MMAT 5322 – Fall 2009
Homework 1– Microstructural Principles
Due Friday, Sep 18, at the start of class

40 points total

Answers in yellow

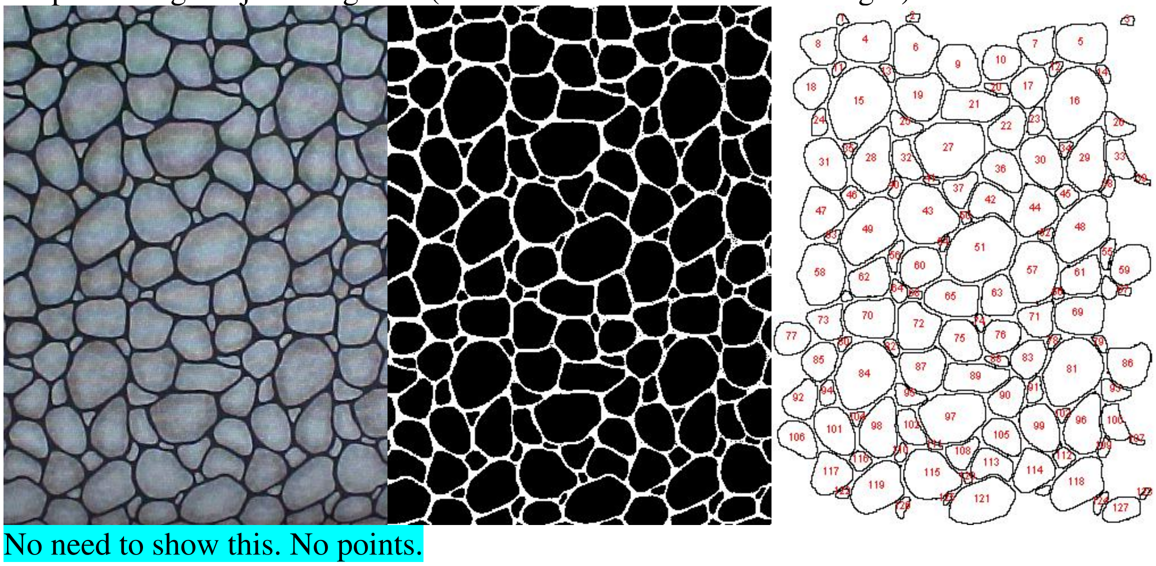
Point designations in blue

Pink identifies possible multiple answers due to some ambiguity

Question 1

For the image below (NOTE: **download** the .jpg file directly from the web site) solve the following. ImageJ is strongly recommended. You may assume that the image, and each pixel, are the same dimensions in x and y:

a. Crop the image to just the grains (throw out the extra info at the edges).



No need to show this. No points.

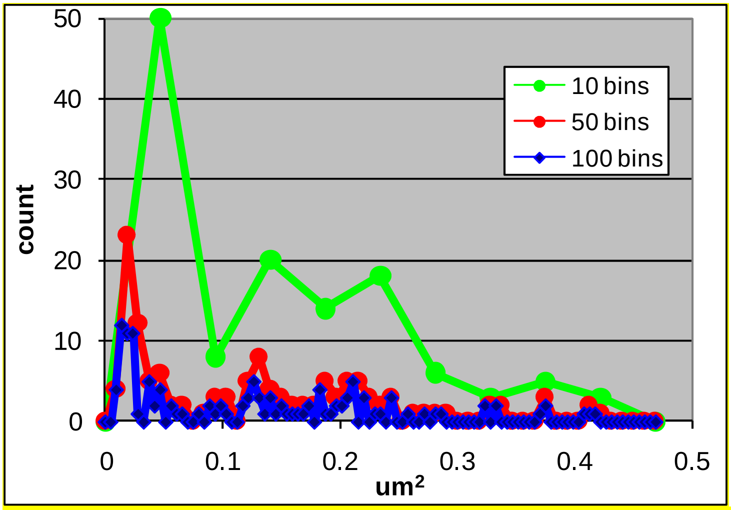
b. What is the average grain size for the entire image (note scale bar)?

0.148 um^2 without edges (0.123 um^2 with edges)

785 pixels^2 without edges (708 pixels^2 with edges)

2 points for correct answer with units properly identified (within 10%: everybody will get slightly different values), 1 if right idea but not quite right (either didn't remove edges or didn't identify units).

