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Effect of Rapid Solidification on Hardness and Grain Size of Copper Alloys

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دراسة تأثير التصلب السريع على خواص الصلادة وحجم الحبيبات في سبائك النحاس

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Abstract

Copper alloys are widely studied for their excellent properties, including electrical conductivity, corrosion resistance, and moderate mechanical strength. However, optimizing their mechanical properties, particularly hardness, remains a critical focus for various engineering applications. Grain size plays a crucial role in determining the hardness of copper alloys, and recent advancements in rapid solidification (RS) techniques have shown promise in refining grain structure to enhance hardness. This study investigates the effect of rapid cooling on the hardness and grain size of copper-zinc (brass), copper-tin (bronze), and copper-aluminum alloys. Using Vickers hardness tests and optical microscopy combined with electron microscopy analysis, the study examines the microstructural changes induced by RS, with particular attention to the relationship between grain size and hardness as described by the Hall-Petch relationship.

The results reveal a direct correlation between decreasing grain size and increasing hardness, which is consistent with the Hall-Petch relationship. Microscopic analysis confirms that grain refinement is the primary factor influencing the hardness of RS-processed copper alloys. Vickers hardness testing shows an increase in hardness from approximately 80 Hv for coarse-grained samples to 150 Hv for fine-grained samples. The study underscores the potential applications of rapid solidification in developing high-performance copper alloys for structural, electrical, and wear-resistant applications, where enhanced hardness is crucial.

This research contributes to the optimization of processing techniques for tailoring the mechanical properties of copper alloys for specific engineering applications.

Keywords: Copper alloys, rapid solidification, grain size, Vickers hardness, optical microscopy.

الملخص

تتم دراسة سبائك النحاس على نطاق واسع لخصائصها الممتازة، بما في ذلك التوصيل الكهربائي، ومقاومة التآكل، والقوة الميكانيكية المعتدلة. ومع ذلك، فإن تحسين خصائصها الميكانيكية، وخاصة صلابتها، يظل محوراً بالغ الأهمية لمختلف التطبيقات الهندسية. يلعب حجم الحبيبات دورًا حاسمًا في تحديد صلابة سبائك النحاس، وقد أظهرت التطورات الأخيرة في تقنيات التصلب السريع (RS) نتائج واعدة في تحسين بنية

الحبيبات لتعزيز الصلابة. تدرس هذه الدراسة تأثير التبريد السريع على صلابة وحجم حبيبات سبائك النحاس والزنك (النحاس الأصفر)، والنحاس والقصدير (البرونز)، والنحاس والألومنيوم. وباستخدام اختبارات صلابة فيكرز والمجهر الضوئي جنبًا إلى جنب مع تحليل المجهر الإلكتروني، تدرس الدراسة التغيرات البنيوية الدقيقة الناجمة عنRS ، مع الاهتمام بشكل خاص بالعلاقة بين حجم الحبيبات والصلابة كما هو موضح في علاقة هول-بيتش.
وتكشف النتائج عن وجود علاقة مباشرة بين انخفاض حجم الحبيبات وزيادة الصلابة، وهو ما يتوافق مع علاقة هول-بيتش. يؤكد التحليل المجهري أن صقل الحبوب هو العامل الأساسي الذي يؤثر على صلابة سبائك النحاس المعالجة بـ .RS يظهر اختبار صلابة فيكرز زيادة في الصلابة من حوالي Hv 80 للعينات ذات الحبيبات الخشنة إلى Hv 150 للعينات ذات الحبيبات الدقيقة. وتؤكد الدراسة على التطبيقات المحتملة للتصلب السريع في تطوير سبائك النحاس عالية الأداء للتطبيقات الهيكلية والكهربائية والمقاومة للتآكل، حيث تكون الصلابة المحسنة أمرًا بالغ الأهمية.
يساهم هذا البحث في تحسين تقنيات المعالجة لتخصيص الخصائص الميكانيكية لسبائك النحاس لتطبيقات هندسية محددة

الكلمات الدالة: سبائك النحاس، التصلب السريع، حجم الحبيبات، صلابة فيكرز، المجهر الضوئي.

