SOLUTIONS

Name (Please PRINT)

U N I V E R S I T Y O F C A L I F O R N I A
Department of Materials Science and Engineering
MSE 104, *Materials Characterization*
Spring Semester 2012
Professor R. Gronsky

Midterm 01

Instructions

This is a CLOSED book exam.
No papers or digital files are permitted.
Answers MUST be written neatly and concisely in the spaces provided.
Calculators ARE permitted, but NOT on smart phones. All phones must be silenced.
There will be NO QUESTIONS permitted during the exam (write comments in your answers).

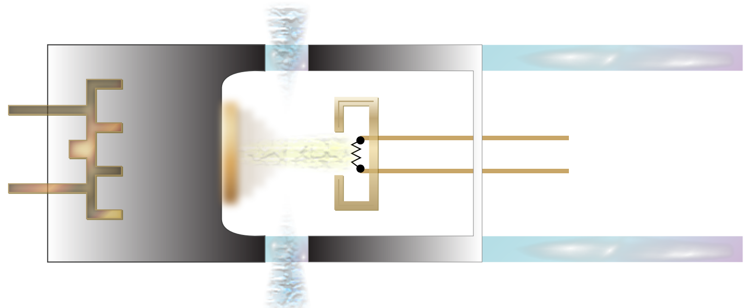
Guidelines

There are 5 equally-weighted questions.
Average pace = 10 minutes per problem.
Show ALL of your work for partial credit, as appropriate.
Read each question FULLY before commencing your answer.
Reference information is found on the last page of this exam, labeled"Reference."

Problem 1(a)

How does the tungsten filament in a tungsten x-ray tube cause the emission of W $K\alpha$ x-rays? Explain, citing the effect of filament current on tube current.

Answer



The filament doesn't emit x-rays, it emits **electrons** that are accelerated through a large potential drop toward the target. When those electrons strike the target, they may be decelerated, generating *Bremstrahlung* ("braking") radiation, a **continuous** spectrum of x-rays, or they may cause ionization of the target, triggering electronic transitions within the target to replace lost core shell electrons, generating **characteristic** x-rays with wavelengths specific to the energy levels of the target.

Filament current, the flow of electrons through the filament, induces resistive **heating**, providing thermal energy to the electrons, enabling them to escape the filament by overcoming the "work function." **Tube current**, the flow of electrons from the filament to the target, **generates x-rays**. With no filament current, there can be no tube current. As the filament current increases, the tube current increases, up to a point (saturation), beyond which the filament lifetime is decreased dramatically. In the extreme, the filament current can be high enough to cause melting of the filament, damaging the tube.

Problem 1(b)

What is the wavelength (to 6 significant figures) of the W $K\alpha$ x-rays emitted from a W tube operated at 60 kV? Explain.

Answer

Before attempting to calculate the weighted average wavelength of $K\alpha$ radiation using the values found in Table I, ([[2 × 0.209210] + 0.213828] / 3 = 0.210749 Å), or calculating the equivalent wavelength associated with the energy of $K\alpha$ radiation from Table I (12.4 / 58.87 = 0.210633 Å) or both, and noting that they are different, and worrying about which is the right answer (!), consider why the **tube voltage** is given in the problem statement.

Begin by calculating the short wavelength limit associated with an x-ray tube running at 60 kV:

$$\lambda_{\mathrm{SWL}} = 12.4 / 60 = 0.206667 \text{ Å}.$$

Now note from Table I that the wavelength associated with the K absorption edge in W is 0.17837 Å. A tube operated at 60 kV therefore **cannot** cause K shell ionization from a W target. With no vacant K shells, there can be no $L \rightarrow K$ transitions, so there can be no $K\alpha$ x-rays emitted from the tube.