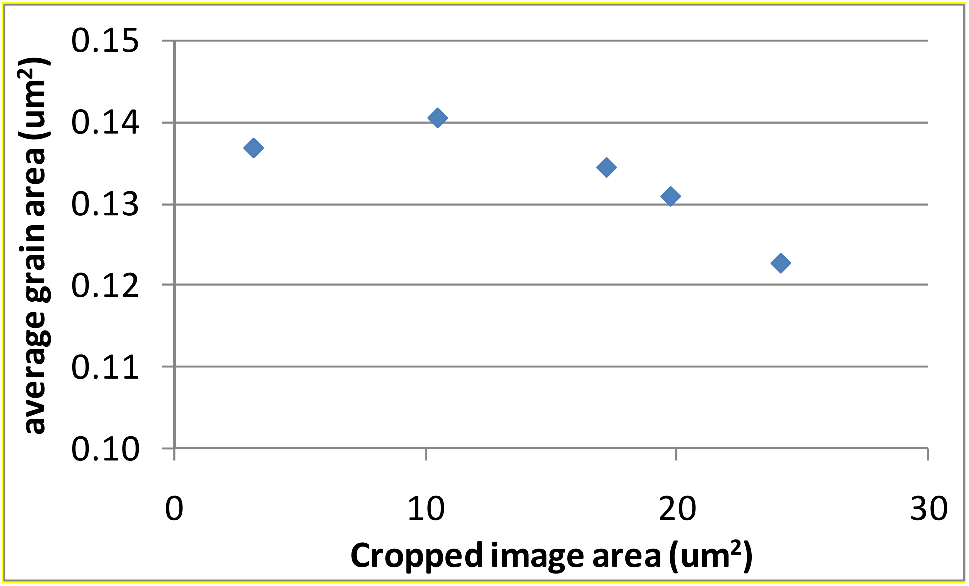
c. Make 3 histograms of the grain sizes, using 10, 50, and 100 bins. Are these bimodal, trimodal, etc? How does bin size matter?



There is a primary grain area, with some grains that are larger suggesting a trimodal distribution when only 10 bins are considered. A larger bin size reveals the details of these peaks, and especially that the third, highest area peak is a broader distribution of small peaks. 10 bins is clearly too few, while 100 bins is unnecessarily large.

3 points, 1 off if wrong values but right idea, 1 off if no discussion of bin size/bimodal/trimodal/etc.

d. How does the average grain size vary as the image size is cropped (plot average grain size as a function of image area, and separately number of grains vs image area). Consider at least 5 areas?



As the image area is cropped, the average grain area values are not the same due to sampling different grains, and different numbers of grains. For a perfectly homogeneous sample, this would not occur.
2 points, 1 for graph, 1 for discussion. Trends will differ for each student, so as long as average area is plotted against image area, at least 1 point is earned.

e. What do (c) and (d) tell you about feature analysis for this sample? In other words, how fine of resolution do I need, and how much sample should I analyze?
Clearly, to fully analyze an image of a sample, measurements at different sizes, and with different fidelity (histogram bins) are crucial to be sure about the sample homogeneity, statistical distribution of features, etc.
1 point for discussion of how number of samples is important to consider (doesn't matter which conclusion was drawn as long as it is consistent with the data set).

Question 2

You need to study a sample with features that are roughly 500 nm tall. You have access to an optical microscope with a 60x oil objective, which has an NA of 1.3 and uses 532 nm light.

a. What is the lateral limit of optical resolution for this setup?
249.6 nm
1 point

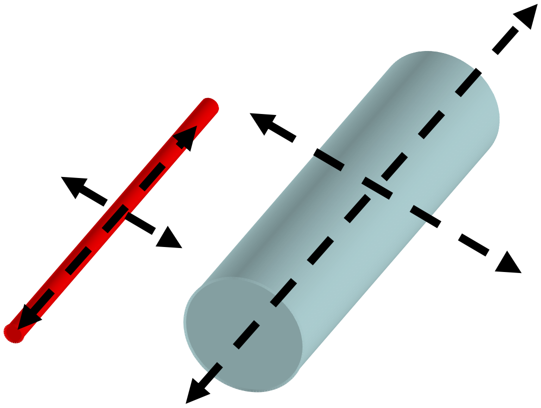
b. You decide to buy a camera for the microscope and want to be able to record images at the optical resolution limit or better. Given the 60x magnification and a standard camera chip size of 1 cm (square), show whether
    i. you need to spend lots of money for a camera with 1024 pixels on a side, or
    In this case, the pixels are each 9.77 um on a side. After a 60x magnification, each pixel is resolving 162.8 nm. This is better than the Raleigh limit of 249.6 nm in part a as we need, but barely. Ideally, there will be multiple pixels (2-10) per Raleigh limit to detect gradients. Another 10x in the optical path would be especially useful, or at least 2x (which is common).
    2 points, 1 for 162.8 nm, and 1 for recognizing this is not enough.
    ii. you can get away with a 512x512 pixel camera.
    Now, the pixels are each 19.5 um on a side. After a 60x magnification, each pixel is resolving 325.5 nm. This is not enough to detect the Rayleigh resolution limit from part a.
    2 points, 1 for 325.5 nm, and 1 for recognizing this is not enough.