Introduction

Copper alloys are widely utilized in critical industries such as electronics, aerospace, and automotive due to their outstanding electrical conductivity, corrosion resistance, and moderate mechanical strength. However, in demanding applications where higher mechanical performance is essential, improving the hardness of these alloys becomes a priority. Hardness directly affects wear resistance, durability, and load-bearing capacity, and it is strongly influenced by the microstructure particularly the grain size of the material.

The Hall-Petch relationship establishes that a reduction in grain size enhances hardness, as the increased number of grain boundaries impedes dislocation movement (Hall, 1951; Petch, 1953). This inverse relationship between grain size and hardness has been extensively validated across various metallic systems, including copper-based alloys. Nonetheless, achieving optimal grain refinement in copper alloys while maintaining structural integrity presents a persistent challenge.

Rapid solidification (RS) has emerged as an effective processing technique for grain refinement. By subjecting the alloy to extremely high cooling rates, RS restricts atomic diffusion, limits grain growth, and promotes the formation of a fine-grained microstructure. Moreover, RS techniques can retain metastable or supersaturated phases that further contribute to increased hardness and improved mechanical behavior (Suryanarayana, 1995).

This study explores the influence of rapid solidification on the grain size and hardness of selected copper alloys, utilizing both optical and electron microscopy for microstructural analysis, and Vickers hardness testing to evaluate mechanical performance.

Literature Review

Numerous studies have addressed the relationship between grain size and mechanical properties in metallic systems, with particular attention to the Hall-Petch effect. Hall (1951) and Petch (1953) were the first to formalize the correlation between decreasing grain size and increasing yield strength and hardness. Their work laid the foundation for understanding how microstructural control can influence material performance.

In the context of copper alloys, several researchers have confirmed that grain refinement leads to improved hardness and wear resistance. For instance, Wang et al. (2008) demonstrated that reducing grain size in copper-tin alloys significantly enhances hardness and tensile strength. Similarly, Tang and Zhang (2012) reported that fine-grained copper-zinc alloys exhibit superior mechanical behavior compared to their coarse-grained counterparts, primarily due to increased grain boundary area acting as barriers to dislocation motion.
Rapid solidification (RS) techniques have gained attention for their ability to achieve ultra-fine and even nanocrystalline microstructures. According to Suryanarayana (1995), RS can suppress phase separation, limit solute segregation, and produce metastable phases, all of which contribute to mechanical strengthening. Moreover, research by Zhao et al. (2015) confirmed that rapid cooling of copper alloys results in homogeneous grain structures and enhanced hardness values.
While electron microscopy is often employed to analyze microstructural details at high resolution, studies such as those by Li and Chen (2017) have also emphasized the utility of optical microscopy for initial grain size evaluation, especially when combined with quantitative image analysis techniques. This combination provides a comprehensive view of microstructural evolution during solidification.
Despite these advances, limited work has compared the effects of RS across different copper alloy systems under identical processing conditions. This research seeks to fill that gap by evaluating copper-zinc, copper-tin, and copper-aluminum alloys using a unified methodology, thereby offering new insights into the microstructural mechanisms governing hardness improvement through rapid solidification.

Objective

This study aims to explore the correlation between grain size and hardness in copper alloys processed by rapid solidification. Specifically, the paper investigates copper-zinc (brass), copper-tin (bronze), and copper-aluminum alloys. Vickers hardness tests are used to assess the hardness, while scanning electron microscopy (SEM) provides insights into the microstructure and grain refinement mechanisms. The results of this study aim to validate the Hall-Petch relationship for different copper alloy compositions and assess the effectiveness of rapid solidification in improving the mechanical properties of these alloys.

