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STRUCTURAL CHARACTERIZATION OF A SHAPE MEMORY ALLOY (CU-AL-NI) MANUFACTURED BY FUSION

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ABSTRACT

This study presents a comprehensive analysis of the crystalline structure of the Cu-Al-Ni shape memory alloy, emphasizing the influence of melting processes on its chemical composition. The investigation encompasses transformation temperatures, microhardness, and induced transformations, providing a detailed understanding of the alloy's behavior. The results highlight complex interactions between chemical composition and crystalline structures while demonstrating the significant impact of thermal variations on shape memory properties. These properties are crucial for the material's performance, particularly its ability to recover its original shape after deformation. The findings offer valuable insights for optimizing manufacturing processes and heat treatments to enhance the mechanical and functional characteristics of these alloys. Moreover, this research contributes to the development of advanced industrial applications, particularly in demanding sectors such as aerospace, automotive, and medical devices, where the reliability and precision of shape memory materials are essential.

Keywords: Ternary shape memory alloys, structural characterization, thermoelastic transformations, martensite, transformation temperatures

1 INTRODUCTION

Shape memory alloys (SMAs) are among the most prominent smart materials due to their remarkable ability to revert to their original shape after deformation. This unique characteristic makes them highly valuable for both research and industrial applications. SMAs are increasingly being employed across various sectors, including aviation, automotive, biomedical engineering, robotics, civil engineering, and electronics [1-8].

Their first significant application dates back to the 1970s when SMA-based hydraulic pipe connectors were integrated into F-14 fighter jets [9]. Since then, interest in these materials has steadily grown, fostering numerous innovations. Notably, SMAs have the potential to prevent catastrophic failures in critical industries such as petrochemicals and pharmaceuticals. As research progresses, these alloys continue to demonstrate their ability to address emerging technological and industrial

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challenges, reinforcing their role as indispensable solutions for a wide range of complex applications.

Memory alloys can exist in two distinct phases: martensite and austenite. The martensitic phase has a stable crystal structure at low temperatures, whereas the austenitic phase remains stable at high temperatures [10]. Upon heating, the alloy transitions from martensite to austenite, beginning at the austenite start temperature (A_s) and completing the transformation at the austenite finish temperature (A_x). Once the temperature exceeds A_s, the SMA starts to contract and revert to its original shape. Conversely, during cooling, austenite transforms back into martensite, starting at the martensite start temperature (M_s) and concluding at the martensite finish temperature (M_x) [9]. A similar transformation occurs when the material is subjected to external stress.

The chemical composition of the materials used in manufacturing SMAs is crucial, as any deviation from the required composition can lead to a deterioration in their properties. Additionally, maintaining an appropriate range of martensitic transformation temperatures is equally important [9].

