# Demonstrating The Effects Of Processing On Structure And Properties Via The Annealing And Strain Hardening Of Copper: Part One - Microstructure

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**Abstract of module:**

An experimental laboratory to demonstrate the structure-properties-processing relationship fundamental to materials science is presented. This part of the exercise correlates structure and processing and has been found to aid in the students' comprehension of the influence of strain hardening and thermal treatment on the resulting microstructure. Students, in teams of two or three, subject pure copper strips to controlled rolling and annealing at various temperatures. Samples are sectioned, mounted and polished in order to determine recrystallization and grain growth temperatures as well as to identify the affects of annealing and cold working.

**Module objective:**

The objective of this part of the laboratory is to explain the relationship between the microstructure of engineering materials and their mechanical properties and how this relationship is influenced by thermal and mechanical processing.

**Student learning objectives:**

This work provides part one of a two part laboratory experiment that aids the student in understanding the affects of annealing and cold work on the properties and microstructure of copper. There are several learning objectives for the students in this part. At the completion of the exercise, it is hoped that the students will be familiar with the use of metallographic sample preparation equipment and an optical microscope and understand the procedures employed to evaluate the microstructures resulting from different annealing temperatures and cold rolling regimens. A correlation should be noted between annealing and cold working conditions and grain size and orientation. The students should be able to define strain and deformation as it relates to structure in deformed materials. The primary intent is to provide the student with an understanding of the effects of strain hardening, recrystallization and grain growth via various processing methods on material structure and properties. Additionally, they should realize that processing methods can be

performed iteratively, since cold worked structure and properties will revert back to their pre-worked state when annealing treatments are applied.

**MatEd core competencies covered:**

0.B Prepare tests and analyze data
1.C Demonstrate Laboratory Skills
4.A Demonstrate effective work with teams
7.H Define strain and deformation
8.E Perform appropriate tests of metallic materials
**16.A** Explain effects of processing on material properties
17.B Describe techniques used in metals processing

**Key words**: annealing, strain hardening, rolling, recovery, recrystallization
**Type of module/mode of presentation**: laboratory
**Time required**: 3 hours for 20 students
**Pre-requisite knowledge**: basic understanding of microstructure of metallic materials, mechanical properties, structure-property relationship in metals
**Target grade level**: college undergraduate

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**List of equipment and supplies needed**

- 1/8 inch (3.175mm) C11000 copper plate or C26000 cartridge brass, cut into ¼ inch (6.35mm) x 3 inch (7.62cm) strips for deflection testing (one per anneal if dividing conditions among groups or one per anneal per group if each group is testing each condition). Samples for part two can be annealed at the same time.
- Heat treating furnaces able to reach 1200°F (649°C). Each furnace should be set at one of several selected temperatures (Not all temperatures given in the procedures need to be done but at a minimum, it is suggested that they be 300°F, 600°F,700°F and 1200°F). Thermocouples may be used to assure heat treating furnaces are accurate and chosen temperatures have been reached before annealing cycle is started.
- Bench top rolling mill
- Optical metallograph with vision software
- Sample preparation equipment; sectioning saw, mounter, polisher, grit paper, polishing cloths and polishing suspensions, etchants

**Curriculum overview and notes to instructor:**

Metallic materials will strain harden to different degrees depending on their ability to plastically deform and work-harden. To obtain a material in a particular shape and size, materials are strain hardened in various ways via cold rolling, extruding, drawing, stamping and forging. Strain hardening at ambient temperature is called cold working. (1) It is important to realize that upon strain hardening, the material microstructure is changed by an increase in the dislocation density and changes in the grain shape. Strength is increased due to inhibition of dislocation motion via dislocation-dislocation interactions. Ductility is decreased and properties become anisotropic. As ductility decreases, toughness, the ability of a material to plastically deform and absorb energy prior to failure, also decreases despite increases in strength. High toughness results from a good combination of strength and ductility. A metal with high strength and high ductility will have greater toughness than one with low strength and high ductility or one with high strength and low ductility. A simple method to measure static or material toughness is by calculating the area under the stress strain curve produced during a tensile test. (2) Annealing at high temperatures can remove the effects of strain-hardening via microstructural changes that generally occur in the following sequence: recovery, recrystallization and grain growth. During recovery, the dislocations reorganize into low energy configurations via diffusion mechanisms and the mechanical properties are virtually unchanged. During recrystallization, new equiaxed grains are formed and the strength is reduced. Grain growth is often avoided due to unfavorable mechanical properties. Strain hardening at high temperature is called hot working and allows the strain hardening and recrystallization processes to occur simultaneously. (3)

**Module procedure:**

The exercise is done in small teams of two to three students. Each group works on one annealing condition and final results are shared.

