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Original Article

Analysis of Microstructure, Recrystallization, and Hardness of Copper Metal Based on Cold Working and Annealing Processes

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Abstract - *The goal of this research is to evaluate the impacts of cold working and annealing on the microstructure of copper strips, study Rockwell's Hardness of copper strips undergoing cold working and annealing, and determine recrystallization temperature. The study shows that an increase in the temperature of annealing decreases the average hardness of the pieces of copper strips. Reduction of thickness from 0 % to 10 % decreases the average hardness of the copper strips. However, as the thickness reduction reaches 30 %, the average hardness would start to increase and continue to increase until a 50 % thickness reduction. The highest hardness is observed in the copper strips that had a 50 % thickness reduction due to rolling. Overall, this shows that the hardness of a material increases as the reduction of thickness increases. The microstructures of copper strips after undergoing cold work rolling show that grains are elongated in the rolling direction. On the other hand, the microstructures of copper strips undergoing annealing show that the grains appear to grow and expand after heating. The grains' rate of growth increases as the temperature of annealing increases.*

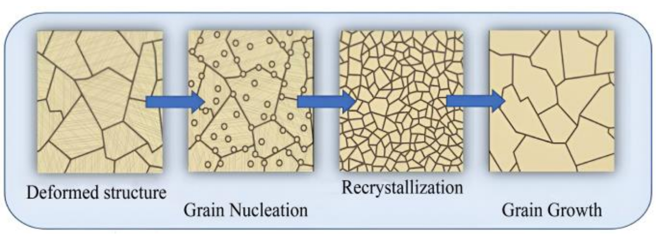
Keywords - *Annealing, Cold working, Hardness, Microstructure, Recrystallization temperature*.

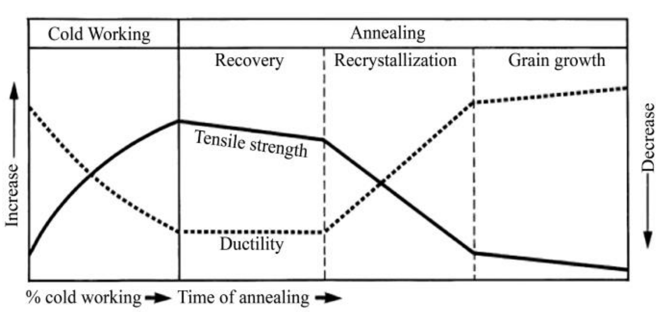
1. Introduction

Metal fabrication techniques in today's industry are typically followed by procedures that result in alloys with the appropriate properties, such as alloying, refining, and frequently heat-treating. Casting, powder metallurgy, welding, and machining are some of the several categories of fabrication techniques; usually, two or more must be used before a component is produced. Forming operations, such as forging, rolling, extrusion, and drawing, are those in which a metal piece's shape is altered by plastic deformation. The material's yield strength must be exceeded for the deformation to be caused by an external force or stress [1, 2]. The plastic deformation of a substance that is metallic under its recrystallization temperature is called cold working. A metal can be shaped by mechanical tension as opposed to heat. Permanent flaws in metals cause their crystal structure to change when they are cold-worked [3]. These flaws lessen the crystal's movement inside the metal structure. Consequently, the metal gains greater resistance against additional deformation [4]. The purpose of cold working is to improve tensile strength and hardness, improve surface finish, control dimensional tolerance and concentricity, improve straightness, and improve the workability of metals. Cold working, however, reduces the malleability of the metal and the ability to change shape without losing strength or breaking [5]. The most popular technique for work hardening is cold rolling. Grain size reduction during compression of the metal can increase strength within grain size limitations [6]. So, it is very urgent to analyze the cold working of a metal. Annealing is a method of heat treatment that makes the metal more ductile and less hard. The annealed material experiences changes in hardness and ductility due to a decrease in dislocations within its crystal structure [7]. In order to avoid brittle failure or to make a material simpler to handle later, annealing is typically done after a material has undergone a cold working or hardening procedure. Other purposes of annealing are to improve the formability and machinability of metals, as well as to eliminate residual stress [8]. There are three phases of annealing: recrystallization, recovery, and growth of grain. A heated furnace is used to increase the metal's temperature throughout the recovery process so as to lower its internal stress. While it is lower than the recrystallization temperature, the heating temperature during the stage of recrystallization is higher than the melting temperature [9]. This eliminates existing pressures and permits the development of new grains. New grains reach their full development during the growth phase. By letting the material cool at a predetermined rate, its growth can be managed [10]. This study is concerned with this process to evaluate the after-impact. The temperature at which recrystallization takes place for a certain material and processing parameters is known as the recrystallization temperature. It is not a fixed temperature, depending on several factors. First, extending the annealing period can lower the metal's recrystallization temperature [11, 12]. Furthermore, the alloy has a higher recrystallization temperature than pure metal. Raising the amount of cold work would lower the metal's recrystallization temperature. Furthermore, the recrystallization temperature is lowered by the decreased cold-worked particle size [13]. The annealing process is divided into three phases:

- Recovery phase - Removing internal stresses and crystal defects from metal, as well as covering all annealing events that take place prior to the emergence of new strain-free grains.
- Recrystallization - The process of recrystallization allows a fresh batch of perfect grains to nucleate and develop in place of the damaged grains until the original grains are completely utilized.
- Grain growth - The metal's microstructure begins to roughen, which could result in less-than-ideal mechanical qualities.

Microstructures are majorly affected by both cold working processes and annealing processes, showed graphically in Figure 1. In cold working, the dislocations of a material, for example, in any metal, are seen to interact and become pinned and then entangled with one another as the surface area is being compressed [14]. Hence, the dislocation motion becomes more difficult and restricted, making its cell to be much more complex when observed under a microscope [15]. As annealing is the vice versa of cold working, when controlled heat is applied over some time, it allows the grain to grow and the recrystallization phase to occur [16].





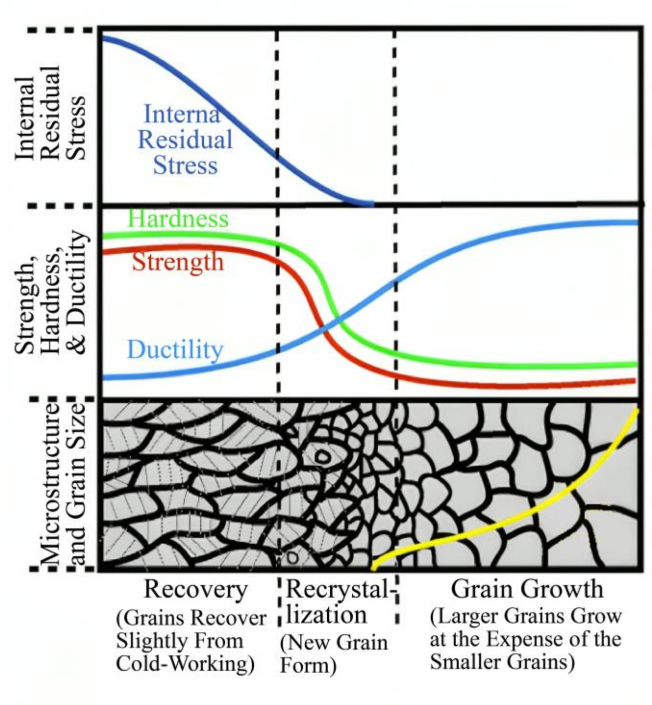


Fig. 1 Stages of annealing (a), the impact of annealing and cold working on internal stress (b), Microstructure and mechanical properties of metal (c)

Several studies are concerned with recrystallization and microstructure analysis; however, this study interconnected the different parameters to visualize the overall impact. The goals of this study are to find the recrystallization temperature of the metal sample (copper), investigate the relationship between thickness reduction, annealing, and mechanical properties, and analyze how the operations of annealing and cold working affect the mechanical properties and microstructure of copper metal.

