Cold roll and anneal of Cu: metallography, microscopy, hardness

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Abstract

The hardness of moderately high purity copper (99.95%) was measured using the Rockwell hardness tester. The effect of cold-working on the hardness of copper and the effect of annealing at different times and temperatures on the hardness of copper were identified. Hardness of copper increases with increasing of cold working. The most suitable annealing temperatures with which to restore the ductile properties of copper was determined to be 350°C, based on microstructural changes. These changes which occur during cold rolling and annealing were observed using the Carl Zeiss optical microscope.

I. Introduction

Copper was the first metal to be used by humans. It was found free in streams and used to make jewelry. Copper is a soft, ductile metal and not useful for tools or weapons. However, the ancients discovered that by beating the copper with stone tools they could make it harder and strong, but less tough. This is what is scientifically called cold working, referring to the fact that the deformation takes place at a temperature well below the melting point, usually close to room temperature, or strain hardening.

Strain hardening (Callister)[1] is the process by which a metal becomes stronger as it is deformed past its elastic limit, resulting in plastic deformation. Although changes in mechanical properties such as tensile strength and ductility are the most noticeable result of permanent deformation, other physical and chemical properties are also affected; e.g., electrical resistivity increases and the resistance to corrosion changes. In fact, strain hardening play a key role in engineering metals since their most mechanical properties change as a function of the amount of strain hardening.

Strain hardening is considered to be a strengthening process, since it increases the tensile and yield strength. Strain hardening is explained by the increase in dislocation density (number of dislocations per cm^2) which occurs when the metal is deformed. The presence of dislocations strains the atomic lattice by slightly displacing atoms from their equilibrium positions. This lattice strain acts to reduce the mobility of the dislocations. The reduced dislocation mobility translates into an increase in the stress required to move the dislocations and, hence, an increase in the strength of the metal (Callister).[1] Permanent deformation is usually expressed quantitatively as percent cold work (percent reduction in cross sectional area, or in the case of cold rolling, in reduction of thickness):

$$\%CW = \frac{t_0 - t_f}{t_0} \cdot 100 \qquad (1)$$

where t_0 is the original specimen thickness and t_f is the final specimen thickness.

However, there are limits to the degree to which metals can be shaped by this process. Repeated cold working makes the metal tougher and more brittle, eventually resulting in fracture when struck. It is often necessary to anneal a metal at regular intervals during the forming process in order to restore ductility and allow for further cold working. The term “annealing”, used loosely for several types of heat treatments applied to any variety of different material systems, is generally associated with some kind of stress relief. The annealing of strain hardened metals has three distinct stages (Callister) [1]: Recovery, Recrystallization and Grain Growth.

II. Experimental Procedure

Specimens were made from moderately high purity copper (99.95%) and were in a fully recrystallized condition with large, equiaxed, strain free grains.

A. Cold rolling

Using a micrometer, the thickness of the Cu bars were measured. The initial hardness of the bars were determined using the Wilson Rockwell Hardness Tester on the F scale, Model # 3JR. The thickness required for 2%, 5%, 10%, 20%, 40% and 60% cold working was calculated using equation 1. After each reduction using Vigor RM 1000- rolling machine, the hardness measured using the Rockwell Hardness Tester as well.

B. Annealing

Three annealing furnaces were pre-heated and held at constant temperature, one each at 270, 310 and 350°C. The treatment started by placing one 60% cold worked Cu sample into each of the furnaces, called "fluidized sand baths", Tecam Fluidized Bath SBS-4. Then the samples were removed from the bath at each of the following cumulative time intervals: 2, 5, 10, and 20 minutes. Upon removal from the furnaces, immediately each sample was quenched in cold water to stop the annealing process. Finally, the hardness of the samples was measured using the Rockwell Hardness Tester.

C. Metallography of Annealed Samples

60% cold worked samples annealed for 20 minutes at one of each of the three temperatures and temperature was sectioned and mounted 600 Grit - Grinding Station, polished using 6 micron diamond polishing wheel and then1 micron alumina polishing wheel , and etched. These samples were examined with the Carl Zeiss optical microscope. The etchant used was 60 mL NH_4OH + 30 mL H_2O_2 (3%). Using the shared digital images, the effects of cold working on grain shape, orientation, and hardness, were examined as well as the effects of annealing and recrystallization on grain size and shape.

