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The Effect of Annealing Conditions on Copper's Brittleness and Powder Production Efficiency

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Recycling copper wire to produce the correct copper powder extends the environmentally friendly alternative to traditional refining techniques. That exploration research uses different annealed environments to regulate the embrittlement and ease of the crunch of the copper wire, with a particular focus on the variations in temperature and the consequences of the atmosphere. The study included heat conductor copper wire in the wind and a synthesis gas mixture (40 % H_2, 60 % N_2) within a temperature range of 250 °C to 850 °C with a duration of either 30 or 60 minutes. Several trials were carried out to measure the influence of the treatment, including the bend test to measure the toughness, the optical microscopy to measure the evolution of the microstructure, and the mass loss measurement to measure the oxidation tiers. The results show that annealing gas at a temperature between 600 °C and 650 °C for 45–60 minutes produces significant embrittlement, which makes the copper more prone to cracking at all right atoms. A microstructural study confirms that the embrittlement detected in the minimization atmosphere is due to excessive grain expansion and the formation of a void caused by hydrogen infiltration. Moreover, oxidation was well reduced under conditions of synthesis gas, with a mass loss of approximately 1–3 %, in contrast to the oxygen record of 10 % above 850 °C. These revelations underline the possibility of controlled annealing and hydrogen embrittlement as a cost-effective and resource-efficient method of producing superior copper powder, which could be highly beneficial for some industrial objectives.

Keywords: Copper Powder Production, Scrap Copper Recycling, Annealing Conditions, Hydrogen Embrittlement, Synthesis Gas Atmosphere, Microstructural Analysis, Brittleness Evaluation,

1 Introduction

Powder metallurgy (PM) has confirmed itself as a key technique in current production, allowing the development of intricate and high-performance components from metallic element powders with minimal material waste [1]. The current method enables excellent dimension control, reduced machining, and simplified material utilization, particularly in the field of automobiles, aeronautics, electronics, and medical devices. [2]. Copper has better thermal and electrical conductivity, corrosion resistance, and mechanical flexibility, which makes it indispensable in electronic components, antifriction components, and heat conductor compositions [3].

The increasing demand for high-quality copper powder has led researchers to explore various production methods, including atomization, electrolytic deposition, chemical reduction, and mechanical comminution [4]. Each of these techniques has its advantages and limitations. Angelo and Subramanian outlined the fundamentals of powder metallurgy. They noted that while atomization and electrolysis are widely used for producing copper powder, they often require high-purity feedstock and energy-intensive setups [5]. Ünal added that atomization offers good control over particle size but is less economical when dealing with lower-value scrap materials [6].

Traditional mechanical grinding methods are typically not suitable for ductile metals like copper, as they tend to deform plastically rather than break into powder [7]. Rojas-Díaz et al. (2020) emphasized that mechanical grinding is highly efficient for brittle materials but less so for ductile ones unless pre-treatment methods are applied [8]. The present article promotes the search for treatments that may embrittle copper to make it more hospitable to mechanical pollution. An individual promising technique is to anneal copper in a hydrogen-containing environment to cause intentional brittleness [9]. Although this process, known as Hydrogen embrittlement, is usually considered to be a negative effect on copper components, it can be used strategically for powder production. Asgharzadeh and Eslami (2019) study the microstructural consequences of hydrogen interaction with the copper complex and show that hydrogen can significantly reduce the ductility of the metallic element by supporting the intergranular crack [10]. Ramazonovich et al. (2021) further detailed by which method copper's mechanical conductivity changes below thermal treatment, demonstrating the possibility of embrittlement subordinate to precise annealing conditions [11].

