

Materials Laboratory Experiment Manual

ChBE 418

Department of Chemical and Biomolecular Engineering

Ohio University

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Materials Science Laboratory Manual
Department of Chemical and Biomolecular Engineering
Ohio University

Laboratory Rules and Instructions
Description of Experiments

Based on the September 1999 Version with minor changes

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To the Student

Welcome to the Ohio University Materials Science Laboratory, Department of Chemical and Biomolecular Engineering, Ohio University. We hope that you find the laboratory an interesting and informative experience. The purpose of this course is to acquaint you with several of the techniques used to modify and test the properties of the various types of materials available to the practicing engineer.

This manual contains descriptions of the experiments that you will perform as part of this course. In addition, the various rules and regulations pertaining to the operation of the laboratory are described.

The experiments in this manual are designed to give you first-hand experience with some of the material treatment and testing techniques that you have learned about in your materials classes and that are used by engineers every day. We ask that you approach the laboratory the way a practicing engineer would. By the end of the quarter, we hope that you will be familiar with the principles of several of the techniques used in materials modification and testing.

To make the most effective use of the time you spend in the materials laboratory, we expect that you will *carefully study each experiment before you come into the laboratory*. To assist you with this, most of the experiments contain several pre-lab questions to which you will be required to answer them in the prelab that is handed in at the beginning of the laboratory period except the first. By following this procedure, you will not need to waste precious time reading the experiment or wondering what to do. Early preparation often saves much time when the formal lab session begins.

Because each experiment section contains pages on which you should record your experimental data, you are not required to maintain a separate laboratory notebook. (You may certainly do so if you wish.) *However*, you will still be responsible for noting and recording all phenomena and observations you make during the course of an experiment. This information will be included in the report you prepare for each experiment.

With careful attention to detail in your preliminary preparations for lab, your performance of the experiments, and your subsequent writing, your materials laboratory experience should be

fruitful. We hope that you will think so at the end of the quarter.

Laboratory Rules

In accordance with the rules of the Stocker Engineering Center, there will be NO SMOKING in the Materials Laboratory. In addition, no chewing tobacco, spitting, etc. is permitted. No food or drink is permitted in the laboratory at any time. Deposit all trash in the receptacles near the door upon entering the laboratory.

SAFETY is each person's full-time responsibility. Plan your work before starting. Always exercise extreme care with chemicals which may be poisonous, corrosive, flammable, and/or explosive. Be careful with glassware. Be careful when using powered equipment, such as the grinders, rolling mill, saws, and polishers. Proper lab attire includes long pants, shirts with at least short sleeves (no tank tops), and close-toed shoes.

Cleanliness is mandatory in the laboratory. All glassware must be cleaned and stored after use. Equipment is to be cleaned and covered after use. Pay special attention to the hardness testers and microscopes, which are delicate, precision instruments. Spilled chemicals should be brushed off and/or wiped away, and the benchtops should be wiped clean. This is particularly important around the polishing tables and sinks. Safety goggles are required.

The course instructor is in charge of the laboratory. In the absence of the instructor, the laboratory teaching assistant is in charge.

Some Expectations

The prerequisite for this course is ChBE 231, and it is assumed that you will make use of what you learned in that course as you move through this course. In particular, a number of the prelab questions you will encounter as you do the various experiments require you to seek information that is not in this laboratory manual. It is expected that you will draw on what you learned in 231 and other courses (such as chemistry) both to answer these questions and to write your laboratory reports. Having sold back to the bookstore your 231 text is not considered any form of excuse. Alden library, with all of its resources, is available to you.

Hardness Testing and the Statistical Significance of Data

Introduction

The property variation most easily followed in this laboratory is the change in hardness associated with some particular treatment, whether it be the kinetics during phasal changes such as age hardening, creep, cold work, or heat treatment. Several of these will be encountered in subsequent experiments. Among the measurements of a material's hardness are the mineral scratch hardness (Moh's) test, the file hardness test, the elastic rebound hardness (Scleroscope) test, and methods which determine a material's resistance to plastic (permanent) deformation or penetration. Devices measuring this latter property are machines such as the Rockwell, Brinell, Vickers, or Tukon hardness testers.

The Brinell, Vickers, and Tukon tests produce numbers which are numerically equal to the load used to produce an impression divided by the surface contact area of that impression. The major differences among the instruments are in the load applied, the method of applying that load, and the size and shape of the indenter. Table 1.1 below shows some of the standard loads and indenters used by these techniques. Others are also in wide use.

The Rockwell tester compares the hardness of materials on the basis of the depth of penetration of a standard indenter under a standard load. Among indenters in use are a diamond Brale with a spheroconical point, as well as 1/16", 1/8", 1/4", and 1/2" polished steel balls. Loads used range from 15 kg to 150 kg. By any of the above tests, an increase in the hardness number indicates an increase in resistance to penetration. Stated

another way, an increase in hardness number indicates a decrease in depth of penetration by the indenter at a fixed load.

Some points to note: First, when selecting a scale, choose one such that the hardness values you obtain fall between 20 and 100. There are no units to Rockwell hardness numbers, and there is no percentage comparison that can be made. In other words, if one material has a hardness of, say, 30 on a particular scale, and, on the same scale, a second material has a hardness of 60, it is not correct to say that the second material is "twice as hard" as the first one. Similarly, there is no such thing as "zero" hardness, even though the tester can supply such a reading and can even give negative values. If you do obtain such readings, change to a different scale. Consult the instructor or TA if this happens.

Second, materials can vary locally from point to point in their degree of homogeneity. Because the hardness test is a very localized one, it is not uncommon for a material to yield hardness readings that vary from point to point (sometimes by as much as five hardness units). Thus, every time a hardness reading is to be made, the customary practice is to obtain several readings (moving the sample after each) and then use the average.

The Wilson Rockwell tester is supplied with a 1/16" steel ball penetrator and is used for testing materials such as brass, bronze, and soft steels and metals. Typical scale designations of this penetrator and others are given for regular scales in Table 1.2. Superficial "T" scales are used for materials similar to those tested on the Rockwell B, F, or G scales, but of thinner gage or where a very small indentation is required.