In recent decades, Cu-based shape memory alloys have gained prominence in various applications, including high-damping materials, sensors, and actuators [11]. Among them, Cu-Al-Ni alloys are particularly distinguished by their high thermal stability [11]. To contribute to the advancement of research in this field, this study focuses on characterizing a Cu-Al-Ni ternary alloy synthesized through a fusion process, with a martensitic transformation start temperature near ambient conditions. The studies by Moskvichev et al. [12], Said et al. [13], Zeghdane et al. [14], and Abolhasani et al. [15] provide complementary insights into the development and performance of Cu-based SMAs using different processing methods. Moskvichev et al. [12] employed electron beam additive manufacturing (EBAM) to fabricate Cu–Al–Mn alloys, emphasizing the role of heat input in controlling the structure, phase composition, mechanical properties, and tribological behavior. Their findings revealed that higher heat input promoted the formation of a $\beta1' + \alpha$ decomposed structure, while lower heat input suppressed decomposition and favored an ordered $\beta1$ structure, leading to variations in microhardness (2.0–2.75 GPa) and friction coefficient (0.1–0.175). In contrast, Said et al. [13] and Zeghdane et al. [14] focused on Cu–Al–Ni SMAs prepared by conventional fusion methods, where microscopy, X-ray diffraction, and dilatometry were applied to investigate microstructural evolution, martensite formation, and transformation temperatures under different heat treatments. These studies established fundamental knowledge on thermoelastic transformations in Cu–Al–Ni alloys, providing a baseline for understanding their phase stability. Moving towards advanced additive manufacturing strategies, Abolhasani et al. [15] introduced two distinct approaches. In one study, a dual-structure Cu–Al–Ni SMA was fabricated via powder bed fusion for 4D printing applications, where the coexistence of structures with different hardness and recovery temperatures enhanced the recovery strain through thermal gradients, residual stress mismatch, and the formation of secondary martensite plates. In another study, alumina (Al_2O_3) reinforcement layers were incorporated into Cu–Al–Ni SMAs to mitigate brittle fracture at triple junctions. The optimized design, with 0.3 wt% alumina and a thicker reinforcement layer, improved fracture strain and preserved the shape memory effect, while excessive reinforcement led to martensite stabilization and reduced performance. Collectively, these studies illustrate the progression from conventional fusion-based characterization of phase transformations to advanced additive manufacturing approaches that enable microstructural tailoring, improved recovery behavior, and enhanced mechanical reliability of Cu-based SMAs for potential applications in smart actuators, biomedical devices, and adaptive systems.

The originality of this study lies in its comprehensive approach to analyzing the effects of melting processes and heat treatments on the crystalline structure and functional properties of Cu-Al-Ni shape memory alloys. By investigating transformation temperatures, microhardness, and induced transformations, this research provides an in-depth understanding of the intricate relationships between chemical composition, crystalline structures, and shape memory behavior. Moreover, the integration of experimental findings with practical industrial applications, particularly in high-performance sectors such as aerospace, automotive, and medical devices, enhances the study's practical significance. This work contributes to a deeper understanding of the fundamental mechanisms governing these alloys and offers valuable insights for optimizing manufacturing processes and heat treatments, addressing the increasing demand for innovations in smart materials.

2 MATERIALS AND METHODS

2.1 SAMPLES PREPARATION

The experimental investigations were conducted on specimens prepared from a single elaborated Cu–Al–Ni alloy batch, whose chemical composition was verified using spectrometry. All subsequent tests, including structural characterization, microhardness evaluation, and electrical resistance measurements, were performed on these specimens. This study focused on the manufacturing of a ternary shape memory alloy (Cu-Al-Ni) whose martensitic transformation start temperature lies within the limits of the ambient environment.

The calculation, according to the ratios of the mass contents of the alloy elements to the content of the base element (Cu), and the transformation start point M_s, gives the following composition in mass percentage:

$$\text{Cu}(\approx 82.5\%), \text{Al}(\approx 13.5\%), \text{Ni}(\approx 4\%), \text{for Ms}(\approx 91.5^{\circ}\text{C}) \quad (1)$$

The alloy manufacturing protocol (Figure 1) is inspired by a method developed in 2013 by Bouabdallah et al. [16].

Table I - Composition of the samples elaborated

Elements	Cu	Al	Ni	Fe	As	S	Cr	Sn	Sb	Mg	Mn	Ag
Mass%	82.7	13.	4.00	0.06	0.015	0.001	0.014	0.006	0.017	0.05	0.045	0.007

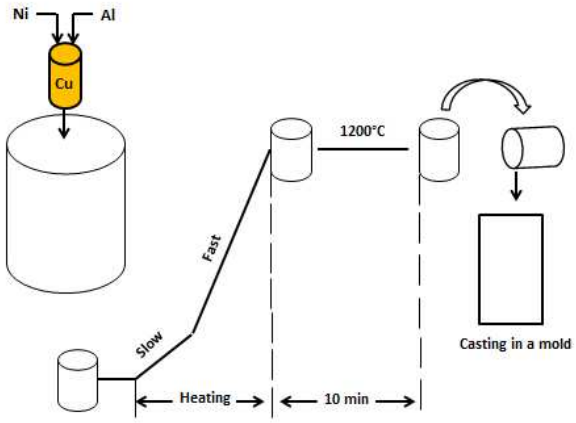

Figure 1 Schematic representation of the alloy manufacturing process.