The transformation process is driven by the interaction between residual oxygen in copper, primarily in the form of Cu_2O, and hydrogen gas during high-temperature annealing. When exposed to elevated temperatures, hydrogen reacts with Cu_2O, reducing it to metallic copper and water vapor, as shown in Equation (1):

$$Cu_2O + H_2 \rightarrow 2Cu + H_2O \qquad (1)$$

As soon as the chemical reaction occurs, water vapor is produced inside the metallic element's microstructure, particularly in the vicinity of grain boundaries. During cooling, the vapor is ineffective in its attempt to escape, developing internal tension that leads to the formation of nothingness and microcracks. The current situation, normally defined as a hydrogen disease, significantly reduces the material's second ductility and makes it brittle. Interestingly, since this importance has traditionally been considered harmful, Schrijvers et al. (2020) state that chemical reactions can be exploited as a productive means of handling and recycling copper under controlled situations.

From a longevity point of view, the use of recycled copper bits as an alternative to virgin elements is in line with modern manufacturing intentions, particularly under the increasing burden of ecological management and provisioning [16]. Moghimian et al. (2021) emphasized the need for closed-loop recycling in metal powder production and highlighted the challenges and opportunities in reusing industrial waste streams [17]. In this context, the brittle conversion of copper from scrap conductors represents a compelling solution [18].

Despite these developments, systematic studies on the influence of annealing temperature, time, and atmospheric composition on copper's brittleness, particularly using waste materials, remain limited [19]. Previous works have addressed the embrittlement mechanism in controlled environments but have seldom linked it directly to powder production potential.

Copper powder is primarily utilized in powder metallurgy, which also uses it to create composite materials and a variety of products for electrical, antifriction, and structural uses [21]. Copper powder, a byproduct of the intermediate copper production process, is subject to particular specifications [22].

Copper and its alloys have been widely used in various industrial applications due to their excellent electrical and thermal conductivity, corrosion resistance, and mechanical properties [23]. The demand for high-performance copper powders has increased in industries such as electronics, additive manufacturing, and metallurgy, where precise control over material properties is crucial [24]. One of the key processing techniques that significantly influences the properties of copper powders is annealing [25].

Annealing is a heat treatment process that alters the microstructure of a material to enhance its mechanical and physical properties. In the context of copper powder production, annealing conditions such as temperature, time, and atmosphere play a vital role in determining particle morphology, grain size, hardness, and electrical conductivity [26]. Optimizing these parameters is essential to achieve desirable properties for specific industrial applications.

Previous studies have shown that variations in annealing conditions can lead to significant differences in the microstructure and properties of copper materials [27]. Understanding the relationship between annealing parameters and copper powder characteristics is essential for improving production efficiency and material performance [28]. This research aims to investigate the effect of different annealing conditions on the properties of copper powders, providing insights into the optimal processing parameters for achieving the desired mechanical and electrical properties [29]. By analyzing the impact of temperature, time, and atmospheric conditions, this study contributes to the advancement of copper powder processing techniques and their application in modern industries [30].

Furthermore, the findings of this study will aid in the development of enhanced manufacturing processes that can be tailored to meet specific industrial requirements. By optimizing annealing conditions, manufacturers can achieve improved material performance, reduced production costs, and enhanced reliability in applications requiring high-quality copper powders. This research not only contributes to the scientific understanding of copper annealing but also provides practical insights for industries aiming to refine their processing techniques and material utilization.

This research aims to bridge that gap by evaluating how specific annealing regimes affect the microstructure and mechanical behavior of copper scrap, ultimately rendering it suitable for powder manufacturing.

This study focuses on copper wire scrap, specifically enameled conductors, as a source material. The objective is to determine the optimal annealing conditions that produce a brittle copper structure capable of undergoing mechanical grinding. By annealing copper in synthesis gas (approximately 40% hydrogen) across a temperature range of 250–850 °C and holding times of 30 to 60 minutes, the study investigates the extent of brittleness achieved through bend testing, metallographic inspection, and grain size analysis.

Through systematic experimentation with annealing media and conditions, this work offers a novel pathway for producing fine copper powder from scrap material, thereby contributing both to metallurgical science and sustainable manufacturing practices.

2 Methodology

The primary material used in this study was scrap copper wire, commonly found in electrical wiring. The wires had enamel insulation and an average diameter of 1.3 mm. These were selected to evaluate the feasibility of direct recycling and powder production without traditional electrolytic or melting-based refining.