Table 1.1. Load/Indenter Combinations for Several Hardness Measurement Methods

	Indenter	Load	Apparent Shape
Brinell	5 or 10 mm ball	500 or 3000 kg	Circle
Vickers	136° pyramidal diamond	10 to 50 kg	Square
Tukon	Knoop diamond	25 to 50,000 g	Rhombus

The 120° diamond sphere/conical penetrator or "Brale" indenter is available as an accessory and is required for testing hardened steels or any hard metals. Typical scale designations for this penetrator are also given in Table 1.2. Superficial

"N" scales are used for materials similar to those tested on the Rockwell C, A, and D scales, but of thinner gage or where a very small indentation is required. Rockwell superficial scale penetrator and loads are given in Table 1.3.

Table 1.2. Rockwell Regular Scales.

Scale Symbol	Indenter	Major Load (kg)
A	diamond	60
B	1/16" steel ball	100
C	diamond	150
D	diamond	100
E	1/8" steel ball	100
F	1/16" steel ball	60
G	1/16" steel ball	150
H	1/8" steel ball	60
K	1/8" steel ball	150
L	1/4" steel ball	60
M	1/4" steel ball	100
P	1/4" steel ball	150
R	1/2" steel ball	60
S	1/2" steel ball	100
V	1/2" steel ball	150

Table 1.3. Rockwell Superficial Scales

Scale Symbol	Indenter	Major Load (kg)
15N	Diamond	15
30N	Diamond	30
45N	Diamond	45
15T	1/16" steel ball	15
30T	1/16" steel ball	30
45T	1/16" steel ball	45
15W	1/8" steel ball	15
30W	1/8" steel ball	30
45W	1/8" steel ball	45
15X	1/4" steel ball	15
30X	1/4" steel ball	30
45X	1/4" steel ball	45
15Y	1/2" steel ball	15
30Y	1/2" steel ball	30
45Y	1/2" steel ball	45

The Experiment

Hardness Testing

(A) From among the available metal, alloy, and polymer samples, choose groups of related materials. They should be related by variation in alloy content, carbon content, alloy base, annealed vs. cold-worked, etc. Measure the hardnesses of the samples on the various Rockwell machines (both the Rockwell Series 500 and 2000 electronic machines). Be sure to use a suitable load/indenter combination. Table 1.4 shows the sample identifications (code number and corresponding sample type) and *suggested* hardness scale to use.

(B) Measure one sample using two different scales. Convert both to a common scale (Brinnell, or convert one to the other) and compare. The values should be similar. Are they? What should you do if they're not?

(C) Measure one sample on all three electronic machines using the same scale and compare. The values should be similar. Are they? What should you do if they're not? Record your data on the data sheets provided, which should be included in the Appendix of your report. *Be sure to include in your discussion a comparison of the various metal samples bearing the relationships discussed in (A), (B), and (C) above.*

Note: In this and other experiments throughout this course, you will sometimes be required to convert your hardness values to different scales. The accepted method for displaying such conversions is to show the converted value and scale followed by the actual value and scale used in the measurement in parentheses. For example, 353 HB (38 HRC). ***Please use this method throughout the course.***

Statistical Significance Study

Choose one sample and measure its hardness twenty times. Record this data on the separate data sheet provided. With this data, calculate the range, mean, mean deviation, standard deviation, variance, and 90% and 95% confidence intervals. A discussion of statistical analysis, including an example of this type of calculation, is provided. Show each part of each calculation in your report, even if you use a computer program or a calculator with built-in statistical functions to make these calculations.

Once you have determined a 90% and a 95% confidence interval for your data, based on the 20 measurements you made, turn things around and calculate the number of measurements necessary to obtain 90% and 95% confidence intervals of ± 1 hardness unit. This will necessarily be an approximation, because the value of N (number of observations) influences both the standard deviation and the value of t_c , the confidence coefficient. Use a value for "nu" of 19. You can then solve for N in the standard deviation expression. Remember to round up to the nearest integer, because fractional observations are not possible.

Effect of Deformed Samples

Obtain a curved sample. The curvature may distort the hardness reading due to its ability to flex when the sample is placed on a large flat anvil with the concave surface down. A "spot" anvil can be used to eliminate this. Record hardness values using both a large, flat anvil and a "spot" anvil. In your report, explain any differences in the readings you obtain.

As you write up this section of the experiment in your report, be sure to discuss what you anticipate will be the effect of the curvature on the reading you obtain. Then note whether your expectation was met, why or why not, and the implications for measuring samples that are damaged in any way in the future.

Table 1.4. Hardness Testing Sample Identification

Identification	Material	Suggested Hardness Scale
801	2024-T4 Aluminum	HR15T or HRE
802	304 Stainless Steel	HR15T or HRB
803	1100-F Aluminum	HR15T or HRH
804	70 Cu - 30 Zn Brass (as-delivered)	HR15T
805	70 Cu - 30 Zn Brass (annealed)	HR15T
806	1017 Steel (cold drawn)	HRB
807	1040 Steel (cold drawn)	HRB
808	Mg AZ31B Magnesium	HR15T or HRE
810	316 Stainless Steel	HR15T
811	4130 Steel (low alloy)	HR15T
812	Copper (99.9%)	HR15T or HRH
813 or 6061	6061-T6 Aluminum	HR15T or HRE
814	7075-T6 Aluminum	HR15T or HRE
909	1095 Steel (hot rolled)	HRB
910 (or 809)	1095 Steel (cold drawn)	HRB
H1	1095 Tool Steel	HRB or HRA
5	95 Cu - 5 Sn Bronze (cold drawn)	HR15T or HRB
6	95 Cu - 5 Sn Bronze (annealed)	HR15T
Green	Polystyrene	HR15Y
Transparent	Lucite	HR15X
White (hard)	ABS	HR30Y or HR15X
PMMA (blue)	PMMA	HR30Y or HR15X
White (soft)	Teflon	HR15Y or HR30Y
Translucent	Polyethylene	HR15Y

