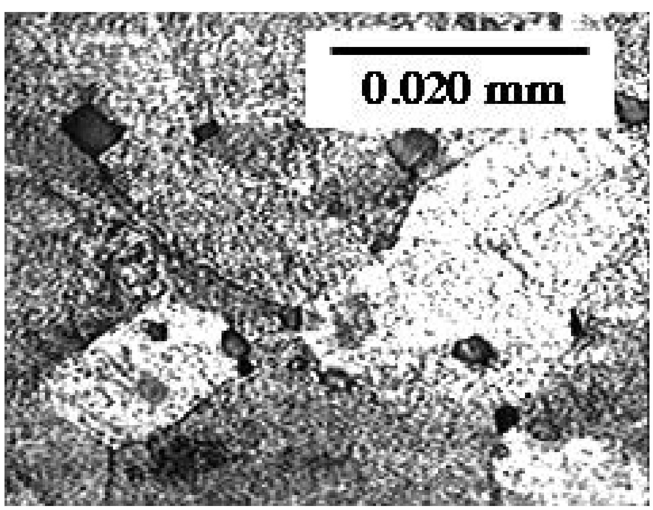
NPRE 432: Behavior of Engineering Materials

(from ME 330)

Lab 3
Cold Work and Annealing



Undeformed free machining brass annealed at 400°C for 2 hours

I. Introduction

Previous laboratories have demonstrated the important material properties of strength and ductility. This laboratory will show how these properties can be controlled in many metals using deformation and thermal processing, such as cold working and annealing, to change their microstructure.

The objectives of this laboratory experiment are:

1. Understand the processes of cold rolling and annealing and the accompanying microstructural phenomena of cold work and softening;
2. Become familiar with standard metallography methods;
3. Investigate the effects of cold work and annealing temperature on the hardness of brass;
4. Observe the corresponding microstructural changes that are responsible for these property changes.

II. Background

It has previously been observed from tensile tests that a metal specimen loaded beyond its yield point will strain plastically. This results in a permanent deformation of the specimen after it is unloaded. When most metals are plastically strained, they will strain harden. In other words, its strength continues to increase as stresses are applied beyond yielding. Recall that the brass specimen in the Tensile Stress-Strain Relations experiment behaved elastically when reloaded after initial yielding and unloading. The brass specimen did not yield again until it reached the load at which it was unloaded. At that load the specimen "remembered" its prior stress-strain behavior, and resumed its previous loading path. After unloading from a stress-strain state where plastic strain hardening occurred, the yield strength of the material is increased. If a specimen is “work hardened” in this manner and then unloaded, another experimenter would obtain different mechanical properties (i.e. higher yield strength and lower ductility).

This experiment reveals three important features of work hardening. First, an application of stress beyond the yield point in processing can increase the yield strength of the material in service. Secondly, a corresponding loss of ductility must accompany the increase in strength. And finally, the original properties of the undeformed material can be restored with the application of heat—a process known as annealing. Figure 1 shows how several properties change with increasing amounts of cold work.