This study used several pieces of equipment to carry out the experimental procedures, as listed in Tab.1

Copper wires were cut into 30 cm lengths. Enamel insulation was not removed manually, as part of the process aimed to demonstrate the benefit of in-situ insulation removal during annealing.

Annealing was conducted in two atmospheres: Air and Synthesis gas (~40% H_2, 60% N_2)

Each batch of copper samples was subjected to the following annealing parameters Tab. 2.

Tab. 1 *Equipment used to carry out the experimental procedure*

Equipment	Purpose
Horizontal Tube Furnace (1200°C max)	Annealing under a controlled atmosphere
Gas Mixer Unit	Preparation of synthesis gas (40% H_2 + 60% N_2)
Thermocouples (K-type)	Real-time temperature monitoring
Ball Mill / Disintegrator	Grinding annealed copper into fine powder
Air Separator + Cyclone Unit	Removal of insulation ash and oxide particles
Metallographic Polishing Kit	Preparation of samples for microscopic analysis
Optical Microscope with Camera	Microstructural observation and grain size measurement
Bend Test Apparatus (ISO/R 398:1964)	Ductility evaluation through controlled deformation
Analytical Balance (0.001g accuracy)	Mass loss and ash content measurement

Tab. 2 *Annealing parameters*

Temperature range	250°C to 850°C (in 50°C increments)
Holding times	30 and 60 minutes
Heating rate	~10°C/min

After annealing, samples were subjected to bend testing to assess ductility loss. Ductility was evaluated using a three-point bending setup according to ISO/R 398:1964, which specifies the bend angle classification method for wire materials. The objective was to evaluate the material's ductility or brittleness following annealing. The results were categorized qualitatively based on the bend angle achieved during the test, as in Tab. 3:

Tab. 3 *Bend Test Results Vs Annealing Conditions*

Atmosphere	Temperature (°C)	Time (min)	Bend Angle (°)	Brittle?
Air	550	30	~90	No
Synthesis Gas	650	45	30–40	Yes
Air	700	60	60–70	Semi-brittle
Synthesis Gas	600	60	40–50	Yes

The strain during bending was calculated using Equation 2:

$$\varepsilon = \frac{t}{2R} \tag{2}$$

Where:

t…Wire thickness (in mm);

R…Bend radius (in mm).

This approach provides a clear assessment of the copper's ductility or brittleness after undergoing various annealing conditions.

We sectioned and polished annealed samples using standard metallographic techniques and etched them using a ferric chloride + hydrochloric acid solution.

We focused on the Grain boundary definition, the Presence of microcracks, and gas bubble inclusions.

Brittle copper samples were placed in a ball mill for 60 minutes at 150 rpm. Afterward, the material was sieved, and particles <100 μm were collected. Ash and oxide flakes were removed using an air separator with a cyclone collector.

Grain size observations are shown in Fig. 1. Fig 1.a shows the Microstructure of unannealed copper with elongated grains due to mechanical drawing.Fig. 1.b for Annealed copper in air at 550°C, moderate grain growth observed with no visible voids, and Fig. 1c shows the copper annealed in synthesis gas at 650°C, significant grain coarsening and intergranular voids visible, consistent with hydrogen embrittlement.

Observations under 100x and 400x magnifications were performed using an optical microscope. The grain size was measured according to EN 16090:2020 Fig. 1, and Tab. 4.