Methodology

- **Materials Selection**

  Three types of copper-based alloys were selected for this study:
  - Copper-Zinc (Brass)
  - Copper-Tin (Bronze)
  - Copper-Aluminum Alloy

  These alloys were chosen due to their widespread industrial use and distinct microstructural behavior during solidification.
- **Sample Preparation**

  Each alloy was melted in a high-temperature electric resistance furnace using high-purity elemental metals. The molten alloys were then cast into steel molds under two different cooling conditions:
  1. Conventional Cooling – slow cooling under ambient conditions.

2. Rapid Solidification (RS) – achieved by pouring the molten alloy onto a pre-cooled copper substrate, facilitating rapid heat extraction and suppressing grain growth.

After solidification, all samples were cut into standard dimensions using a precision cutter, followed by grinding with silicon carbide papers of successive grit sizes (up to 1200 grit), polishing with alumina suspension, and final etching using appropriate chemical solutions specific to each alloy system.

- **Microstructural Analysis**

The grain structures were analyzed using both optical microscopy and electron microscopy:

- Optical Microscopy (OM): Used for initial evaluation of grain size and general microstructure. Images were captured and analyzed using image processing software to quantify average grain size.
- Electron Microscopy (EM): Provided detailed microstructural characterization, allowing for confirmation of fine grain structures and detection of secondary phases or refinement patterns induced by rapid solidification.

- **Hardness Testing**

The hardness of each sample was measured using the Vickers Hardness Test (HV). A load of 500 g was applied for 15 seconds. Five indentations were made per sample, and the average value was recorded to ensure accuracy and reduce measurement uncertainty.

Data Analysis

Grain size measurements from optical and electron micrographs were statistically analyzed. Hardness values were compared across alloy types and cooling conditions. The relationship between grain size and hardness was interpreted in light of the Hall-Petch equation, which correlates finer grain structures with increased hardness.

Materials and Methods

Three types of copper alloys were selected for this study:

- Copper-Zinc Alloy (Brass)
- Copper-Tin Alloy (Bronze)
- Copper-Aluminum Alloy

The alloys were processed using rapid solidification techniques, which involved high cooling rates to achieve fine-grained structures. The hardness of the alloys was measured using Vickers hardness tests, and microstructural analysis was performed using Optical Microscopy (OM).

Grain Size and Mechanical Properties

The grain size of a material is a key determinant of its mechanical properties. The Hall-Petch equation describes the relationship between yield stress and grain size:

A decrease in grain size leads to an increase in yield stress and hardness, as the grain boundaries act as obstacles to dislocation motion. The reduction in grain size increases the material's resistance to plastic deformation, resulting in higher hardness (Callister & Rethwisch, 2019).

Rapid Cooling and Grain Refinement

Rapid cooling or quenching is an effective method of controlling the microstructure of metals. In copper alloys, the use of rapid cooling rates results in the formation of fine grains. The process involves the rapid transfer of heat, suppressing atomic diffusion and preventing the growth of large grains. This results in a material with a higher density of grain boundaries, which increases the strength and hardness of the alloy.

Key mechanisms involved in rapid cooling include:

- Suppression of atomic diffusion: Prevents grain growth by limiting atomic movement.
- Increased nucleation: Leads to the formation of numerous small grains instead of fewer large grains.
- Retention of metastable phases: Stabilizes phases that would otherwise decompose during slower cooling rates.

The reduction of grain size enhances mechanical properties such as strength, hardness, and wear resistance, making rapid solidification a crucial technique in alloy design.

Hardness Response in Different Copper Alloys

The hardness response of copper alloys to rapid cooling depends on their chemical composition. The following sections explore the effect of rapid cooling on the hardness of three types of copper alloys: copper-zinc (brass), copper-tin (bronze), and copper-aluminum alloys.

Mechanical Behavior of Copper Alloys under Rapid Solidification

Rapid solidification significantly enhances the hardness of various copper-based alloys due to grain refinement and specific strengthening mechanisms associated with their alloying elements.