Before the lab:

Furnaces should be pre-heated.

Strips are divided into lots for heat treating at suggested temperatures of 300°F (149°C), 500°F (260°C), 600°F (316°C), 700°F (371°C), 800°F (427°C), 900°F (482°C), and 1200°F (649°C). If heat treating at all temperatures cannot be performed, conduct anneals at 300°F (149°C), 600°F (316°C), 700°F (371°C), and 1200°F (649°C) to capture the effects.

Students place samples in furnaces and allow furnace to return to temperature. Samples are held at temperature for one hour, removed from the furnace and air cooled for 10 minutes. Some samples heat treated at 1200°F (649°C) are rolled using a bench top rolling mill until hardness reaches the level of the as-received condition; about five passes in the mill will suffice. Here a hardness tester can be used if available. If not a scratch test may used.

Optical microscopy samples are sectioned from the end of the deflection samples, mounted and polished using 240 grit, 480 grit paper, 600 grit paper, and a series of colloidal suspensions (9 micron, 6 micron and 1 micron) on polishing cloths. Each polishing step takes about three minutes. The polished samples are etched for 3-5

seconds using a mixture of 70 mL water, 5g $Fe(NO_3)_3$, and 25 mL HCl and then examined using a metallograph.

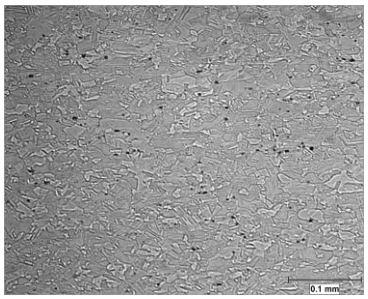
**Safety procedures:**

Etchants are mixed under a fume hood in glass containers, pouring the acids into the water, one at a time. Rubber gloves and eye protection are necessary to protect the students. In the event of a spill, contain the spill and place cleanup materials in a separate container, designated as hazardous waste. If acid gets on skin or in someone's eyes, rinse thoroughly and seek medical attention. Refer to the MSDS for each acid for more details. Acid mixtures are stored for later use in a locked acid cabinet. They may not be poured down the drain. For disposal, contact your hazardous material office.

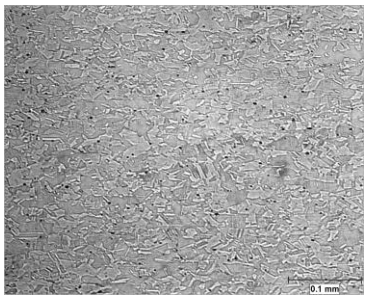
**Supporting handouts and materials:**

Representative micrographs for various processing conditions are shown below.

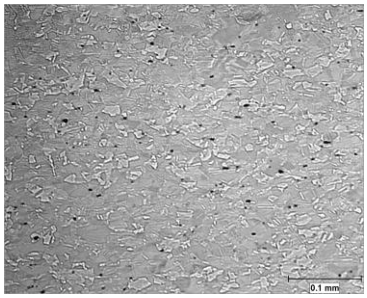
As-received plate, most likely in the H02 condition as inferred from measured properties (4), may have had some cold work performed on it to optimize properties. This is beneficial as some minimum value of cold work is required for recrystallization to occur. The expected recrystallization temperature is typically one third to one half of the absolute melting temperature, with the exact value depending on the amount of initial cold work and material impurities. (5) The corresponding expected range in recrystallization temperature for Cu is 355-763$^{o}$F (180-406$^{o}$C). Some grain elongation in the as-received condition persists until the 600$^{o}$F (316 $^{o}$C) annealing temperature. The specimen annealed at 700$^{o}$F (371$^{o}$C) shows an equiaxed grain structure, supporting that recrystallization has taken place. Annealed samples when rolled multiple times (five times in this example) demonstrated properties approaching those for the as-received samples (which were most likely in the cold worked state).



