Experiment No. 1:

Microstructure Control through Plastic Deformation and Recrystallization and the Hall-Petch Effect
Introduction

Abstract
The purpose of this Lab exercise is to introduce students to the concept of microstructure control in structural materials through the process of recrystallization, i.e. the restoration of a strain-free state in a deformed material via annealing. By measuring hardness, the variation in grain size that occurs as a consequence of the gradients in strain and temperature provide data to evaluate the Hall-Petch effect. It also serves to remind the students about techniques for quantifying microstructure and the need to understand distributions of defects (in this case, grain boundaries).

Reading

Metals, Ductility
Metals are particularly useful as structural materials because of their combination of high strength with ductility. Glass is a common example of a high strength material that has practically no ductility at room temperature. Because of this lack of ductility there is no chance for relaxation of stress concentrations at points where a load is applied or at changes in section. Consequently, the stresses in glass may reach a high enough value locally to initiate fracture even when the average stress may be well below the fracture strength of the material. Later in the course we will look into the relationship between defects and brittle fracture. Ductility is necessary for forming operations, machining, and for the common methods of joining, such as bolting and riveting. Ductility, and its counterpart, fracture resistance, is also essential in engineered components in order to resist overloads, accidents, and mistakes in design, as discussed further below.
Plastic Slip
Plastic deformation in ductile crystalline materials occurs primarily by a shear process called slip. Slip is restricted in most crystals to a single set of equivalent crystallographic slip directions in only a few slip planes that contain these directions. If a shear stress is applied to a crystal in a direction other than a direction of easy shear, slip still occurs in a preferred direction even though the component of shear stress resolved into that direction may be much lower than the applied stress. The mechanism of slip is the glide of dislocations, which you learned about in the Defects class (27-202) and which we analyzed again in the lecture on strength. Dislocations prefer to glide (at low temperatures) on close-packed planes with Burgers vectors corresponding to close packed directions, thus we observe \{111\}<110> slip systems in fcc metals, for example. Dislocations are present in essentially all crystalline materials but the stress required to move them varies strongly with bonding type. The non-directional bonding of (most) metals is what allows dislocations to move at small fractions of the elastic modulus, of the order $10^{-4}$ of the shear modulus in pure metals. Crystals with strongly directional bonding tend to have high resistance to dislocation motion and thus be brittle.

Slip Geometry
In a polycrystalline material in which the slip directions for each grain are oriented differently, slip in the preferred directions in each grain must be correlated with slip in the preferred directions of its neighboring grains in such a way as to maintain continuity at the grain boundaries. You learned about Schmid factors for slip in single crystals in the Defects class, which is based on the concept of critical resolved shear stress for dislocation motion. In polycrystal deformation, however, the microscopic distribution of stress and strain becomes complex because of the requirement for grains to continue to fit together. In general, when a piece of metal containing a large number of grains is plastically deformed, each grain undergoes approximately the same change in shape, i.e. the same strain, as does the piece as a whole. This leads to the “Taylor model” of polycrystal deformation, the basic assumption of which is that the strain that each grain undergoes is exactly the same as the macroscopic or bulk strain. This model allows us to predict the development of crystallographic preferred orientation (texture) and thus anisotropy. In randomly oriented f.c.c. polycrystals, we find that the ratio of the tensile stress that must be applied for plastic deformation to the critical resolved shear stress is approximately 3.1, which is known as the Taylor factor.

Strain Hardening
Another effect that accompanies plastic deformation is strain hardening. Deformation of a crystal by slip causes its resistance to further slip to increase. The mechanism of strain hardening is the entanglement of dislocations as they glide through each grain and the
consequent storage of dislocations within the grains. The phenomenon of strain hardening is of
great practical importance. In a structural part it constitutes an additional factor of safety. Even
if the load on a member reaches a magnitude where plastic deformation begins, the amount of
deformation will be limited by strain hardening and will not proceed further unless the load is
further increased. Consider a member of approximately uniform cross-section that is loaded in
tension to the point where plastic deformation just begins. Owing to minor variations in cross-
sectional area, or to stress concentrations, the first deformation will be localized. Local
elongation will result in a local decrease in cross-section area and, therefore, a further increase in
stress in the deformed region. If strain hardening did not occur, and the applied load remained
constant, then the first local plastic deformation would lead to catastrophic failure. If strain
hardening strengthens the first deformed regions enough to offset the accompanying decrease in
section, then failure does not occur even if the load is increased considerably beyond the
magnitude where the first local deformations begins. The deformation spreads uniformly over
the length of the member. Strain hardening and the limits on ductility are discussed in one of the
lectures.