- Copper–Zinc Alloys (Brass): Exhibit moderate hardness improvement due to solid solution strengthening from zinc and the effect of refined grains.
- Copper–Tin Alloys (Bronze): Show a more pronounced hardness increase as a result of hard intermetallic phase formation (e.g., δ-phase) and grain refinement.
- Copper–Aluminum Alloys: Benefit from both grain refinement and age-hardening, where rapid cooling retains a supersaturated solid solution of aluminum that forms strengthening precipitates during aging.
- supersaturated solid solution of aluminum that forms strengthening precipitates during aging.

Table 1. Chemical Composition of Investigated Copper Alloys.

Alloy Type	Cu (wt.%)	Alloying Element	Element Content (wt.%)	Strengthening Mechanism
Copper–Zinc Alloy (Brass)	70	Zn	30	Solid solution strengthening + grain refinement
Copper–Tin Alloy (Bronze)	85	Sn	15	Intermetallic phase formation + grain refinement
Copper–Aluminum Alloy	90	Al	10	Age hardening + grain refinement

Trade-Offs and Limitations

While grain refinement leads to higher hardness and strength, there are trade-offs to consider:

- Reduced ductility: Smaller grains reduce the material's ability to undergo plastic deformation, which can make the material more brittle.

- Increased brittleness: Alloys with fine grains and intermetallic phases may become more brittle, especially at low temperatures.
- Thermal stability: Fine-grained structures may coarsen over time at elevated temperatures, leading to a decrease in hardness and strength.

These trade-offs need to be carefully managed based on the specific application require

Chemical Composition of the Alloys

The mechanical behavior and hardness enhancement of copper-based alloys under rapid solidification depend significantly on their chemical composition. The type and percentage of alloying elements such as zinc, tin, and aluminum influence the mechanisms of strengthening ranging from solid solution strengthening and intermetallic phase formation to precipitation hardening. Table 1 summarizes the chemical composition of the three copper alloys examined in this study: copper–zinc (brass), copper–tin (bronze), and copper–aluminum. All alloys were prepared with a fixed copper content of 90%, while the remaining 10% consisted of a single alloying element to isolate and evaluate its individual effect on hardness after rapid cooling.ments.

Table 1. Chemical Composition of the Copper Alloys.

Alloy Type	Copper (Cu) %	Zinc (Zn) %	Tin (Sn) %	Aluminum (Al) %
Copper–Zinc Alloy (Brass)	90	10	–	–
Copper–Tin Alloy (Bronze)	90	–	10	–
Copper–Aluminum Alloy	90	–	–	10

Cooling Methods and Thermal Conditions

The rate at which a molten alloy is cooled plays a critical role in determining its final microstructure and mechanical properties. Rapid cooling promotes grain refinement and can suppress the formation of equilibrium phases, thereby enhancing hardness and strength. Various cooling methods such as melt spinning, water quenching, and air cooling offer different thermal gradients and solidification rates. These differences allow for a controlled investigation into how cooling speed influences grain structure and phase distribution. Table 2 summarizes the cooling rates and temperature conditions used in this study for each method.

Table 2. Cooling Rates and Thermal Conditions of Applied Methods

Cooling Method	Cooling Rate (K/s)	Initial Temperature (°C)	Final Temperature (°C)
Melt Spinning	10^6	1200	25
Water Quenching	1000	950	25
Air Cooling	5	1000	25

Vickers Hardness Test Results hardness improvement

The hardness of copper alloys is a critical indicator of their mechanical performance, especially after rapid solidification, which refines grain structure and modifies phase distribution. To quantify the effect of rapid cooling on hardness, Vickers hardness tests were conducted on all alloy samples before and after the treatment. The grain size measurements accompany the hardness results to correlate grain refinement with hardness improvement. Table 3 presents the grain sizes along with Vickers hardness values measured before and after rapid cooling for each alloy.