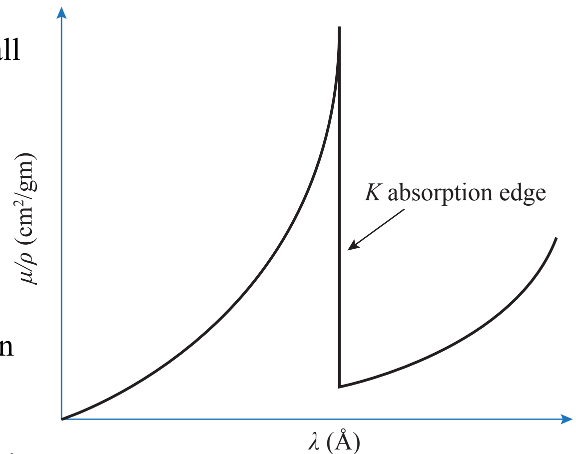
Conclusion: $K\alpha$ radiation is **NOT EMITTED**. (No need to calculate anything more!)

Problem 2(a)

Experimentalists know that hard x-rays are generally more difficult to absorb than soft x-rays, and that heavy elements are generally better absorbers than light elements. Why therefore does vanadium absorbs Cu $K\alpha$ radiation (λ = 1.542 Å) more easily than Cr $K\alpha$ radiation (λ = 2.291 Å), and that Co $K\alpha$ radiation (λ = 1.790 Å) is more readily absorbed by vanadium (Z = 23) than niobium (Z = 41)? Explain.

Answer

The apparent anomaly here is due to the presence of **absorption "edges"** in the mass absorption coefficients of all elements. When the wavelength of the incident radiation is sufficient to cause ionization, or the stripping of electrons from bound orbitals within the absorber, there is a marked increase in absorption as ionization occurs. Table II illustrates this apparent anomaly in all four of its rows.



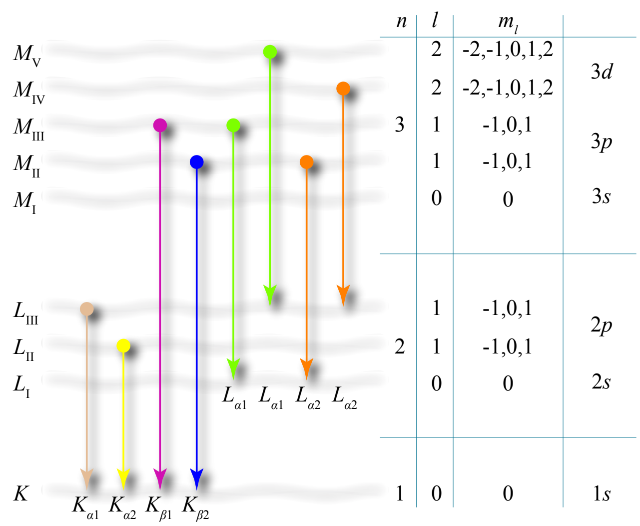
Vanadium absorbs more hard radiation (Cu $K\alpha$) than soft radiation (Cr $K\alpha$), and is a better absorber of Co $K\alpha$ radiation than an element with much higher atomic number (Nb) because V has an K absorption edge between 2.0 and 2.3 Å, as revealed in the first row of Table II. Radiation with slightly shorter wavelengths is therefore very readily absorbed.

Problem 2(b)

The production of K-L_{II}-L_{III} Auger electrons diminishes the fluorescence yield of $K\alpha$ radiation from magnesium (Z = 12) by 97%. Explain.