Hardness Testing Data Sheet
(include in Appendix of your report)

Investigator _____ Date _____

Material Comparison Tests

Sample No.	Sample ID No.	Material	Rockwell Scale	Measured Hardness Values			Average
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							

Testing of A Curved Sample

	Rockwell Scale	Measured Hardness Values			Average
Concave Down with Flat Anvil					
Concave Down with Spot Anvil					
Sample material _____					

Use of Two Different Scales on the Same Hardness Testers

	Rockwell Scale	Measured Hardness Values			Average
Scale 1					
Scale 2					
Scale used for comparison _____		Converted hardness from Scale 1 _____			
		Converted hardness from Scale 1 _____			
Sample No. _____		Sample Material _____			

Use of Two Different Hardness Testers

Machine ID	Rockwell Scale	Measured Hardness Values			Average
1					
2					
Sample No. _____		Sample Material _____			

Note: You may reuse some data in the Material Comparison Tests

Statistical Study

Investigator _____		Date _____	
Sample Materials (including ID #) _____		Hardness Scale _____	
Measurement No.	Hardness	Measurement No.	Hardness
1		11	
2		12	
3		13	
4		14	
5		15	
6		16	
7		17	
8		18	
9		19	
10		20	

Range of Sample _____	Mean of Sample _____
Mean Deviation of Sample _____	Std. Dev. of Sample _____
Variance of Sample _____	
90% Confidence Level of Sample _____	
95% Confidence Level of Sample _____	
<p>Now, using a value of $n = 19$ (assigned for this laboratory section), calculate the number of observations necessary for</p>	
90% confidence interval of ± 1 _____ (observations)	
95% confidence interval of ± 1 _____ (observations)	

Note: A sample calculation must be included in your report as an appendix

Statistical Evaluation of Hardness Testing Data

You have obtained the following ten values of hardness on a particular sample:

Test No.	Hardness Value
1	35.0
2	32.0
3	32.5
4	33.0
5	32.9
6	34.0
7	34.0
8	34.6
9	34.0
10	34.0
total	336.0

The range is 32.0 to 35.0. The arithmetic mean is

$$\text{Arithmetic Mean} = \bar{x} = \frac{\text{total}}{N} = \frac{336.0}{10} = 33.6$$

The mean deviation is given by

$$\begin{aligned} \text{M.D.} &= \frac{\sum |x - \bar{x}|}{N} = \frac{|35.0 - 33.6| + |32.0 - 33.6| + |32.5 - 33.6| + |33.0 - 33.6| + |34.0 - 33.6| + |33.0 - 33.6| + |32.9 - 33.6|}{10} \\ &\quad + \frac{|34.6 - 33.6| + |34.0 - 33.6| + |34.0 - 33.6|}{10} = \frac{8.0}{10} = 0.80 \end{aligned}$$

Now, the variance, s^2 , of sample population is given by the sum of the squares of the individual deviations divided by the total number of measurements as follows

$$s^2 = \frac{\sum(x_i - \bar{x})^2}{N - 1}$$

which equals 0.913 in this instance.

The standard deviation, s , is just the square root of the variance

$$s = \sqrt{s^2} = \sqrt{0.913} = 0.956$$

We now wish to determine the smallest number of hardness readings which will give us a mean value (33.6), which represents the mean established from the 10 readings within certain confidence levels. Let us select 95% and 90% confidence levels and use the following formula:

$$\bar{x} \pm t_c \frac{s}{\sqrt{N - 1}} \quad (\text{Eq. 1})$$

where t_c , confidence coefficients, are selected from Table 1.5 at the end of this section. For a 95% confidence level, select $t_{0.975}$ (0.25, or 2.5% at each tail of the curve), and for a 90% confidence level, select $t_{0.95}$. Reading the value under t_c corresponding to degrees of freedom ($= N-1$), for $= 9$, $t_{0.975} = 2.26$ and $t_{0.95} = 1.83$ in Table 1.5.

Therefore,

$$\bar{x} \pm t_{0.975} \frac{s}{\sqrt{N - 1}} = 33.6 \pm \frac{(2.26)(0.956)}{\sqrt{10 - 1}} = 33.6 \pm 0.72$$

and

$$\bar{x} \pm t_{0.95} \frac{s}{\sqrt{N - 1}} = 33.6 \pm \frac{(1.83)(0.956)}{\sqrt{10 - 1}} = 33.6 \pm 0.58$$

which tells us that 95% of the time, the true value of the mean will be within ± 0.72 units, and 90% of the time, the true value of the mean will be within ± 0.58 units of the measured mean, when 10 readings are taken.

If we are willing to accept a maximum of ± 1.0 units as the required level of accuracy, what is the minimum number of readings required to get a 95% confidence level and what is the minimum number of readings required to get a 90% confidence level? The number of observations required in an experiment to compare mean values must obviously depend on the precision of the observations. If (as is usual), the standard deviation of a large sample, is not known accurately when the experiment is planned, it is impossible to predict precisely what the number of observations should be.

If we make the assumption that the standard deviation, s , for a small sample ($N < 30$) is equivalent to σ , the standard deviation of the population, we can calculate a value of N from Eq. 1 above. Our assumption would be much better if $N = 30$. We now select a value of t_c table 1.5. Assuming normal distribution or an infinite number of samples, $\nu = N - 1 = \infty$, and the value for $t_{0.975}$ for a 95% confidence level will be 1.96. Using the arbitrarily acceptable mean value ± 1.0 , we set Eq. 1 equal to ± 1 and solve for N .

$$\pm 1.0 = t_{0.975} \frac{s}{\sqrt{N-1}} = 1.96 \frac{0.956}{\sqrt{N-1}}$$

$$N-1 = \left[\frac{(1.96)(0.956)}{1.0} \right]^2 = (1.87)^2 = 3.5$$

Thus, use $N = 3.5 + 1 = 4.5 \approx 5$ readings.