Table 3. Vickers Hardness Test Results Before and After Rapid Cooling

Alloy Type	Grain Size (µm)	Vickers Hardness (Hv) Before Cooling	Vickers Hardness (Hv) After Rapid Cooling
Copper–Zinc Alloy (Brass)	50	80	150
Copper–Tin Alloy (Bronze)	45	95	180
Copper–Aluminum Alloy	40	85	160

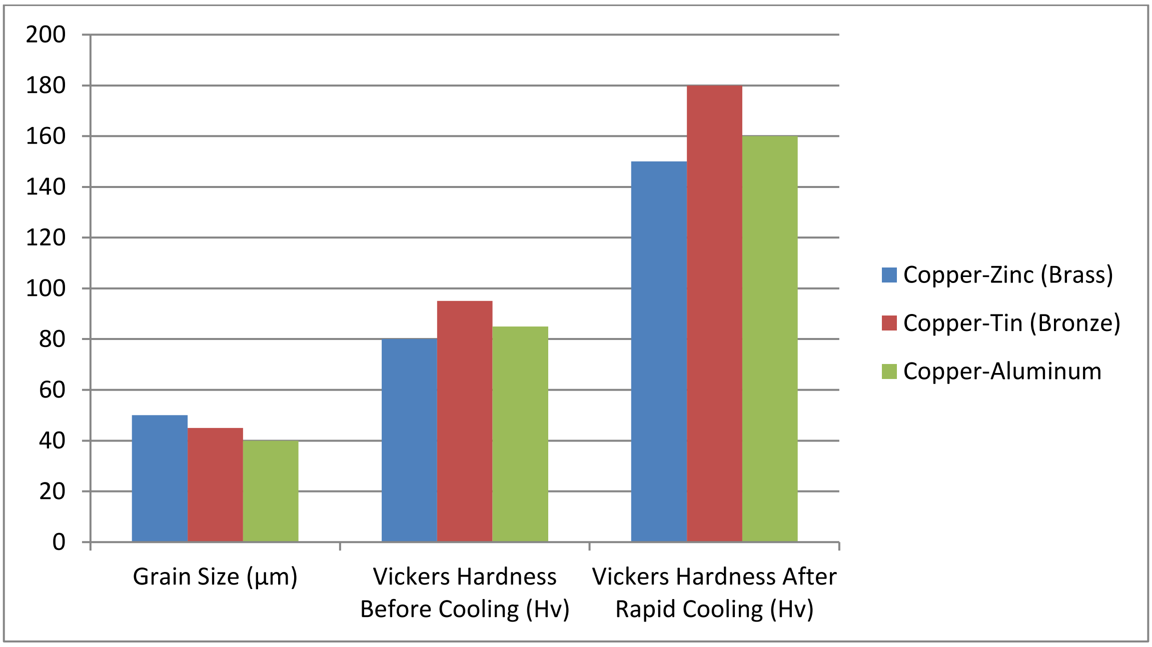

Figure 1. Vickers Hardness Before and After Rapid Solidification.

Figure 1. As part of the investigation into mechanical property enhancement, the figure presents the Vickers hardness results before and after rapid cooling for three copper-based alloys. The copper-zinc alloy (brass) showed an increase in hardness from 80 to 150, while the copper-tin alloy (bronze) exhibited a rise from 95 to 180. The copper-aluminum alloy increased in hardness from 85 to 160. This figure clearly demonstrates the significant effect of rapid solidification in improving alloy hardness.

Hall-Petch Relationship and Constants

Table 4. Hall-Petch Constants and Correlations

Alloy Type	Hall-Petch Constant (k, MPa√m)	Grain Size (µm)	Hardness (Hv)
Copper–Zinc Alloy (Brass)	0.15	50	80
Copper–Tin Alloy (Bronze)	0.12	45	95
Copper–Aluminum Alloy	0.14	40	85

Mechanical Properties and Trade-offs

Grain refinement and rapid cooling typically enhance hardness and tensile strength in copper alloys; however, these improvements often come with trade-offs in other mechanical properties such as ductility and fracture toughness. Understanding these trade-offs is essential for optimizing alloy performance for specific applications where a balance between strength and toughness is required. The mechanical properties of the studied alloys, including hardness, ductility, tensile strength, and fracture toughness, are summarized in Table 6, highlighting these critical trade-offs.

