

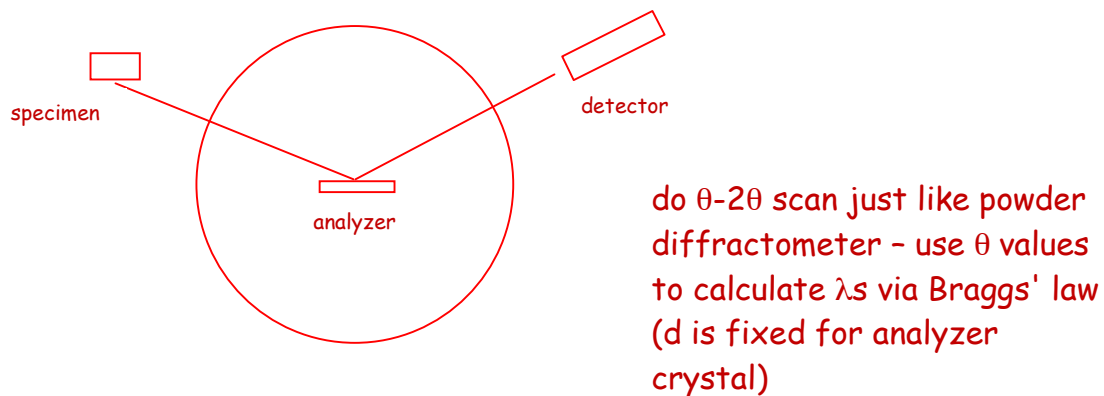
3rd homework answers

1. Explain the difference between wavelength dispersive (WDS) and energy dispersive (EDS) spectroscopies.

Uses analyzing crystal and counter to do a θ - 2θ scan

Uses solid state detector to do an energy "scan"

Show a diagram of a WDS instrument and explain how it works.



Which type is used in an SEM? Explain why.

EDS -faster, smaller, no moving parts

2. Name the three components of a thermionic electron gun and give their function.

emitter - gives off electrons

anode - accelerates electrons

Wehnelt cap - concentrates electron beam

3. List four desirable features of the SEM.

Magnification range 15x to 200,000x

Resolution of 50 Å

Excellent depth of focus

Relatively easy sample prep

4. In the SEM, what are the effects of increasing the voltage on the electron gun. Explain each of the effects.

Resolution increased (λ decreased)

Penetration increases

Specimen charging increases (insulators)