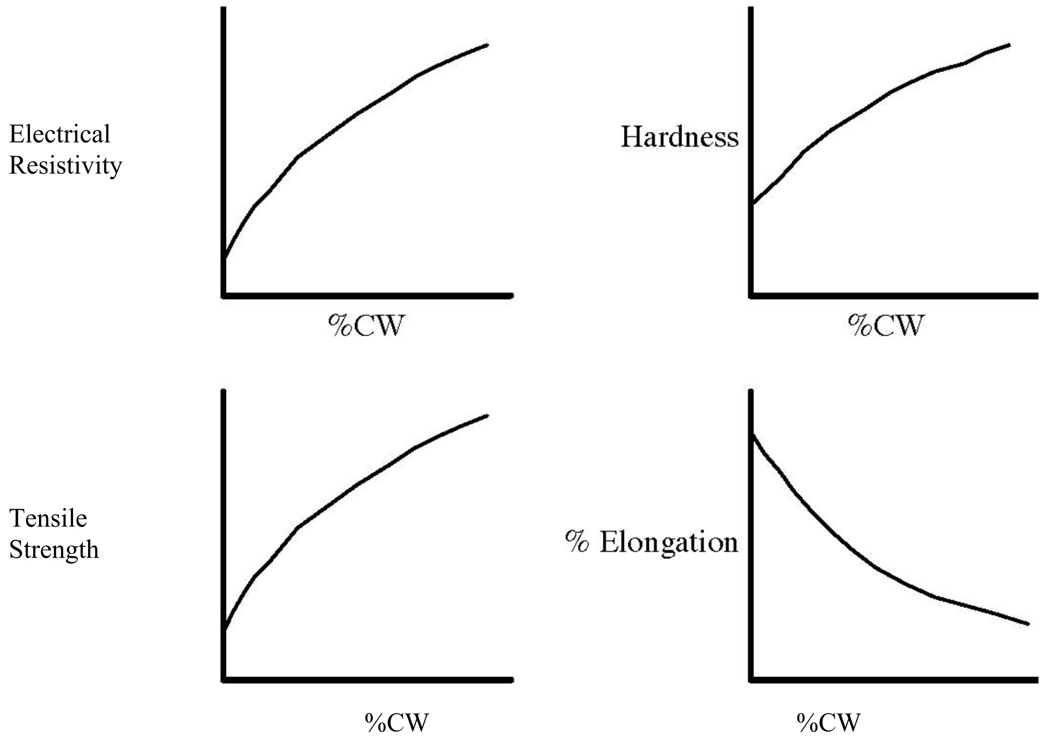


Figure 1: Changes in Material Properties with Cold Work

III. Cold Working

Many wrought metal products such as sheet metal, plates, and bars are produced by deformation processes such as rolling and drawing. When deformation of a material occurs below the temperature needed for recrystallization, it is called "cold working."

As expected, rolling a material changes its geometry. As the workpiece passes through the rollers, the thickness decreases provided the rollers apply sufficient pressure to the material. Since the volume of material must be conserved, the reduction in thickness is accompanied by an increase in length. One might expect the material to be squeezed equally outward in all directions, becoming longer *and* wider, such as pressing on a mound of clay. The cylindrical rollers apply a compressive load equally across the width, but do not produce a significant stress distribution in that direction. Rather, the stresses are principally compressive through the thickness and tensile (due to friction) in the rolling direction. This effect is similar to that observed for a rolling pin.

A. Rolling and Drawing Processes

At a commercial rolling mill, plates and sheets are rolled at high speed from semi-finished shapes (slabs, blooms, or billets) that remain hot from the previous casting process. This is accomplished by compressing the material between two rotating rolls. As the thickness is reduced, the length increases dramatically, and the speed of the material exiting from the rolls is higher than the speed entering. A typical plate mill has a large number of reduction rolls in succession on the production line.

It is quite common for the rolls to be rather small in diameter, and are usually reinforced by larger diameter rolls. The mill in the ME 330 laboratory is much smaller than commercial rolling machines and consists of only two rolls. This mill is termed "two-high" because it consists of two rolls that appear to be stacked on top of one another. A mill with one set of reinforcing rolls appears to have four stacked rolls, and is called "four-high."

Drawing is a similar process used to produce wire and rod products such as concrete reinforcing bars. Unlike rolling which uses flat cylindrical rolls, drawing relies on matching grooves in the rolls to reduce the diameter of the rod. The grooves are usually shaped to produce round rod or wire. However, square, rectangular, and hexagonal bar stock is also drawn.

By studying rolling, it is possible to understand the principles that govern a wide range of manufacturing steps that require plastic deformation. Other forming processes of interest include die-forming a sheet metal component and forging. Like strain hardening in simple tension, rolling and drawing have limits as well. Although the limits are the same (i.e. the UTS), necking is not observed due to the multiaxial loading. As a result, after the limit on cold work has been exceeded there is a strong tendency for the specimen to crack or tear when it is drawn or rolled further.

B. Quantifying Cold Work

The percent cold work for a material is defined by the initial and final cross sectional area, A_o and A_f respectively, as

$$\%CW = \frac{A_o - A_f}{A_o} * 100 \quad (1)$$

Sometimes this parameter is called the percent cold reduction, %CR. For the specific case of rolling, the percent cold work can be expressed by an initial thickness to and a final (after rolling) thickness t_f as:

$$\%CR = \frac{t_o - t_f}{t_o} * 100 \quad (2)$$

Notice that the percent cold work is similar to the reduction in area parameter used for an indication of ductility in a tension test.

