LABORATORY EXERCISE #1
STEEL DENSITY AND MICROSTRUCTURE

OBJECTIVES

- Utilize the procedures for grinding, polishing and etching steel metallurgical samples to reveal the microstructure.
- Observe the grain structure of low carbon steel at a magnification in excess of 4000X displayed on a video monitor.
- Experimentally determine the densities of steel samples from measurements of true mass and measurements of apparent mass when samples are submerged in water.
- Analytically determine the theoretical density of body-centered cubic (BCC) iron based on a crystal structure model.
- Perform a statistical test of hypothesis comparing sample densities from measurements with the theoretical density of iron.

PROCEDURES

Metallography

- Obtain a steel sample from the lab instructor.
- Grind one end of the sample using the grinding platforms, starting with the coarse grit paper and ending with the fine grit paper.
- Polish the same end of the sample, first with the wheel containing 0.30-micron alumina polishing compound and then with the wheel containing 0.05-micron alumina polishing compound. Make sure the sample is cleaned with clear water before each polishing step to avoid contamination of the polishing wheels.
- Etch the polished end of the sample for about 15 – 20 seconds in NITAL (3% nitric acid in methanol). Rinse the sample immediately in clear water and dry it with a blow dryer.
- Place your sample under the microscope and focus on a clear area that shows the grain structure of the metal on the video monitor. Try to identify several different types of grains by coloration and try to identify grain boundaries. Sketch several grains of the microstructure.

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Density

- Use a balance to measure the true mass of your sample, M_T, in grams.
- Use a length of fine thread and a small piece of tape to suspend the sample from the pan of the balance into a beaker of water. Make sure that the sample is completely submerged and not touching the sides of the beaker.
- Record the mass of the submerged sample, M_S, in grams.
- Return your sample to the instructor.
- Obtain the values of M_T and M_S from all other groups in your lab section such that a statistical sample for the density can be generated.

DATA ANALYSES

- Using the mass data for each sample, calculate the true densities of the steel samples as follows:

$$\rho = \left(M_T / (M_T - M_S) \right) \rho_{H2O}$$

where: ρ_{H2O} = density of water = 1.0 g/cm^3

- Using one unit cell as a basis, calculate the theoretical density of body-centered cubic iron, ρ_{Fe}, at room temperature.
- Use your sample data to test the hypothesis that the density of the steel used in this lab equals the theoretical density of iron, calculated above, versus the alternative that the density of this steel is not equal to that of iron. Use small sample methods (student's "t" distribution) and a 5% level of significance. Note that, as stated above, this is a two-sided test of hypothesis. Make sure that you clearly state your hypothesis and the alternative hypothesis, and state your conclusion in a clear and concise sentence.

REPORT

Write a short format lab report (tech brief) that includes the following elements:

- A brief statement of the objectives of the exercise.
- A detailed description of the sample preparation process for steel metallographic specimens.
- A sketch of several grains of the steel microstructure observed, identifying several grain boundaries.
- A detailed description of the experimental procedure used to determine the densities of the steel samples.
- A table summarizing the values of M_T, M_S and ρ for all samples measured in your lab section.
- A summary of the test of hypothesis based on the density data and the theoretical density of iron. The hypotheses tested and the meaning of the results of the test must be stated clearly.
- A brief summary of the results of this exercise.
- An appendix containing the calculations for the theoretical density of iron at room temperature.
- An appendix containing the calculations for the test of hypothesis.

APPENDIX "A"
STATISTICAL RELATIONSHIPS

SMALL SAMPLE CONFIDENCE INTERVAL FOR THE POPULATION MEAN, μ

K = desired percent confidence

$\alpha = (1. - K/100.) / 2.$

n = sample size

ν = degrees of freedom = (n - 1)

$t_{\alpha,\nu}$ = normalized probability distribution variable (from table*)

$\overline{X}$ = sample mean = $\sum X_i / n$

S = sample standard deviation = $\{ \sum (X_i - \overline{X})^2 / (n - 1) \}^{0.5}$

THEN

$$\overline{X} - (t_{\alpha,\nu} S / \sqrt{n}) \leq \mu \leq \overline{X} + (t_{\alpha,\nu} S / \sqrt{n})$$

* Note: "t – tables" are not all assembled in exactly the same manner . . . . be careful!