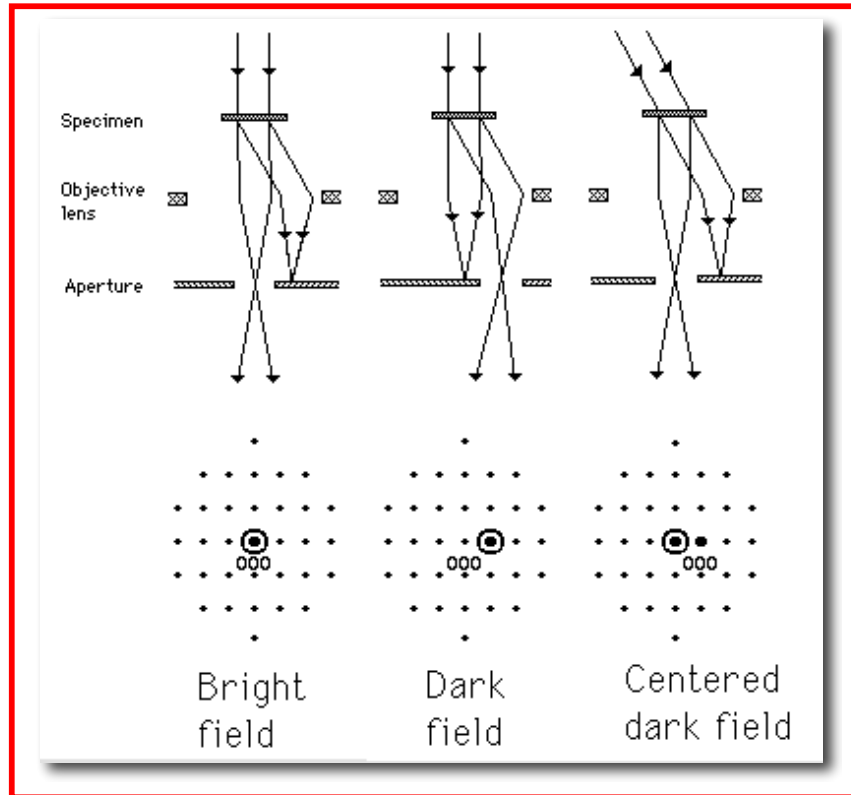
Specimen damage increases

Image contrast decreases

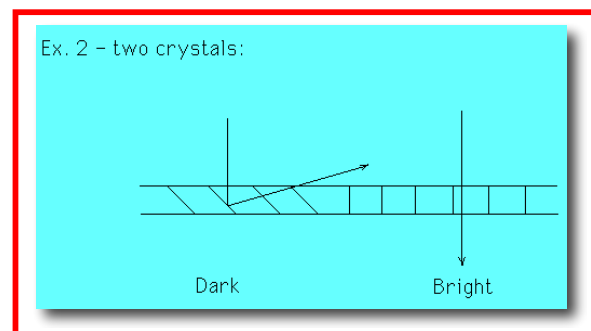
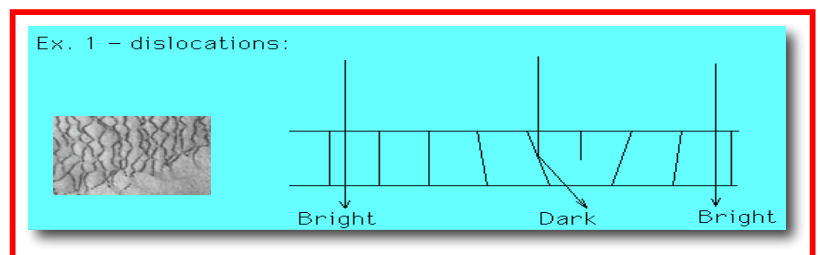
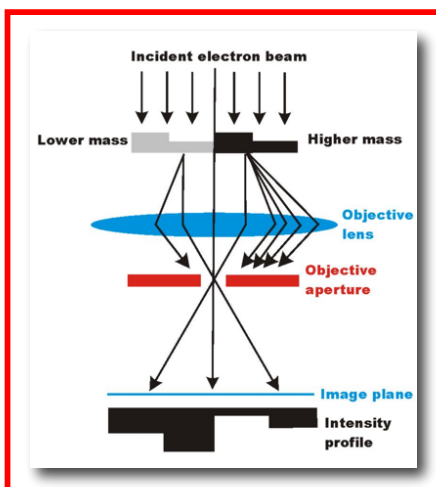
5. Which of the two, SEM or TEM, will give better spatial resolution in the composition determined by either WDS or EDS? Explain why. Diagrams of the specimen regions for the two cases will be helpful.

TEM.....SEM has larger interaction volume - TEM specimen very thin

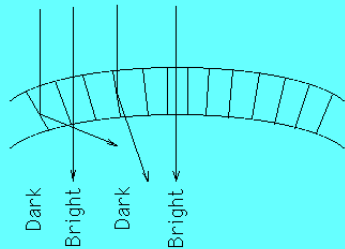
6. Use diagrams to explain the difference between the production of BF, DF, and CDF images in the TEM.



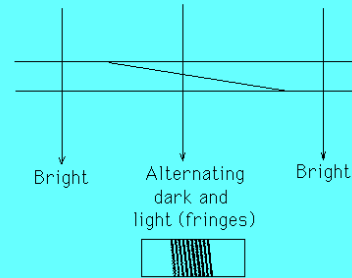
7. Use diagrams to illustrate four sources of contrast for TEM images.