C. Microstructural Changes During Rolling

During the rolling process, each grain's volume is approximately conserved. Since grains cannot be moved from their original locations, each grain must be reduced in the thickness direction and elongated in the direction of rolling. Microscopic examination of the material as shown in Figs. 4(a), (c), and (e) shows clear evidence of this distortion of the grains.

Work hardening is mainly attributed to the entanglement of dislocations, which are line defects in the crystal structure. When plastic strains are imposed on the metal, the dislocations move through the crystal. Dislocation movement is prevented when obstacles such as precipitates, grain boundaries, or another dislocation (dislocation tangles) are encountered. When a dislocation is prevented from moving, the stress required to cause further deformation in the crystal structure increases. This effect is observed macroscopically as an increase in the stress required to further deform the specimen.

More than 90% of the energy expended in cold-working a metal is converted into heat. The cold-worked bar specimen is noticeably warmer immediately after rolling. The remainder of the energy is stored in the metal as strain energy, primarily at dislocation tangles and grain boundaries. When the metal's internal energy is raised, it is no longer at its lowest possible energy state. The locations where the strain energy is stored are the sites where changes occur first, provided enough additional energy is added. Since these sites have greater energy associated with them than the surrounding metal, they are thermodynamically favored over other locations. At room temperature, the energy required to begin moving atoms is greater than the energy available at these favored sites. Recall from thermodynamics that the temperature of a material simply reflects a greater level of energy associated with each individual atom. Thus, at elevated temperatures each atom has more energy. Raising the temperature of the specimen, adds energy to allow the transformation back to a lower energy state. This treatment using elevated temperature is called annealing.

IV. Annealing

After cold working a material, the ductility of the specimen has been reduced due to its inverse relationship with strength. If a large reduction is desired, such as in drawing wire or forming a complex component, annealing can be used to regain lost ductility. After annealing, the metal will have properties close to those of the original undeformed material.

A. Annealing Stages

Annealing takes place in a deformed microstructure in three distinct stages: recovery, recrystallization, and grain growth. The microstructure and property changes are summarized in Fig. 2.

During the recovery stage of annealing, there is little change in the size or shape of the distorted grains. However, the residual stresses in the highly strained grains are relieved and some dislocations are removed. This stress relief is accompanied with little appreciable change in the physical properties of the material. Additionally, a partial reduction in the crystalline distortion occurs.

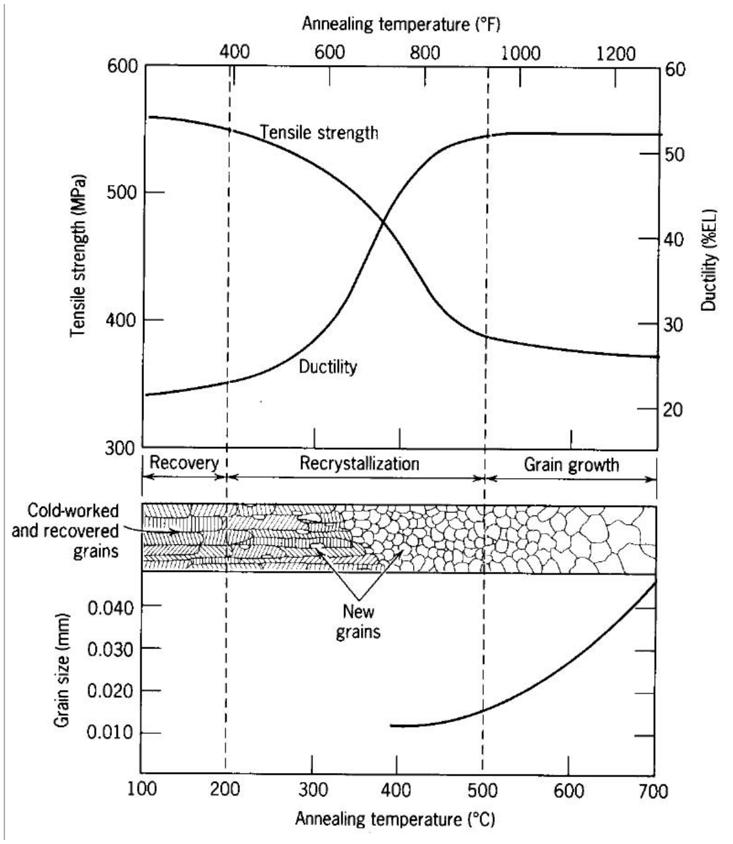


Figure 2: Microstructure and Property Changes During Annealing (Fig 7.20, Calister, 6^{th} Ed.)