For a 90% confidence level [Hint: Do not use $t_{0.90}$ below! $1 - (1 - 90\%)/2 = 0.95$]

$$\pm 1.0 = t_{0.95} \frac{s}{\sqrt{N-1}} = 1.645 \frac{0.956}{\sqrt{N-1}}$$

$$N-1 = \left[\frac{(1.645)(0.956)}{1.0} \right]^2 = (1.57)^2 = 2.47$$

Thus, use $N = 2.47 + 1 = 3.47 \approx 4$ readings.

Table 1.5 Percentile Values (t_p) for Student's t Distribution (with degrees of freedom)

	$t_{0.995}$	$t_{0.99}$	$t_{0.975}$	$t_{0.95}$	$t_{0.90}$
1	6.31	3.08	1.78	1.38	1.00
2	9.92	6.96	4.30	2.92	1.89
3	5.84	4.54	3.18	2.35	1.64
4	4.60	3.75	2.78	2.13	1.53
5	4.03	3.36	2.57	2.02	1.48
6	3.71	3.14	2.45	1.94	1.44
7	3.50	3.00	2.36	1.90	1.42
8	3.36	2.90	2.31	1.86	1.40
9	3.25	2.82	2.26	1.83	1.38
10	3.17	2.76	2.23	1.81	1.37
11	3.11	2.72	2.20	1.80	1.36
12	3.06	2.68	2.18	1.78	1.36
13	3.01	2.65	2.16	1.77	1.35
14	2.98	2.62	2.14	1.76	1.34
15	2.95	2.60	2.13	1.75	1.34
16	2.92	2.58	2.12	1.75	1.34
17	2.90	2.57	2.11	1.74	1.33
18	2.88	2.55	2.10	1.73	1.33
19	2.86	2.54	2.09	1.73	1.33
20	2.84	2.53	2.09	1.72	1.32
21	2.83	2.52	2.08	1.72	1.32
22	2.82	2.51	2.07	1.72	1.32
23	2.81	2.50	2.07	1.71	1.32
24	2.80	2.49	2.06	1.71	1.32
25	2.79	2.48	2.06	1.71	1.32
26	2.78	2.48	2.06	1.71	1.32
27	2.77	2.47	2.05	1.70	1.31
28	2.76	2.47	2.05	1.70	1.31
29	2.76	2.46	2.04	1.70	1.31
30	2.75	2.46	2.04	1.70	1.31
40	2.70	2.39	2.02	1.68	1.30
60	2.66	2.36	2.00	1.67	1.30
120	2.62	2.33	1.98	1.66	1.29
∞	2.58	2.33	1.96	1.645	1.29

Cold Work and Annealing of Alloys

Introduction

When a material is subjected to a stress in excess of its "elastic limit," it deforms permanently, or "plastically," which is indicative of a property termed "ductility." The plastic deformation of metals occurs by a process called slip, which occurs along many planes which are favorably oriented for the slip process. Orientation with respect to the applied stress, dislocation densities, and interplanar spacings are all factors which determine if a particular "slip system" will be operative. During the deformation process, the slip that occurs on any one plane will usually involve only a small number of interatomic spacings, while the overall change in shape will be due to the sum of this process occurring on a very large number of such planes. Certain structural irregularities, one of which is the "Frank-Read" source, may contribute to multiplication of the number of dislocations which move across an individual plane. With increases in the number and complexity of dislocations, pile-ups and entanglements increase with increasing degree of deformation.

When deformation is accomplished at temperatures below one-half to one-third of the absolute melting point ($^{\circ}\text{K}$ or $^{\circ}\text{R}$), the process is referred to as "cold work." The hardness, yield point or strength, and tensile strength all increase with the degree of deformation until a maximum value of the property is approached. Yield strength increases more rapidly than tensile strength, and at the limit of deformation, the two are equal. Electrical conductivity and ductility are properties which decrease to minimum values with increasing deformation. In most cases, an almost undetectable change occurs in both the moduli of elasticity and rigidity, which is referred to as a "modulus defect" and which increases to a maximum value with increasing deformation.

Most of the energy expended in deformation is transformed into heat, but a small percentage is stored in the crystal lattice in the form of strain energy. Much of the lattice between slip planes is undistorted and still relatively perfect with most of the strain energy highly localized at sites on the slip planes. These sites of high strain energy are the points at which the nuclei of new strain-free grains form and grow during that portion of the annealing process known as "recrystallization."

When the cold-worked material is heated at relatively low temperatures, relief of certain small defects (micro-strains) in atomic configuration occurs, and the hardness and tensile strength may increase slightly. The electrical conductivity returns to its original, higher value, and the modulus defect decreases toward zero. At some higher temperature, the hardness, ductility, and tensile strength return rather abruptly to their original (pre-deformation) values. The low temperature process is not accompanied by any detectable microscopic change, but the higher temperature process is accompanied by the appearance of small colonies of new, distributed, "equi-axed" grains which are scattered through the highly deformed, cold-worked grain structure. The small new grains will, with increasing time and/or temperature, grow in size (and sometimes in number) from atoms which diffuse from the highly deformed grains.

The low temperature process, during which the electrical conductivity regains its original value and the modulus defect decreases to zero, is termed "recovery." The higher temperature process, during which the engineering properties are returning to normal, is termed "recrystallization." At temperatures above the recrystallization range, or for longer annealing periods in that range, marked grain growth may occur. A few materials, when annealed at somewhat higher temperatures, will exhibit a behavior referred to as "secondary grain growth,"

during which some grains grow to abnormally large size.

In the cases of steel and other multi-phase alloys and allotropic elements which suffer a solid-solid transformation, hot and cold working operations are separated by the transformation temperature. In these alloys, refinement of structure (not grains) can be accomplished in many cases by controlling the cooling rate from above the transformation temperature. In the single phase alloys and non-allotropic pure metals,

the recrystallization temperature separates hot and cold-working operations. For materials such as the "alpha" brasses, which do not undergo an allotropic change, cold work plus some recrystallization may be the only way in which a segregated, cored, coarse-grained, cast structure can be both homogenized and refined in grain size.