c. The 532 nm light source breaks one day. The only thing you have available is a violet-ish source (422 nm) Do you get better or worse lateral resolution?
The lateral resolution is now 198 nm. Generally, lower wavelength gives better resolution due to the 1/lambda relationship in the Rayleigh limit.
1 point for better resolution (no need to calculate the value).

d. Can you ***simultaneously*** focus on the top of the 500nm features, and the substrate beneath, with either source (or both)? (You may assume a collection angle of 60 degrees for each case)?
The depth of field with 532 nm is 144.1 nm, while the depth of field for 422 nm is 198 nm. Both are much less than the 500nm features you want to detect, so NO you cannot simultaneously focus on top and bottom of the features.
2 points, 1 for depth of field values, 1 for noting this isn't enough for 500 nm features.

Question 3

You have an optical microscope with a 100x air lens with a numeric aperture of 0.90, and you are using **680 nm light**.



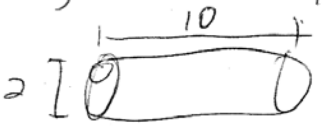
You use this scope to image nanotubes and microtubes. The nanotubes have a diameter of 10 nm, are straight, and have a length of 100 um. The microtubes have a diameter of 5 um, are straight, and have a length of 100 um.
You should first determine the Raleigh resolution limit, which is 460.9 nm.

a. Assuming your tubes are isolated from each other, draw a single plot showing the cross section (as sketched) of the intensity as a function of position:
    a. along the microtubes
    b. along the nanotubes.
b. Draw another single plot sketching the intensity as a f(position) for:
    a. perpendicular to the microtubes
    b. perpendicular to the nanotubes

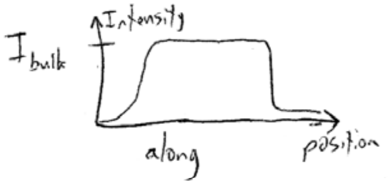
NOTE: Each diagram should be drawn with respect to the intensity you would detect for an infinitely large sample (ie do you reach this intensity or not?).
See below for a sketch. Generally, the microtube is resolved with an intensity on the structure of some bulk value, and Gaussian edges extending beyond the edges of the structure itself (it should thus appear slightly wider than the 100 um length and 5 um diameter). The nanotube can be detected, but its intensity is substantially less than the bulk intensity, its length is determined correctly except for the unavoidable Gaussian tails (as with the microtube), and the cross-section is purely Gaussian and appears at least as wide as the Raleigh resolution (i.e. it is NOT resolved laterally).

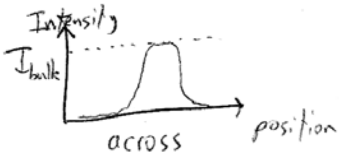
The answer is similar to the one below, except with slightly different dimensions.
8 points total: 2 points each section. For full credit, each section should show correct intensity, correct feature dimensions, and Gaussian tails (extending beyond feature dimensions).

b) For a microtube with dimensions of ($10\,\mu m_{\ell} \times 2\,\mu m_{di}$)

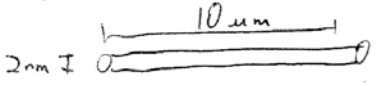


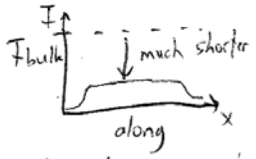
the tubes can be resolved.

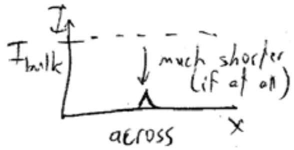




c) For a nanotube:







note: an intensity decrease is also acceptable, esp. for dark field (which you might use to enhance the detection).