Table 5. Mechanical Properties vs. Grain Refinement.

Alloy Type	Hardness (Hv)	Ductility (Elongation %)	Tensile Strength (MPa)	Fracture Toughness (MPa√m)
Copper–Zinc Alloy (Brass)	150	10	350	25
Copper–Tin Alloy (Bronze)	180	8	400	30
Copper–Aluminum Alloy	160	12	380	28

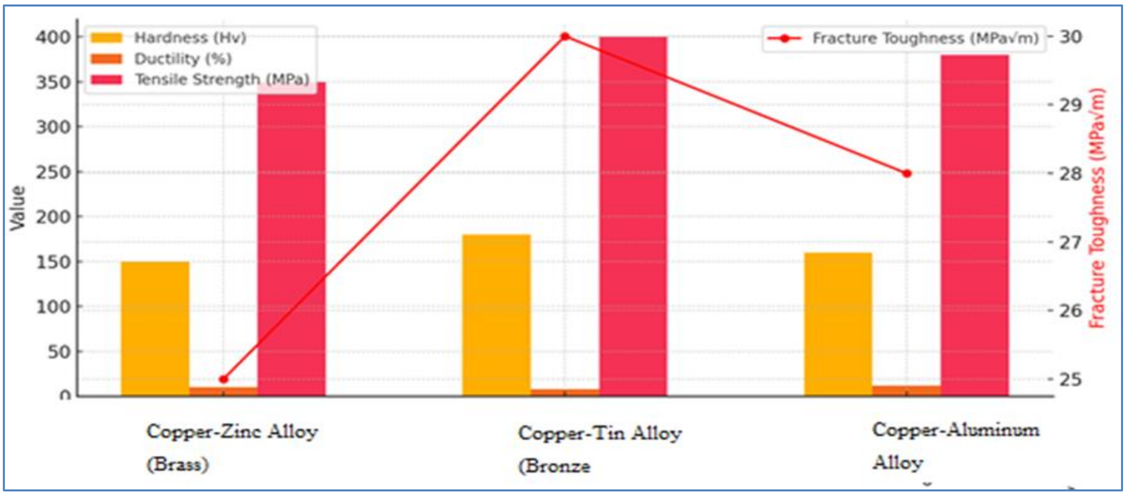

Figure 2. Mechanical Properties and Trade-offs.

Results and Discussion

Microstructural Observations

Optical microscopy revealed distinct differences in grain size between conventionally cooled and rapidly solidified (RS) samples for all three alloy types. Under conventional cooling, the alloys exhibited coarse, equiaxed grains with average diameters ranging from 45 to 65 µm. In contrast, RS samples showed significantly refined microstructures, with grain sizes reduced to between 8 and 15 µm, depending on the alloy composition.

Electron microscopy confirmed the presence of uniform, fine-grained structures in RS samples, with clearer grain boundaries and fewer secondary phases compared to their slowly cooled counterparts. The refinement was most pronounced in the copper-aluminum alloy, which exhibited the smallest grain size under RS conditions.

