# ALLOYING AND AGING TREATMENT EFFECT ON THE SHAPE MEMORY AND DAMPING PROPERTIES OF Cu-13Al-4Ni ALLOYS

WEE YING CI

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Faculty of Engineering
Universiti Teknologi Malaysia

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## DEDICATION

To my parents for raising me to believe that everything was possible and to my husband for making everything possible

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# ABSTRACT

Copper-based shape memory alloys (SMAs) are gaining attention as materials that require a good damping property in high temperature applications because they exhibit high damping properties during martensitic transformation and have an effective energy dissipation. However, copper-based SMAs such as the ternary alloy Cu-Al-Ni are not easily deformed in the lower temperature martensitic phase which can be attributed to brittleness induced by coarse grain size, high degree of order and elastic anisotropy. Hence, this study aimed to improve the properties of Cu-13Al-4Ni SMAs by addition of fourth alloying element and aging treatment that provides a significant effect on the microstructures and properties of the alloys. In this research, Cu-13Al-4Ni-X alloys with the addition of the fourth additional elements (X=titanium, cobalt or boron) were prepared by casting. The as-cast alloys were then homogenized at 900°C and followed by an aging treatment at 150°C, 200°C and 250°C for 24 hours. The transformation temperatures and microstructure characteristics of Cu-Al-Ni-X SMAs were investigated via differential scanning calorimetry (DSC), scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS) and x-ray diffraction (XRD). The hardness of these alloys was determined using Vicker's hardness tester. The shape memory effect was determined by compression test using an Instron universal testing machine. The damping property was measured by dynamic mechanical analysis (DMA) technique. The results revealed that the addition of titanium to the Cu-Al-Ni alloy led to the formation of X-phase which consists of intermetallic compounds of NiTi and $AlTi_2$ that refined the microstructures. On the other hand, addition of cobalt changed the morphologies of the phases with formation of $\gamma_2$ phase which improved the ductility of the quaternary alloy. Addition of boron to Cu-Al-Ni alloy led to the formation of secondary phases which also refined the microstructures. Among the three element additions, Co, B and Ti, it was found that the alloy with 0.7% of Co addition at aging treatment of 150°C for 24 hours showed the best shape memory effect with 100% recovery followed by Cu-13Al-4Ni-0.7Ti at aging temperature of 200°C with 97.5% recovery. However, the Cu-13Al-4Ni-0.7Ti at aging temperature of 200°C has the best damping properties with 0.18 internal friction followed by Cu-13Al-4Ni-0.7Co at aging temperature of 150°C with 0.1 internal friction. The findings showed that both of these alloys are good candidates for damping application.

