MM 216 - PHYSICAL METALLURGY

REPORT

PROJECT TITLE : EFFECT OF ANNEALING ON MECHANICAL PROPERTIES

Objective:

Study of change in microstructure and hardness of cold worked copper and Cryo rolled copper after annealing.

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Abstract:

This study examines the influence of annealing on the microstructure and hardness of copper subjected to two different deformation techniques: conventional cold working and cryogenic rolling. The primary objective is to compare how annealing modifies grain structure and mechanical properties in each processing condition. Initially, both cold working and cryo-rolling introduce significant strain and dislocation density, resulting in increased hardness and elongated grain structures. Upon annealing, recovery and recrystallization occur, leading to notable changes in grain morphology and a reduction in hardness. In cryo-rolled copper, discontinuous recrystallization is observed below 350 °C, with certain grain orientations exhibiting preferential growth. At higher temperatures, abnormal grain growth occurs due to the presence of a wide grain size distribution. In contrast, conventionally cold-worked copper undergoes complete recrystallization around 300 °C, followed by grain coarsening at elevated temperatures. In both cases, annealing transforms the microstructure from a heavily deformed state to one with equiaxed grains, while also decreasing hardness and enhancing ductility. These results demonstrate that annealing has a profound impact on both microstructural evolution and mechanical properties, with distinct behaviors depending on the initial deformation method.

Key words:

Annealing, Cold working, Hardness, Microstructure, Recrystallization temperature. Electron Backscatter Diffraction(EBSD), Nanostructured materials, Thermomechanical processing, Grain growth, Cryogenically-rolled copper

Introduction:

The processing of copper, as with other metallic materials, typically involves a series of fabrication techniques aimed at achieving specific material properties. These processes often include alloying, refining, and heat treatments, with cold working and cryogenic rolling being among the most prevalent deformation techniques. Cold working refers to the plastic deformation of metal at temperatures below its recrystallization temperature (RT), which results in the introduction of high dislocation densities and strain into the crystal structure. This leads to an increase in strength and hardness, though at the expense of ductility and malleability. Conversely, cryogenic rolling, which involves deformation at sub-zero temperatures (typically below -150°C), has gained increasing attention due to its ability to produce ultra-fine grain structures and alter the fundamental properties of metals.

In both cold working and cryogenic rolling, significant internal stresses and a high dislocation density are introduced into the material, resulting in a non-equiaxed, elongated grain structure. Such deformation processes significantly affect the mechanical properties of the material, with hardness being increased and ductility reduced. The application of heat treatment, specifically annealing, is crucial to reverse or modify the effects of these deformation processes. Annealing is a heat treatment process typically performed to relieve internal stresses, restore ductility, and alter the material's microstructure.The annealing process is generally divided into three primary stages: recovery, recrystallization, and grain growth. During the recovery phase, the metal undergoes a reduction in internal stresses, with minimal changes to the microstructure. The recrystallization phase follows, where new strain-free grains nucleate and grow, replacing the deformed grains. Finally, at elevated temperatures, the process of grain growth may occur, which involves the coarsening of the recrystallized grains, leading to a reduction in the material's strength.

The recrystallization temperature, at which the onset of recrystallization occurs, is influenced by several factors, including the extent of prior deformation and the material's composition. In particular, the cold work introduced by either conventional cold working or cryogenic rolling can significantly lower the recrystallization temperature compared to that of the non-deformed material. This results in distinct recrystallization behaviors between materials subjected to different deformation techniques. Cryo-rolled copper, for instance, is prone to discontinuous recrystallization, especially at lower temperatures (below 350°C), while conventionally cold-worked copper tends to undergo a more typical recrystallization process followed by grain coarsening at higher temperatures.This study aims to investigate the influence of annealing on the microstructure and hardness of copper subjected to two distinct deformation techniques: conventional cold working and cryogenic rolling. Specifically, the research will focus on the evolution of the grain structure and the hardness properties during annealing. Through this investigation, the study will assess the extent of grain refinement, grain morphology, and the associated mechanical property changes induced by annealing in each processing condition.In cryo-rolled copper, discontinuous recrystallization is expected to occur below 350°C, with preferential growth of certain grain orientations. At temperatures above this threshold, abnormal grain growth is anticipated due to the wide distribution of grain sizes present in the deformed structure. In contrast, conventionally cold-worked copper will undergo complete recrystallization around 300°C, followed by grain coarsening at higher annealing temperatures. The microstructural transformations in both cases are expected to lead to a transition from a heavily deformed, elongated grain structure to a more equiaxed grain morphology, accompanied by a decrease in hardness and an improvement in ductility.