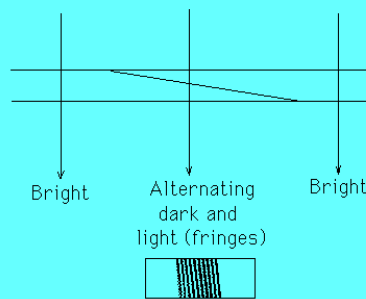
Ex. 3 – bend contours:



Ex. 4 – thickness fringes



Ex. 4 – thickness fringes

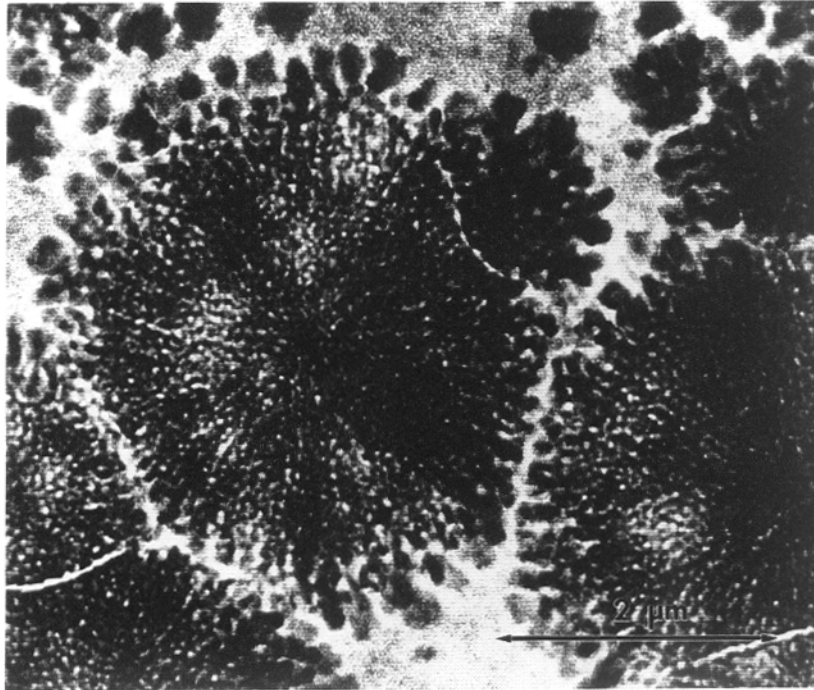


8. The micrograph shown below is a transmission electron microscope (TEM) image from a lead zirconate titanate (PZT) film produced by solution processing. The film is of uniform thickness, and is viewed face-on in the TEM (with the thin direction parallel to the electron beam). The phases present, and, in some cases, their orientations, are very important in controlling the ferroelectric behavior of the material and, thus, its use in memory, sensor, and actuator applications. The desired phase is $\text{Pb}(\text{Zr}_x\text{Ti}_{1-x})\text{O}_3$ (perovskite), but incomplete reactions or other problems can sometimes leave other compounds such as Pb_2O , TiO , $\text{Pb}_{\sim 2}(\text{Zr}_x\text{Ti}_{2-x})\text{O}_{\sim 7}$ (pyrochlore), or amorphous phases in the films. The crystalline compounds listed here all have (approximately) cubic crystal structures; the Bravais lattice and lattice parameter for each are listed below:

Pb_2O : primitive cubic, $a_0 = 5.38 \text{ \AA}$

TiO : face-centered cubic, $a_0 = 4.18 \text{ \AA}$

$\text{Pb}_{\sim 2}(\text{Zr}_x\text{Ti}_{2-x})\text{O}_{\sim 7}$ (pyrochlore): face-centered cubic, $a_0 = 10.40 \text{ \AA}$
 $\text{Pb}(\text{Zr}_x\text{Ti}_{1-x})\text{O}_3$ (perovskite): primitive cubic, $a_0 = 4.04 \text{ \AA}$