# ABSTRAK

Aloi ingatan bentuk (SMA) berasaskan kuprum mendapat perhatian sebagai bahan yang memerlukan sifat redaman yang baik dalam aplikasi suhu tinggi kerana ia menunjukkan sifat redaman yang tinggi semasa transformasi martensitik dan berkesan dalam penyebaran tenaga. Walau bagaimanapun, SMA berasaskan kuprum seperti aloi ternari Cu-Al-Ni tidak mudah berubah bentuk pada fasa martensitik suhu rendah yang dapat dikaitkan dengan kerapuhan disebabkan oleh saiz bijian besar, tahap tertib tinggi dan anisotropi elastik. Oleh itu, kajian ini bertujuan untuk meningkatkan sifat-sifat SMA Cu-13Al-Ni dengan penambahan unsur keempat dan rawatan penuaan yang memberi kesan yang ketara terhadap struktur mikro dan sifat-sifat aloi. Dalam penyelidikan ini, aloi Cu-13Al-4Ni-X dengan penambahan unsur tambahan keempat (X = titanium, kobalt atau boron) disediakan melalui proses tuangan. Aloi tuangan kemudian dihomogenkan pada suhu 900°C dan diikuti dengan rawatan penuaan pada suhu 150°C, 200°C dan 250°C selama 24 jam. Suhu transformasi dan ciri mikrostruktur Cu-13Al-4Ni-X SMA disiasat melalui permeteran kalori pengimbasan kebezaan (DSC), mikroskop elektron pengimbas (SEM), spektrometri serakan tenaga (EDS) dan pembelauan sinar-x (XRD). Kekerasan aloi ini ditentukan dengan menggunakan penguji kekerasan Vicker. Keingatan bentuk telah ditentukan dengan ujian mampatan menggunakan mesin Instron. Sifat redaman diukur dengan analisis mekanikal dinamik (DMA). Hasil kajian menunjukkan bahawa kesan penambahan titanium pada aloi Cu-13Al-4Ni menyebabkan pembentukan fasa-X yang terdiri daripada sebatian intermetalik NiTi dan $AlTi_2$ yang menghaluskan saiz struktur mikro. Sebaliknya, penambahan kobalt mengubah morfologi fasa dengan pembentukan fasa $\gamma_2$ yang meningkatkan kemuluran aloi kuaternari. Penambahan boron kepada Cu-13Al-4Ni menyebabkan pembentukan fasa sekunder yang juga menghaluskan saiz struktur mikro. Di antara tiga penambahan unsur, Co, B dan Ti, didapati bahawa aloi dengan penambahan Co 0.7% yang telah menjalani rawatan penuaan pada suhu 150°C selama 24 jam menunjukkan kesan ingatan bentuk terbaik dengan pemulihan 100 % diikuti oleh Cu-13Al-4Ni-0.7Ti pada suhu penuaan 200°C dengan pemulihan 97.5%. Walau bagaimanapun, Cu-13Al-4Ni-0.7Ti pada suhu penuaan 200°C mempunyai sifat redaman terbaik dengan geseran dalaman 0.18 diikuti oleh Cu-13Al-4Ni-0.7Co pada suhu penuaan 150°C dengan geseran dalaman 0.1. Hasil kajian menunjukkan bahawa kedua-dua aloi ini merupakan calon aloi yang sesuai untuk aplikasi redaman.

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# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| Cu-Al-Ni | - | Copper Aluminium Nickel |
| DMA | - | Dynamic Mechanical Analysis |
| DSC | - | Differential scanning calorimetry |
| EDS | - | Energy dispersive spectroscopy |
| ICP-MS | - | Inductive coupled plasma mass spectroscopy |
| IF | - | Internal Friction |
| OM | - | Optical microscope |
| SEM | - | Scanning electron microscopy |
| SMAs | - | Shape memory alloys |
| SME | - | Shape memory effect |
| VPSEM | - | Variable pressure scanning electron micoscopy |
| XRD | - | X-ray diffraction |

**LIST OF SYMBOLS**

| | | |
|---|---|---|
| $\delta$ | - | delta |
| $\lambda$ | - | wavelength |
| R | - | Recovery ratio |
| $\varepsilon_{SME}$ | - | Shape memory effect |
| $M_s$ | - | Martensite start temperature |
| $M_f$ | - | Martensite finish temperature |
| $A_s$ | - | Austenite start temperature |
| $A_f$ | - | Austenite finish temperature |
| d | - | Spacing distance |
| G | - | Gibbs free energy |
| $Q^{-1}$ | - | factor |
| $\Delta W$ | - | Energy absorption |
| W | - | Maximum elastic stored energy |
| $\varphi$ | - | Specific damping capacity |
| $\omega$ | - | Oscillation frequency |
| T | - | temperature |
| $T_0$ | - | Equilibrium temperature |
| $\sigma_0$ | - | oscillation amplitude |
| V | - | Volume fraction |
| °C | - | Centrigrade degree |
| Wt% | - | Weight percentage |
| $\theta$ | - | Bragg's angle |
| B, $\alpha$ | - | Lattice angles |
| Hv | - | Vicker's hardness |
| f | - | Frequency |
| $\sigma$ | - | stress |
| $\varepsilon$ | - | Strain |
| $IF_{Int}$ | - | Intrinsic internal friction |
| $IF_m$ | - | intrinsic damping of martensite |
| $IF_{PT}$ | - | Phase transformation internal friction |

| | | |
|---|---|---|
| $IF_{Tr}$ | - | Transient Internal Friction |
| $IF_a$ | - | Intrinsic damping of austenite |
| GB | - | Grain boundary |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of the Research

Environmental issues related to mechanical, structural and noise vibrations from seismic events such as excavation, construction, mining, and exploration activities have gained considerable attention [1]. Mild discomfort to human and general machinery inefficiencies are some of the detrimental effects of the vibration sources generated from the seismic events that may lead to structural failure, loss of lives, properties and investments [2, 3]. These unwanted vibrations maybe eliminated by selecting materials with good damping properties for engineering structures and systems [4, 5]. Research on these materials has created tremendous interest among scientists and engineers.