2. Related Research Work

The impact of annealing and cold rolling on low alloy steel's grain refinement was discussed earlier in the research. The purpose of the rolling reduction was to distribute carbides and create stress energy that would be used to refine the grain during the annealing process [17]. Re-crystallization is expected to occur at temperatures with longer soaking times and the least degree of grain coarsening due to the dispersion of carbides, especially vanadium carbides [18]. Another research work also investigated the alloy's microstructure and characteristics both before and during annealing. The findings demonstrated that coarsening happened following periodization and that the alloy's microstructure eventually became uniform with an extension of the annealing duration and temperature. Furthermore, the microstructure underwent fusion and engulfment, causing the microstructure's size to first drop and then rise. The alloy's fracture strain and strength both first increased before declining. The alloy's microstructure was improved, and its characteristics were enhanced by the development of annealing twins [19].

Additionally, casting was utilized in another work to produce the copper-based alloys Al-bronze and α-brass, each containing 10% aluminum and zinc. Following that, the specimens experienced varying different types of deformation before being cold-rolled. The resulting samples were then aged for four hours at temperatures as high as 500 °C. Optical characteristics, differential scanning calorimetry, electrical resistivity, micro-hardness testing, and microstructure inspection with an optical microscope characterized the samples.

According to the findings, solid-solution hardening was the cause of the Cu based alloys' hardening. Al addition caused the aged alloy, which was not only brittle but also hard by nature, to generate different intermetallic copper aluminates, which accelerated the hardness during aging [20]. Another research work examined how different heat treatment temperatures affected the manganese bronze alloy's mechanical properties and microstructure. Nucleation and grain development followed the reduction of accumulated energy in the form of crystal imperfections brought about by the processes of rearrangement and dislocation annihilation. In the mechanical property analysis, yield strength, tensile strength, elongation, and Vickers hardness were all taken into account. The microstructure of the materials was examined using both optical and scanning electron microscopy. The findings indicated that as the annealing temperature increased, elongation increased, and Vickers hardness, tensile, and yield strengths decreased [21].

Similar research also conducted an analysis on copper alloys that combine high electrical and thermal conductivity with greater strength, thanks to the addition of chromium, hafnium, and scandium. To meet the needs of growing material, it is meant to contrast the precipitation process under temperature exposure. This study focuses on alloying elements whose solubility in copper decreases with temperature and whose maximum solubility is 1 weight percent [22]. Additionally, another same work discussed about Cu-Fe-P alloys.

Those deformed at cryogenic temperature (CT) develop a complex microstructure with Strain Localizations (SLs), heterogeneous deformation, and recrystallized and deformed grains. Both Electrons Back-Scattered Diffraction (EBSD) and High-Resolution Transmission Electron Microscopy (HR-TEM) were used to analyze the microstructural evolution of the CR specimens [23]. Besides, this research examined how pure copper will deform plastically when compressed at room temperature and during varied annealing times. To describe the solder-affected copper materials and examine potential modifications or enhancements resulting from work-hardening and thermal treatments, several properties, such as the hardness of the specimen, thermal stability, conductivity, and structure, were selected in this context [24]. Another potential study discussed and investigated the formation of intermetallic compounds during the alloying additions and heat treatment procedures of Al 4% Cu alloy with the addition of Ni and Sn. Many tests were performed after manufacture, such as optical microscopy, x-ray diffraction (XRD) analysis, Scanning Electron Microscopy (SEM) photographs, and hardness evaluations. Besides, this work examined how substructures affected conductivity and hardness in cold-rolled copper after partial annealing. After annealing at a lower temperature, substructures were seen; however, the sample that was annealed at a higher temperature largely lacked substructures. As a result, it is suggested that the restoration mechanism at lower temperatures envisions copper recovery; at higher temperatures, however, partial recrystallization occurs [25, 26, 27]. Many of the studies discussed about the characteristics of different copper-based alloys, not copper metals, separately. Most of the research works discussed separately about different attributes in different works. The novelty of this research is to evaluate the copper metal and evaluate fractional crystallization, microstructure as well and hardness, utilizing both annealing and cold working processes.