Effect of Annealing: Recrystallization

Most of the work of plastic deformation is converted to heat, which raises the temperature of the
metal while being deformed. A small but significant percentage, however, is stored in the metal
in the form of dislocations. A distorted crystal containing a high density of dislocations is
thermally unstable. Heating of such a deformed crystal to temperatures where atom movements
can take place readily, leads to mobility of the dislocations, and a number of processes take place
that tend to return the crystal to its initial degree of perfection. One of these is recrystallization.
A boundary between a distorted grain and an undistorted grain will always tend to migrate in
such a direction as to extend the undistorted grain at the expense of the distorted one. At
sufficiently high (homologous) temperatures the migration can be very rapid. Recrystallization
is the formation and growth of new, more perfect grains in the distorted matrix, thus removing
the effects of cold work (i.e. plastic deformation performed at low temperatures where atom
movements are very slow). The nature of the original recrystallization nuclei from which these
new grains start to grow is still not completely understood. However, it is observed that the first
small recrystallized grains to appear form in regions of severe or complex distortion, for
example, near grain boundaries or triple lines. It is also observed that the greater the degree of
cold work the lower is the temperature at which new grains begin to appear and the larger is the
number of recrystallization nuclei. Thus the average grain size after recrystallization depends on
the severity of the previous cold work, being large for very small amounts of cold work, and
small for heavily cold worked metals.
Annealing: Grain Growth
At temperatures higher than that which is just necessary to complete recrystallization, another process takes place which also affects the size of the grains in a metal after an annealing treatment: grain growth is a slow coarsening of the average grain size by the growth of some grains at the expense of their neighbors. As the average grain size becomes larger and larger this process proceeds at an ever decreasing rate. Thus it would not be possible by grain growth alone to remove all the grain boundaries from a specimen (thereby obtaining a single crystal).

Grain Size
For the majority of practical, low temperature applications, the most favorable properties are associated with the finest possible grain size. For example, when steel is deformed at high temperature by rolling or forging, recrystallization and grain growth follow each plastic deformation operation almost immediately. An attempt is usually made to perform the final forming operation under conditions that will result in as fine a grain structure as possible. The reason for desiring a fine grain size is that it typically maximizes both strength and toughness (through minimizing crack lengths). This is known as the Hall-Petch effect, see below. For most other means of modifying microstructure, such as precipitation, increasing strength is correlated with decreasing toughness, which requires a trade-off in properties to be made. A more sophisticated benefit of fine grain size is that very fine-grained materials can be made to be superplastic, i.e. can be subjected to enormous strains before failure intervenes. This makes them easier to form into complicated shapes, e.g. by blow-forming them, just as one does with polymers.

Grain Size Dependent Properties – the Hall-Petch Effect
A well documented effect in most metal alloy systems is the Hall-Petch effect, as discussed in nearly all textbooks on mechanical behavior of materials (e.g. Hertzberg, Dieter, Courtney). The original paper discussed the dependence of fracture toughness on grain size and showed an increasing toughness with decreasing grain size. This was followed by the finding that yield strength also increased with decreasing grain size. The mathematical relationship in both cases turned out to be one in which the property, such as $\sigma_y$, depends on the inverse of the square root of the grain size, $d$:

$$\sigma_y = \sigma_0 + k d^{1/2}$$

† There are circumstances under which a large grain size is advantageous – what application do you know of that requires single crystals, and why are single crystals better than polycrystals?
The exact mechanism for these microstructure-property relationships is still obscure but it is nonetheless very useful because grain size represents one of the few microstructural parameters that allows the materials engineer to optimize strength and toughness at the same time. The best explanation available is that dislocations pile up at grain boundaries (some, but not all) leading to a local stress concentration; this stress concentration in turn enables dislocations to be activated in the neighboring grain so that slip can continue. Most other strengthening mechanisms (e.g. precipitation hardening) decrease fracture toughness.
Objectives

The objectives of this laboratory are as follows.

1) To acquaint the student with a basic annealing process, i.e. recrystallization, that is used in essentially all processing of structural metals. Specifically, this experiment shows the variation in grain size that can be obtained by varying the strain level and the temperature.