Answer

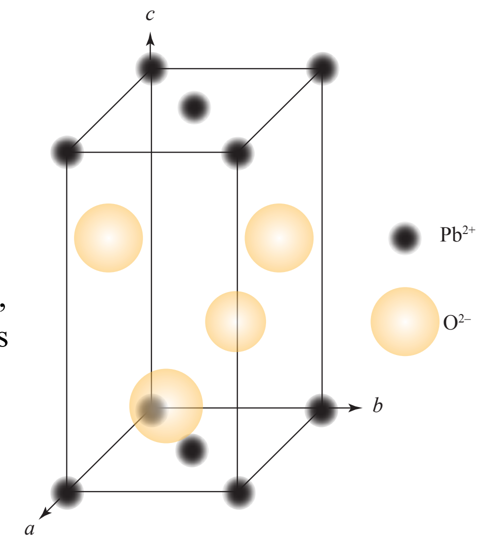
The production of a K-L_{II}-L_{III} Auger electron begins with a K shell ionization event (first index K) followed by an $L_{II}\rightarrow K$ transition (second index L_{II}). At this point it would be expected that a $K\alpha_1$ x-ray photon would emerge and be counted, representing high "fluorescence yield" under photon illumination energetic enough for K shell ionization. However, there is another possible outcome. The energy released by the $L_{II}\rightarrow K$ transition might instead be transferred to an L_{III} electron (third index L_{III}). No x-ray photon is counted (yield is zero) when such an electron, called an Auger electron, is ejected. In light elements like Mg, Auger electron generation is prominent because of fewer electrons in orbitals above the L shell.



Problem 3(a)

An experimentally-observed solid phase of lead oxide is thought to be isostructural with the B10 *Structurbericht* classification (International Union of Crystallography), having cations at 0,0,0 and ½,½,0, and anions at 0,½¼, and ½,0,¾, in a tetragonal cell with $c/a = 2.0$. Sketch the unit cell and specify its Bravais lattice and motif.

Answer



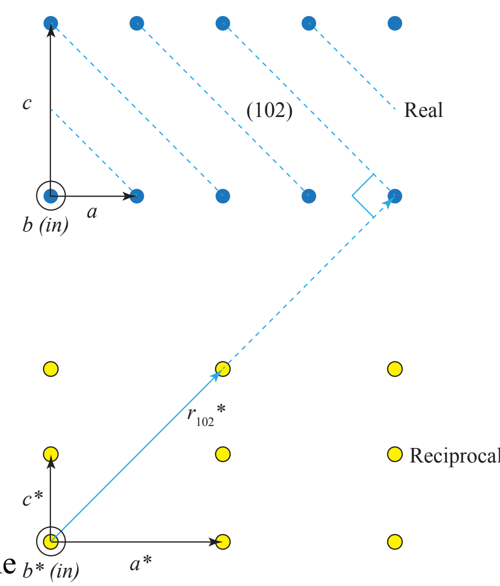
The unit cell is shown here to be populated by 2 Pb^{2+} ions (8 × ⅛ + 2 × ½) and 2 O^{2-} ions (4 × ½), preserving its 1:1 stoichiometry, as required.

The Bravais lattice is therefore a SIMPLE TETRAGONAL lattice with a 4 ion motif, Pb^{2+} at 0,0,0 and ½,½,0, and O^{2-} at 0,½¼, and ½, 0,¾, as given in the problem statement. Drawn to scale, the *c* axis is twice as long as the *a* (= *b*) axis.

Problem 3(b)

Now construct the reciprocal lattice corresponding to your Bravais lattice from above and show the explicit correspondence between r_{102}^* and the (102) family of lattice planes in the B10 structure.

Answer



Consistent with the sketch in (**a**) above, a projection of the real space lattice shown here has the *b* axis pointing into the page, *c* vertical, and *a* to the right. Only the **lattice** is required in this sketch (no ion positions are needed), scaled with $c/a = 2$ as specified in the problem statement.