Question 4

a. Calculate the deBroglie wavelength for a 500 keV TEM experiment and a 50 keV SEM. Account for relativistic effects in both cases—do they matter?

TEM: **1.42 pm with relativity**, 1.73 pm without, so 2.4% error if ignoring relativity.

SEM: **5.36 pm with relativity**, 5.48 pm without, so 22% error if ignoring relativity.

Beyond the relativity values, it may be also/separately be argued that the relativity correction DOES NOT MATTER because other parameters dominate the actual instrument resolution such as spherical and chromatic aberration, with a 10-100x greater error than that imposed by the relativistic or simplified equations.

4 points: 1 for each value with relativity, AND:

> 2 pts for showing values that indicate relativity does matter (OR 2 pts for saying it doesn't matter specifically because of other much greater sources of error).

Now, for the TEM case only:

b. Determine the ideal alpha angle in degrees, assuming Cs=6 mm. What is the error caused by spherical aberration, and the error due to diffraction effects (i.e. diffraction limited resolution)? What is the overall total error in this IDEAL case?

If you assume Cs=6mm, as the question asked:
Ideal angle = 0.252 degrees (0.004 radians)
At the ideal angle, spherical aberration=diffraction effects, which are each 0.513 nm.
The overall error is thus 0.725 nm.

OR, If you assume Cs=0.6mm, which the book gives as standard
Ideal angle = 0.353 degrees (0.0062 radians)
At the ideal angle, spherical aberration=diffraction effects, which are each 0.141 nm.
The overall error is thus 0.199 nm.

1 point for ideal angle (radians or degrees), and no credit for error calculations due to ambiguity about whether alpha is determined in radians or degrees.

c. Now, alpha is actually 1 degree (i.e. NONIDEAL). In this case, what is the error caused by spherical aberration, the error due to diffraction effects (i.e. diffraction limited resolution), and the total error?
If Cs=6mm:
At a 1 degree angle, spherical aberration=31.899 nm.
diffraction effects=.049 nm.
The overall error is thus 31.900 nm.
OR, If Cs=0.6mm:
At a 1 degree angle, spherical aberration=3.190 nm.
diffraction effects=.050 nm.
The overall error is thus 3.190 nm (actually slightly larger than the aberration itself, but only if more decimal places are considered, which is physically not meaningful).

3 points for spherical error, 1 point for diffraction error, and 1 point for overall error.

d. What is the depth of field for the ideal case, and for the 1 degree (nonideal) case?
If Cs=6mm:
Ideal depth of field (d_{object}) is 102 nm, while 1 degree depth of field is 1828 nm.
OR, If Cs=0.6mm:
Ideal depth of field (d_{object}) is 32.3 nm, while 1 degree depth of field is 183 nm.
Not graded due to ambiguity about alpha units.

e. For a magnification of 100,000, what is the depth of focus for the ideal case (only).
If Cs=6mm:
Ideal depth of focus (d_{image}) is 1020 <u>meters</u>
1 degree depth of focus is 18277 m, but the question didn't ask for this.
OR, If Cs=0.6mm:
Ideal depth of focus (d_{image}) is 322.6 <u>meters</u>
1 degree depth of focus is 1828 m, but the question didn't ask for this.
Not graded due to ambiguity about alpha units.

<u>Question 5</u>

What optical microscopy method would you use to study the following and why:

a. Porous alumina substrates.
b. Distributed tin-oxide nanorods.
c. Transparent polymers with embedded rods of transparent (but different n) second phases.
d. Light-sensitive cells stained with fluorescent dyes.
e. Identifying x, y, and z positions of fluorescing 2^{nd} phases in glass.
f. Grain distribution in ceramics with an anisotropic index of refraction.
g. Surface roughness on highly porous titanium.
h. the orientation of a lithographic step on a surface (stepping up, or stepping down).

a. *Bright field, interference, stereographic*
b. *Dark field (better contrast for sub-resolution features), interference*
c. *Phase imaging, DIC, maybe polarization*
d. *Fluorescence, confocal*
e. *Z-stacks, deconvolution, confocal*
f. *Polarization*
g. *Interferometry*
h. *Interferometry, oblique illumination*

1 pt for each letter. NOTE: each does not require multiple answers (several answers are simply shown where they might make sense).
–½ point for any other methods mentioned that do not make sense.