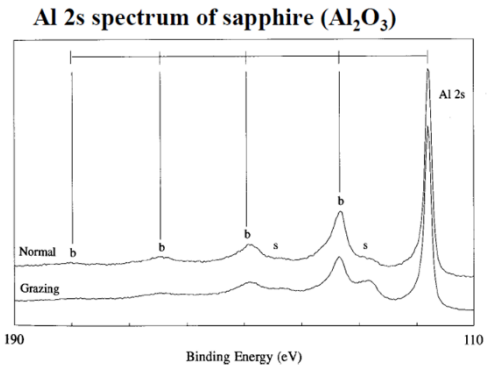
Materials Characterization Final exam (Chapter 2.2-3)

Department of materials science and engineering, National United University Jan. 7, 2025 9:00 – 11:00

(A) Select the correct answer from the list (45%)

1. The detection limit for XRF is around (a) 10 at% (b) 0.1 at% (c) 1-100 ppm (d) 1-100 ppb (e) 1-100 ppt
2. Which of the following is not the event about X-ray interactions with matter? (a) photoelectric effect (b) Compton effect (c) Rayleigh scattering (d) sputtering (e) pair production
3. For XRF quantification, the fluorescence intensity would depend on the interatomic distances separating the excited atoms in the crystal lattice (e.g. $\alpha-\text{quartz}$ and $\beta-\text{quartz}$). The effect is called (a) absorption effect (b) enhancement effect (c) particle-size effects (d) mineralogical effects (e) surface effects
4. Regarding the secondary target used in XRF, which of the following is not true as compared to direct tube excitation? (a) avoid the intense Bremsstrahlung continuum (b) low-powered tube is required (c) more sophisticated and expensive (d) a better selectivity and an improved sensitivity (e) can select a secondary target that has characteristic lines just above the absorption edges of the elements of interest in the sample
5. Which of the following analyzing crystal can detect the highest X-ray energy? (a) LiF (200) (b) LiF (220) (c) LiF (420) (d) PET (e) multilayer
6. The (a) convex lens (b) concave lens (c) polycapillary focusing optics (d) collimators (e) electromagnetic lens collect X-rays from the divergent X-ray source and direct them to a small focused beam at the sample surface with diameters as small as tens of μm.
7. Which of the following tool usually equipped with an electron gun source and a WDS detector? (a) EDS (b) XRF (c) EPMA (d) XPS (e) Auger
8. What are the incident source and detecting signal for the XPS analysis, respectively? (a) photon, photon (b) photon, electron (c) electron, photon (d) electron, electron (e) ion, electron
9. In the right figure, what causes the loss of a specific amount of kinetic energy evenly? (a) contamination (b) plasmon (c) charging (d) core electron (e) unpaired 3d or 4f electrons



10. Corrections for charging can be accomplished by the use of reference standards. The reference standard is usually (a) O 1s (b) N 1s (c) C 1s (d) Al $2p_{3/2}$ (e) Cu $2p_{3/2}$
11. To study the valence band structure and work function, you may use (a) UPS (b) XPS (c) ARXPS (d) AES (e) EDS
12. The detector with which AES frequently equipped is (a) cylindrical mirror analyzer (b) concentric hemispherical analyzer (c) spherical sector (d) proportional counter (e) photomultiplier tubes
13. The Roland circle and analyzing crystal equipped with the XPS instrument is used for (a) setting pass energy (b) monochromator (c) detecting kinetic energy of electrons (d) enhancing the X-ray intensity (e) neutralizing charging effect
14. Which of the following is not true? (a) spin-orbit splitting increases with Z (b) magnitude of spin-orbit splitting decreases with distance from nucleus (c) withdrawal of valence electron charge decreases binding energy (d) binding energy $2p_{1/2} > 2p_{3/2}$ (e) intensity ratio $2p_{1/2} : 2p_{3/2} = 1:2$
15. Which of the following tools is more accurate in determining the composition of a material? (a) X-ray photoelectron spectroscopy (XPS) (b) Energy dispersive spectroscopy (EDS) (c) X-ray diffractometer (XRD) (d) X-ray fluorescence (XRF) (e) Auger electron spectroscopy (AES).

(B) Answer the following questions (55%)

16. In XRF, what detector will you use to detect the intensity of characteristic X-ray from light element and heavy element, respectively? Describe how these detectors work. (12%)

17. The table shows the effective layer thickness for limestone pressed pellet for XRF analysis. Explain why Ti $K_{\alpha 1}$ exhibits a smaller effective layer thickness than Ca $K_{\alpha 1}$? (8%)

Table I: Effective layer thickness for limestone pressed pellet (with permission from Bruker)

Compound	Line	Concentration (%)	Energy (keV)	Layer Thickness (µm)
Fe_2O_3	Fa KA1	0.722	6.40	174
MnO	Mn KA1	0.016	5.89	139
TiO_2	Ti KA1	0.016	4.51	66
CaO	Ca KA1	30.12	3.69	104
K_2O	K KA1	0.103	3.31	77
SO_3	S KA1	0.000	2.31	27
P_2O_5	P KA1	0.004	2.01	19
SiO_2	Si KA1	1.130	1.74	13
Al_2O_3	Al KA1	0.277	1.49	8
MgO	Mg KA1	21.03	1.25	7
Na_2O	Na KA1	0.029	1.04	4
CO_2		46.37		

18. How do you use X-ray photoelectron spectroscopy to differentiate Cu^0, Cu^{1+}, and Cu^{2+} in a copper-based material? Why? (8%)

19. Describe how to correct the charging effect in X-ray photoelectron spectroscopy. (8%)

20. Explain the kinetic energy of the X-ray photoelectron is determined by $KE = h\nu - BE - \Phi_{spec}$ rather than $KE = h\nu - BE - \Phi_{sample}$. ($KE$ is kinetic energy, $h\nu$ is X-ray energy, BE is binding energy, Φ_{spec} is spectrometer work function, Φ_{sample} is sample work function) (10%)

21. Following is an XPS spectrum, using Al Kα as incident source (1486.6 eV), obtained from a silver sample (BE for Ag $4p_{3/2}$ at 57.6 eV, 4s at 96.9 eV, $3d_{5/2}$ at 368.0 eV, $3d_{3/2}$ at 373.4 eV). The work function of Ag sample is 4.7 eV. The work function of XPS instrument is 4.2 eV. (a) What will be the kinetic energy for $4p_{3/2}$, 4s, $3d_{5/2}$, and $3d_{3/2}$ photoelectrons? (4%) (b) Ag MNN (BE = 1128.8 eV) is not an XPS peak. Calculate its kinetic energy. Explain where does this peak from. (5%)