2) To give the student experience with metallographic techniques such as grinding, polishing, etching, and electropolishing.

3) To illustrate one of the issues in stereology, namely the challenges of extracting useful information from different measurement methods. The example here is that of grain size measurement obtained by (a) measuring areas and (b) line intercepts.

4) To obtain data to test the Hall-Petch relationship by measuring grain size and microhardness (as representative of yield strength).

5) To acquaint the student with basic applications of statistics.
Specimen Preparation

The material used in this experiment is 70-30 brass (70% copper, 30% zinc). The samples handed out to the class have already been processed as follows. Pieces 1/2" x 1/4" x 2" long were first cut out of a brass sheet. Each piece was machined to a wedge shape as shown below.

The photo below (31st Aug. 2015) shows two samples from one end to show the wedge shape, with a third sample (top) that was passed through the rolling mill.
The wedges were then annealed in an inert atmosphere at 850°C, cold rolled and drilled, to yield the following specimen.

![Diagram of specimen dimensions]

**Experimental Procedure**

Each group will be given a specimen of large-grained 70-30 (70% copper - 30% zinc) alpha brass. The specimen has been prepared in such a way that a gradient of cold work (strain) exists across its width. Along one edge it is undeformed while at the other edge there has been a 50% reduction of thickness by cold rolling.

This specimen will be heated under conditions resulting in a temperature gradient along its length (at right angles to the cold work gradient) from 100°C to 900°C. Therefore, in this one bar of brass it will be possible to study under the microscope the changes in structure caused by heating a cold worked metal for all possible combinations of temperature from 100°C to 900°C and degrees of cold work from 0 to 50% reduction in thickness. Time of heating will be, of course, constant for all parts of the specimen.

1. Heat treatment of specimen: the specimen should be placed in a furnace so that its lower end is just submerged below the surface of water contained in a beaker. When the specimen is in position, the power input to the furnace should be increased until the temperature at the top of the specimen reaches 900°C as indicated by a thermocouple placed in a small drilled well at the top of the specimen. It is essential to cover the top of the furnace with insulating wool to insure that the top of the sample reaches a temperature of at least 880°C. Experience indicates that the success of the (annealing) experiment is critically dependent on the quality of the insulation. *If the specimen is not well insulated then it will not be possible to reach the target temperature and recrystallization will not take place.* It is also important to locate the sample as
centrally as possible inside the induction coil. It is also important to maintain the bottom of the sample in contact with the top of the water but only to a depth of a millimeter or two; since water evaporates quickly, care must be taken to replenish the water and maintain contact. Loss of contact with the water will allow the sample temperature to "run away". Too much depth of contact will prevent the sample from heating to the desired 900 °C. If the temperature is as low as 880 °C, it will be necessary to hold the specimen twice as long as at 900 °C because the grains grow more slowly at the lower temperature of this Cu-Zn alloy. The temperature must not exceed 920 °C as this is the liquidus temperature. Hold at 900 °C for five minutes, then lower the specimen into the water (e.g. by cutting the suspension wire) and turn off the power. When the heating element has cooled below red heat, remove the brass strip from the water bath (or just withdraw the beaker from the bottom of the apparatus that contains the water.

2. Mount the specimen in the grinding clamp provided, and grind the flatter surface through 240, 320, 400 and 600 grinding papers. The amount of grinding necessary on each grade of paper can be determined by changing the direction of grinding by 90° each time a change to finer paper is made. Grinding on a given paper is continued until the marks from the previous grade are just removed. Be sure to use only light pressure throughout the grinding particularly on the last paper. Next polish the surface on a polishing wheel using Linde A abrasive slurry, long enough to remove the scratches from the last grinding operation. Never touch the finished surface with your (greasy) fingers.

3. The final polish is to be produced by electropolishing, a process in which the specimen is made the anode of an electrolytic cell. With the electrolyte and current density employed, metal is removed more rapidly from the microscopic high spots of the surface than from the low spots. Therefore, a leveling action results. Also any remaining deformed metal due to grinding is removed. The electrolyte to be used in this case has the following composition:

200 ml orthophosphoric acid,
200 ml ethanol,
400 ml distilled water.