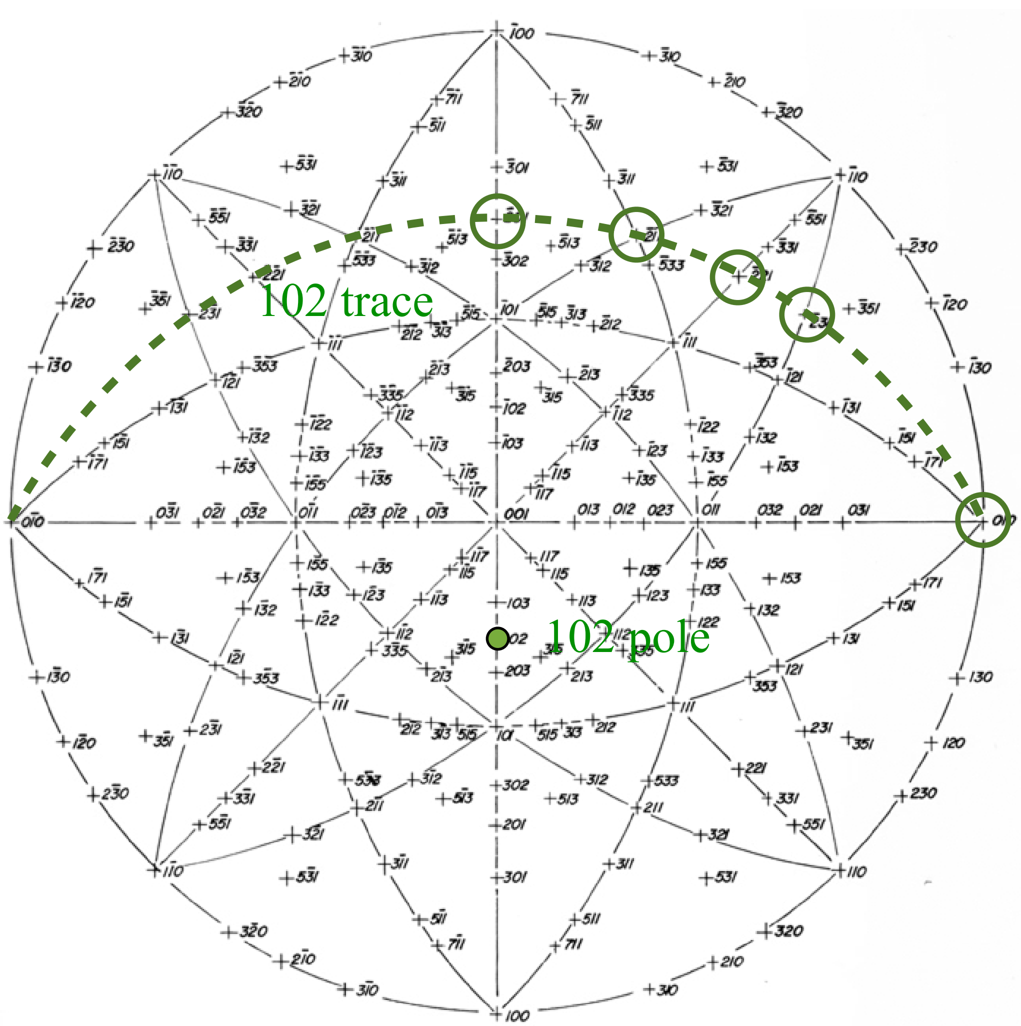
Consequently the reciprocal lattice has its fundamental lattice translation vectors parallel to their corresponding vectors in real space, but with reciprocal magnitudes, that is $a^*/c^* = 2$.

To complete the problem, the (102) family of planes is shown in real space to intersect the *a* axis at 1, the *b* axis ∞, and the *c* axis at ½, comprising parallel members with the same interplanar separation distance, shown edge-on in this projection. Finally, the reciprocal lattice vector r_{102}^* is shown to be the normal to the (102) family of planes, with a magnitude equal to the reciprocal of the interplanar spacing. This is shown here by the relative magnitudes of a^*, c^* and r_{102}^*.

Problem 4

Another observed solid phase of lead oxide is known to be cubic.
(**a**) Use the following standard cubic stereographic projection to show the location of the (102) family of planes in this structure by two indicators: (**i**) pole and (**ii**) trace.
(**b**) Now use your construction to identify three non-parallel lattice directions lying in the (102) family of planes, confirming your results by appropriate calculation.

Answer



(**a**) The 102 **pole** is the **normal** to the (102) family of planes, and it is found plotted on the projection at 26.6° south (recall from several homework problems). The trace of the (102) planes is the location of a **planar intersection** with the projection sphere, found at 90° from the pole along an equatorial line. In this case the N-S meridian will do. The **trace** is the great circle shown as a dotted line here.