During recrystallization, the highly distorted grains are replaced by new equiaxed and stress-free grains. The nucleation sites for the new grains are typically the dislocation tangles and grain boundaries where higher strain energies are stored. In most metals, the initial effect of recrystallization is to transform a single distorted grain into a number of smaller, equiaxed grains. Recrystallization results in a decrease of the dislocation density, lower strength, and increased ductility.

If heating is allowed to continue, grain growth occurs. The diffusion laws are such that larger grains grow faster than smaller ones. Therefore, the small grains are consumed by the larger, faster growing grains. Excessive grain growth can detrimentally affect the material properties and produce a rough surface appearance on components formed from sheet metal.

Annealing is a function of both time and temperature. However, the relationship is typically the product of temperature and the logarithm of time, which implies that variations in time have much less effect than variations in temperature. It is standard to anneal for one hour, varying only the temperature. The temperature used for annealing depends on the thickness, composition, and geometry of the component. This is true for tempering steel as well, since the mechanics of the process are similar.

B. Activation Energy

In general there is a minimum annealing temperature for annealing to have an effect, roughly one-third to one-half the melting point. This temperature must be calculated on an absolute temperature scale such as Rankine or Kelvin. A different temperature would be obtained if the Celsius or Fahrenheit scales were used. The presence of a minimum temperature indicates the process is governed by an Arrhenius Rate Equation,

$$rate = Ae^{\frac{-Q}{RT}} \tag{3}$$

In the case of recrystallization during annealing, by convention, the recrystallization rate, RR, is inversely proportional to the time required for 50% recrystallization to occur:

$$RR \propto \frac{1}{t_{50\%}} = Ae^{\frac{-Q}{RT}} \tag{4}$$

where Q is the activation energy for recrystallization and $t_{50\%}$ is the time. In the case of grain growth during annealing, a relationship between grain size and time is desired. The equation takes the form

$$d = A\, t^n\, e^{\frac{-Q}{RT}} \qquad (5)$$

where d = average grain diameter
A = frequency factor
Q = activation energy for grain growth
R = universal gas constant
T = temperature (absolute scale)
t = time
n = exponent (0.3 - 0.5 for most alloys)

The activation energy Q can be determined by measuring the average grain diameters after annealing identically cold-worked samples for the same time at different temperatures (both above the recrystallization temperature). Then, the activation energy can be found from the slope by plotting the logarithm of the grain diameter versus the inverse of the temperature. Rearranging the above equation for the two tests gives

$$\ln\left(\frac{d_1}{d_2}\right) = \left(\frac{Q}{R}\right)\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \qquad (6)$$

From this relationship it is possible to find the activation energy Q, which is a property of the material.

The activation energy of a cold-worked material can be provided by any source. Ordinarily the energy comes from an external source: the temperature increase while annealing. But there is another important source of energy: the strain energy stored in dislocation tangles and crystal distortion. Thus for a greater cold work, which increases the amount of strain energy, a lower temperature is necessary to reach the activation energy. This effect is observed in actual experiments, i.e. lower minimum annealing temperatures are required to anneal specimens with increasing amounts of cold work.

V. Materials Processing Applications

The principles of cold work and annealing can be applied to a large number of practical manufacturing problems. The microstructure and property changes are summarized in Fig. 4.

A. Increasing Strength

As noted earlier, cold work can increase the yield strength of the metal. Rolling and drawing (i.e. applying a multiaxial state of stress) can increase both the yield strength and the ultimate strength of the material. For example, cold-drawn mild steel rods can have strengths nearly five times that of annealed mild steel. However, there is a corresponding loss in ductility. Many materials such as mild steel, brass, and aluminum have sufficient ductility that even after moderate amounts of cold working, adequate ductility remains. For some metals, cold working is the only method available for increasing the strength.