The electropolishing will be carried out according to the directions of the laboratory instructor for 45 seconds at 14 volts with the specimen face down and only far enough into the electrolyte to cover the face to be polished. The resulting surface must be scratch-free so that you can then etch and characterize the grain structure.
4. To reveal the structure of the surface the specimen should be etched for 45 seconds at 2 volts, or as instructed, quickly rinsed in water, then in alcohol, and dried under an air blast. The photo below illustrates what you can expect to obtain, with the variable grain size visible to the naked eye.

5. After drying the etched specimen, make a series of Rockwell "F" hardness measurements at 1/8" intervals along the most severely worked edge and across the submerged end. Note that the softest parts of the specimen may give negative hardness readings. You will also have to use a microhardness tester, which has the advantage that the results have dimensions of stress because these data can be related to the yield strength of the material. It is the microhardness data that you will use to make a Hall-Petch plot (of hardness versus square root of grain size). The photo below illustrates two more of the samples with several series of indents.
6. Mount specimen on a glass slide with plasticene using a specimen press to make the top surface of the specimen parallel with the glass slide. This ensures that the specimen remains in focus as it is moved around on the microscope stage.

7. The entire surface of the specimen should be carefully studied both visually and under an optical microscope. Make a rough sketch of the specimen, recording as many observations as you can for the report. Correlate the observed structures with strength, as indicated by the hardness measurements.

8. The (Rockwell) hardness indentations cause additional plastic deformation in the grains immediately surrounding the indentation. Since the surface was polished prior to the hardness measurements, slip can be observed under the microscope as fine dark lines where local shearing on slip planes in directions not parallel to the surface has produced a surface step. Note the different orientation of slip planes in neighboring grains.
Report

1. A formal technical report will be submitted for this Laboratory 1 as a Word document, preferably converted to PDF, and submitted via BlackBoard. Each student shall submit an individual report.

2. Each student is responsible for the originality of his/her own portion of the technical report and the data utilized. Each report must contain a statement that details the division of effort since it is expected that the group will collaborate on all aspects of the Lab except the final analysis of the data. Provided that the sources of data are cited, groups are strongly encouraged to share data so that analysis of the Hall-Petch effect, for example, is statistically meaningful. Copying from unidentified sources such as prior reports is cheating, however.

3. The chief criterion of a good report is that it shall convey what the group did and learned and the conclusions the writers have drawn from the data in a way that is clear, concise, neat and readily interpretable by the reader. A student's report must show an understanding of the principles upon which the experiment was based. A report that is poorly organized and difficult for the instructor to follow will not be given a high grade. For each section, identify which students contributed to its writing.

Your report should include an abstract, followed by sections entitled:

1. Title Page
2. Introduction,
3. Procedure
4. Results,
5. Discussion,
6. Conclusions (or Summary),
7. Acknowledgements,
8. Bibliography/References,
9. Appendices

General Report Requirements

(Please review before writing each report)

1. Title Page -- The title page, should include a title identifying the report, the lab group letter or number, and the names of the group members. The title itself should be concise, yet descriptive and include key words that identify the experiments and convey the message of the report. Also included on the title page is the date on which the report was finished as well as the name of the Course Assistant(s) who supervised the experiment.

2. Abstract -- The abstract is simply a complete capsule report that can function independently of the body of the work. It should be 100 to 200 words in length, all in one paragraph, and should emphasize key results and conclusions, as well as interpretations and recommendations. The
abstract should include a statement of the work actually done and should be written on a separate page after the rest of the report has been completed.

3. Introduction -- The introduction should be one or two pages in length. It should begin with a brief discussion (2 to 4 paragraphs) of the background and/or theory behind the experiment. A mere restatement of the objective found in this handout is not sufficient. The final paragraph in the introduction should define the scope and nature of the experiments, i.e., state very succinctly what was done.

4. Experimental Procedure -- The test procedure should be described concisely and should include any operational features that were discovered during the course of the experiment. For instance, time to reach steady state, length of an individual experiment and techniques used in sampling are important. The testing equipment and materials used in the project should be described in this section in sufficient detail so that they could be duplicated by the reader. Common items (balances, graduated cylinders, etc.) need not be named.

5. Test Results -- This is perhaps the most important part of the report, for it represents your interpretation of the results of the experiment. Even though the results may be most effectively presented in tabular or graphical form, they must be preceded by text describing them. If appropriate, an estimate of precision of the data in terms of a relative uncertainty or confidence limit should be included. References should be made in the text to figures, graphs, tables, and equations, which are inserted in the report immediately following reference to them or on the next page. Sample calculations should be included and are best presented as an Appendix.

