Multidisciplinary approach to NPs characterization

Doctoral School, MSE-674

Exam, 09.01.2025

Name _____ Surname _____ Sciper # _____

Correct: 5; Wrong: -1; No answer: 0. Minimum grade: 60 points.

Multiple correct answer possible; Open book; 30 minutes.

Section 1

Q1: What values must be indicated when reporting a PSD?

✓ an average size value with the reference base (such as N, V)

☐ the powder density

☐ the crystallographic phase

✓ a span value

☐ an average size value

Q2: What are the physicochemical parameters that influence colloidal stability?

✓ dielectric properties of the solid

✓ temperature

✓ ionic strength

✓ pH

✓ particle size

Q3: What is the physical phenomenon considered in particle-particle electrostatic interaction in solution?

☐ attractive Coulombic attraction

☐ repulsive Coulombic repulsion

✓ repulsive overlap of EDL

✓ osmotic pressure

☐ overlap of polymeric chains

Q4: Where is the zeta-plane located?

☐ within the solid surface

☐ on the solid surface

☐ at the end of the Stern layer

✓ somewhere in the diffuse layer

☐ in the solution bulk

Q5: What is the physical phenomenon considered in photon correlation spectroscopy to evaluate the PSD?

✓Brownian motion

☐ gravitation sedimentation

✓light scattering

☐ surface potential

☐ surface charge density

Section 2

Q6: in X-ray Powder Diffraction the position of diffraction peaks

✓ Is sensitive to the X-ray wavelength.

☐ Is sensitive to the particle size.

☐ Is sensitive to the crystallite size.

✓ Is sensitive to the unit cell dimensions.

☐ Is sensitive to the different elements.

Q7: Peak broadening originates from

☐ Particle size

✓ Crystallite size

☐ Low density of the material

✓ Setup geometry

✓ Microstrain

Q8: Scherrer's equation

☐ Can be applied on high-angle peaks only.

☐ Is valid only for Lognormal crystallite size distributions.

☐ Is exact if the instrumental resolution contribution is deconvoluted from the peak broadening.

✓ Assumes microstrain-free crystallites

☐ Relates the peak height to the particle size

Q9: Microstrain

□ Is the same as strain on microscopic crystallites.

✓ Can be caused by defects.

□ Is present on nanoparticles only.

□ Cannot be evaluated together with crystallite size.

✓Causes a peak broadening with a different theta dependency with respect to crystallite size.

Q10: When doing a refinement

□ The end of the refinement is reached when R_{wp} is smaller than 3.

□ It is irrelevant to have a sensible starting model.

✓ You always get a number.

□ You should always keep peak-shape functions fixed.

✓ You should know the instrumental contribution to evaluate correctly the crystallite size.

Section 3

Q11: What determines the intensity of scattered radiation in SAS?

☐ The wavelength of the incoming radiation

✓ The contrast between the scattering particles and the surrounding medium

☐ The temperature of the sample

☐ The polarization of the radiation beam

☐ The size of the sample holder

Q12: What is a key advantage of SAS techniques in structural analysis?

☐ Ability to study samples only in crystalline form

☐ High resolution for atomic-level structure determination

✓Non-invasive characterization of structures in their native environment

☐ High sensitivity to thermal conductivity variations

☐ Exclusive applicability to metallic samples

Q13: How can the overlap of form and structure factors affect a SAS measurement?

☐ It simplifies data analysis

✓ It can obscure contributions from the shape and size of individual particles

☐ It only occurs in homogeneous systems

☐ It is irrelevant in most scattering experiments

☐ It eliminates the need for data corrections

Q14: Why is q-range calibration critical in SAS experiments?

□ To match scattering results across different instruments

□ To isolate isotropic scattering from anisotropic scattering

□ To improve the spatial resolution of 2D scattering patterns

✓ To ensure accurate particle size determination

□ To reduce noise in high-q measurements

Q15: How can SAS synchrotron experiments accommodate samples that are unstable under radiation?

□ By reducing the beam intensity during measurements

✓ By using short exposure times with frame averaging

□ By aligning the beam to avoid high-scattering regions

□ By increasing the sample temperature during analysis

□ By utilizing smaller sample-to-detector distances

Section 4

Q16: for which techniques in electron microscopy, inelastic scattering events are a mandatory prerequisit?

✓ Secondary electron imaging in SEM

✓ Energy dispersive X-ray spectroscopy in SEM or TEM

☐ electron diffraction in TEM

✓Electron energy loss spectroscopy in TEM

☐ High-angle annular dark field STEM

Q17: for which of the following tasks would you use the SEM?

☐ find out, whether Pd-nanoparticles of 1nm size are on top or inside CeO_2 host crystallites of 50-100nm

✓ check with EDX the composition of the surface of a screw

☐ determine the crystal structure of nanoparticles of 20 nm diameter

✓Get an overview on the morphology and size distribution of 500-700nm sized particles

☐ provide an atomically resolved image of a thin, crystalline membrane

Q18: which of the following parameters influence, whether lattice periodicity can be resolved in parallel illumination HRTEM?

☐ the beam diameter

✓ the thickness of the specimen

✓ the acceleration voltage

✓ the spherical aberration of the microscope lenses

✓ the specimen orientation

Q19: you have a mixture of Ta and TaO_2 nanoparticles of 200-300nm diameter and of 200-500nm sized SiO_2 particles. Which of the following conditions would you use to show which of the particles are metallic and which are oxides?

✓ SEM: Energy dispersive X-ray spectroscopy (EDX or EDX)

☐ SEM: off-axis SE image at 20keV

✓ SEM: BSE

✓ TEM: Electron energy loss spectroscopy

☐ TEM: High-resolution TEM imaging

Q20: what are the strengths of transmission electron microscopy?

☐ its capability to easily provide statistically relevant answers about large amounts of nanoparticles

✓ It can provides information from individual particles

☐ it is especially helpful in case of very thick pieces of material

✓it has the capability to provide information about the chemical composition, the crystallographic structure as well as the morphology and the size of the same small piece material

☐ the resolution which can be reached in imaging is nearly identical with the small wave length of the beam electrons in TEM