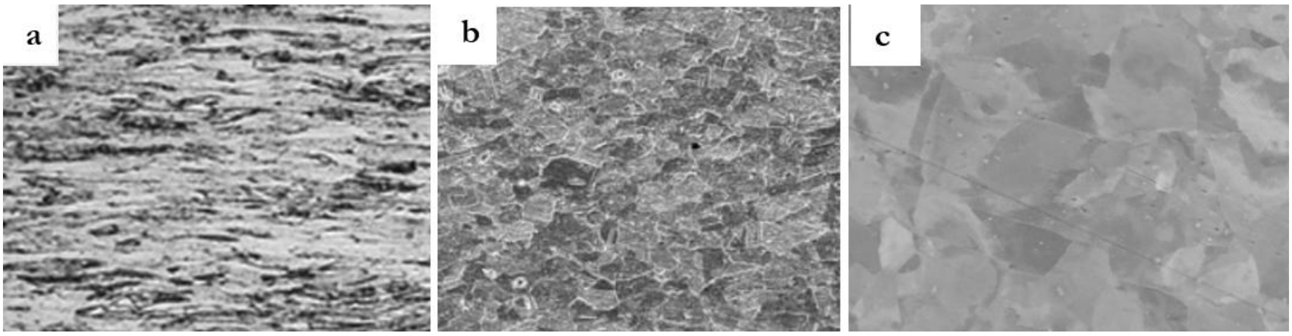

Fig. 1 *Influence of cold working, annealing temperature, and atmosphere on grain size.*

Tab. 4 *Grain size growth*

Condition	Across Fibers (mm)	Along Fibers (mm)	Notes
Before Annealing	0.001–0.015	0.01–0.02	Elongated grains
Annealed in Air @ 550°C, 30min	0.017–0.030	0.015–0.030	Slight coarsening
Annealed in Gas @ 650°C, 45min	0.080	0.050	Microcracks present, recrystallized grains

3 Results and Discussion

Bend tests were conducted on annealed copper specimens, and the results confirmed a clear correlation between annealing conditions and the material's ductility/brittleness Fig. 2.

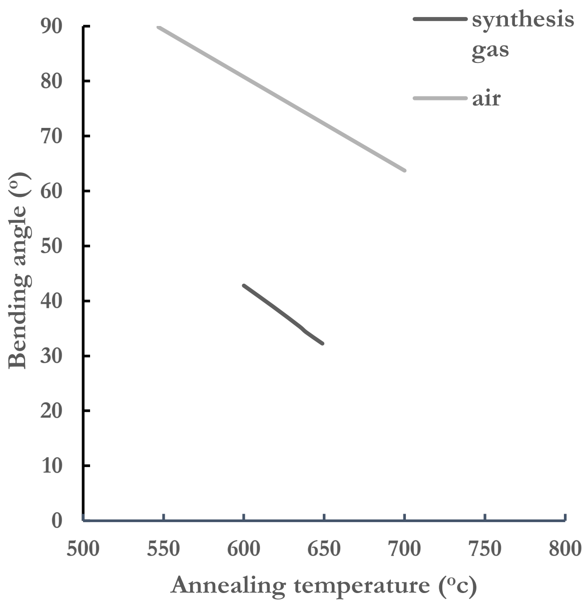

Fig. 2 *Influence of annealing temperature and atmosphere on brittleness*

- Air Atmosphere: At 550°C for 30 minutes, the copper sample retained its ductility with a 90° bend angle, indicating minimal embrittlement. However, at 700°C for 60 minutes, the bend angle was reduced to 60–70°, categorizing the material as semi-brittle.
- Synthesis Gas Atmosphere: At 650°C for 45 minutes, samples exhibited extreme brittleness, fracturing at 30–40°. Similarly, at 600°C for 60 minutes, the bend angle remained 40–50°, confirming embrittlement.

These results suggest that a reducing atmosphere (H_2 + N_2) promotes embrittlement more effectively than an oxidizing environment (air), particularly at higher temperatures.

The microstructural analysis reveals that the embrittlement in synthesis gas annealing correlates with the formation of microcracks and grain boundary voids. This aligns with the hydrogen embrittlement theory, where trapped hydrogen-induced voids weaken intergranular cohesion.