B. In-Process Recovery

Unfortunately, there is a limit on the amount of cold work for a metal before it becomes too brittle. This effect is especially important when manufacturing a complex component by cold-working, such as stamping from sheet metal. During the manufacturing of a complex part, it is possible to recover original properties (lower strength and increased ductility) by annealing in order to make further cold work easier and to increase formability. It is common practice to compute the amount of cold work during each manufacturing step, thus planning in advance where annealing will be necessary.

C. Hot Work

An obvious step beyond the recovery process is to keep the workpiece above the recrystallization temperature during plastic deformation. This process is known as hot work to make it distinct from cold work, where the deformation step has a measurable effect (i.e. increased strength, reduced ductility). The difference between hot work and cold work is the temperature of the workpiece. If the material is above the recrystallization temperature during the deformation process, it is called hot working.

D. Grain Size Control

If annealing is allowed to progress through recovery and recrystallization, but is stopped before grain coarsening occurs, the grain size of the material will have been reduced. Thus, it is possible to use cold work and annealing to control the grain size in a metal that cannot otherwise be controlled. In fact, with careful planning it is even possible to use hot work to control the final grain size.

A class of materials growing in importance is the high-strength, low-alloy (HSLA) steels. These steels are typically used in the normalized (grain-refined) condition for the best toughness. Many of these steels rely on dynamic recrystallization (hot work) to obtain grain refinement. During the rolling or drawing process, the temperature and deformation rate are carefully controlled to obtain not only the required shape, but also the grain size appropriate to the normalized condition. This process eliminates the need for a separate heat treatment step, significantly reducing the cost of producing the steel.

IV. Experiments

A. Materials

In this exercise, several small free machining brass bars (62wt.% Cu, 35wt.% Zn, 3wt.% Pb) will be rolled. They will be cold rolled to achieve several steps of cold work ranging from 10% to 75%. A diagram of the original specimens is shown in Fig. 3. All the specimens were annealed at 450°C for 1 hour, and cooled at 0.5°C/min before testing. (See the cover photograph of the lab manual for the initial microstructure.) The specimens, originally wedge-shaped, have lines inscribed on the sides to indicate locations corresponding to approximately 10%, 20%, 30%, and 40% cold work at an final thickness of 5.0 mm. Each group will roll two wedges to a final thickness of approximately 5.0 mm. One of these wedges will be rolled again to halve the thickness to about 2.5 mm. Some groups will trade their specimen(s) for previously heat treated (annealed) specimens and put theirs in the furnace for 1 hour due to lab time constraints.

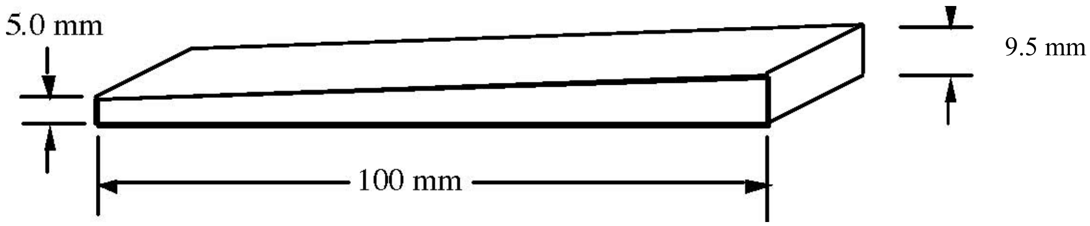


Figure 3: Schematic of Brass Cold Work Specimen

Hardness measurements (Rockwell B) will be taken for the 5.0 mm and 2.5 mm thick specimens (some may have been heat treated). Hardness data is supplied on the class web site for specimens that have been annealed at other temperatures. Thus, the effects of annealing can be evaluated, and the combined effects of annealing after cold work can also be considered.