III. Results

The hardness of the three Cu samples was found to increase with increasing cold working. This trend is shown in Figure 1. The relationship of temperature versus pressure is roughly logarithmic. Good agreement is absorbed between the harnesses of the three samples of copper.

Also included in the Appendix are the values of hardness measured for copper after cold working, and the standard deviation.

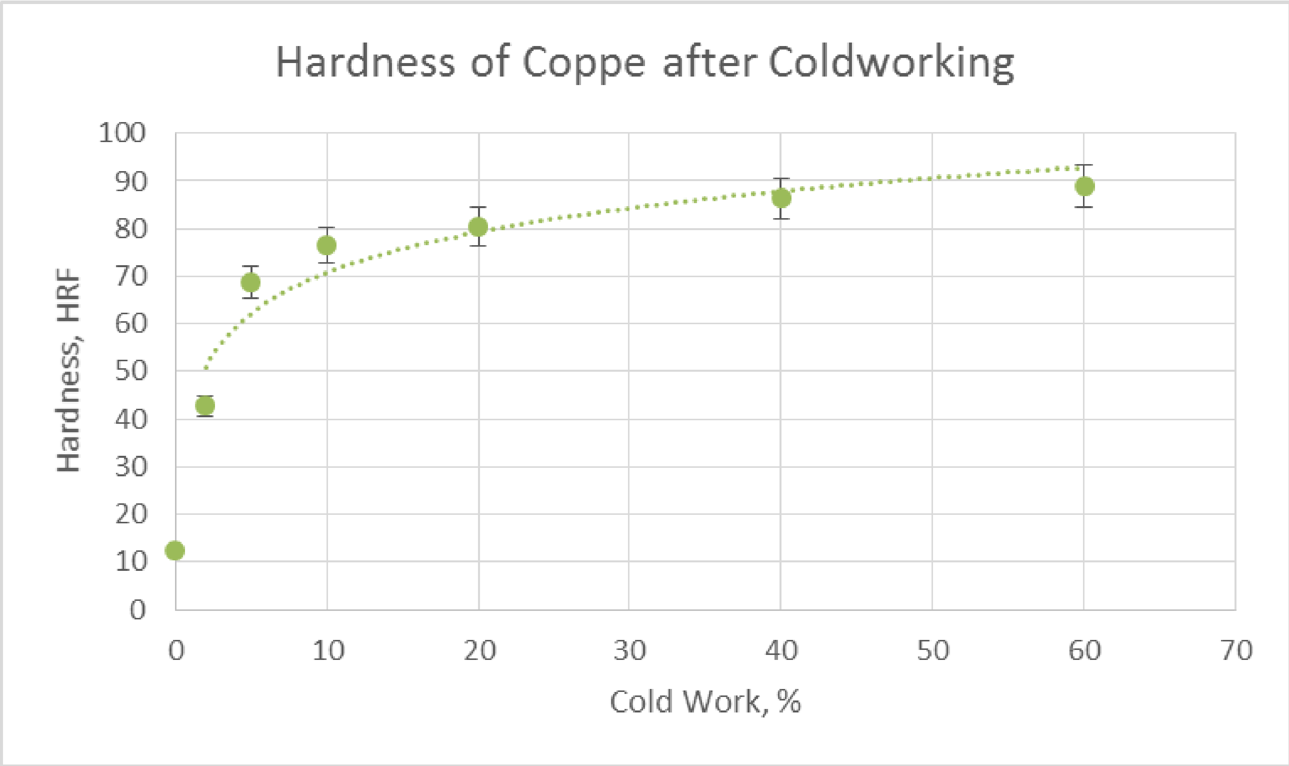


Figure 1. Hardness as a function of actual % cold work.

The hardness of annealed copper tends to decrease as it is hold at a constant temperature for a period of time, as demonstrated in Figure 2.

