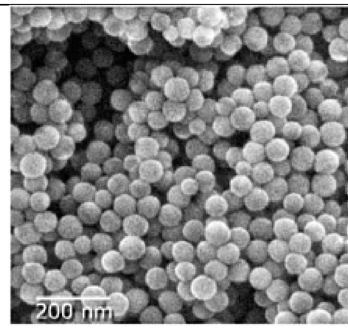
$$I_{\mathrm{sc}}(K) \cong \frac{A}{K^n}$$

Electron microscopy (total of points: 34)

a)

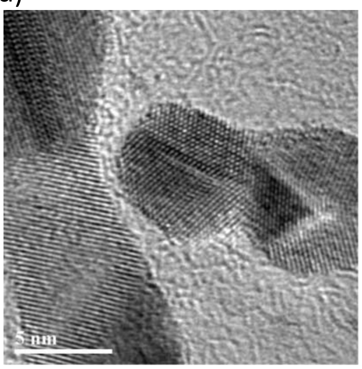


b)

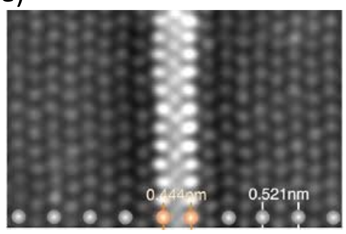


c)

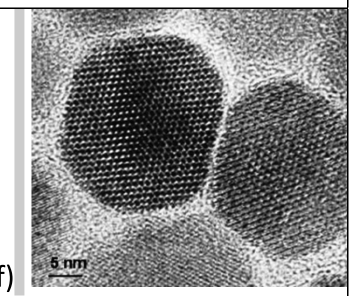
d)



e)



f)



1. By which of those three methods have the images been acquired: Bright-field phase contrast transmission electron microscopy (BF-TEM), dark field scanning TEM (DF-STEM), or scanning electron microscopy (SEM)? (3 P)

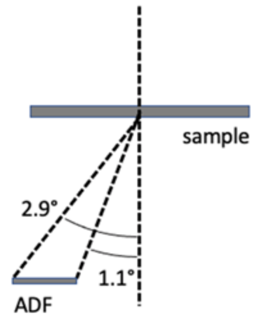
2. In the lecture FIB milling for sample preparation was covered. In the guest lecture from Jessi van der Hoeven FIB was used in combination with SEM to obtain a 3D image of the sample ('slice and view'). In the lecture, the FIB/SEM was also used to prepare thin slices ('lamellae') of thick samples to image them in a TEM. Please list each two advantages for Slice-and-view and lamellae TEM, respectively. (4 P)

3. The new materials science transmission electron microscope (TEM) in Utrecht can be operated at voltages ranging from 80 kV to 300 kV. You want to investigate a 300 nm thick organic specimen. At which voltage would you operate the microscope for this sample and why? (2 P)

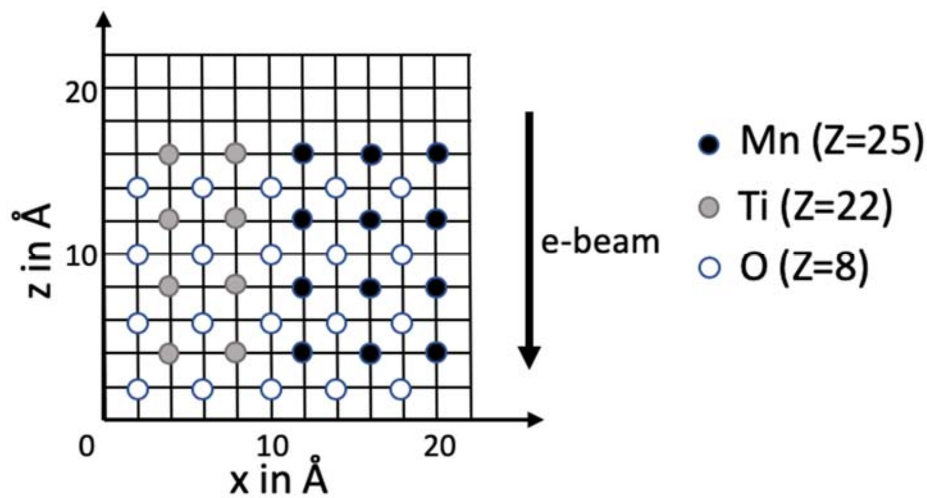
4. Now you want to use the same TEM to image a very thin (~20 nm) slice of metal. Please provide one reason to operate the TEM at 80 kV and one reason arguing for 300 kV. (4 P)