B. Procedure–Cold Work Tests

SAFTEY WARNING*: The use of the rolling mill in this experiment can be very hazardous. Although safety precautions have been taken, the possibility of injury still exists. Keep your hands and fingers away from the moving parts of the machinery except as directed. Pay close attention to the instructions given by the laboratory assistants. If you are unsure of the operation of the equipment, STOP! Wait for instructions or clarification.*

(1) Take three hardness readings on the edge of the undeformed wedges to establish a base hardness. Also measure the original thickness at each of the four locations (i.e. I, II, III and IV). Record the data on the lab data sheet and your lab notebook.
(2) Roll each specimen to its appropriate thickness, either 5.0 mm or 2.5 mm. With the digital calipers, measure the thickness of the rolled specimen at each location. Record these data in the lab data sheet and your lab notebook.
(3) Make at least three hardness measurements on the rolled surface at each of the marked locations for the as rolled or annealed test specimens (depending on your group assignment. Again, record the lab data sheet and your lab notebook.
(4) Enter the data into the lab section summary at one of the computer stations.
(5) Roll the two 2.5 mm bars (as-rolled and annealed) to a final thickness of approximately 2.0 mm. Continue rolling the bar until final failure. As the bar is rolled, make observations and measurements as necessary. Note the differences between the as-rolled and annealed bars and approximate thickness at failure
(6) Observe the annealed specimens being removed from the oven if time permits. Return the lab data sheet to Dr. J. Williams so that a master data summary can be created and data for specimens annealed will be available for the next ME330 lab section.

C. Procedure–Metallography

(1) Observe the microstructures of representative brass samples that have been previously prepared. The etchant used for the free machining brass samples consisted of 15 ml H2O, 2.5 ml HCl, and 1 g FeCl. Make observations and sketches as necessary. Also refer to the microstructures shown in the cover photo and in Figure 6. (Note that the annealing temperature after rolling in the microstructures in the figures may differ from that performed in this lab experiment.) Some additional microstructural photos will be posted to the laboratory web site to aid in the lab writeup.

References:

Flinn, R.A., and P.K. Trojan, Engineering Materials and Their Applications, Third Edition, Houghton Mifflin, 1986, Chapter 3.
Boyer, H.E., and T.L. Gall (Editors), Metals Handbook: Desk Edition, American Society for Metals, 1985, Section 26
. Callister, W.D., jr., Materials Science and Engineering: An Introduction, Wiley, 1985, Chapters 7 and 10.
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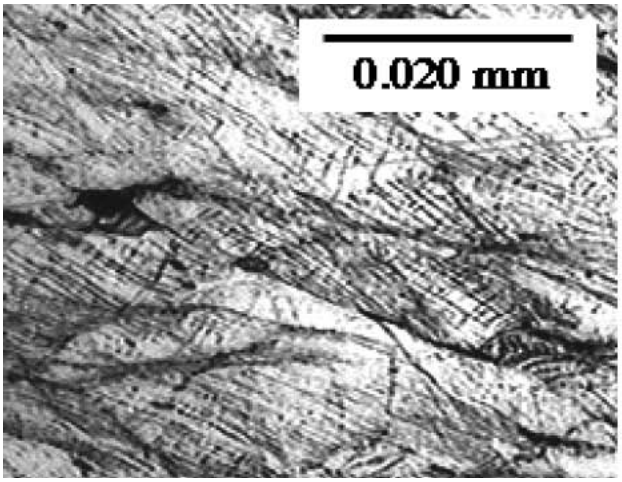
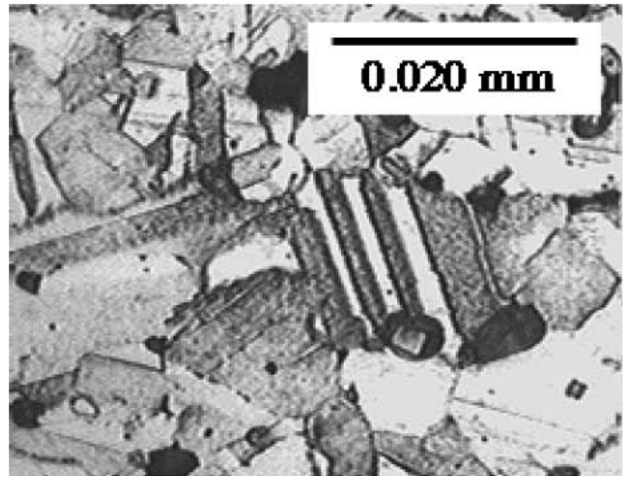
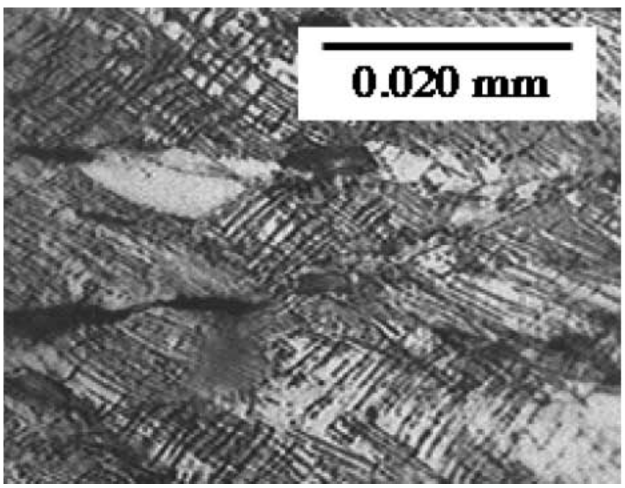