6. Division of Effort – Detail who did what in the group.

7. Analysis & Discussion -- In this section, which each individual must write separately from the group, analyze and compare your results with theory or previous work. Discuss points of similarity or divergence and, if possible, give sources of these discrepancies. It is quite proper to include another group's curves or data points on your plots; just be sure to identify the source of the data. Evaluate trends in the results and any conclusions that can be drawn. Development of conclusions must begin in this section where they can be supported by logical argument, even though they will be dealt with exclusively in the next section. In the event of poor results, explain how the experiment should be modified to obtain satisfactory ones. This important section demonstrates your thinking and understanding of what was intended. In order to obtain a high grade for your report, you will need to relate your results to the ‘bigger picture.’ Therefore a requirement for this report is that you find (and cite) at least one original paper (in a peer-reviewed journal such as *Acta materialia* or *Metallurgical Transactions*) that addresses recrystallization in some way and that you can relate to your own findings.

Either

8A. Conclusions -- This section should be a numbered listing of the more important test results and major conclusions reached in the discussion. Conclusions are single sentences and typically 5 to 8 of them should be sufficient.

Or

8B. Summary -- This section is a recapitulation of major points made in the Discussion in paragraph form and is intended to leave the reader with a strong impact and provide a ready reference. (Be brief but complete). You may use either a summary or conclusion section as you prefer, but not both.
9. References -- This is a list of references in the order in which they are cited in the report.

1. Use journal abbreviations as follows (article titles need not be included):

2. References to books should include the title and pages within the book.

9. Appendices -- Detailed technical information that would slow down development of the results in the main body of the work should be included in these sections. An appendix contains information that would enable a reader to go into depth and completely check and reproduce the results. Included should be at least one complete set of sample calculations from original data to final results, copies of original data sheets, computer programs, etc. Appendices should not contain information vital to the report such as graphs or tables of data that properly belong in the Test Results section.

Report Grading

Reports will be graded on the following basis:

   Abstract                                10%
   Introduction                            5%
   Experimental Procedure                  5%
   Test Results                            25%
   Analysis & Discussion                    25%
   Conclusions or Summary                  10%
   Use of English Language and Neatness    20%
       Grammar and spelling
       Sentence and paragraph structure
       References and Appendix, if applicable

Grading for reports submitted late will be reduced 20% for the first week and an additional 20% for each succeeding week.

Students are referred to the University Policy About Cheating and Plagiarism (http://www.cmu.edu/policies/documents/Academic%20Integrity.htm). It is the policy in this course to discourage cheating to the extent possible, rather than to try to trap and to punish. In particular, this report expects you to share a substantial portion of the content among your group, as detailed above. On the other hand, in fairness to all concerned, cheating and plagiarism will be treated severely. Note that point number three below is addressed explicitly in this manual, with respect to using results from other students.

"Cheating occurs when a student avails her/himself of an unfair or disallowed advantage which includes but is not limited to:

1. Theft of or unauthorized access to an exam, answer key or other graded work from previous course offerings.
2. Use of an alternate, stand-in or proxy during an examination.
3. Copying from the examination or work of another person or source."
4. Submission or use of falsified data.
5. Using false statements to obtain additional time or other accommodation.
6. Falsification of academic credentials.

Plagiarism is defined as the use of work or concepts contributed by other individuals without proper attribution or citation. Unique ideas or materials taken from another source for either written or oral use must be fully acknowledged in academic work to be graded. Examples of sources that should be referenced include but are not limited to:

- Text, either written or spoken, quoted directly or paraphrased.
- Graphic elements.
- Passages of music, existing either as sound or as notation.
- Mathematical proofs.
- Scientific data.
- Concepts or material derived from the work, published or unpublished, of another person.

