INFLUENCE OF ALLOYING ELEMENTS AND AGING TREATMENT ON THE PHASE TRANSFORMATION AND SHAPE MEMORY BEHAVIOR OF Cu-Al-Ni SHAPE MEMORY ALLOYS

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To:

My beloved family

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ABSTRACT

Nickel-Titanium (Ni-Ti) shape memory alloys (SMAs) have been used in many engineering and medical applications. However, their use is limited, due to their low transformation temperatures, difficulties in processing and high cost of the raw materials. As an alternative material to Ni-Ti alloys, copper-based alloys are successfully being used. Among copper-based SMAs, Cu-Al-Ni alloys are used in a wide range of applications, particularly if high temperatures are required. However, Cu-Al-Ni SMAs also have limitations such as very low ductility and low shape recovery strain. Therefore, this research aims to enhance the ductility and shape memory effect of Cu-Ni-Al by alloying additions and aging heat treatment. The base metal, Cu-Al-Ni, was cast without and with different amounts of the fourth alloying elements, namely, titanium (Ti), manganese (Mn) and cobalt (Co). The modified and unmodified alloys were homogenized and aged at 373 K, 423 K and 523 K for 24 and 48 hours. Phase transformation and microstructural changes were characterized using techniques such as optical microscopy, field emission scanning electron microscopy (FESEM), energy dispersive spectrometry (EDS), differential scanning calorimetry (DSC), x-ray diffractometry (XRD) and transmission electron microscopy (TEM). The tensile properties and hardness were determined using a universal Instron tensile machine and Vicker's hardness test machine, respectively. The shape memory test was performed using a specially designed tensile machine equipped with a heating tape. The results revealed that the alloying elements and aging treatment were found to control the phase morphology, orientations and grain size along with the formation of precipitates, thereby improving the shape memory characteristics, ductility and hardness. The volume fraction, size and distribution of the precipitates are mainly dependent on the type and amount of alloying element as well as the condition of aging treatment. The Cu-Al-Ni with the addition of 0.76 wt. % Ti and age treated showed complete recovery after the shape memory test. This may be attributed to the high volume of X-phase precipitates and grain refinement that led to the restricted mobility of martensite variant interfaces and dislocations. It was found that the alloy with 1.14 wt. % of Co gave the best overall improvement in terms of the transformation temperatures, ductility and shape memory recovery. These improvements were mainly due to the exceptionally high content of the gamma-2 (γ_2) phase in the microstructures of the modified alloy. Furthermore, the ductility of the Cu-Al-Ni SMAs increased from 1.65 to 7.0 % when 1.14 wt. % Co was added and the alloy undergone aging treatment where the fracture surfaces showed more ductile features and less brittle cleavages. It was also found that Cu-Al-Ni SMAs with 1.14 wt. % Co obtained full shape recovery after being aged at 523 K for 48 hours.