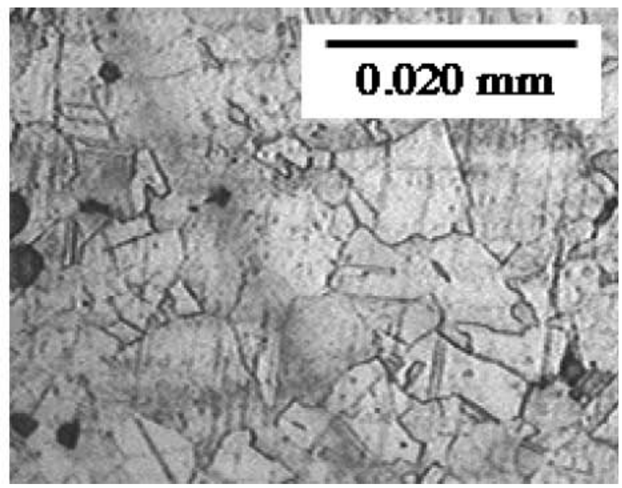
(a) 5 mm thickness, as rolled, Loc. II



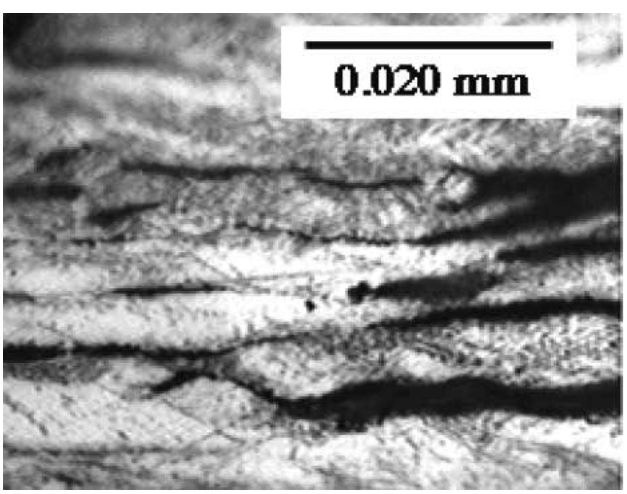
(d) 5 mm thickness, annealed, Loc. II



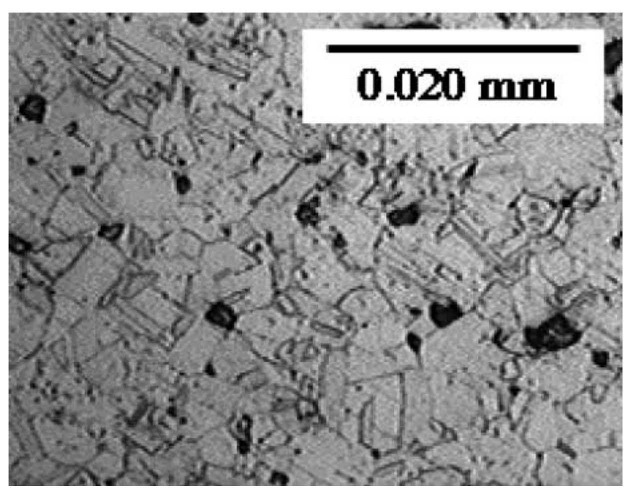
(b) 5 mm thickness, as rolled,Loc. IV



(e) 5 mm thickness, annealed, Loc. IV



(c) 2.5 mm thickness, as rolled, Loc III



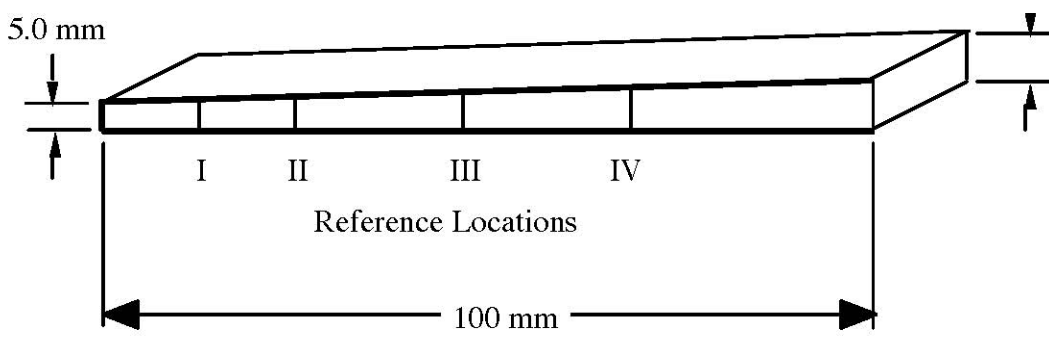
(f) 2.5 mm thickness, annealed, Loc. III

Figure 4 (a-f). Brass Rolling Microstructures (Annealing Temperature, 400^{o}C)

Data Sheet
Cold Work and Annealing
Rockwell B Hardness Tests

					Hardness, Rockwell B scale				
Final Thickness (mm)	Location	t_o (mm)	t_f (mm)	% CW	No Anneal 25°C	350°C	400°C	450°C	500°C
5.0	I								
5.0	II								
5.0	III								
5.0	IV								
2.5	I								
2.5	II								
2.5	III								
2.5	IV								

Note: Melting Temperature of Brass ~ 950 ^{P}C



Brass Cold Work Specimen Data Sheet

(One data sheet required for each specimen)

Annealing Temperature

Section performing rolling Date
(*For Groups A & B only*)

Time annealed wedge placed in furnace (Set timer also.)

Circle Your Group:

Group	Specimen Condition	Target Thickness
A	Rolled and Annealed	5.0
B	Rolled and Annealed	2.5
C	As Rolled	5.0
D	As Rolled	2.5

Specimen Dimensions (mm)

	Location			
	I (thinnest)	II	III	IV (thickest)
Initial				
Final				

Original Hardness (RB)