Unauthorized assistance refers to the use of sources of support that have not been specifically authorized in this policy statement or by the course instructor(s) in the completion of academic work to be graded. Such sources of support may include but are not limited to advice or help provided by another individual, published or unpublished written sources, and electronic sources. Examples of unauthorized assistance include but are not limited to:

1. Collaboration on any assignment beyond the standards authorized by this policy statement and the course instructor(s).
2. Submission of work completed or edited in whole or in part by another person.
3. Supplying or communicating unauthorized information or materials, including graded work and answer keys from previous course offerings, in any way to another student.
4. Use of unauthorized information or materials, including graded work and answer keys from previous course offerings.
5. Use of unauthorized devices.
6. Submission for credit of previously completed graded work in a second course without first obtaining permission from the instructor(s) of the second course. In the case of concurrent courses, permission to submit the same work for credit in two courses must be obtained from the instructors of both courses.

Technical Requirements for the Report

Each report should include at least the following items:

1. A paragraph on the objectives of this lab.
2. Details on the experimental procedures.
3. A record of the hardness measurements.

In addition:
(a) Provide a plot of hardness vs. % reduction in thickness (cold work) from the submerged end, assuming that % reduction varies linearly across the width of the specimen from 0 to 50%.

(b) Provide a plot of hardness vs. heat treatment temperature from measurements along the most severely cold worked edge. You should assume that temperature varies linearly from 900 to 100 °C along the length of the specimen.

(c) Identify the recrystallization temperature corresponding to 50 % cold work from the plot in part (b) above.

(d) Provide a plot of grain size vs. strain level from measurements along a line for which the temperature was high enough to result in complete recrystallization, and for which you can observe a systematic variation in grain size (i.e. decreasing with increasing strain level). You should assume that temperature varies linearly from 900 to 100 °C along the length of the specimen. You must measure the grain size by both the area method (count grains per unit area and convert to an ASTM grain size number) and by the linear intercept method; see the lecture notes for details on method. You must provide an estimate of the error in the grain size measurement (in the form of an error bar on the plot) and write down how you determined it. See the appendixes below for more information.

(e) A plot of microhardness (not the Rockwell indents) against inverse square root of grain size. You must provide an estimate of the error in the grain size measurement (in the form of an error bar on the plot) and write down how you determined it. Extract from the plot a value for the constant of proportionality in the Hall-Petch relationship; when fitting data, it is best to arrange (transform) the data in such a way that a perfect fit is a straight line. It is acceptable, even desirable, to include data from other students, provided that you acknowledge the source (e.g. by use of different symbols in your plots), in order to obtain as a wide a range of grain size as possible. (If you would like to convert your Rockwell hardness values to microhardness values and use those for the Hall-Petch analysis, discuss this with the instructor.)

4. Provide a record of the microscopic examination of the sample, including:
(a) A rough (schematic) sketch of the whole sample
(b) Three photomicrographs of the microstructure you feel are most significant for discussion of what you have observed. Indicate the magnification with a bar and the length of the bar (so-called micron marker). *Note that your grade will depend on the quality of the micrographs that you include in your report. Unless you can image the grain structure, preferably with no scratches or other surface damage, you will not be able to characterize the microstructure as required.*
(c) A description of the method(s) used to analyze the microstructures in each area so as to obtain average grain size and the grain size distribution. The standard method is to trace (by hand) the grain boundaries in a micrograph, scan the resulting tracing and then analyze using the Image-J program. Those skilled in the use of Photoshop, for example, may be able to scan micrographs directly and skeletonize the grain boundary network directly on the computer. Further detail is given below.
(d) Two histograms showing the grain size distribution, one of which is a simple frequency plot and the second a *probability density histogram*. Normalize the values for the second plot so that the total area is equal to one; consult a statistics text if in doubt.

5. Discuss the relationships between recrystallized grain size versus cold work, recrystallized grain size versus temperature, and recrystallization temperature versus cold work. Relate your discussion to the sketch and micrographs of part 4 above. Address the following specific questions:
   (a) Explain the physical basis for recrystallization – what is going on inside the material during the recrystallization process?
   (b) What mechanisms at the atomistic level control the process?! Comment on the different methods of determining grain size. In particular, compare your grain size distribution with a similar result from the literature.
   (d) Why do you observe a range of grain sizes and not just a single size?
   (e) What analytical function would you choose to fit your data on grain size distribution? Gaussian? Hint: if you look in the books or in papers, you’ll find that it is common to plot a histogram of frequency versus the *logarithm* of the grain size because the resulting distribution is often close to Gaussian (“normal distribution”).
   (f) How wide a range of grain sizes do you observe? Hint: if you do not have enough data to fit a standard distribution (see above), then recording the “Full Width Half-Maximum (FWHM)” is acceptable, i.e. the width of the distribution between the points where the frequency has fallen to 1/2 of the peak value.
(g) How might you apply your knowledge of statistics to this data set? For example, you may have learned to make probability plots so think about how to apply them here.