By exploring the interplay between cold working, cryogenic rolling, and annealing, this study seeks to contribute to a deeper understanding of the microstructural evolution and mechanical property changes that occur during heat treatment processes. The findings will offer valuable insights into how different deformation histories influence the final material properties, which is crucial for tailoring the performance of copper in various engineering applications where a balance between strength, ductility, and hardness is essential.

Materials and Methodology:

In this study, 99.9 wt pct pure copper, supplied as a hot-rolled bar with an initial thickness of 3 mm, was chosen as the material for investigating the effects of different deformation and annealing processes on its microstructure and hardness. This material was selected due to its high purity and common use in industrial applications where thermal and mechanical treatments are employed to enhance its properties. The copper samples were processed using two distinct methods to explore the influence of varying deformation conditions: cold-working and cryogenic rolling.

For the cold-working process, the copper bar was subjected to rolling with thickness reductions of 10%, 30%, and 50%, producing a total of 12 samples per reduction. Additionally, 4 samples were kept unrolled as controls. The samples were then divided into four groups and furnace-annealed at temperatures of 200°C, 300°C, and 600°C for 30 minutes, with a natural cooling process. To analyze the microstructure, the annealed samples were polished using sandpapers (400, 600, and 1200 grit), etched with sulfuric acid, and examined under an optical microscope at magnifications of 5x and 10x. The hardness of the samples was measured using both Rockwell hardness testing with a 15 kg load and Vickers microhardness testing with a 50 g load, performed at multiple points on each sample to obtain average values.

For the cryo-rolled copper, the material underwent a severe "ABC" deformation and was subsequently cryogenically rolled to achieve a total thickness reduction of 90% (true strain = 2.3), with each pass reducing the thickness by 10%. To maintain cryogenic conditions, both the workpiece and the rolls were soaked in liquid nitrogen for 20 minutes before each pass, and the copper sample was immediately reinserted into the nitrogen after each rolling pass. This process ensured that the temperature of the copper remained low, with minimal temperature increase during the deformation (approximately 1-4°C). After the cryogenic rolling, the samples were furnace-annealed over a temperature range from 50°C to 950°C for 1 hour, including 10 minutes for heat up. Additionally, isothermal annealing was carried out at 150°C and 450°C for various time intervals. Following each heat treatment, the samples were quenched in water to preserve the microstructures. For microstructural characterization, electron backscatter diffraction (EBSD) was used, with the samples prepared using conventional metallographic techniques and electropolishing in a 70% orthophosphoric acid solution at an applied voltage of 5V.

Observations were conducted in the longitudinal and rolling planes, focusing on the mid-thickness of the sheets. The EBSD data was collected using a Hitachi S-4300 SE field-emission scanning electron microscope (SEM) equipped with a TSL OIM™ EBSD system, and the grain size was quantified using both the linear intercept method for cryo-rolled and partially recrystallized samples and the grain-reconstruction method for fully recrystallized samples. Hardness was again measured using Rockwell hardness (15 kg load) and Vickers microhardness (50 g load), with at least 10 measurements taken per sample to ensure accuracy.

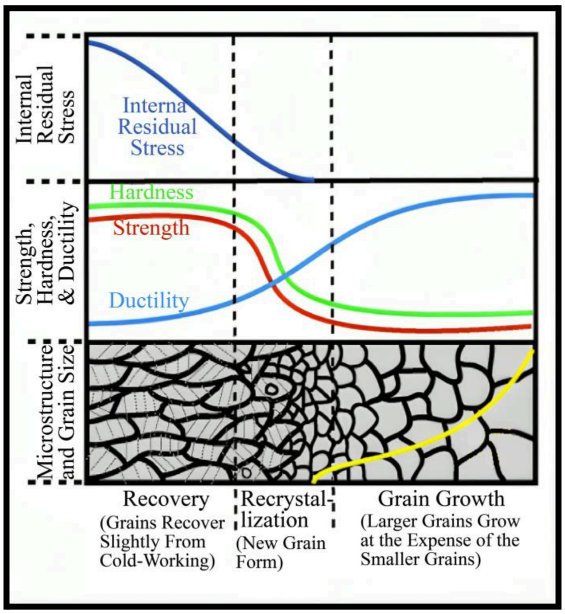
Results and Discussion:

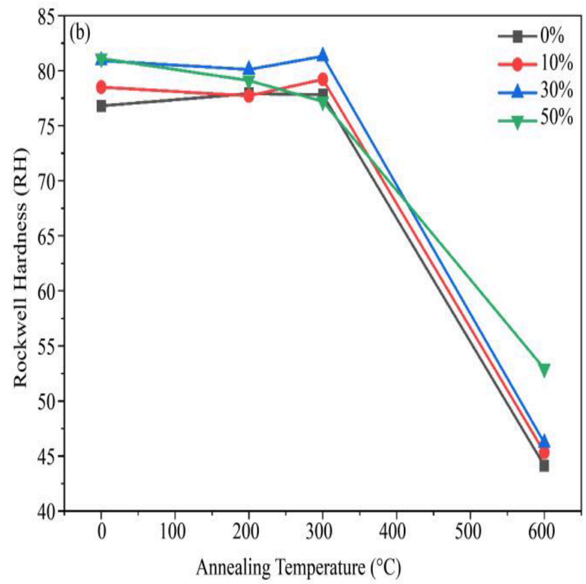
The evolution of microstructure and hardness in copper samples subjected to cold working and cryogenic rolling was systematically studied as a function of annealing temperature. The findings demonstrate a strong correlation between deformation history, thermal treatment, and the resulting mechanical and microstructural properties.

Cold worked Copper:

The microstructural and hardness evolution in cold-worked copper was studied using samples reduced by 10%, 30%, and 50% thickness, followed by annealing at 200 °C, 300 °C, and 600 °C. In the as-cold-worked state, optical microscopy revealed elongated grains aligned along the rolling direction, especially at higher reductions. These grains exhibited high aspect ratios and internal distortions, indicative of a high dislocation density and stored strain energy. Hardness increased with deformation: ~76 RH for 10% reduction, ~79 RH for 30%, and ~81 RH for 50%, due to work hardening from dislocation accumulation.

Annealing at 200 °C initiated recovery processes. Subgrains began to form within the deformed structure, and limited nucleation of recrystallized grains occurred in the heavily deformed 50% samples. However, grain refinement was still incomplete, and hardness decreased only slightly. The structure remained largely deformed, especially in the lower reduction samples.





At 300 °C, recrystallization was nearly complete across all deformation levels. Micrographs showed uniform, equiaxed grains replacing the elongated deformation structures. The LAB content dropped significantly, and strain-free HABs dominated the microstructure. Hardness dropped substantially to ~52 RH (10%), ~50 RH (30%), and ~55 RH (50%), indicating a reduction in internal stresses and restoration of ductility.

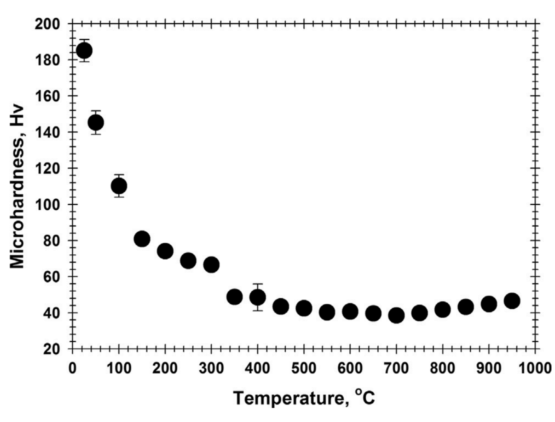
At 600 °C, grain growth was evident. Grains coarsened and became more rounded and equiaxed. The microstructure showed fewer grain boundaries per unit volume, suggesting active boundary migration. Hardness values further decreased to ~45 RH (10%), ~46 RH (30%), and ~53 RH (50%). The slightly higher hardness in the 50% reduced sample may be due to residual substructures that delayed grain coarsening.