The decrease in bend angle in Fig. 2 corresponds with the presence of intergranular voids seen in Fig. 1c, confirming hydrogen embrittlement

Microstructural observations were performed to assess the grain morphology and crack formation Fig. 3

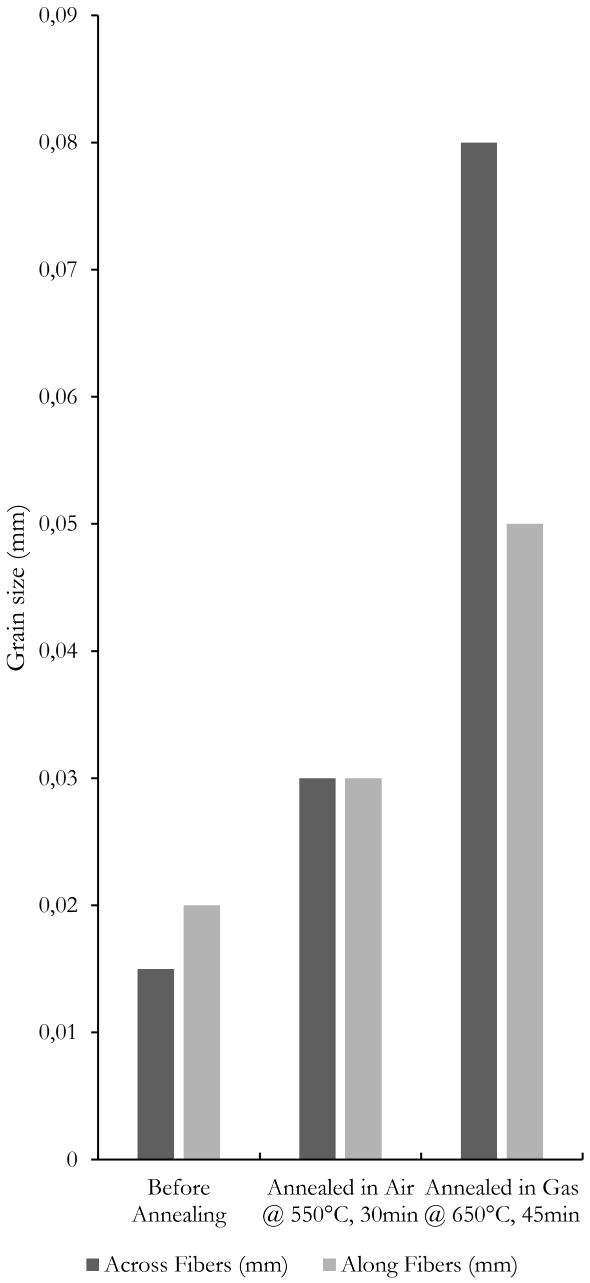

***Fig. 3** Grain size according to annealing conditions.*

In both atmospheres, grain size increases with temperature, which is expected as higher annealing temperatures promote grain growth. The increase in grain size is more pronounced in the Synthesis Gas Atmosphere, particularly beyond 600 °C. This suggests that the synthesis gas environment enhances grain coarsening, potentially due to a reduction in surface energy or changes in diffusion mechanisms.

Atmospheric Influence: Under an Air Atmosphere, the grain size exhibits a steady and moderate increase. Under Synthesis Gas Atmosphere, the grain size sharply rises after 600 °C, reaching values significantly higher than those in air. This behavior suggests that the synthesis gas environment may facilitate grain boundary movement, leading to accelerated growth at elevated temperatures.

Pre-Annealing Structure: The grains were elongated because of the mechanical processing of the wire.

Air-Annealed Samples: Showed moderate grain growth, indicating typical recrystallization behavior.

Synthesis Gas-Annealed Samples: Exhibited excessive grain growth with voids and cracks along grain boundaries, explaining the drastic reduction in ductility.

The observed embrittlement is attributed to hydrogen diffusion into the grain structure, forming high-pressure voids that propagate into cracks upon mechanical stress.

Post-annealing and milling, the chemical composition of the final copper powder was analyzed using X-ray fluorescence (XRF). The results showed that the powder maintained a high purity level with the following typical composition:

- Copper (Cu): 99.5–99.8 wt.%
- Oxygen (O): 0.1–0.3 wt.% (primarily due to residual Cu_2O from the scrap material)
- Trace elements (Fe, Ni, Zn): <0.05 wt.% total

The low oxygen and trace impurity levels confirm the effectiveness of synthesis gas in minimizing oxidation during annealing. This composition meets the requirements for most powder metallurgy applications involving high-conductivity copper.