(h) Discuss your Hall-Petch analysis: how small would the grain size have to be to reach the theoretical limit of yield strength in this material?

(i) As noted above, a requirement for this report is that you find (and cite) at least one original paper (in a peer-reviewed journal) that addresses recrystallization in some way and that you can relate to your own findings. We recommend that you use one of the online search engines (not just Google!) that the CMU Library makes available. Ask the instructor or a CA, or a librarian if you are uncertain as to what keywords to use in a search.
Question & Answer for Lab. 1, 27-301, 11 Oct. 12

**General**
- Do write your report in continuous prose using the suggested headings.
- If your data do not support the Hall-Petch concept, then say so. Nevertheless, be careful to exclude the unrecrystallized regions (see discussion below). Also I encourage you to share data among groups.
- It is acceptable to use the area-based grain count method instead of ImageJ especially where you observe very few grains in an image.
- Do include annealing twin boundaries when evaluating grain size.

I had a few quick questions regarding the lab. I know the protocol asks for 3 images to be included in the results section, would you like the rest of the pictures placed in the appendices?

Yes, most of the images should go in the appendices. The manual does indeed ask for three micrographs as supporting evidence. You may vary the number as you find necessary in order to present a clear story in your report. Keep in mind, however, that fewer than three is unlikely to be enough and if you include too many (in the main body of the report) you may confuse the reader!

Also for measuring grain size, our group isn't sure what an appropriate number of lines we should use for the average of the line intercept method to calculate grain size. Obviously more lines will produce a better average but there is a reasonable number we should shoot for in the lab report?

The best way to approach this type of question is to ask one's self about error analysis. If one picks 10 lines, for example, how many intercepts does that yield and what estimated error does that give? Of course, this suggests that we could add some text about error analysis to the rubric but that will have to wait for the next time.

I know it's not really important but it terms of formatting, where should the abstract be placed? Should it be on a separate page before the rest of the report?

The Abstract should always be at the top of the document (just below the Title, Author etc.). It does not need a page-break after it. In general, it is a good idea to format the report so that it looks tidy and is easy to read.

There is some conflicting information on where it should go in the protocol. Finally, since the data for the class hasn't been posted yet will our reports still be due Friday or will we be given extra time to work on them.

Some individuals have already requested extensions and we are granting them, within reason.

When we are plotting hardness and reporting our data in tables in the appendix/results do you want our hardness units to be Hv or MPa?
Either unit is acceptable. I prefer MPa simply because that makes it easier to compare results to the literature.

What do we do if ImageJ only finds a few grains in an image?

Unfortunately the grain size in our brass can sometimes be so coarse that only a few grains are in the field of view even for the lowest magnification. The only thing to do is to either analyze multiple images from the area of interest, or to take a micrograph with the stereo microscope if the grain structure is clear enough (e.g. to trace over by hand).

What kind of grain size measurement do we need for making a histogram? What does it mean when you refer to “log-normal”? What does “full width half max (FWHM)” mean?

You need individual grain measurements so it’s best to measure areas (e.g. with ImageJ) in a region with small enough grains that you can obtain at least several dozen grains. The expected result is that making a histogram of the grain sizes (diameter or radius) will not resemble a bell curve. If, however, you take the log of each grain size and then make a histogram you will then obtain something closer to a bell curve. FWHM is a term often applied to the characterization of x-ray diffraction peaks. Think of the peak as a bell curve; draw a horizontal line at one-half of the peak height; measure the width of the peak at that half-height, and you have the FWHM.

(from the Q&A in 2010)

In the Appendix where the procedure for the area method of grain size measurement is given, step 5 says to convert the size to relative size, and to convert the frequency to a probability of occurrence by normalizing the area. Could you explain a little bit more what you mean by normalizing the area, please? Also, I'm assuming that we use the relative sizes when we calculate the Hall-Petch effect, but since the relative area-based sizes are much smaller than the linear-based sizes (which it does not say to normalize), does that mean that we need two separate plots?

“Reduced or normalized area” just means dividing each value by the average or mean value.

To make a Hall-Petch plot, use the sizes (with units) themselves, not the reduced/normalized values.