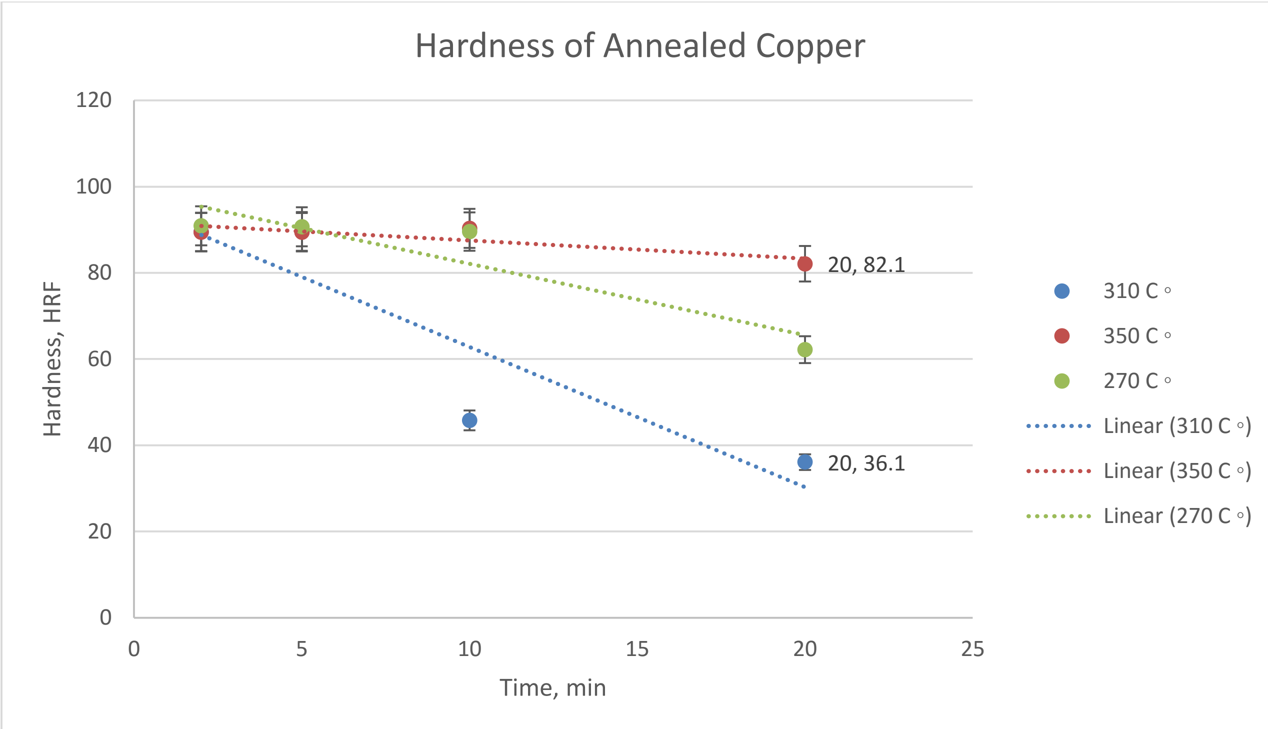
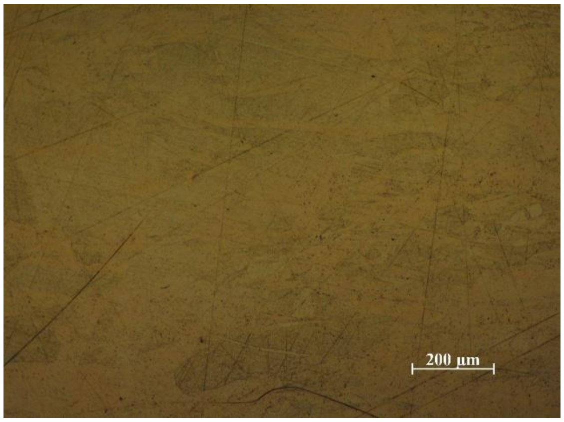
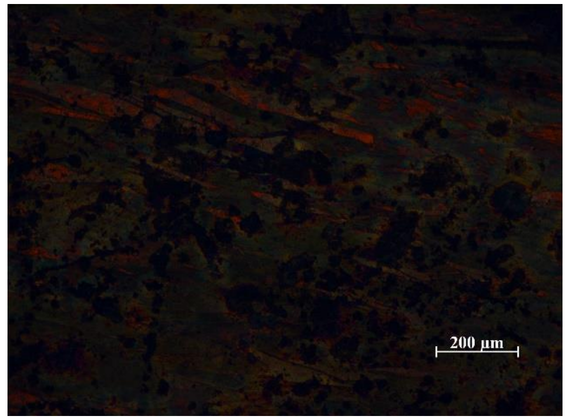