Larger grain size and void formation due to hydrogen diffusion suggest increased internal energy and residual stresses. This could affect downstream processes such as sintering, conductivity, or mechanical stability. Addressing hydrogen content could improve the reliability of copper powder for high-performance applications

After annealing, the remaining ash (insulation residue and oxides) was separated and weighed. Mass loss (%) was calculated using Equation 3, Mass loss percentage:

$$\text{mass loss\%} = \left(\frac{\text{m initial} - \text{m final}}{\text{m initial}}\right) x100 \quad (3)$$

Where:

$m_{initial}$…Initial mass of the specimen (in grams);

m_{final}…Final mass of the specimen after annealing (in grams).

Mass loss due to oxidation and decomposition was measured post-annealing. The percentage mass loss was calculated. The results are illustrated in Fig. 4.

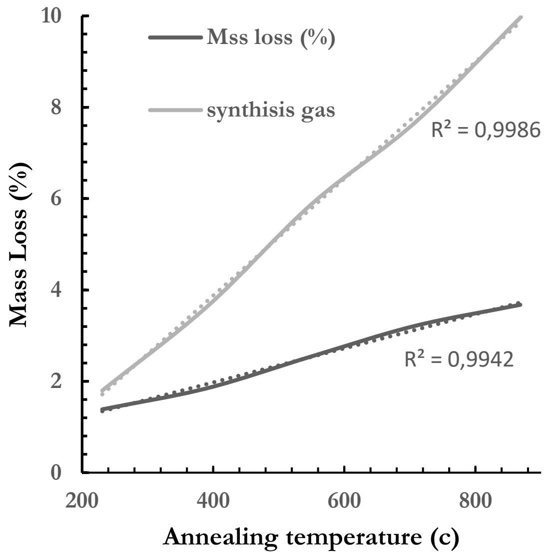

Fig. 4 *Mass Loss (%) vs annealing temperature*

Air Atmosphere: Mass loss increased with temperature, reaching ~10% at 850°C due to oxide formation and insulation combustion.

Synthesis Gas Atmosphere: Minimal mass loss (~1–3%) was observed, confirming reduced oxidation.

This indicates that annealing in a reducing atmosphere effectively suppresses oxidation while facilitating embrittlement.

After annealing, brittle samples were ground in a ball mill for 60 minutes. The resultant powder particle size was <100 μm after sieving.

Air-annealed samples produced larger and less uniform particles, likely due to retained ductility.

Synthesis gas-annealed samples generated finer, more uniform powder, confirming that embrittlement enhances the grindability of copper.

Thus, annealing in synthesis gas at 600–650°C for 45–60 minutes provides optimal conditions for copper powder production.

While the primary focus was on brittleness and grain morphology, future work should evaluate the retained hydrogen content and its impact on purity. As hydrogen can influence mechanical performance, especially in downstream sintering or electrical applications, understanding residual hydrogen levels is critical

4 Conclusion

In this study, it was observed that grain growth becomes more noticeable in the Synthesis Gas Atmosphere, especially at temperatures above 600 °C. This suggests that the reducing environment accelerates grain coarsening, likely due to changes in the diffusion processes.

Trends in Mass Loss:

- Mass loss tends to increase with temperature in both the air and synthesis gas atmospheres.
- In the Air Atmosphere, significant oxidation leads to a mass loss of nearly 10% by 850°C, pointing to considerable material degradation.
- On the other hand, the Synthesis Gas Atmosphere shows only minimal mass loss, indicating that it effectively prevents oxidation even at elevated temperatures.

Atmospheric Effects:

- The data show that when exposed to an oxidizing atmosphere (like air), grain growth is controlled but at the expense of higher material degradation.
- In contrast, a reducing atmosphere (such as synthesis gas) fosters quicker grain growth but keeps mass loss to a minimum, making it ideal for applications that require strong oxidation resistence.

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