APPENDIX "B"
STATISTICAL RELATIONSHIPS

SMALL SAMPLE TEST OF HYPOTHESIS CONCERNING THE POPULATION MEAN, μ

Null hypothesis = H_0 : population mean = μ = claimed value = μ_0

Alternative hypothesis = H_a : $\mu \neq \mu_0$ (for a two-sided test, see below for a one-sided test)

K = desired percent level of significance (maximum probability of rejecting a true hypothesis)

$\alpha = (K / 100.) / 2.$ (for a two-sided test only . . . see below for a one-sided test)

n = sample size

ν = degrees of freedom = (n - 1)

$t_{\alpha,\nu}$ = normalized probability distribution variable (from table)

$\overline{X}$ = sample mean = $\sum X_i / n$

S = sample standard deviation = $\{ \sum (X_i - \overline{X})^2 / (n - 1) \}^{0.5}$

$t_{calc} = | (\overline{X} - \mu_0) / (S / \sqrt{n}) |$

THEN:

* Reject H_0 if $t_{calc} > t_{\alpha,\nu}$

* Reserve judgment, i.e., not enough evidence to reject H_0, if $t_{calc} < t_{\alpha,\nu}$

NOTE THAT FOR A ONE – SIDED TEST:

H_a: ($\mu > \mu_0$), or ($\mu < \mu_0$), then this becomes a one-sided test of hypothesis, and

$\alpha = K / 100.$

APPENDIX "C"
STATISTICAL RELATIONSHIPS

SMALL SAMPLE TEST OF HYPOTHESIS CONCERNING TWO POPULATION MEANS, μ_1 AND μ_2 (ASSUME EQUAL POPULATION VARIANCES AND UNPAIRED DATA)

Null hypothesis = H_0: $\mu_1 = \mu_2$ (or: $(\mu_1 - \mu_2) = 0$)

Alternative hypothesis = H_a: $\mu_1 \neq \mu_2$ (or: $(\mu_1 - \mu_2) \neq 0 \rightarrow$ two - sided test)

K = desired percent level of significance

$\alpha = (K / 100.) / 2.$ (for a two-sided test only . . . see below for a one-sided test)

n_1 = sample size #1, n_2 = sample size #2

v = degrees of freedom = $n_1 + n_2 - 2$

$t_{\alpha, v}$ = normalized probability distribution variable (from tables)

$\overline{X}_1$ = mean of sample #1 = $\Sigma X_{1,i} / n_1$

$\overline{X}_2$ = mean of sample #2 = $\Sigma X_{2,i} / n_2$

S_1 = standard deviation of sample #1 = $\{\Sigma (X_{1,i} - \overline{X}_1)^2 / (n_1 - 1)\}^{0.5}$

S_2 = standard deviation of sample #2 = $\{\Sigma (X_{2,i} - \overline{X}_2)^2 / (n_2 - 1)\}^{0.5}$

S_p = pooled standard deviation = $\{((n_1 - 1)S_1^2 + (n_2 - 1)S_2^2) / v\}^{0.5}$

$$t_{calc} = \left| (\overline{X}_1 - \overline{X}_2) / (S_p \sqrt{(1/n_1 + 1/n_2)}) \right|$$

THEN:

* Reject H_0 if $t_{calc} > t_{\alpha, v}$
* Reserve judgment, i.e., not enough evidence to reject H_0, if $t_{calc} < t_{\alpha, v}$

NOTE THAT FOR A ONE – SIDED TEST:

H_a: $(\mu_1 > \mu_2)$ or $(\mu_1 < \mu_2)$, then this becomes a one-sided test of hypothesis, and