Figure 2. Hardness of copper versus annealing time as a family of three curves for different temperatures: 270 C ◦, 310 C ◦ and 350 C ◦.

The slopes of hardness measured varies with different temperatures. When copper is hold at 350 °C, the hardness decreases from 89.4 to 82.1 DPH. At 310 °C, the hardness decreases from 89.9 to 36.1 DPH. At, 270 °C, the hardness decreases from 90.9 to 62.2 DPH. The collected data shows that the annealed copper at 310 °C has a dramatic change in hardness after 10 minutes of treatment, while the sample annealed at 350 °C has the lowest affected hardness. The hardness of the Cu sample annealed at 270 °C lies in between the other two. Specific values can be found in Table 2 in the appendix.





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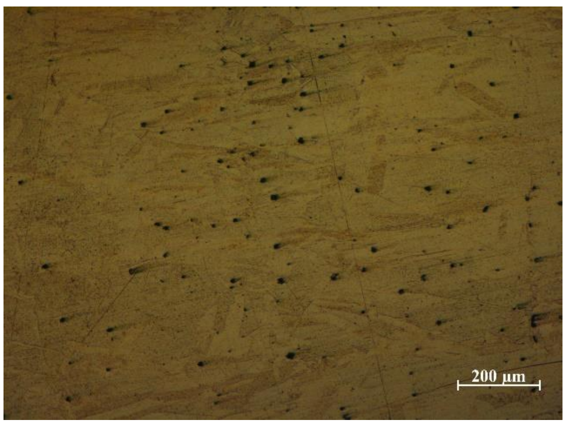

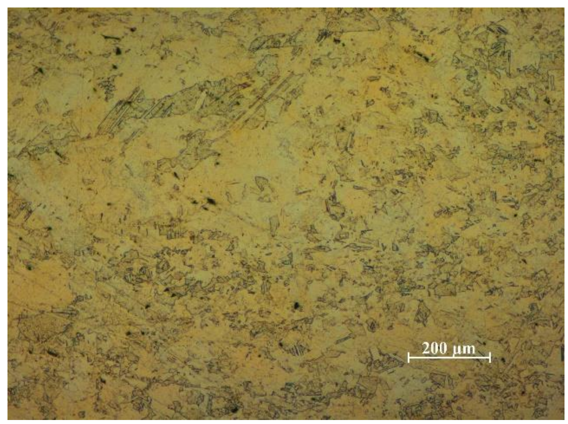

c d

Figure 3. Microstructural changes during annealing coldworked copper (a): showing 60% coldworked copper, (b): annealed coldworked copper for five minutes at a temperature of 270 C ◦., (c): annealed coldworked copper for five minutes at a temperature of 310 C ◦., and (d): annealed coldworked copper for five minutes at a temperature of 350 C ◦.

The microstructural changes in 60% coldworked copper shows a sharp increase in dislocations density as seen in (a). After copper has been strain hardened, both (b, c) show some stress relief. Annealing, or more specifically recrystallization, is observed as in Figure 3. (d), where there are still high dislocation densities in the grain; new strain-free grains nucleate and grow to replace the strained grains.