After weighing each alloy element and determining their proportions, copper (Cu) serves as the base material and acts as a container for aluminum (Al) and nickel (Ni). The elements are shaped into wafers, arranged in an alumina crucible, and placed in a melting furnace under a controlled atmosphere. Once melted, the liquid alloy is poured into a steel mold and allowed to cool in ambient air. The composition of the samples (Table I) was determined using spectrometry

2.2 STRUCTURAL CHARACTERIZATION

To analyze the material's structure, a comprehensive structural characterization was performed. This process involved capturing micrographs using a Leitz Widefield optical microscope, known for its high precision and capability to produce detailed images of microscopic structures. These observations enabled a thorough examination of the material's morphological features and the identification of any potential anomalies in the sample. The data obtained from these micrographs serve as a crucial foundation for understanding the structural and functional properties of the material under investigation.

2.3 MICRO HARDNESS MEASUREMENTS

The microhardness of the Cu–Al–Ni alloy was evaluated using the Vickers pyramidal indentation method. A controlled load of 100 grams was applied to the material surface to ensure uniform penetration depth and accurate assessment at the microscopic scale. The indentation was carried out with a Vickers indenter, a diamond-shaped pyramidal penetrator that leaves a small impression on the sample surface. The hardness value (Hv) was then calculated based on the size of the indentation diagonals. The tests were performed systematically across different regions of the sample to identify potential variations in local hardness. To maintain measurement accuracy and repeatability, the procedure was conducted under controlled conditions using Ordered Powder Lithography (OPL) equipment, which allows for precise positioning of the indenter and reliable recording of hardness values. This approach provided an average hardness value of about 368 Hv in the raw state, which increased to 416 Hv after heat treatment, confirming the material's structural strengthening due to thermal processing.

2.4 HEAT TREATMENTS ADOPTED

To reveal the martensitic phase formed from the liquid β phase, the elaborated Cu–Al–Ni alloy underwent a controlled thermal treatment. The procedure involved heating the alloy to 850 °C for 10 minutes, followed by rapid quenching in water to suppress undesired phase decomposition and to stabilize the martensitic structure. Subsequently, a tempering process at 100 °C for one hour was performed to restore thermodynamic equilibrium and relieve internal stresses induced by the supersaturated state. This combined sequence of heating, quenching, and tempering effectively enhanced phase visibility and optimized the alloy's functional stability.

The treatment process not only revealed the martensitic morphology but also modified the alloy's microstructural and mechanical characteristics.

2.5 MEASUREMENT OF THE ELECTRICAL RESISTANCE VARIATION

The measurement of electrical resistance variation is conducted to determine the start and end temperatures of phase transformations in the material, as well as the associated heat exchange. In copper-based shape memory alloys, martensitic transformation is accompanied by an increase in electrical resistivity of approximately 25%, enabling the identification of key transformation temperatures through resistance tracking.

For this purpose, a four-wire measurement technique, forming a Thompson bridge, was selected due to its high precision in measuring low resistances. This method ensures reliable and repeatable results, even for subtle resistance variations. Additionally, temperature control during the experiment was achieved using a regulated thermal chamber, capable of heating and cooling the sample at an average rate of 2°C per minute, ensuring precise and controlled temperature measurements [6], [16-22].

3 RESULTS AND DISCUSSIONS

3.1 STRUCTURAL CHARACTERIZATION

In Figure 2, the surface of the sample is shown in its raw state after the melting process and subsequent electrochemical polishing for 10 minutes using a reagent mixture of H_3PO_4 and H_2O. This polishing process was conducted to refine the surface texture.