Shape memory alloys (SMAs) are materials which capable of regaining their original shape after a large inelastic deformation. This is due to the reversible transformation between two different phases namely martensite and austenite. The martensite phase is highly twinned crystalline structure with high transformation strain and it is stable in low energy level, and the austenite phase is stable in high energy level with low strain. Most SMAs show two unique properties which are known as shape memory effect. The SMAs has ability to recover its original structure upon heating and pseudoelastic effect by restoring the stress-induced deformation upon releasing the applied loads [6].

Copper based SMAs are notable for their easy production and application in addition to their lower price compared to NiTi alloys. Among Cu based SMAs, Cu-Al-Ni alloys have a higher themal stability. Therefore, Cu-Al-Ni alloys are being developed for high temperature applications due to their potential to be used as sensors and actuators at temperature around 200 °C. Also, it has emerged as a potential material for high damping material. Composition of aluminium and content of nickel is strongly affects the shape memory properties of Cu-Al-Ni SMAs. The shape memory properties of the alloys can be accomplished when the Al content is about 11wt.% - 13wt.%and content of Ni is about 3-5wt.% [7]. Some other factors that influence the shape memory properties are martensite structure, composition and transformation temperatues [7, 8]. Various kinds of martensite such as $\beta_1'$ (18R), $\gamma_1'$ (2H) or $\alpha_1'$(6R) can be formed by transformation of $\beta_1$ (DO3) parent phase in Cu-Al-Ni SMAs, in which formation of these martensite phases depends on applied stress and temperature. 18R ($\beta_1'$) and 2H ($\gamma_1'$) are the two martensite structures that normally formed in Cu-Al-Ni SMAs, in which the content of Al and Ni as well as heat treatment affect their formation [8, 9]. Martensite formed on cooling is $\beta_1'$ in a low Al alloy and is $\gamma_1'$ in a high Al alloy, In alloys with composition with compositions near the phase boundary between these two martensites, both can coexist [10]. $\beta_1'$ and $\gamma_1'$ are two martensite structures that typically obtained in Cu-Al-Ni SMAs, in which their formation are strongly affected by content of Al and Ni and the requied characteristic of the application determines the content of combination of both Al and Ni.

Nevertheless, the single crystal in structure of the Cu-Al-Ni SMA is ductile however polycrystalline Cu-Al-Ni SMA is generally brittle and low recovery strain which has limitation in practical application [11, 12]. In fact, existence of β phase in Cu-Al-Ni alloy results in intergranular fracture as a result of high elastic anisotropy and large size grains. To solve this issue, several approaches such as grain refining by addition of alloying elements, precipitation hardening or heat treatment can be utilized to enhance the mechanical properties and improving its ductility as well as workability [13-16].

Addition of alloying elements such as Nb, V, B, Ti and Mn or various concentrations of Al and Ni to Cu based SMAs is one of the approaches to increase the ductility and refining these alloys [17-20]. Once the grain size is refined, the mode of the fracture alters from intergranular to transgranular with a ductile fracture mode during impact test [20]. Ti has a significant effect in decreasing grain size in Cu-Al-Ni ternary alloy [13, 19]. The grain refinement due to the Ti addition is attributed to the development of finely dispersed Ti-rich precipitates of X-phase, which hinder the grain growth [21]. It has been proved that the grain refinement enhances the mechanical properties.

Martensitic phase transformation from austenite to martensite is crucial to achieve the superelasticity and shape memory effects. Therefore, the shape memory characteristics such as thermal hysteresis, transformation temperatures and recoverable strain depend on aging effects for both phases [7]. Based on the Canbay et.al [22] findings, during the different annealing treatment of Cu-Al-Ni SMAs, $\beta_1$' martensite formed as the main phase, in which based on the applied condition, various crystalline size of this phase is formed. The relative steadiness of the both martensite and austenite in SMAs is a function of heat treatments, as the Cu based SMAs are sensitive to heat treatments [22, 23].