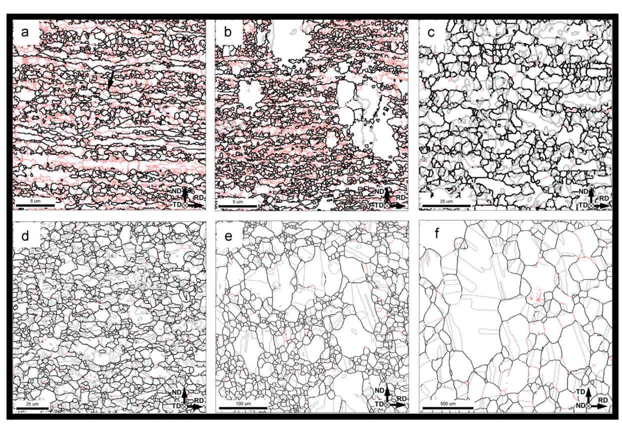
<u>Cryo rolled copper:</u>

The cryo-rolled copper samples, processed to a 90% reduction at liquid nitrogen temperature, showed a distinctly different behavior during annealing due to the extremely high stored energy from suppressed dynamic recovery and severe plastic deformation.

In the as-cryo-rolled condition, the microstructure was highly strained, with ultra-fine, elongated grains and a high fraction of LABs. EBSD maps confirmed dense subgrains, high dislocation content, and significant texturing. These samples exhibited the highest hardness among all conditions due to intense work hardening and minimal recovery during rolling.

Annealing at 50 °C led to a significant drop in hardness. This rapid softening, despite the low temperature, indicates the early onset of recovery and discontinuous recrystallization. A bimodal grain structure emerged, consisting of a mix of coarse, newly formed grains and fine, deformed ones. LABs began converting to HABs as grain boundaries became mobile, and hardness decreased dramatically.





At 150 °C, the sample appeared nearly fully recrystallized. Equiaxed grains dominated the microstructure, and the LAB fraction dropped sharply. Annealing twins began to form, reflecting active grain boundary migration. Hardness values stabilized at lower levels, signaling the transition to a strain-free structure.

From 300 °C to 450 °C, microstructural changes were minor. The grains remained equiaxed, and hardness values plateaued. This temperature range represented the completion of recrystallization and a thermally stable state.

However, above 450 °C, abnormal grain growth initiated. Grain size became highly inhomogeneous, and a second drop in hardness was recorded. This stage saw the reappearance of bimodal grain distributions and a slight increase in LABs due to internal substructuring of growing grains.

At 600 °C and beyond, grains became extremely coarse, with sizes exceeding several hundred microns. The structure became fully recrystallized and dominated by HABs, with a high density of annealing twins. Hardness values continued to decline but showed a slight recovery in the 750–950 °C range, likely due to twin boundary strengthening mechanisms that offered some resistance to dislocation motion.

Conclusion:

Annealing of cold-worked and cryo-rolled copper reveals distinct microstructural and hardness changes: cold-worked copper exhibits increased hardness with thickness reduction (peaking at 50%), while annealing reduces hardness as grains grow and become equiaxed, particularly above 300°C. Cryo-rolled copper (90% reduction) shows extreme microstructural instability, with recrystallization initiating at 50°C and completing by 150°C, causing rapid hardness loss. Below 350°C, discontinuous recrystallization favors grains with (55;30/60;0), Brass-R, and Dillamore orientations, while Brass and Goss orientations resist recrystallization; above 300°C, abnormal grain growth dominates due to broad grain-size distributions (1–40 µm). Cryo-rolled copper's low-temperature recrystallization and abnormal grain growth contrast with conventional cold-worked copper's gradual softening, highlighting its heightened sensitivity to annealing. The cryo-rolled copper responds much more sensitively to annealing compared to cold-worked copper. In both conditions, prolonged annealing at higher temperatures leads to significant grain growth and softening, but the grain coarsening is more pronounced and occurs earlier in cryo-rolled copper.

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