ABSTRAK

Aloi ingatan bentuk (SMA) Nikel-Titanium (Ni-Ti) telah digunakan dalam banyak aplikasi kejuruteraan dan perubatan. Walau bagaimanapun, kegunaan aloi tersebut adalah terhad disebabkan suhu penjelmaan rendah, kesukaran dalam pemprosesan dan kos bahan mentah yang tinggi. Sebagai bahan alternatif kepada aloi Ni-Ti, aloi berasaskan kuprum telah berjaya digunakan. Aloi Cu-Al-Ni adalah antara SMA berasaskan kuprum digunakan dalam pelbagai aplikasi, terutamanya jika suhu yang tinggi diperlukan. Walau bagaimanapun Cu-Al-Ni SMA juga mempunyai kekurangan seperti kemuluran yang sangat rendah dan terikan pemulihan yang rendah. Oleh itu, kajian ini bertujuan untuk meningkatkan kemuluran dan kesan ingatan bentuk Cu-Ni-Al melalui tambahan unsur mengaloi dan rawatan haba penuaan. Logam asas Cu-Al-Ni dituang tanpa dan dengan jumlah berbeza unsur mengaloi keempat, iaitu titanium (Ti), mangan (Mn) dan cobalt (Co). Aloi diubahsuai dan tidak diubah suai telah dihomogenkan dan dilakukan rawatan penuaan pada suhu 373 K, 423 K, dan 523 K selama 24 dan 48 jam. Penjelmaan fasa dan perubahan mikrostruktur telah dicirikan dengan menggunakan teknik seperti kemikroskopan optik, kemikroskopan elektron imbasan pancaran medan (FESEM), spektrometri serakan tenaga (EDS), permeteran kalori pengimbasan kebezaan (DSC), pembelauan sinar-X (XRD) dan kemikroskopan elektron penghantaran (TEM). Sifat tegangan dan kekerasan ditentukan masing-masing dengan mesin Instron tegangan semesta dan mesin ujian kekerasan Vicker. Ujian ingatan bentuk telah dilakukan dengan menggunakan mesin tegangan direka khas yang dilengkapi dengan pita pemanasan. Keputusan kajian mendedahkan bahawa penambahan unsur mengaloi dan rawatan penuaan didapati mengawal morfologi fasa, orientasi dan saiz bijian bersama dengan pembentukan mendakan, oleh itu ciri ingatan bentuk, kemuluran dan kekerasan didapati bertambah baik. Pecahan isipadu, saiz, dan taburan mendakan terutamanya bergantung kepada jenis dan jumlah unsur mengaloi serta keadaan rawatan penuaan. Cu-Al-Ni dengan penambahan 0.76 % berat Ti dan telah melalui rawatan penuaan menunjukkan pemulihan lengkap selepas ujian ingatan bentuk. Ini boleh dikaitkan dengan jumlah yang tinggi mendakan fasa-X dan penghalusan bijian yang membawa kepada mobiliti terhad antara muka varian martensit dan kehelan. Kajian ini mendapati bahawa aloi dengan 1.14 % berat Co memberi peningkatan keseluruhan yang terbaik dari segi suhu penjelmaan, kemuluran dan pemulihan ingatan bentuk. Peningkatan ini terutamanya disebabkan oleh kehadiran yang sangat tinggi daripada fasa gamma-2 (γ_2) dalam mikrostruktur aloi yang diubah suai. Kemuluran Cu-Al-Ni SMA meningkat daripada 1.65% kepada 7.0 % apabila 1.14 % berat Co unsur mengaloi ditambah dan aloi menjalani rawatan penuaan di mana permukaan patah menunjukkan lebih ciri mulur dan kurang belahan rapuh. Hasil kajian juga menunjukkan bahawa Cu-Al-Ni SMA dengan 1.14 % berat Co mencapai pemulihan bentuk penuh selepas melalui rawatan penuaan pada suhu 523 K selama 48 jam.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Cu-Al-Ni	-	Copper Aluminum Nickel
DSC	-	Differential scanning calorimetry
EDS	-	Energy dispersive spectroscopy
FE-SEM	-	Field emission scanning electron microscopy
HRTEM	-	High resolution transmission electron microscopy
ICP-MS	-	Inductive couple plasma- mass spectroscopy
OM	-	Optical microscope
SADP	-	Selected area diffraction pattern
SEM	-	Scanning electron microscopy
SMAs	-	Shape memory alloys
SME	-	Shape memory effect
TEM	-	Transmission electron microscopy
XRD	-	X-ray diffraction

LIST OF SYMBOLS

(hkl)	-	Miller indices
$\Delta H^{M\rightarrow\gamma}$	-	Enthalpy of Martensite → Austenite
$\Delta H^{\gamma\rightarrow M}$	-	Enthalpy of Austenite → Martensite
$\Delta S^{M\rightarrow\gamma}$	-	Entropy of Martensite → Austenite
$\Delta S^{\gamma\rightarrow M}$	-	Entropy of Austenite → Martensite
ϵ^{f}	-	Fracture strain
ε_{p}	-	Strain after heating above Af
ε_{r}	-	Residual strain
ε_{SME}	-	Strain recovery ratio by shape memory effect
σ^{f}	-	Fracture stress
a, b, and c	-	Lattice parameters
a.u	-	arbitrary unit
A_f	-	Austenite finish temperature
A_s	-	Austenite start temperature
B	-	Full width at half maximum
d	-	Spacing distance
G	-	Gibbs free energy
Hv	-	Vicker’s hardness
K/min	-	Kelvin per minute
M_f	-	Martensite finish temperature
M_s	-	Martensite start temperature
^{o}C	-	Centigrade degree
T	-	Temperature
T_o	-	Equilibrium temperature