3. Materials and Methods

The 3 mm thick copper bar was cold-worked by rolling to decrease its thickness by 10 %. 30 % and 50 % using the roller. 12 pieces were rolled for each respective thickness reduction, and 4 pieces were left unrolled. The copper pieces were divided into 4 untreated groups, 200 °C, 300 °C and 600 °C annealing temperature. All sets were heated to their respective annealing temperature for 30 minutes by using a furnace. After 30 minutes, the copper pieces are left to be cold. The resin mixture was prepared by mixing 30 ml epoxy resin and 7 ml hardener using a small syringe. Then, copper pieces in each set were arranged based on the thickness and placed in a moulding holder. The epoxy resin was poured into the moulding holder of each set and allowed to be solidified for 24 hours.

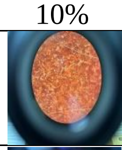
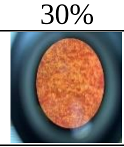
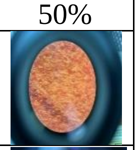
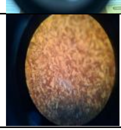
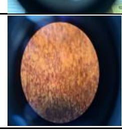
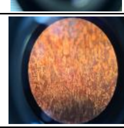
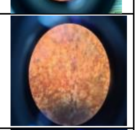
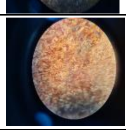
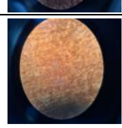
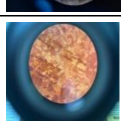
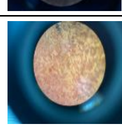
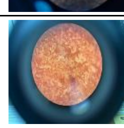
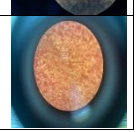
After the epoxy resin was solidified, all sets were taken out from the moulding holder. All the sets were polished by using a grinding and polishing machine with grade sandpaper 400, 600, and 1200 to remove the stretch and oxide layer. Sulfuric acid was used to etch each pair, revealing the metal's microstructure. Each copper piece's microstructure was examined using an optical microscope at 5x and 10x magnification. Pictures of the microstructure were taken as a result of this experiment. Rockwell Hardness Machine was turned on, and the Load knob of the machine was adjusted to set 15 kg of force to ensure constant force was applied to all specimens. The copper plate was placed on the platform and the “up” button was pressed to bring the plate close to the indenter. The start button was pressed to start the testing, and the hardness measurement was taken from the machine. This step was repeated 3 times to get the average value. The procedures were repeated for other copper plates. All data was recorded and tabulated.

4. Results and Discussion

4.1. Analysis of Microstructure

Physical characteristics, including strength, toughness, ductility, hardness, and corrosion resistance, are influenced by a material's microstructure. The various issues in or out of the structure are what essentially control how a material's microstructure affects its mechanical and physical qualities. The microstructures of copper strips after undergoing cold work rolling show that grains are elongated in the rolling direction. On the other hand, the microstructures of copper strips undergoing annealing show that the grains appear to grow and expand after heating. As the annealing temperature rises, the grain growth rate also rises. It would be observed from the microstructure of the copper sample that a larger amount of cold work done (indicated by the percentage of thickness reduction) causes the appearance of grain size to be smaller and more elongated, which follows the rolling direction, which is shown in Table 1. This is a result of the internal stress being exerted by the cold work on the copper atoms hence creating dislocations in the microstructure and increasing the dislocation density in the sample. After the cold working process, the average grain size decreases, whereas the area of grain boundary per unit volume rises. A higher amount of cold work would leave the grains elongated more in the rolling direction.