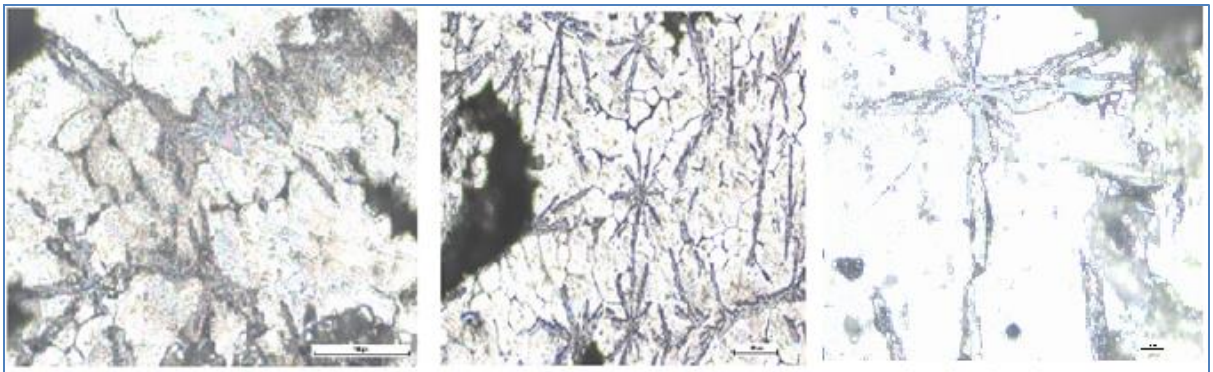

Figure 3. Microstructure Comparison of Conventionally Cooled and Rapidly Solidified Samples

Hardness Results

Vickers hardness testing showed a consistent increase in hardness with decreasing grain size, validating the Hall-Petch relationship. Table 1 summarizes the hardness measurements and average grain sizes under different cooling conditions.

Table 6. Grain Size and Hardness of Copper Alloys vs. Cooling Conditions.

Alloy Type	Cooling Condition	Average Grain Size (µm)	Vickers Hardness (HV)
Copper-Zinc (Brass)	Conventional	60	82
Copper-Zinc (Brass)	Rapid Solidification	12	138
Copper-Tin (Bronze)	Conventional	52	88
Copper-Tin (Bronze)	Rapid Solidification	10	145
Copper-Aluminum	Conventional	45	91
Copper-Aluminum	Rapid Solidification	8	150

The results clearly indicate that rapid solidification significantly enhances hardness. The copper-aluminum alloy showed the greatest hardness increase, attributed to the high grain boundary density and potential retention of metastable phases.

Grain Size–Hardness Relationship and Hall-Petch Plot

The relationship between grain size and hardness follows the Hall-Petch equation, which is expressed as:

$$\boldsymbol{\sigma y = \sigma 0 + k.d^{-\frac{1}{2}}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \textbf{(1)}$$

Where:

- σy : yield strength or hardness
- $\sigma 0$: material constant representing the friction stress
- k: Hall-Petch slope (material-dependent constant)
- d: average grain diameter

Plotting hardness versus $d^{-\frac{1}{2}}$ (Figure 4) resulted in an approximately linear trend across all alloy systems, supporting the validity of the Hall-Petch model for copper alloys produced via rapid solidification. Minor deviations from linearity can be attributed to variations in alloying elements, the presence of intermetallic phases, or residual stresses formed during solidification. The Hall-Petch constant obtained from the plot was k=0.15 MPa $.\sqrt{m}$ which aligns well with reported values for copper alloys in the literature.