Pre-Lab Questions

(40% of your prelab score)

1. Distinguish between elastic and plastic deformation.
2. How is cold work performed and what happens to a metal sample's physical properties after it is cold worked?
3. Describe what happens to its physical properties when a cold worked metal sample undergoes an annealing process.
4. One of the alloys typically studied in this experiment is 1100 aluminum. Find out its chemical composition and melting point. As always, you must cite your source.

The Experiment

The object of this experiment is to determine the change in an engineering property (in this case, hardness) and relate these changes to the degree of cold work producing that change. Following the cold-working operation, the material will undergo an annealing operation at several temperatures and times to determine the temperature ranges of recovery, recrystallization, and grain growth. From the overall change in hardness during cold-work, determine the degree of cold-work necessary for 1/4, 1/2, and 3/4 of the overall change, and compare, where possible, with standard literature values.

The laboratory section will be divided into squads, and each squad will be provided with a different alloy chosen from among brasses, bronzes, low carbon steels, etc. These alloys will be in the form of bar stock 1/4" thick, 1/2" or 3/4" wide, and of variable length from 4 to 6 inches.

Each squad will be provided with two samples of the alloy in different conditions. One piece will be in the condition in which it was shipped from the factory, and thus will be termed the "as-received" sample. (Why wouldn't its percent cold work be zero?) The second piece will have been previously annealed to a relatively soft state in order to undergo the cold-working operation. Measure the hardness using an appropriate scale (Table 1.4).

On the accompanying data sheets, notice that the only measurement to be made on the as-received sample is its hardness value. Once you have prepared a hardness curve for your alloy, you can determine the amount of cold-work the material had received prior to shipment. This value should be reported in your lab report.

For the annealed specimen, record the initial dimensions (width and thickness) and hardness and record this data as the zero-percent cold-worked entry. The hardness at the assigned terminating cold-work value is also the room temperature entry on the annealing data sheet.

Remember, the reduction in area calculations always refer to the original cross sectional area prior to any cold work. Initially, carry out the cold work in several 2-to-3% reduction increments followed by larger reductions of 5-to-8% each until approximately 50% reduction is reached. At this point, have six to eight pieces, each about one inch long, cut (the T.A. will do this) from the specimen for annealing at various temperatures (which will vary from metal to metal and will be given to you in class). Set one piece aside and continue to cold-work it in 2-to-4% increments (record hardness values as you progress) until either the specimen fails in brittle fracture or the rolling mill rollers come into contact. A plot of percent cold-work vs. hardness will look something like Figure 2.1.

When you make your plots, do not connect dots. As we learned in Experiment No. 1, hardness measurements invariably show scatter. For the purposes of these plots, the best approach is to draw a smooth curve through the points. If you are making your plot with a computer, use the program's freehand drawing tool to draw the curve. If the program you're using doesn't allow this, draw the curve by hand after you've printed the graph.

The cut-off pieces are to be annealed at various temperatures for several different time periods as

shown on the attached data sheets. The particular temperatures to be used depend on the particular alloy you are investigating, and this information will be furnished to you in the lab. At the end of each time period, the samples, with the exception of the steels, can be quenched in water. Steel samples that have been annealed above 720°C must be first cooled in air for several minutes to avoid the formation of martensite. Figure 2.2 shows typical annealing curves for different amounts of initial cold work at fixed times. Since all of the samples to be annealed in this experiment will have the same amount of initial cold work, the plots that you construct will look more like those shown in Figure 2.3.

Now, in contrast to the curves you drew on the hardness-vs.-cold work plots, for the annealing curves, when you make your plots, you do want to connect the dots.

NOTE:

Typical hardness values for some steels can be found in Vol. 1 of both the 9th and 10th Editions of the Metals Handbook. Values for aluminum and copper-based alloys (brasses, bronzes, etc.) can be found in Vol. 2 of both the 9th and 10th Editions of the Metals' Handbook.

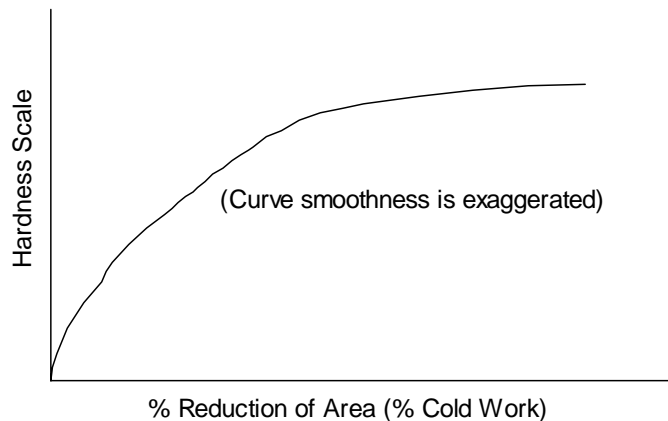


Figure 2.1. Typical curve of hardness vs. percent cold work.