Wt.%	-	Weight percentage
α, β	-	Lattice angles
θ	-	Bragg's angle
λ	-	Wavelength
G.B	-	Grain boundary

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Research

Shape memory alloys (SMAs) and their associated thermoelastic martensitic transformation have been the subject of extensive studies for many years, since they have many technological applications utilizing the shape memory effect [1, 2]. Typical for shape memory material is a unique, unconventional correlation of temperature, stress and strain that crystallographic reversible thermo-elastic martensitic transformation forms its basis. Since technological applications impose reliability requirements on commercial SMAs, the MT (martensitic transformation) in particular need to be highly reproducible [3]. As a result, controlling the martensite type and the MT temperatures becomes a basic point for determining the SMA's thermo-mechanical and hysteresis properties. Since extreme sensitivity is shown towards concentration changes by MT temperatures, controlling quaternary or ternary alloys' concentration with a lot of precision is difficult as the composition of the alloy is very vital in the determination of the MT temperatures. Parameters such as structure ordering and grain size are depended upon by phase transformation temperatures but their importance can not be compared to that of the alloy composition. It is also worthy to note that the martensite type which is obtained is dependent of the intermetallic alloy's degree of order [4] that also is independent of the concentration of the alloy [5].

When compared to the many SMAs which are available, the most commercially attractive alloys which can be employed in applications which are practical are the Cu-based SMAs. That is because they exhibit reasonable shape memory effect, offer the possibility of being used at high temperatures, have better thermal stability and are not expensive. It should also be noted that the Cu-Al-Ni alloys' applications have restrictions since they are associated with brittle inter-granular cracking and poor workability and thus used where very small shape changes are required [6]. Their grain size which is large (1-3 mm), high elastic anisotropy ratio (~14) and the high degree of order which they are associated with is responsible for the material failure through inter-granular fracture which they exhibit [7-9]. The difficulty of stress concentration is enhanced by these factors at the grain boundaries and as a result promoting fracture. Hence, poor mechanical properties are exhibited by these alloys (fracture strain 0.5% and fracture stress 150 MPa). It has been established by Husain [10] and Sugimoto et al [11] that within Cu-Al-Ni shape memory alloys with a basis on β when subjected to stress, the existent elastic anisotropy within the allows is responsible the polycrystalline samples' stress concentration at the grain boundaries. High stress concentration can result due to the large grain size. The martensitic strain's orientation dependence also determines the elastic anisotropy. The alloys' stress relaxation properties can also be reduced by the spinodal transformation and the existent ordered phases [12, 13].

Research work pertaining to Cu-Al-Ni SMAs is of considerable significance since these alloys possess higher transformation temperatures [14] and higher damping capacity [15, 16], especially at high temperatures, compared with even Nitinol. That makes them to be well suited when being employed in engineering applications like actuators. They fit well in actuators that operate within high temperatures, bridges' damping elements, buildings, oil well applications and other structures. It should be remembered that when Cu-Al-Ni alloys are subjected to very high temperature service conditions, they show some susceptibility to post-quench aging and as a result lead to a change in their mechanical properties, martensitic phases and transformation temperatures with the time of operation [17, 18].

The material's deformation within the martensitic state (below M_f) is influenced by the shape memory effect. A number of effects can take place over the deformation which may lead the macroscopic shape change within Cu-Al-Ni SMAs. Usually, self-accommodating plate variants are contained within the thermoelastic martensitic structure. The variants rearrange and coalesce through the intervenient boundaries' movement when the deformation takes place. Development of mechanical twins may take place during this process. As a result, in response to the stress which is applied, formation of the most favorably oriented martensite variants occur. Additionally, when responding to the deformation, martensite → martensite transformations which involve changes in the stacking sequence form: taking place of austenite → martensite transformations occurs[19, 20]. There have been wide studies regarding ro the Cu-Al-Ni SMAs' shape memory effect within the bending test [20-22] though not much has been done when studies these properties under the tensile test [23, 24].

