**MSE 528 - Cold Work, Recovery, Recrystallization and Grain Growth**

Objective
To study the effects of cold working on the microstructure and mechanical properties of 70/30 Cartridge Brass.

Background
A small percentage of the energy expended in plastically deforming a material remains stored in the metal as an increase in internal energy. Changes are produced in both its physical and mechanical properties. Principally, there is a marked increase in hardness and electrical resistivity with the amount of cold working.

Microstructurally, this increment in internal energy is associated with an increase in the dislocation density as well as the density of point defects, such as vacancies and interstitials. For most metals, the dislocation density increases from the values of $10^6$-$10^7$ lines/cm$^2$ (typical of the annealed state) to $10^8$-$10^9$ after a few percent deformation and up to $10^{11}$-$10^{12}$ lines/cm$^2$ after heavy deformation.

At a more macrostructural level, the grains become markedly elongated in the direction of working and heavily distorted. This distortion is evident from a bending of annealing twins and from unevenness **in etching caused by local strain inhomogeneities.** While the increased hardness and strength that result from the working operation can be important, it is often **necessary to return the metal to its initial condition by annealing**. This usually means holding the cold worked metal at a temperature above about **1/3 of the absolute melting point** for a period of time. The annealing treatment is divided into three distinct regions:

1. **Recovery:** This usually occurs at low temperatures and involves motion and annihilation of point defects as well as annihilation and rearrangement of dislocations resulting in the formation of subgrains and subgrain boundaries (e.g., tilt and/or twist low-angle boundaries). A distinctive feature of the recovery process is that it does not involve any change in the grain structure of the cold-worked metal, the only changes taking place are the **dislocation arrangements within the existing grains**. Small **changes in hardness** that are sometimes observed during recovery can be attributed to the **decrease in the dislocation and point defect density** and to the growth of the subgrains.

2. **Recrystallization**: If increased thermal activation is available (i.e., if the temperature is raised) nucleation and growth of strain-free grains in the deformed matrix will take place. As these grains grow, the dislocations in the matrix are annihilated at the boundaries of the newly-formed grains. **Strength and hardness decrease considerably** and ductility increases. The **lowest temperature** at which stress-free grains appear in the structure of a previously plastically deformed metal is termed the **recrystallization temperature**. This depends upon the grain size, the severity of plastic deformation, and the presence of solute atoms or second phase particles. The **recrystallization temperature is usually 1/3-1/2 the absolute melting point** of the material.

3. **Grain Growth:**  If a recrystallized material is further annealed at the same temperature or at a higher temperature **grain growth usually occurs**.  Boundaries between annealed grains migrate and larger grains grow by an increase in the average grain size (or a decrease in the ASTM grain size number, n).  Grain growth depends on the fact that the **grain boundary energy of the material is reduced due** to the decrease in grain boundary area for a given volume of material. The effect of recovery, recrystallization and grain growth on grain size, internal stress and strength (or hardness) of a plastically deformed material is illustrated schematically in **Figure 5-1.**
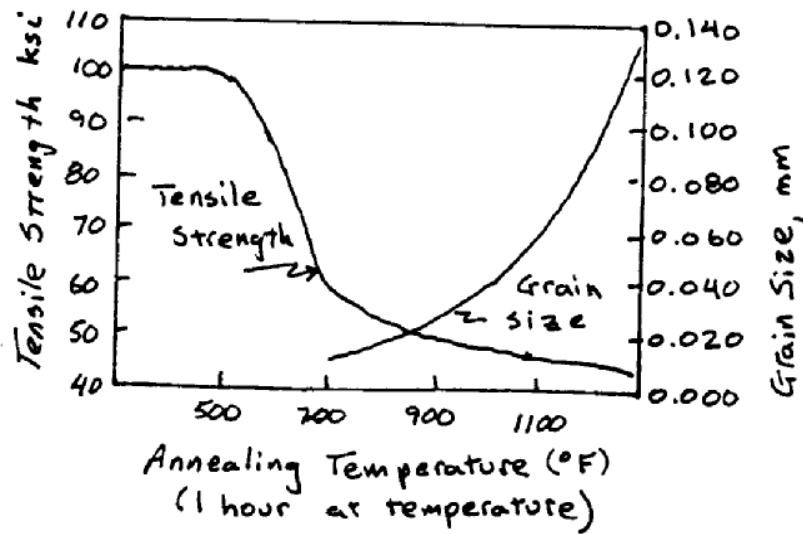


Figure 5-1. Tensile Strength and Grain Size as a Function of Annealing Temperatures for Plastically Deformed Material.