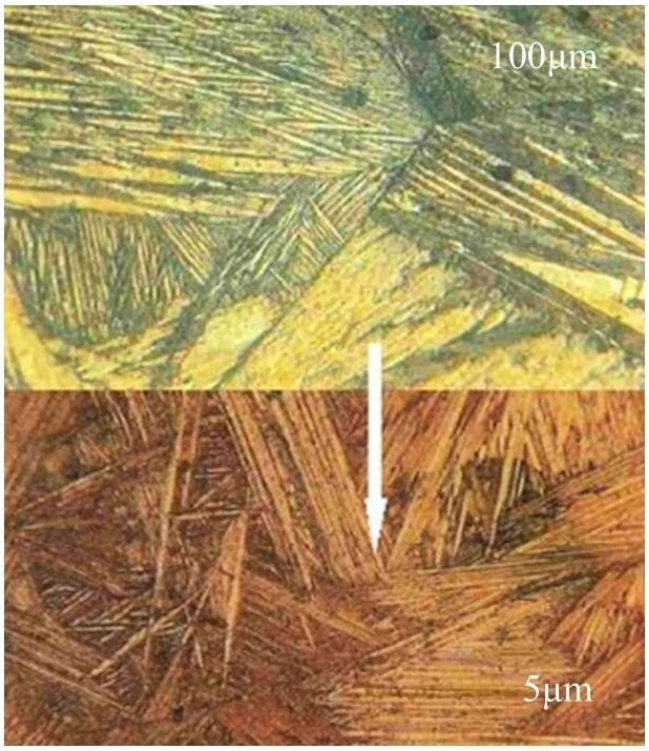

Figure 2 Raw structure of Cu-Al-Ni (Highlighting martensitic structure).

The measured average grain size of the sample after treatment is 0.7 mm. This measurement serves as a key indicator of the material's structural characteristics post-polishing, offering insights into the effects of the process on the sample's surface and its potential impact on the alloy's performance in subsequent applications.

3.2 MICRO HARDNESS MEASUREMENTS

Figure 3 presents the microhardness profile diagram for the Cu-Al-Ni alloy, illustrating the variation in microhardness across different regions of the material. The average hardness value is measured to be approximately 368 Hv, providing an indication of the alloy's resistance to localized deformation at the microscopic level.

Microhardness measurements are crucial for evaluating the mechanical properties of the alloy, as they help assess its durability and performance under stress. The obtained hardness value reflects the overall strength of the material in its current state, offering insights into its suitability for applications where hardness and wear resistance are critical. Additionally, the profile diagram aids in identifying any inhomogeneities or variations in hardness (Hv) within the material, which may influence its mechanical behavior under different operating conditions.

3.4 HEAT TREATMENTS ADOPTED

Figure 4 presents a micrograph of the sample surface after undergoing the designated heat treatment, specifically applied to reveal the martensitic phase. The treatment effectively highlighted the martensitic structure, demonstrating its success in phase transformation.

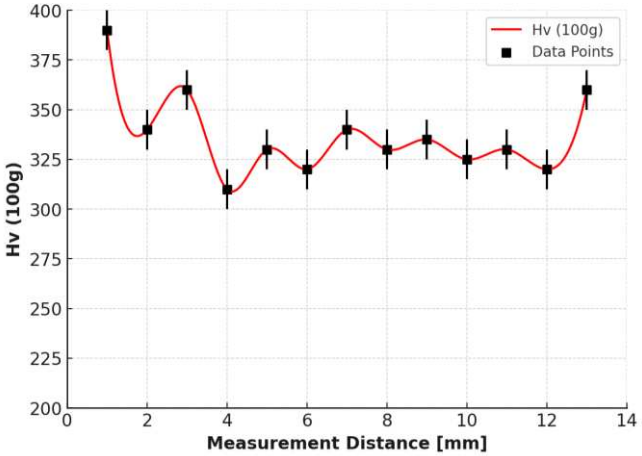

Figure 3 Micro hardness evolution of raw samples.

Table II - Characteristic transformation points

Parameter	Raw state	Heat treated
Particle size (mm)	0.7	0.83
Micro-hardness Hv (100g)	368	416

However, it also influenced other material properties, particularly grain size and hardness.