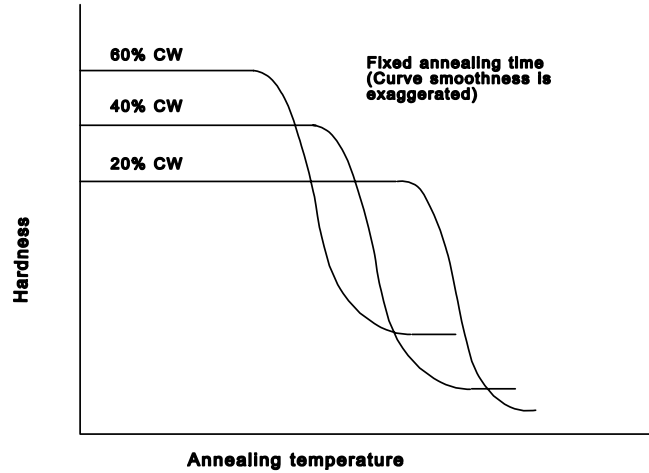


Figure 2.2. Hardness vs. annealing temperature for a fixed annealing time and differing amounts of initial cold work. *(No need to use plot this kind of figure in your report.)*

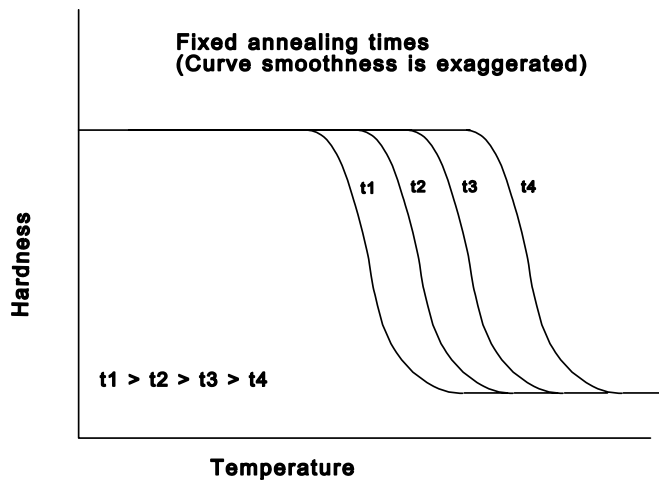


Figure 2.3. Hardness vs. annealing temperature for different annealing times and identical amounts of initial cold work. *(Plot this kind of curve in your report instead of Figure 2.2.)*

The Heat Treatment of Steel

Introduction

The properties that can be developed in a steel by heat treatment are due to two very important solid state reactions. The first reaction is that of "austenite" decomposing into ferrite and cementite at a temperature which is about 723°C (1333°F) and depends upon both the kinds and amounts of additional alloying elements present. This reaction is similar to the eutectic reaction of a liquid transforming into two solids and is termed a "eutectoid," or eutectic-like, reaction. The engineering properties of the steel are very sensitive to the manner in which the cementite (also called Fe₃C, iron carbide, or simply "carbide") is dispersed in the ferrite with regard to shape, size, and volume fraction. All of these variables can be controlled by varying carbon content. Additional alloying elements also have varying degrees of control over shape, size, and volume fraction of the cementite.

The second reaction is the transformation of austenite to "martensite." Unlike the first reaction, where the fcc austenite decomposes into a bcc ferrite containing less than 0.025 w/o dissolved carbon and Fe₃C (cementite), and in which the growth of either ferrite or carbide is a diffusion-controlled reaction, the martensite reaction is a diffusionless change of the fcc austenite to the body-centered tetragonal martensite with carbon content identical to that of the original austenite.

The eutectoid decomposition will begin and end isothermally at any temperature below the eutectoid temperature. A temperature-dependent nucleation period precedes the appearance of cementite or ferrite produced by the eutectoid decomposition, and this transformation can be suppressed by rapid cooling. The temperature at which martensite will begin to form is strongly dependent upon the carbon and alloying element content of the austenite. (Recall the Isothermal Transformation diagrams from ChBE 231!) The fraction of austenite transformed to martensite depends upon the

temperature attained, which must be below an upper critical value called the martensite start temperature, M_S. Below a lower critical temperature, called the martensite finish temperature, M_F, the austenite will have transformed completely into martensite. At intermediate temperatures (between M_S and M_F), only a portion of the austenite undergoes this transformation.

The formation of martensite produces very high hardness in steel, but in plain-carbon steel, the rate of cooling necessary to produce this structure is extremely high. Although the surface of the sample may be cooled rapidly, the interior cools at a slower rate and may not develop martensite. The addition of alloying elements to a steel can allow development of martensite at lower cooling rates or the formation of more martensite at a fixed cooling rate. By suitable addition of alloying elements, a steel can be made which will produce martensite throughout the section when air cooled. The ultimate hardness value depends almost entirely on both the carbon content of the martensite and the amount of martensite formed. The ease (defined as the necessary cooling rate—the lower the rate needed the greater the "ease") with which martensite is formed is termed the "hardenability."

The Jominy Test

The Jominy test is designed to determine the hardenability of a steel by cooling one end of an austenitized bar with a jet of cold (75°F) water. A bar 1 in. (25 mm) in diameter and 3.5 in. (88 mm) long is suspended by an attached collar over the jet which releases water at a controlled flow rate and temperature. Hardness values are obtained at positions along a "flat" ground onto the side of the bar and are plotted against the distance from the quenched end (D_{qe}). Since some inhomogeneous or variable concentration of carbon and alloying elements is possible and is expected from point to point, two flats are ground, and hardness values are

averaged for equivalent D_{qe} values. The flatter this "Jominy" or "hardenability" curve, the higher the hardenability of the steel regardless of the absolute

magnitude of the hardness (within reason) as long as martensite has been formed at the quenched end. If martensite has not been formed, the test indicates that water of that particular temperature is inadequate as a quenching medium.

Pre-Lab Questions

1. Define or explain the following:
(a) spheroidite, (b) cementite, (c) pearlite, (d) martensite, (e) tempering.
2. What is the difference between tempering and annealing?
3. A steel contains 40 w/o (%wt) primary ferrite and 60 w/o pearlite at 723°C. What is the carbon content of each component and what is the carbon content of the steel?
4. In the discussion above, it was noted that if a steel is quenched to a temperature below the martensitic start temperature (M_s) but above the martensitic finish temperature (M_f), only a portion of the austenite is transformed to martensite. What happens to the rest of the austenite?

The Heat Treat Experiment

In this experiment, you will correlate the heat treatments and resulting properties for a medium to high-carbon steel. The steel will have been "austenitized" at a temperature between 790 and 950°C (1450 and 1750°F) for 45 minutes to 1 hour. (Be sure to note in your report the exact temperature that was used.) To avoid "decarburization," some type of controlled atmosphere will have been maintained in the furnace. Most likely this will be a carbonaceous chamber which reacts with oxygen in the furnace before the steel samples can. Other methods of preventing decarburization include covering the samples with charcoal or by maintaining a balanced atmosphere of CO and CO₂ in the furnace. These gases tend to neither deposit carbon into the steel nor extract carbon from the steel.