IV. Discussion

The curve of hardness as a function of actual % cold work shown in figure 1follows the accepted trend for metals, given the fact that Aluminum, silver and gold have the same crystal structure as copper and exhibit the same characteristics. [2, 3] Hardness of copper increases with increasing cold work. Other important mechanical properties such as tensile strength and yield strength increase with the increase of cold working, while ductility, toughness and impact strength decrease. Elastic modulus neither increases nor decreases with the increase of cold working.[2]

However, the three curves of hardness at the different temperatures of cold worked copper versus annealing do not follow the trends of other metals. Many studies have shown that the effect of temperature is much greater on restoring mechanical properties than the effect of time on the process of annealing metals. [1, 3] several errors could explain the differences: precision errors in annealing time; errors in hardness measurements and imprecision in labeling each sample. The bias errors might arise from the variations of temperatures as they were not held constant. Given that the temperature ranges are large, the calibration curves may not be as precise.

Nevertheless, the microstructures in figure. 3 indicate that the samples (b) and (c) are transferring from the recovery stage to recrystallization, where there is some dislocation motion and a reduction in the overall number of dislocations. Some physical properties such as electrical conductivity revert to values close to their pre-cold worked values. Mechanical properties, however, are not significantly altered, although residual stresses can be relieved. [1] Furthermore, the pre-cold worked condition of the metal is restored by this annealing treatment and the property changes are the reverse of strain hardening. After copper has been strain hardened, annealing, or more specifically recrystallization, will returned it to a soft and ductile condition. [3] Based on the metallographic results and hardness data, full recrystallization has occurred at 350 °C. During recrystallization, where the large cold worked grains are gradually replaced by a fine network of strain-free grains. [1, 3] The results observed in the experiment agrees with theoretical values. The effect of the temperature necessary for recrystallization to occur is a function of the composition of the metal and the amount of cold work; the higher the purity or greater the amount of cold work, the lower will be the recrystallization temperature. Literature confirms the experimental result to be accurate by stating that the recrystallization temperature for most metals is generally between one third and one half of the melting temperature. [2] The melting temperature for copper is 1080 °C, and one third of that is 360 °C, which is very close to the experimental value. [4]

V. Conclusions

Overall, the experiment succeeded in showing that hardness of moderately high purity copper (99.95%) increases as cold working increases, using the Rockwell hardness tester. From the hardness tests, it is asserted that annealing beyond the recovery stage has an effect on hardness. Other mechanical properties are restored as well one the material is fully recrystallized. The effect of cold-working on the hardness of copper and the effect of annealing at different times ranging from 5 to 20 minutes and three constant temperatures (270, 30 and 350°C) on the hardness of copper were measured with and observed with. Differences existed in the experimental graphs of hardness of copper versus annealing time and the theoretical trends. These differences, however, can be accounted for by experimental error. Instead, accurate results were derived from microstructural examination. The most suitable annealing temperature with which to restore the ductile properties of copper was determined to be 350°C. Microstructural changes which occur during cold rolling and annealing were observed using the Carl Zeiss optical microscope. Further experiments of cold rolling and annealing of copper should include further analysis of the grain growth and copper alloys in the strain hardening process.

References

[1] W.D. Callister Jr., Materials Science and Engineering: An Introduction, Seventh Edition (Wiley, New York, 2007).

[2]L. Reynolds, Cold roll and anneal of Cu, MSE 255 experiment description, 2016.

[3]W.D. Jia, K. T. Ramesh, and E. Ma, Appl. Phys. Lett. 81, 2002.

[4]J.R. Dives, Concise Metals Engineering Data Book, (ASM International, Ohio, 1997).

Appendix

Table.1: Hardness measurements of copper after cold working

Percent Cold work	Hardness, DPH	The Standard Deviation
0% CW	12.27	1.568
2%CW	42.73	2.937
5%CW	68.82	0.988
10%CW	76.51	1.229
20%CW	80.54	1.198
40%CW	86.32	0.345
60%CW	88.82	0.849

Table. 2: Hardness of Annealed Copper at Different Temperatures

Time	310 C ◦	350 C ◦	270 C ◦
2 min	89.5	89.4	90.9
5 min	89.6	89.4	90.7
10 min	45.7	90.3	89.5
20 min	36.1	82.1	62.2