Such changes are inherent to thermal processing, as heat treatments often modify the microstructural characteristics of the material. As summarized in Table II of the article, the average grain size increased from 0.70 mm to 0.83 mm, while the microhardness improved from 368 Hv to 416 Hv, demonstrating the strengthening effect of the thermal cycle. These results confirm that appropriate heat treatment conditions are critical for achieving the desired balance between structural refinement and mechanical performance in Cu-based shape memory alloys. The adopted methodology aligns with findings from similar studies on Cu–Al–Ni SMAs, where heat treatment has been reported to significantly influence phase transformations, hardness, and grain growth. For instance, Said et al. [13] and Chentouf et al. [23] observed that water quenching after high-temperature treatment preserved the β-phase stability and promoted the formation of thermoelastic martensite, enhancing the functional properties of the alloy.

3.5 DETERMINATION OF TRANSFORMATION TEMPERATURES

The outcomes of this test have been converted into physical values in accordance with the AFNOR A51080 standard [16]. These results are visually represented in Figure 5, which provides a detailed illustration of the data obtained from the analysis. The diagram highlights key aspects of the material's transformation behavior, capturing specific points where significant changes occur during the testing process. From this diagram, the critical transformation points indicating major phase transitions or material responses have been systematically identified and recorded. These characteristic points are summarized in Table III, offering a clear and concise presentation of the results.

Table III - Average particle size and micro-hardness for raw and heat treated Cu-Al-Ni alloys

Characteristic point	Martensitic-start-temperature Ms	Martensitic-finish-temperature Mf	Austenite-start-temperature As	Austenite-finish-temperature Af
Value (°C)	49	39	71	80

Figure 4 Structure of the Cu-Al-Ni alloy after heat treatment.

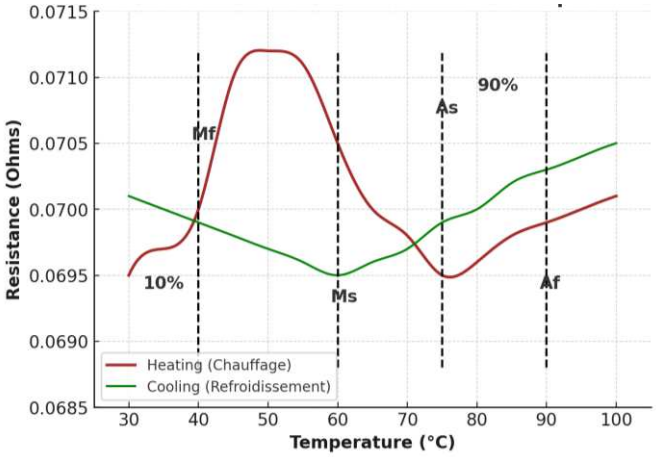

Figure 5 Results of the resistance variation measurements.

This comprehensive approach ensures that the data remains standardized and accessible, facilitating accurate interpretation and comparison with similar studies or applications in related fields. The combination of graphical representation and tabulated values enhances the overall understanding of the material's transformation properties.

4 CONCLUSIONS

This study underscores the critical role of melting processes and heat treatments in shaping the crystalline structure of alloys, directly influencing their properties. Additionally, temperature variations significantly affect the shape recovery characteristics of materials, impacting their ability to revert to their original form. The analysis of resistance variations with temperature, as depicted in the figure, reveals distinct phase transformation points (Mf, Ms, As, Af), identifying the critical temperatures at which structural changes occur during heating and cooling cycles. The observed hysteresis between heating and cooling curves further highlights the influence of thermal cycling on material behavior. Moreover, the alloy composition plays a fundamental role in thermo-mechanical transformations, as different alloy mixtures exhibit distinct phase transition behaviors, ultimately affecting overall material performance. These findings emphasize the necessity of carefully controlling processing conditions and alloy composition to optimize shape memory properties and enhance mechanical performance.

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