(**b**) Directions lying in the (102) plane can be read directly from the projection as **poles lying in the (102) trace**. These are circled on the projection. For any three of these (as required by the problem statement), confirmation of their orthogonal orientation relative to the (102) plane normal, can be provided by taking their dot products with [102], all of which should be zero (0).

$$[\bar{2}01]\cdot[102] \;=\; [\bar{2}11]\cdot[102] \;=\; [\bar{2}21]\cdot[102] \;=\; [\bar{2}31]\cdot[102] \;=\; [010]\cdot[102] \;=\; 0$$

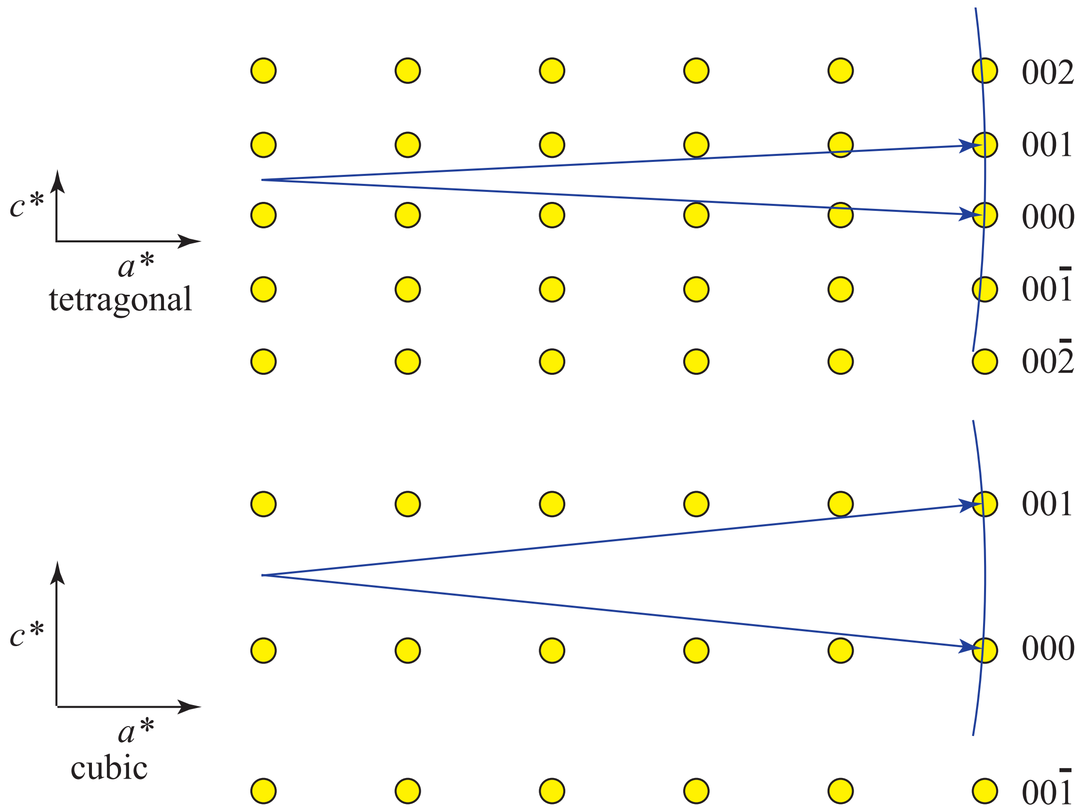
Problem 5

(**a**) Sketch a reciprocal lattice / Ewald sphere construction for 001 diffraction from both PbO phases above, namely, (**i**) a tetragonal crystal with $c/a = 2$, and (**ii**) a cubic crystal with $c = b = a$. Assume the $a_{\text{tetragonal}}$ // a_{cubic} and $c_{\text{tetragonal}}$ // c_{cubic}, the a parameter is exactly 1Å in both cases, and the radiation is monochromatic W $K\alpha$.