Six samples, each about 1/2" x 1/2" x 3/4", of each steel to be studied will be available in the furnace.

All six samples are then cut slowly in half using a water-cooled cut-off wheel with a high flow of cold

The six samples are to be cooled as follows: two are quenched in room temperature tap water, two are quenched in Tenaxol A® or RL® (to be determined in class), one is allowed to cool in room temperature air, and one is allowed to cool with the furnace (furnace cool) down through at least 650°C (1200°F). (The Tenaxols are polymer-water dispersions having quenching properties similar to low viscosity petroleum oils, but without the fire hazard. Their use provides a cooling rate between those of water and air.) One each of the water, Tenaxol A, and Tenaxol RL-quenched samples is then immediately placed in a tempering furnace at an intermediate temperature (400 to 600°C—again, record the exact temperature used in your report) for a period of 1 hour. After each sample has cooled, and before any further processing is done, mark the samples on both ends indicating the steel type, type of quench, and whether tempered or not.

water. Failure to supply sufficient coolant or cutting at too great a speed may result in "burning," leaving

dark brown to blue arcs on the freshly cut surface. The teaching assistant will perform all cutting operations. Again, be sure that each half of each sample is adequately marked for identification, and store one of the halves for later use in the metallography experiment.

On the remaining six halves, spray tool and diemakers layout blueing on the cut face. Once the blueing is dry, scribe two diagonals, and then obtain hardness readings at the center and then at the four equivalent diagonal positions every 3/32" until the corners are reached (Figure 4.1). Using the average of the hardness at the four equivalent positions, plot symmetrical "U" curves of the hardness traverse from corner to corner. A "U" curve is simply a plot of hardness vs. position across the cut face. Put the coordinate for the center of the cut face at the center of your x-axis, and then simply plot the hardness for the various points out from the center to each side of the center point value. The result is a curve showing the hardness from one edge to the other. It's called a "U" curve because, in many instances, the slower cooling rate experienced by the center vs. the edge of the sample results in a lower hardness at the center. Its appearance is thus in the shape of a U.

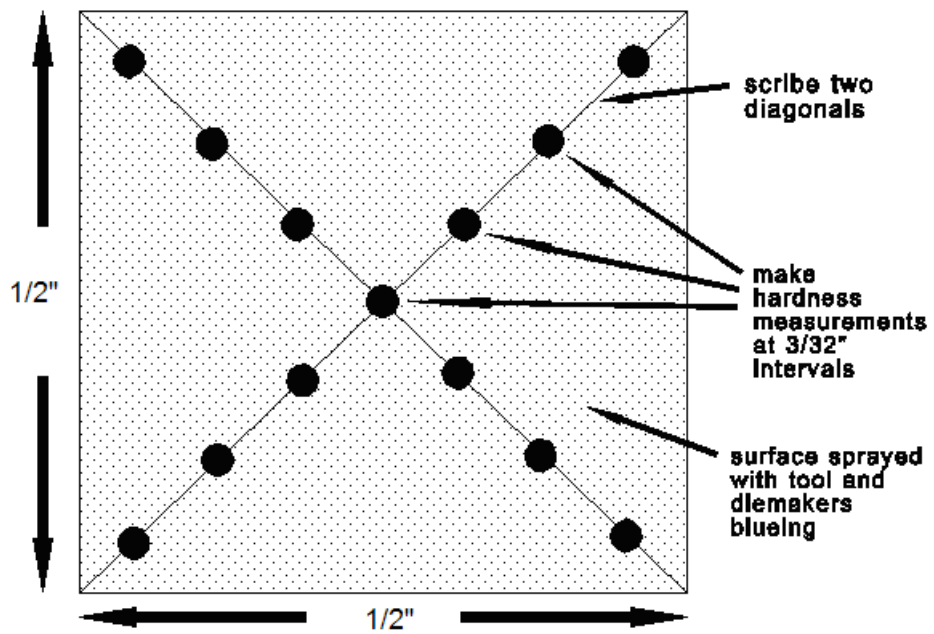
For the surface hardness, it is often better to turn the sample on its side and take a hardness reading into the center of one of the uncut surfaces. This is because the samples will tend to tip, thus giving low readings as the corners are approached along the diagonals.

The Jominy Experiment

One or more Jominy bars will have been placed in the same austenitizing furnace as described above.

The bars will consist of common plain carbon and low alloy steels and will usually be chosen to compare carbon or alloy content. The bars are removed, one at a time, and placed in the cooling fixture. The cooling water flow should be turned on and adjusted to the correct temperature (75°F) prior to placing the Jominy bar in the fixture. You may wish to practice transferring the bar from the furnace to the cooling fixture once or twice with cool bars to improve your skill at this operation.

Once the bar has cooled sufficiently that it can be handled (about 20 minutes), prepare it for hardness readings by grinding flats in two locations along the side of the bar. (You will be shown how in lab.) A special fixture is available to support the bar while hardness measurements are made. Begin at the quenched end and take hardness readings at increasing distances from the quenched end as shown on the data sheets. Average the readings for the two flats for equivalent D_{qe} values, and plot these results in the standard manner, which is to plot hardness versus distance from the quenched end. Note that it is difficult to obtain accurate readings near the end of the bar because of a tendency for the bar to tip. Arrange the bar in the fixture to maximize the support under the point where the indentation is made. Compare your results on two different bases. If there are two or more plain carbon steels with differing carbon content, plot these on the same graph. If there are two or more bars with the same nominal carbon content but differing alloy type and amount, plot these on the same graph. From the literature, you should be able to locate hardenability curves for the steels you have studied. As part of your report, compare your experimentally-obtained curves with those from the literature.