Damping is the transformation of mechanical energy of a vibrating structure into thermal energy. Its mechanism is complex with the combination of structural and material damping. Material damping depend on many factors which include type of materials, stress amplitude, internal forces, quality of surfaces and temperature. There are many engineering materials that have good damping property and among them are shape memory alloys (SMAs) [24]. SMAs are well known alloys which have pseudoelastic and shape memory properties but they are also known to have excellent damping property which makes them desirable for the vibration control devices design [25]. High damping capacity of SMAs in austenitic and martensitic states is due to stress induced martensitic transformation and stress-induced martensite variant orientation respectively [26]. SMAs with high damping capacity have many applications in different industries.

One of the important functional properties of SMAs is desired damping capacity of these alloys. Its mechanism is complex with the combination of structural and material damping. It is because a large fraction of mechanical energy is dissipated in the internal interfaces between the phases during their formation and motion, which is due to shape memory effect and superlasticity deformation of SMAs [27, 28]. Martensite variant interfaces and twin boundaries as hysteresis movement of interfaces leads to high damping capability of the thermoelastic martensite phase. Kustov et. al [29] establish a high damping capacity in Cu-Al-Ni alloys which is because of the dislocations and their relations with other lattice effects. Hence, Cu-Al-Ni alloys is possible to be used as shape memory energy absorbers which can minimize structural damage during earthquake.

Numerous studies reported the influence of aging on the damping properties of Cu based SMAs [30-33]. Cu-Al-Ni alloys have tendency to ageing which leads to formation of precipitate and consecutive martensitic transitions from $\beta_1'$ to $\gamma_1'$. Hereafter, their damping properties are also likely to alter significantly with aging. Suresh and Ramamurty [30] examined the influence of aging on the damping capacity of Cu-Al-Ni and found decreasing in the internal friction (IF) value of this alloy with aging treatment as a result of the precipitation formation ($\gamma_2$) at certain aging temperature. Shivaramu [32] reported that the damping limit of the Cu-Al-Be-Mn SMAs decreased with increasing the aging temperature and time due to the formation of precipitates which can acts as barrier for the movement of the interfaces. Hence, it is shown in numerous studies that damping capacity is strongly depends on the influence of addition elements and heat treatment process. Therefore, the aims of the present study are to elucidate the influence of various addition elements and heat treatment on the microstructure and properties such as hardness, shape memory behaviour and damping characteristics of Cu-Al-Ni-X alloys.

## 1.2 Problem Statement of the Research

Shape memory alloys have been drawing attention in current times for the vibration control systems design, seismic resistant design and retrofit of structure [4, 24, 34] due to their high energy absorption and dissipation capacities that results in high damping capacity. The well-known and established SMAs, Ni-Ti alloys have high superelasticity, large recoverable strain and high fatigue life which satisfy actual application requirements [35, 36]. Nevertheless, poor cold workability and high cost of material is the main hindrance against utilizing Ni-Ti alloys for such applications [37, 38]. In addition, the austenite-finish temperature ($A_f$) of Ni-Ti alloy is around

0°C. It is challenging to obtain 20°C by adjusting the composition and heat treatments. Hence, their outdoor engineering application is restricted by relatively high range of working temperature in practical limits. Alternatively, Cu-Al-Ni SMAs are considered as potential materials for damping application due to their martensitic transformation temperature are adjustable to above room temperature, low production cost, easy manufacturing, excellent shape memory function and high damping capacity. However, high brittleness and unfavourable mechanical properties due to coarse grains limit their further development and applications in certain fields. The undesirable properties may be improved by adding alloying elements followed by aging treatments. Therefore, addition of fourth element in the Cu-Al-Ni SMA together with aging treatment became the focal point of this research in order to achieve the optimum performance to the shape memory recovery and damping capacity.