(**b**) Can a Laue experiment be used to decide which crystal structure is the correct one? How? Be specific, using your sketches as illustrative guides.

Answer

(**a**) The sketches here show (**i**) above and (**ii**) below. Note that the wavelength of the radiation source is approximately 0.2 Å, making the radius of Ewald's sphere about 5 times a^*. For an exact 001 diffraction condition, Ewald's sphere must contact both 000 and 001 reciprocal lattice points.



(**b**) Yes. A Laue experiment uses **polychromatic radiation** incident on a **single crystal** to satisfy Bragg's Law for a large number of lattice planes in many different orientations. The corresponding reciprocal lattice / Ewald sphere construction will look like those above, but with a multitude of Ewald spheres, all pinned to the origin. Consequently the Laue pattern for a **cubic** crystal illuminated by an x-ray beam along [100] will show **equally-spaced spots along both orthogonal [001] and [010] directions** in the plane of the pattern, but a tetragonal crystal will show **twice as many spots along the [001] direction, with half the spacing, as those along the [010] direction.**

The sketch above also shows that even for monochromatic radiation, if the wavelength is short enough, Ewald's sphere will intersect BOTH the 001 and 002 reflections in the tetragonal crystal, enabling the ½ spacing along the c* direction to be identified.

Reference

Table I: X-Ray Wavelengths (in Å) and Energy of $K\alpha$ Emission (from Cullity & Stock, Appendix 7)

Element	E ($K\alpha$) [keV]	$K\alpha_2$ (strong)	$K\alpha_1$ (v. strong)	$K\beta_1$ (weak)	K edge
29 Cu	8.04	1.544390	1.540542	1.392218	1.38059
42 Mo	17.44	0.713590	0.709300	0.632288	0.61978
74 W	58.87	0.213828	0.209010	0.184374	0.17837

Table II: Mass Absorption Coefficients (μ/ρ) in cm^2/gm (from Cullity & Stock, Appendix 8)

Absorber	24 Cr		27 Co		29 Cu		42 Mo	
	$K\alpha$ 2.291 Å	$K\beta$ 2.085 Å	$K\alpha$ 1.790 Å	$K\beta$ 1.621 Å	$K\alpha$ 1.542 Å	$K\beta$ 1.392 Å	$K\alpha$ 0.711 Å	$K\beta$ 0.632 Å
23 V	75.06	501.0	332.7	254.7	222.6	168.0	25.24	18.07
26 Fe	113.1	86.77	56.25	345.5	304.4	233.6	37.74	27.21
28 Ni	145.7	112.5	73.75	56.05	48.83	282.8	47.24	34.18
41 Nb	431.9	336.4	222.9	170.4	148.8	112.3	16.96	81.22

$$\lambda[\text{Å}] = \frac{12.40}{\text{V[kV]}} \qquad I_x = I_0 e^{-(\mu/\rho)\rho x} \qquad \frac{1}{d^2} = \frac{h^2+k^2}{a^2} + \frac{l^2}{c^2} \qquad \mathbf{a}* = \frac{1}{V}(\mathbf{b}\times\mathbf{c})$$

$$\frac{\mu}{\rho} = k\lambda^3 Z^3 \qquad d_{hkl} = \frac{a_0}{\sqrt{h^2+k^2+l^2}}$$

Electron Charge	e	1.602×10^{-19} coulomb
Electron Mass	m	9.109×10^{-31} kg
Velocity of Light	c	3×10^{8} m/sec
Planck's Constant	h	6.626×10^{-34} joule sec
Boltzmann's Constant	k	1.381×10^{-23} joule/K
Conversion Factors		1 cal = 4.186 joule 1 erg = 10^{-7} joule 1 eV = 1.602×10^{-19} joule 1 nm = $\times 10^{-9}$ m = 10 Å

Problem	Possible Points	Score
1	20	
2	20	
3	20	
4	20	
5	20	
TOTAL	100	