Table 1. Microstructure of copper under optical microscope

	Reduction			
°C	0%	10%	30%	50%
Un-treated				
200 °C				
300 °C				
600 °C				

The function of grain size expresses how the annealing temperature affects the microstructure of the copper sample. Based on the observations made on the microstructure for copper samples annealed at 200 °C, it is found that there are formations of a new set of strain-free and equiaxed grains. This is a depiction of a transition from the recovery stage to the recrystallization stage. At this stage, smaller nuclei that develop into new grains expand until they consume all of the parent material. At 300 °C, the grains are all now equiaxed and less deformed. This indicates the end of the recrystallization stage [28]. At 600 °C, the average grain size increases, whereas the area of grain boundary per unit volume decreases. At this point, there is no more formation of new equiaxed grain towards the end of the stage. The mechanical properties that were altered after cold working are now restored to their pre-cold worked values. In this case, the metal becomes weaker, has lower tensile strength, and is softer, but it now has a higher ductility.

4.2. Analysis of Hardness

Cold working produces crystallographic defects or dislocations across the atomic plane of the copper. After cold working, dislocation density increases and entangles with one another, causing dislocation motion to be more difficult. Hence, the dislocations pile up, forming obstacles, causing more repulsion with each other, and therefore resulting in higher resistance (hardness) to deformation. The hardness of copper materials is listed in Table 2.

Table 2. Hardness in different thickness reduction and annealing temperatures

% of Thickness Reduction	Annealing Temperatures	Hardness (RH)			
		1	2	3	Average
Un-Rolled	Un-treated	77.6	76.4	76.4	76.8
	200 °C	77.8	77.8	78.2	77.9
	300 °C	77.6	78.0	77.8	77.8
	600 °C	44.0	44.6	43.8	44.1
10% Reduction	Un-treated	78.4	79.2	78.0	78.5
	200 °C	78.0	76.8	78.4	77.7
	300 °C	79.6	77.8	80.2	79.2
	600 °C	47.6	44.2	44.2	45.3
30% Reduction	Un-treated	81.2	79.6	82.0	80.9
	200 °C	81.6	80.0	78.8	80.1
	300 °C	82.6	80.2	81.2	81.3
	600 °C	45.6	46.2	46.8	46.2
50% Reduction	Un-treated	80.0	81.8	81.6	81.1
	200 °C	79.6	79.8	78.0	79.1
	300 °C	75.2	75.6	80.8	77.2
	600 °C	53.4	53.6	51.8	52.9

Hardness increases as the percentage of thickness reduction (deformation) increases which is shown in Figure 2. So, in other words, the yield stress is proportional to hardness and increases with the degree of cold work. As the mechanical properties of materials are distorted, hardness increases. When the hardness of the metal increases, its tensile strength also increases but its ductility decreases. The hardness of the copper that was annealed at 600 °C, however, is very much lower than other copper plates. The residual stress and recrystallization temperature decrease as the percentage of cold work increases [29]. The atoms may have increased internal tension due to dislocation as the temperature rises. The microstructure becomes unstable as a result of this. When the temperature rises, the copper atom may be relocated to a more stable matrix to reduce its stress to attain stability.

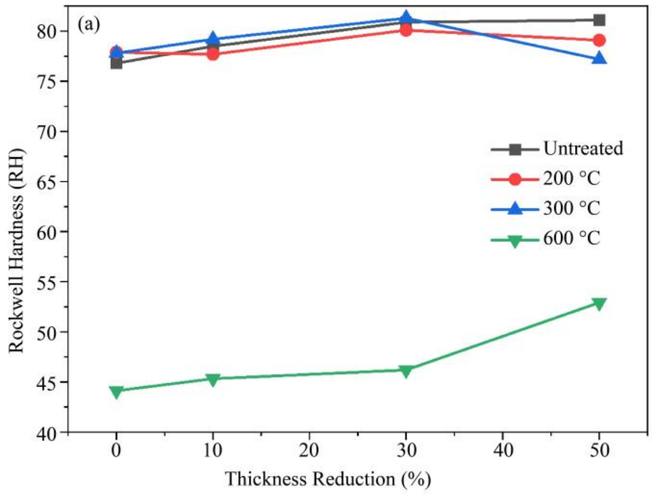


Fig. 2 Rockwell Hardness, RH vs. Thickness Reduction (%)