Heat Treatment of Steel Data*	Date _____
Alloy _____	Data Recorder _____
Hardness Scale _____	Austenitizing Temp _____
Method of Cooling _____	Method of Cooling _____
Hardness at surface _____ ; at center _____	Hardness at surface _____ ; at center _____
at 3/32" _____	at 3/32" _____
at 3/16" _____	at 3/16" _____
at 9/32" _____	at 9/32" _____
Averages	Averages
3/32 " _____ 3/16" _____ 9/32" _____	3/32 " _____ 3/16" _____ 9/32" _____
Method of Cooling _____	Method of Cooling _____
Hardness at surface _____ ; at center _____	Hardness at surface _____ ; at center _____
at 3/32" _____	at 3/32" _____
at 3/16" _____	at 3/16" _____
at 9/32" _____	at 9/32" _____
Averages	Averages
3/32 " _____ 3/16" _____ 9/32" _____	3/32 " _____ 3/16" _____ 9/32" _____

*Include in the Appendix of your report.

"Hardness at surface" means hardness at side surfaces.

"at center" means at the center of the freshly cut surface.

Additional Data Sheet:

Heat Treatment of Steel Data		Date _____	
Alloy _____		Data Recorder _____	
Rockwell Scale _____		Austenitizing Temp _____	
Method of Cooling _____		Method of Cooling _____	
Hardness at surface _____ ; at center _____		Hardness at surface _____ ; at center _____	
at 3/32" _____		at 3/32" _____	
at 3/16" _____		at 3/16" _____	
at 9/32" _____		at 9/32" _____	
Averages		Averages	
3/32 " _____ 3/16" _____ 9/32" _____		3/32 " _____ 3/16" _____ 9/32" _____	
Method of Cooling _____		Method of Cooling _____	
Hardness at surface _____ ; at center _____		Hardness at surface _____ ; at center _____	
at 3/32" _____		at 3/32" _____	
at 3/16" _____		at 3/16" _____	
at 9/32" _____		at 9/32" _____	
Averages		Averages	
3/32 " _____ 3/16" _____ 9/32" _____		3/32 " _____ 3/16" _____ 9/32" _____	

Jominy Test Data				Include in Appendix of Your Report
Alloy _____ Hardness Scale _____				Recorder _____ Date _____
D _{qe}	Flat #1	Flat #2	Average	
1/16"				
1/8"				
3/16"				
1/4"				
5/16"				
3/8"				
7/16"				
1/2"				
9/16"				
5/8"				
11/16"				
3/4"				
13/16"				
7/8"				
15/16"				
1"				
1&1/8"				
1&1/4"				
1&1/2"				
1&3/4"				
2"				
2&1/4"				
2&1/2"				

Metallography

Atomic and Molecular Grain Structure

The atomic arrangement in metals and alloys is of too small a scale to be visible using the light microscope. The separation between atoms is on the order of $2 - 5 \text{ \AA}$ ($2 - 5 \times 10^{-10} \text{ m}$) and can be resolved only by radiation of comparable or shorter wavelength. The light microscope does, however, resolve many important details concerning the orientation of blocks, or "grains" of atoms, the shapes of these grains, and the various solid phases present in the metal specimen. In addition, information concerning the previous treatment of the specimen can often be obtained from a microscopic view.

If the metal has been cold-worked, the grains may be deformed from their equi-axed shape to a distorted shape, or they may be fragmented. If a material has undergone a particular heat treatment, the microscopic examination may detect the type of heat treatment or the effectiveness of that heat treatment.

The preparation of the sample surface is very important if an accurate view of the microstructure is to be obtained. Scratches only 1 or 2 microns ($1 \mu\text{m} = 10^{-6} \text{ m}$) deep will appear as canyons under the microscope, and small pits will look like craters. Either of these surface defects can obscure or obliterate the true grain structure.

For ease in handling, specimens are often mounted in acrylic or Bakelite resins, especially if the samples are small. (Larger specimens may be more easily handled without mounting, and certain advantages accrue in electro-polishing and etching when the samples are not mounted.)

In preparation procedures, it is important to avoid overheating the specimens any more than is absolutely necessary. Heat treated steels may undergo tempering, precipitation-hardened alloys may overage, and cold-worked alloys may be annealed if subjected to too high a temperature. Abrasive cut-off wheels and grinding belts can

produce a very high surface temperature, and care must be exercised to avoid these heat effects.

The general procedure in specimen preparation is to first cut off a sample of suitable size using the water-cooled abrasive wheel. [This likely will have already been done as part of a previous experiment. Any cutting needed will be done by the instructor or TA.] After mounting the specimen with an assumed flat surface exposed, the scratches from the abrasive wheel are removed by hand polishing using successively finer abrasive grits. This operation is performed most rapidly and can be followed visually if the sample is rotated 90° between each grit size. In this fashion, the scratches from the preceding grit are replaced by finer scratches at right angles to the previous set. The polishing operation is complete when none of the scratches from the previous set are visible. Motion in this operation should be in one direction only (toward or away from you), otherwise two surfaces which are non-planar and roof-shaped will be produced. This procedure is carried out through the progressively finer grit sizes of 240, 320, 400, and 600.

The last stages of polishing involve cloth-surfaced wheels and liquid suspensions of polishing compounds having particle size ranges from 15 to $0.05 \mu\text{m}$. These last steps will produce a plane, mirror finish free from scratches if care and extreme cleanliness were maintained during the polishing operations.

Examined under a microscope at this stage, the mirror surface will exhibit no detail. During the grinding and polishing processes, the metal surface layers have been smeared and deformed, and this deformed layer (often many atoms thick) must be removed before the surface details become apparent.

No mechanical polishing can remove this deformed layer. Rather, a chemical reagent called an "etchant" is utilized to dissolve this layer without additional deformation. After the deformed layer has been removed, the same etchant or another reagent will attack the various structural features at varying

μm alumina is sufficient as the final polishing suspension for steels, whereas 0.5 μm or 0.03 μm alumina will generally be necessary for copper and aluminum samples.