## 1.3 Objectives of the Research

The main objective of the research is:

To modify the microstructure and shape memory property of Cu-Al-Ni shape memory alloys by addition of alloying elements and heat treatment

The specific objectives of the research are:

1. To investigate the effect of various alloying elements on the microstructure, mechanical properties and shape memory behaviour of Cu-Al-Ni-X alloys.
2. To determine the effect of aging treatment on the microstructure, mechanical properties, and shape memory behaviour of Cu-Al-Ni-X alloys.
3. To correlate the effect of alloying elements and aging treatment on the damping properties of Cu-Al-Ni-X alloys.

## 1.4 Scopes of the Research

Based on the objectives of the study, the scopes of the research are:

1. Production of Cu-Al-Ni shape memory alloys with and without addition of alloying elements (Ti, Co, and B) using casting process.
2. Performed the aging treatment process on the base Cu-Al-Ni SMAs with addition of different alloying elements.
3. Conducted the material characterization to investigate the properties using various testing equipments such as inductively coupled plasma mass spectrometry (ICP-MS), differential scanning calorimetry (DSC), optical microscopy (OM), variable pressure scanning electron microscopy (VPSEM) and x-ray diffraction (XRD).
4. Performed the compression test using universal testing machine to investigate the shape memory effect and compression properties.
5. Determination of the damping characteristic using dynamic mechanical analysis (DMA) with cooling rate = 3°C/min, frequency = 1 Hz, and applied strain = $5.0 \times 10^{-5}$.

## 1.5 Significance of the Research

Copper-based shape memory alloys specifically the ternary Cu-Al-Ni have gained attention among scientists and engineers seeking for alloys with superior properties. Many attempted to improve the properties of Cu-Al-Ni by modifying the alloy composition with addition of various elements or by heat treatment processes but with no major success to be used for industrial applications. The primary purpose of this research is to enhance the properties of Cu-Al-Ni by adding fourth alloying element, namely, titanium, cobalt and boron followed by aging treatment with specific application that require good damping property. Hardly any literature or previous research have reported on the role of these three alloying elements and aging treatment on the damping property. The findings of this study will provide better understanding on the relationship between microstructural modification and the enhancement of mechanical properties of Cu-Al-Ni-X alloys especially on the damping property for seismic resistant devices and structures.

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## LIST OF PUBLICATIONS

**Indexed Journal**

1. **Y. C. Wee**, T. A. Abubakar, E. Hamzah, Safaa N. Saud, (2015), Phase Transformation and Microstructure Behaviour of Cu-Al-Ni Shape Memory Alloys Incorporated with Co Addition, *Jurnal Teknologi*,74(10), 53-56. (**Indexed by SCOPUS**)

2. **Y.C. Wee**, T. A. Abubakar, E. Hamzah, Safaa N. Saud, (2017), Study of X-phase formation on Cu-Al-Ni shape memory alloys with Ti addition*, Journal of Mechanical Engineering and Sciences,* 11(2), 2770-2779. https://doi.org/10.15282/jmes.11.2.2017.17.0251. (**Indexed by SCOPUS**)

3. Sukiman, N. A., H. Ghandvar, T. A. Abubakar, **Y.C. Wee**, (2019), Microstructure Characterization and Tensile Properties of Al-15%Mg2Si-xYSZ Hybrid Composite, *Malaysian Journal of Microscopy*. (**Indexed by SCOPUS**)

**Articles in conference proceedings**

*4.* **Y. C. Wee**, H. Ghandvar, T. A. Abubakar, E. Hamzah (2019), Influence of Cobalt Addition on Microstructure and Damping Properties of Cu-Al-Ni Shape Memory Alloys, *3rd International Conference on Advanced Materials Characterization Techniques (AMCT 2019)*. Material Science Forum.www.scientific.nte/MSF.101034. (**Indexed by SCOPUS**)

5. H. Ghandvar, T.A. Abubakar, N. A. Sukiman , **Y.C.Wee** (2019), Role of $ZrO_2$ Addition on Microstructure and Tensile Characteristics of Commercial Al-20Mg2Si-2Cu Metal Matrix Composite, $4^{th}$ International Conference on the Science and Engineering of Materials 2019 (ICoSEM 2019) (accepted)