Measurement of Hardness

Throughout this experiment, **hardness measurements will be made using a Rockwell hardness tester**.  A **1/16" steel ball indenter** with either a 60 or **100 kg load** corresponds to the Rockwell F or **Rockwell B scales**.  Hardness value may be calculated from the load applied divided by the surface area of the indentation.  However, the instrument you will use is calibrated to read hardness values directly.  **Consult with the instructor before operating the hardness tester**.  You should run a <u>hardness test on the test standard</u> for the particular scale being used before and after each set of measurements.  **At least three hardness** measurements should be made on a smooth surface of each specimen to achieve statistical significance. If your **hardness values fall below 20 or above 100, you need to change hardness scales.**

Procedures

You are provided with one rectangular strip of 70/30 brass (Cartridge Brass).  The strip is fully annealed.

On the **fully-annealed strip (THICKER brass sample)**, perform the following:
1. Measure the initial **thickness** and **hardness**
2. **Reduce the thickness 50%** by many small rolling steps. Make sure to **measure the thickness and hardness after each pass through the rolling mill**. Tighten the rolls only a small amount at each step or a **safety pin will break.**
3. **Plot a curve of hardness vs. percent reduction in thickness.**

On the cold-rolled sample **(flattened, slightly warped piece of brass)**, perform the following:
1. Measure the **hardness** using **Rockwell B** scale
2. Cut the specimen into **eight pieces** about 1/2 in. long
3. Heat the eight pieces for **30 minutes** at the following temperatures:

| specimen | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|
| Temperature °C | 250 | 300 | 350 | 400 | 500 | 600 | 700 | reference |
| Temperature °F | 480 | 570 | 660 | 750 | 930 | 1110 | 1290 | |

After heating, quench the specimens in cold water.
4. **Measure the hardness** values of each of the eight specimens **after** the above **heat treatment** using the **B scale.**
5. In Excel, p**lot a curve of hardness vs. annealing temperature.**

Microstructural Investigation
Grain size determination is described in Appendix F (on website). You will be provided with a brass sample; you may have to grind, polish and etch the sample. Examine the sample with the optical microscope noting **grain size and shape**. Determine quantitatively, the **grain size** for the samples **that were annealed at 700, 600 and 500 °C temperatures**. Be careful in handling polished samples, as they are very susceptible to scratching. Take **photographs of the polished and etched samples** microstructure. **Use the Buehler Omnimet software to measure and label several grains for each anneal temperature.**

Lab book
No report is required; however the data and questions below should be **included in your notebook.**
1. In Excel, plot a curve of **(R$_B$) hardness vs. percent reduction in thickness** (on x-axis).
2. In Excel, plot **(R$_B$) hardness** (on y-axis) **vs. annealing temperature** (on x-axis).
3. Plot the grain size number (n) vs. temperature for the three highest annealing temperatures.
4. **Photograph the microstructures at each of the different temperatures of annealing, include in your lab book**.

**Glossary of Terms**
Understanding the following terms will aid in understanding this experiment.

**Annealing** -A heat treatment used to eliminate part or all of the effects of cold working.

**Cold working** - Deformation of a metal below the recrystallization temperature. During cold working, the number of dislocations increases, causing the metal to be strengthened as its shape is changed.

**Dislocation density** - The number of dislocations per unit volume in a material.

**Grain growth** - Movement of grain boundaries by diffusion in order to reduce the amount of grain boundary area. As a result, small grains shrink and disappear while other grains become larger.

**Interfacial energy** - The energy associated with the boundary between tow phases.

**Matrix** - Typically the first solid material to form during cooling of an alloy. Usually, the matrix is continuous and a second phase precipitates from it. However, in some complex alloys, the matrix is more difficult to define.

**Nucleation** - First stage of a phase transformation, such as precipitation.

**Preferred orientation** - An alignment of grains, inclusions, or other microstructural features in a particular direction of plane in material as a result of its processing.

**Plastic deformation** - Permanent deformation of the material when a load is applied, then removed.

**Recovery** - A low-temperature annealing heat treatment designed to eliminate residual stresses introduced during deformation without reducing the strength of the cold worked material.

**Recrystallization** - A medium-temperature annealing heat treatment designed to eliminate all of the effects of the strain hardening produced during cold working. Nucleation and growth of a new stress-free microstructure occurs. Recrystallization must be accomplished above the recrystallization temperature.

**Recrystallization temperature** - The temperature above which the effects of strain hardening are eliminated during annealing. The recrystallization temperature is not a constant for a material but depends on the amount of cold work, the annealing time, and other factors. At this temperature atomic mobility becomes high enough to allow recrystallization. This temperature is usually one-third to one half the melting temperature (in degrees Kelvin).

References
1.      Van Vlack, Elements of Materials Science and Engineering, Chapter 6
2.      Barrett, Nix and Tetelman, The Principles of Engineering Materials
3.      ASM Handbook, Vol. 2, Heat Treating and Cleaning of Metals
4.      Flinn and Trojan, Engineering Materials and Their Applications, Chapter 3