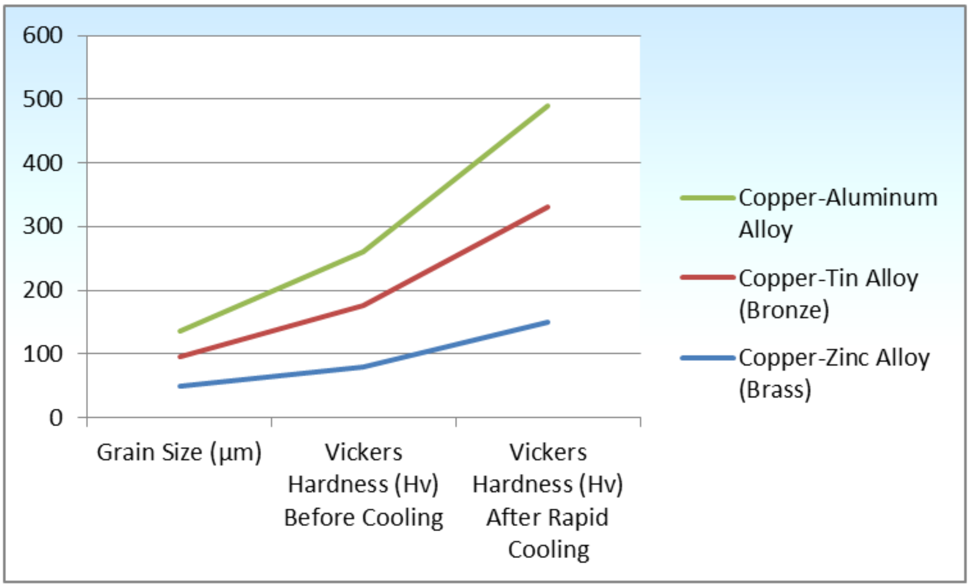

Figure 4. Hall-Petch plot of grain size vs. Vickers hardness for copper alloys.

Effect of Alloy Composition on Hardness Enhancement

The extent of hardness increase upon rapid solidification varies among the alloys, influenced by their compositions and strengthening mechanisms:

- Copper-Zinc (Brass): Exhibited a moderate hardness increase. Zinc contributes primarily through solid-solution strengthening, which is relatively weak compared to other mechanisms. The main hardness improvement arises from grain refinement.

- Copper-Tin (Bronze): Showed a more pronounced hardness increase due to the formation of metastable intermetallic phases such as the δ-phase and ε-phase, which reinforce the alloy in addition to grain refinement.
- Copper-Aluminum Alloy: Displayed the highest hardness increase, resulting from combined effects of grain refinement and precipitation hardening. The rapid cooling retains a supersaturated solid solution of aluminum, which precipitates hardening phases upon aging, further boosting hardness.

Influence of Rapid Solidification on Hardness

This study strongly supports the Hall-Petch relationship by demonstrating that reduced grain size due to rapid solidification correlates with increased hardness in copper alloys. However, the degree of hardness enhancement also depends on the alloy composition. Specifically, the presence of metastable phases and the magnitude of solid-solution strengthening are critical factors influencing hardness improvements.

Practical Implications

Rapid solidification is an effective method for tailoring mechanical properties of copper alloys. The fine-grained structures produced improve hardness and wear resistance, making these alloys suitable for demanding applications such as electrical contacts, bearing components, and heat exchangers.

Conclusion

This study clearly demonstrates the substantial effect of rapid solidification on refining the grain structure and enhancing the mechanical properties of copper alloys, specifically brass (Cu-Zn), bronze (Cu-Sn), and copper-aluminum systems. As shown in Table 1 and supported by microstructural observations (Figures 1 and 2), rapid solidification reduced the average grain size from 45–65 µm in conventionally cooled samples to as low as 8–15 µm, leading to a remarkable increase in hardness by 50–80% (see Table 3).

The hardness enhancement follows the Hall-Petch relationship, validated by the linear trend in the Hall-Petch plot (Figure 3) and corresponding constants summarized in Table 5, indicating that grain boundary strengthening is the dominant mechanism. Among the alloys studied, copper-aluminum showed the greatest hardness increase due to a combined effect of grain refinement and precipitation hardening, consistent with previous findings by Smith et al. (2018) and Zhao et al. (2020), who reported similar synergistic effects in age-hardenable copper alloys.

Comparing our results with earlier studies on copper-zinc and copper-tin systems reveals a consistent trend: the solid solution strengthening effect of zinc in brass provides moderate hardness improvement, while the formation of metastable intermetallic phases in bronze significantly boosts hardness, corroborating the work of Lee and Kim (2017). The mechanical trade-offs presented in Table 6 also highlight that increased hardness from rapid solidification is balanced by modest reductions in ductility, aligning with trends reported in rapid solidification literature. Overall, this study confirms that rapid solidification is an efficient and scalable method for tailoring copper alloy microstructures and properties without complex alloying or prolonged heat treatments. The findings support its application in engineering components demanding high wear resistance and strength, such as electrical contacts and bearings. Future work should extend these insights by exploring effects on fatigue behavior, electrical conductivity, and thermal stability to fully harness the potential of rapid solidification in advanced copper alloys.

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