5. A transmission electron microscope (TEM) is equipped with an annular dark field (ADF) detector, which covers an angular range of $1.1° < \theta < 2.9°$ (see schematic below). The TEM is operated at 57 kV (corresponding wavelength of the electrons is 5×10^{-12}

m). The sample is crystalline. Which (approximate) range of Bragg reflections will be recorded (characterized by lattice spacings *d* in Å)? (2 P)



6. You want to image a sample with a transmission electron microscope (TEM). Below is a simplified two-dimensional representation of the part of the sample that is imaged (a slice of the real 3D scenario): in the field of view Mn (Z=25), Ti (Z=22) and O (Z=8) are positioned in columns. The image created by the electron beam is then one-dimensional along the x-axis (corresponding to a line of the complete 2D image). (Total 6 P)



   a. Suppose you image with the TEM in high-angle annular dark field (HAADF) STEM mode. What would the approximate relative intensities I_{HAADF} of columns imaged on the detectors be? Please assign the relative values of the three column types for the different elements. (2 P)

   b. Now you calculate the Fourier transform of the acquired image. You observe a pattern with maxima in the amplitudes. To which spatial frequencies (in $Å^{-1}$) do these peaks correspond? (2 P)

   c. Now you image the same sample in bright field mode (BF-TEM) with phase contrast induced by operating the TEM with a slight defocus. You also Fourier transform the image and compare the amplitudes to those of the HAADF image. What do you observe? (2 P)

7. You now want to verify the different elements from question 6. You exploit the difference in inelastic scattering to distinguish Mn and Ti using spectroscopic approaches. Suppose the incident electron transfers energy to an electron in the K-shell for Mn and Ti, respectively. The work function is this K-shell electron is E_K (Total 9 P)
   a. Please describe briefly
      i. the principle of energy-dispersive X-ray spectrometry (EDX or EDS) (2 P)
      ii. the shape of the observed local maximum/maxima in the spectrum (1 P)
      iii. how it/they relate(s) to E_K for above inelastic scattering event. (1 P)

   b. Please describe briefly
      i. the principle of energy electron loss spectroscopy (EELS) (2 P)
      ii. the shape of the observed local maximum/maxima in the spectrum and (1 P)
      iii. how it/they relate(s) to E_K for above inelastic scattering event. (1 P)

   c. You now want to detect oxygen as well. Would you prefer EELS or EDX? (1 P)

8. You now aim to get a 3D image of the sample from question 6 (in the simplified cartoon the 2D slice) using electron tomography. (Total: 4 P)
   d. You image the sample in the TEM using a perfect specimen holder that allows tilting the sample from -90° to +90°. 2D images of the sequentially tilted sample are acquired using HAADF. In the resulting reconstruction, what would the relative intensities of Mn, Ti and O be (see also question 1)? (1 P)

   e. Unfortunately, the perfect specimen holder is broken, and you repeat your measurement with a holder that can only tilt -70° to +70°. You Fourier transform the resulting reconstruction and compare it to the previous one obtained using the perfect holder. What is the difference between the two Fourier transforms? (2 P)

   f. How does the *real-space* reconstruction from the imperfect holder differ qualitatively from the reconstruction obtained with the perfect holder? (1 P)