Measure hardness at *any* 3 locations on the *edge* of the wedge. Space measurements at least 5 diameters from the faces of the wedges and from other indentations and scratch marks.

Original Hardness (R_B)			

Final Hardness (RB)

Perform 3 hardness tests across the *width* of the strip at each location. Enter *all three* into the spreadsheet.

		Location			
I	II			III	IV

Speed cracks noted: Location (mark) Thickness

Roll to failure: Type specimen (circle): As-Rolled Annealed

Location (Mark) Thickness

Brass Cold Work Specimen Data Sheet

(One data sheet required for each specimen)

Annealing Temperature

Section performing rolling Date
(*For Groups A & B only*)

Time annealed wedge placed in furnace (Set timer also.)

Circle Your Group:

Group	Specimen Condition	Target Thickness
A	Rolled and Annealed	5.0
B	Rolled and Annealed	2.5
C	As Rolled	5.0
D	As Rolled	2.5

Specimen Dimensions (mm)

	Location			
	I (thinnest)	II	III	IV (thickest)
Initial				
Final				

Original Hardness (RB)

Measure hardness at *any* 3 locations on the *edge* of the wedge. Space measurements at least 5 diameters from the faces of the wedges and from other indentations and scratch marks.

Original Hardness (R_B)			

Final Hardness (RB)

Perform 3 hardness tests across the *width* of the strip at each location. Enter *all three* into the spreadsheet.

		Location			
I	II			III	IV

Speed cracks noted: Location (mark) Thickness

Roll to failure: Type specimen (circle): As-Rolled Annealed

Location (Mark) Thickness