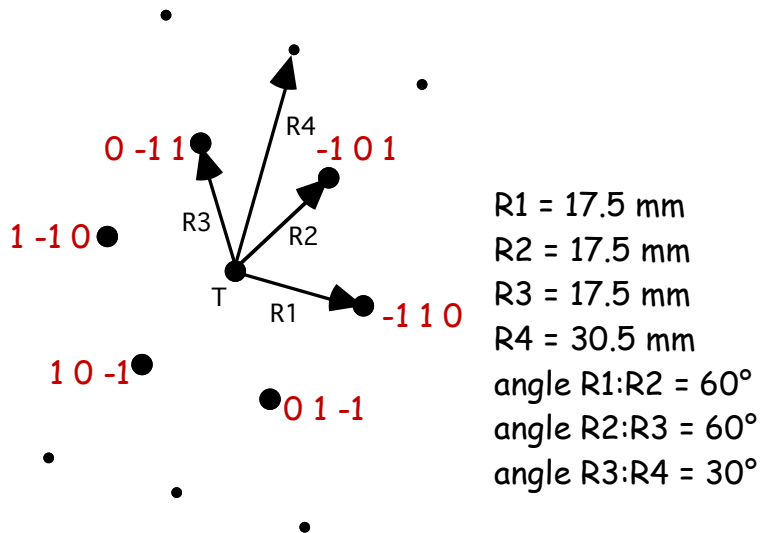


In the area shown, there are two distinct kinds of regions: the dark roughly circular patches, and the lighter material in between. The diffraction patterns shown on the next page (simulated, but similar to the real ones, which don't reproduce well; transmitted beam indicated by "T") are taken at an accelerating voltage of 200 kV and a camera length of $L=2000 \text{ mm}$ from one of the dark circles (A) and from a light area between such circles (B). Answer the following questions based on these data. Show your work and/or explain your reasoning in each case. (In analyzing the diffraction patterns, use the marked values of the measurements even if the measured spacings and angles come out a little differently on the sheet.) Here are some equations that you may or may not find useful:

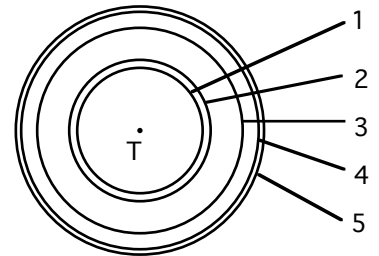
angle ϕ between planes $(h_1 \ k_1 \ l_1)$ and $(h_2 \ k_2 \ l_2)$
in cubic crystal systems:

$$\lambda_r (\text{in nm}) = \frac{1.2264}{\sqrt{V_0 + 0.97845 \times 10^{-6} V_0^2}} \quad \text{for } V_0 \text{ in volts}$$

A:



B:



$R1 = 8.4 \text{ mm}$
 $R2 = 9.6 \text{ mm}$
 $R3 = 13.6 \text{ mm}$
 $R4 = 15.8 \text{ mm}$

(a) From the diffraction pattern (A), determine whether the dark circular area is the desired perovskite phase, one of the other three phases listed, or something else.

Assume cubic - then zone axis obviously $[111]$ type so examining the zero level of a primitive reciprocal lattice down this direction, we can index the single crystal pattern by inspection as shown above taking cross product for any 2 reflections verifies that zone axis is $[111]$ type

camera constant is $0.02508 \times 2000 = 50.16 \text{ mm-}\text{\AA}$ thus $d = 50.16/17.5 = 2.8663 \text{\AA}$, and a is $2.8663 \times 1.414 = 4.053 \text{\AA}$ Perovskite

For polycrystalline pattern, since cubic, pattern is obviously F cubic, and second reflection is (200). Then, $d = 50.16/9.6 = 5.225 \text{\AA}$ and $a = 10.45 \text{\AA}$ verify: for (111), $d = 50.16/8.4 = 5.971 \text{\AA}$, and $a = 1.732 \times 5.971 = 10.34 \text{\AA}$ Pyrochlore

(b) From the diffraction pattern (B), determine whether the material in the lighter regions is the desired perovskite phase, one of the other three phases listed, or something else.

(c) Determine the orientation of the phase in the dark circles by fully indexing diffraction pattern *A* and then finding the zone axis direction. (This crystallographic direction must then be perpendicular to the film surface, at least for this grain, since it is parallel to the electron beam direction!)