A number of attempts have been made in improving mechanical properties. Ductility is the property within Cu–Al–Ni SMAs that has been put into consideration by using different techniques to do grain refining such as adding small amounts of quaternary elements, such as Zr, V and Ti and including aging treatment [22, 25-28]. Though properties like ductility and strength were improved after that, some mechanical properties still remained unsatisfactory to be used for some targeted commercial applications more so when a full shape recovery was necessary. A number of studies have been done on different aging aspects of the alloys and the influence they have on their shape memory properties [17]. The martensite stabilization phenomenon was given special attention since it is normally associated the reverse transformation temperatures (A_s and A_f)'s increase once deformation has taken place. Though, not many studies were done on the aged alloys' thermo-mechanical response [23, 29, 30]. Thus, this research aims to study the effect of alloying elements and aging treatments on the phase transformation behavior and properties of Cu-Al-Ni SMAs.

1.2 Problem Statement

Since Cu-Al-Ni shape memory alloy has many advantages, which has attracted more attention from the scientists and researchers, their applications are limited due to the low recovery strain and high brittleness. Their damaging properties may be improved by adding specific alloying elements followed by aging treatments. An investigation into the structure and property relationship is necessary to ensure the correct compositions of Cu-Al-Ni SMAs, which gives the optimum performance that can achieve 100 % of the shape recovery.

1.3 Purpose of the Research

The purpose of this research is to find ways to improve the poor properties of Cu-Al-Ni SMA in terms of its low recovery strain (40-50 %) and high brittleness (1-1.2% fracture strain). The research induces understanding the mechanisms and transformation behavior of the Cu-Al-Ni SMAs and identifies the influence of alloying elements on the structure and properties of the alloys. The output of this research is expected to improve the mechanical properties and reduce the limitation usage for many industrial applications, e.g., actuators and sensors that will be helpful to prevent the destruction of such structures as buildings and bridges. The effect of various alloying materials such as Mn, Co, and Ti with different additions amounts along with applying different aging conditions of 373 K, 423 K, and 523 K for 24 and 48 hr on the phase transformation behavior of Cu-Al-Ni SMAs are investigated using various material characterization techniques. This study is expected to provide the phase transformation characteristics of each alloy in accordance with their microstructural observations, transformation temperatures and mechanical properties such as tensile, shape memory effect, and hardness.

1.4 Objectives of the Research

The objectives of the research are as follows:

1. To determine the effect of different amounts of alloying elements (Ti, Mn, and Co) on the structure and properties of the Cu-Al-Ni shape memory alloys.
2. To investigate the effects of aging temperature and aging time on the structure and properties of Cu-Al-Ni shape memory alloys.
3. To establish the correlation between the phase transformation characteristics of Cu-Al-Ni SMA, with respect to the concentration of alloying elements and aging treatment.

1.5 Scopes of the Research

The scope of the research is as follows:

1. Preparation of the samples by casting commercially pure raw metals (Cu 99.999 %, Al 99.999, and Ni 99.995 %) with and without alloying elements e.g., Ti 99.99 %, Mn 99.99%, and Co 99.95% by using an induction furnace.
2. Analysis of the as-cast samples by using inductively coupled plasma mass spectrometry (ICP-MS) for composition analysis, differential scanning calorimetry (DSC) for phase transformation temperature, and Vicker's test for hardness measurement.
3. Aging treatment of the as-cast samples under different condition of temperature and time.
4. Microstructural and phase analysis of the as-cast and aged samples using field emission scanning electron microscopy (FESEM), X-ray diffraction

(XRD), and energy dispersive spectroscopy (EDS).

5. Perform the tensile test on the as-cast and aged samples in order to determine the fracture stress and strain by using an Instron 5982-type universal testing machine.
6. Perform shape memory test on the as-cast and aged samples using specially designed tensile test machine.
7. Detailed analysis of selected samples after shape memory test by using transmission electron microscope (TEM).

1.6 Significance of the Research

The basic aim of this research is to provide significant information on the behavior of the Cu-Al-Ni SMAs with and without alloying addition and aging treatments. Thus, the results of this research will benefit the automotive, civil and aerospace industries using the shape memory alloys in terms of improved properties such as ductility and reduction in brittleness. However, it gives the allowance for these materials to be used for higher temperatures without losing its shape memory effect. In addition, the outcomes of this research will contribute to providing a sustainable material with better properties and extensive earthquake applications.

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