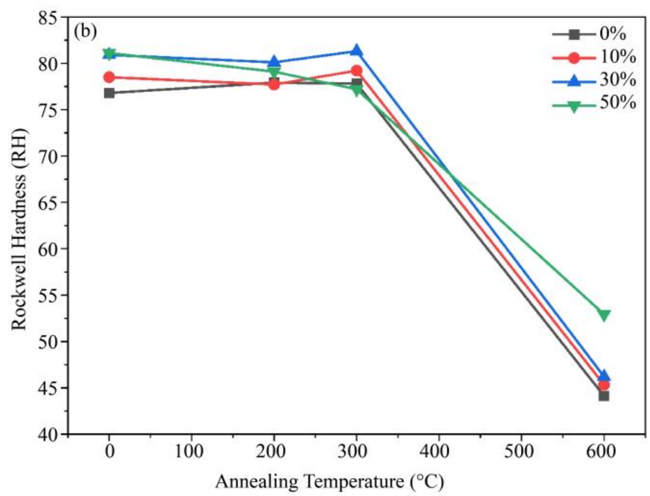


Fig. 3 Rockwell hardness, RH vs. Annealing Temperature (°C)

Therefore, the recrystallization temperature is lower when the percentage of cold working is larger. As the heat treatment temperature rises, the hardness of the copper plate reduces which is shown in Figure 3. If the annealing temperature increases, the grain boundary area per unit volume would be dropped as a result of a decrease in hardness. Reducing the strain from cold working requires heating the copper plate to a temperature below the recrystallization temperature, which reduces the frequency of dislocation. The metals recover more quickly at higher annealing temperatures, increasing ductility and decreasing hardness [30]. Copper metal loses hardness when it is annealed.

The tensile strength of copper metal falls as its hardness increases while its ductility rises. Considering the results of the Rockwell Hardness Test, the hardness of copper is inversely proportional to its annealing temperature. After 300 °C, the Rockwell hardness of all specimens decreases. This implies that at high annealing temperatures, the hardness of copper will decrease regardless of its microstructure [31, 32]. After 300 °C, all specimens experienced a significant decrease in hardness because the annealing temperature had already exceeded the recrystallization temperature. Copper's recrystallization temperature is approximately 300 °C.

5. Conclusion

In conclusion, an increase in annealing temperature decreases the average hardness of the pieces of copper strips. Reduction of thickness from 0 % to 10 % decreases the average hardness of the copper strips. However, as the thickness reduction reaches 30 %, the average hardness would start to increase and continue to increase until a 50 % thickness reduction. The highest hardness is observed in the copper strips that had a 50 % thickness reduction due to rolling. Overall, this shows that the hardness of a material increases as the reduction of thickness increases. The microstructures of copper strips after undergoing cold work rolling show that grains are elongated in the rolling direction. This makes the material anisotropic, having a larger stiffness along the rolling direction than in the transverse direction. However, the microstructures of copper strips undergoing annealing show that the grains appear to grow and expand after heating. An increase in annealing temperature causes the grains to develop faster.

Acknowledgements

This study was urgently needed to find out the temperature at which recrystallization occurs, as well as to examine the effects of cold working and annealing on the microstructure of copper strips and their Rockwell's Hardness. The study's authors watched all relevant activities and collected data in the university laboratory. Since the study didn't require a significant amount of funding to be completed, we haven't mentioned any financial sponsors.

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