$\alpha = (K / 100.)$

APPENDIX "D"
STATISTICAL RELATIONSHIPS

LINEAR REGRESSION BY THE METHOD OF LEAST SQUARES

n = number of data point pairs

X_i = values of the independent variable

Y_i = values of the dependent variable

$\overline{X}$ = mean value of $X_i = \sum X_i / n$

$\overline{Y}$ = mean value of $Y_i = \sum Y_i / n$

$\overline{XY}$ = mean value of products = $\sum(X_i Y_i) / n$

$\overline{X^2}$ = mean value of $X_i^2 = \sum(X_i^2) / n$

NOTE: $\overline{X^2} \neq (\overline{X})^2$

THEN THE LINEAR REGRESSION EQUATION IS GIVEN BY:

$$Y = a + bX$$

WHERE:

$$b = \text{slope} = (\overline{XY} - \overline{X}\,\overline{Y}) / (\overline{X^2} - (\overline{X})^2)$$

$$a = \text{intercept} = \overline{Y} - b\overline{X}$$

TEST OF HYPOTHESIS FOR MORE THAN TWO SAMPLES USING ANALYSIS OF VARIANCE (ANOVA)

Null hypothesis = H_0: $\mu_1 = \mu_2 = \mu_3 = \ldots\ldots$ (Population means are equal for all treatments)

Alternative hypothesis = H_a: Not all population means are equal for all treatments.

Level of significance = maximum probability of rejecting a true hypothesis (for "F"- table).

σ_B^2 = variance between samples.

σ_W^2 = variance within samples.

$F_{Calc} = \sigma_B^2 / \sigma_W^2$ = calculated value of the "F" statistic.

F_{Crit} = Critical value of the "F" statistic for selected level of significance (from "F" - table).

$F_{Calc} > F_{Crit}$: Reject H_0.

$F_{Calc} < F_{Crit}$: Reserve judgment concerning H_0, i.e., not enough evidence to reject H_0.

Where:

k = number of treatments

n_c = number of data values for each treatment (not necessarily all the same value)

$N = \Sigma\, n_c$ = total number of data values

x_i = individual data values

$T_c = \Sigma\,(x_i)$ for $i = 1$ to n_c (total of data values for each treatment)

$T_a = \Sigma\,(x_i)$ for $i = 1$ to N (total of all data values = $\Sigma\, T_c$)

$T_s = \Sigma\,(x_i^2)$ for $i = 1$ to N (total of squares of all data values, $\neq T_a^2$)

$SST = \Sigma(T_c^2 / n_c) - T_a^2 / N$

$SSE = T_s - \Sigma(T_c^2 / n_c)$

$\sigma_B^2 = SST / (k-1)$

$\sigma_W^2 = SSE / (N-k)$

$$\left.\begin{array}{l}\text{Degrees of freedom in the numerator} = k-1\\ \text{Degrees of freedom in the denominator} = N-k\end{array}\right\}\ \text{For locating } F_{Crit} \text{ in the "F" - table}$$

LAB REPORT GUIDELINES

- All reports must be written in the English language.
- All statements must be written using proper sentence structures with subjects and verbs.
- All words must be spelled correctly.
- All sentences must utilize proper punctuation.
- Always refer to what was done using the past tense.
- Do not use pronouns or the names of persons.
- Do not use "run-on sentences". Minimize the use of words such as "and then".
- Do not write procedures like an instruction manual. You are not telling someone else what to do. You are describing what you did. Write the procedures using a passive voice.
- When appropriate, all numerical information must be accompanied by proper units.
- All tables must be numbered and labeled, e.g., Table 1, Density Data.
- All graphs must be numbered and labeled, e.g., Figure 2, Stress versus Strain.
- All tables and graphs must include proper labeling to identify the meaning of numerical information and associated units.
- In general, tables and graphs should be discussed in the body of your report. Don't flop a page full of numbers before the reader and then say nothing about what they represent or where they came from.
- Reports must be written and assembled in a concise and logical manner. A stranger with minimal understanding of the subject matter should be able to read your reports and deduce the essence, and perhaps even some of the details, of what you did and what happened. You are not relieved of the obligation to write complete documents because you think that readers already know what you would say. Assume nothing.
- Imagine that your reports are for the benefit of your employer, who signs your paychecks, but may know little or nothing about the details of your work. If you were the employer you would want reports to be accurate, complete and understandable. You have to assume that others expect no less.