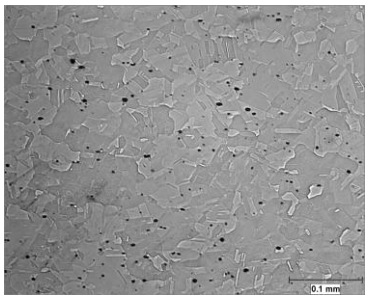
Copper As-received
Elongated grains seen



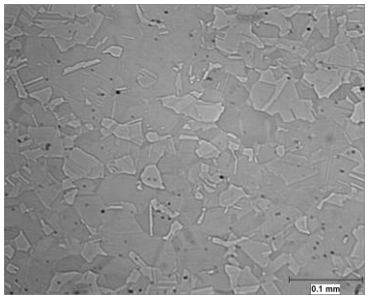
Annealed 300°F (149°C)
No change



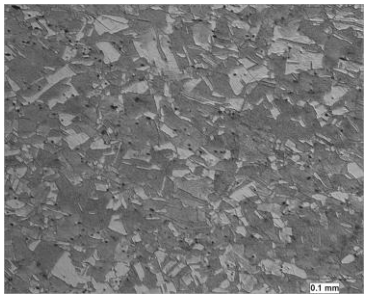
Annealed 600°F (316°C)
Softened, but little change



Annealed 700°F (371°C)
Some small new crystals evident, more equiaxed grains and grain growth also noted)



Annealed 1200°F (649°C)
(pronounced grain growth)



Annealed 1200°F (649°C) and rolled (deformation noted, microstructure upset and grain size reduced)

**References:**


1. W. D. Callister, Jr., *Fundamentals of Materials Science and Engineering*: *An Integrated Approach*, 2nd ed., John Wiley and Sons, Inc, 2005. pp. 190-198.
2. D.R. Askeland, P.P Phule, *The Science and Engineering of Materials*, 5th ed., Thomson, 2006, pp. 289-290.
3. 2008 ASM Handbook, H. Baker, et al, editors, Vol 2, *Properties and Selection: Nonferrous Alloys and Special-Purpose Materials,* ASM International, Materials Park, Ohio, 1992. pp. 265-266.
4. W.D. Callister, Jr. and D.G. Rethwisch, *Fundamentals of Materials Science and Engineering: An Integrated Approach,* John Wiley and Sons, Inc, 3rd edition, 2008. pp 263-265.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***Evaluation packet***

Student evaluation questions (discussion or quiz):

1. How does the microstructure compare for the cold rolled condition and the as-received condition? How has the microstructure changed with rolling?
   *After annealing causes recystallization and subsequent grain growth, cold rolling, breaks up the microstructure and returns it to the condition (cold worked to plate form) of the as received material.*
2. What is the effect of annealing on the microstructure? *At temperatures below the recrystallization temperature, there is no change. When held at or above the recrystallization temperature for a long enough time, new grains nucleate until cold worked grains are no longer evident. If held for a longer time, grain growth will occur.*
3. At which temperature would you suspect that recrystallization has taken place? What fraction of the melting temperature does this temperature correspond to*? Recrystallization has occurred somewhere between 600$^o$F (316$^o$C) and 700$^o$F (371$^o$C), less than one half the absolute melting temperature.*
4. Under what conditions will you see significant grain growth? *At higher temperatures such as 1200°F or above the recrystallization temperature for longer annealing times.*

Instructor evaluation questions:

1. For what grade level and engineering program is this appropriate?
2. Was the level and rigor of the module what you expected? If not, how can it be improved?
3. Is the demonstration difficult to recreate? Did the demonstration work as presented? Did it enhance student learning and generate interest among the students?
4. Was the background material sufficient for the students? The instructors?
5. Please provide your input on how this module can be improved, including comments or suggestions concerning the approach, focus and effectiveness of this activity in your context.

Student evaluation questions:

1. Was the laboratory procedure defined clearly?
2. Were the objectives well defined? Was the relevance to your course of study explained?
3. Was the instructor able to explain the objectives and outcomes clearly?
4. Was the instructor able to answer student questions?