MICROSTRUCTURES AND PROPERTIES OF Ti-51at.%Ni, Ti-23at.%Nb AND Ti-30at.%Ta SHAPE MEMORY ALLOYS FABRICATED BY MICROWAVE SINTERING FOR BIOMEDICAL APPLICATIONS

MUSTAFA KHALEEL IBRAHIM

UNIVERSITI TEKNOLOGI MALAYSIA

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MUSTAFA KHALEEL IBRAHIM

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Universiti Teknologi Malaysia

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ALHAMDULILLAH

All Praise for Allah, Creator of This Universe
Thanks for The Precious Iman & Islam You Blessed on Me
Thanks for All the Strength and Knowledge You Granted on Me
And, Peace Be Upon the Holy Prophet Muhammad SAW.
Thanks

I dedicated this work to,
My mother, whose sacrifice,
My Father, whose support and encouragement during his life,
and
All my family, whose love and patience
led to achieving my doctoral degree

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ABSTRACT

Titanium-nickel (Ti-Ni) shape memory alloys (SMAs) have been widely used for biomedical applications. However, Ni is recently known as a toxic element that can cause hypersensitivity on human body. Therefore, the development of Ni-free SMAs for biomedical applications is crucial. The best candidate to substitute Ti-Ni alloy is β type Ti alloys composed of nontoxic elements. The purpose of this research is to investigate the possibility of Ni-free Ti alloys, namely, titanium-niobium (Ti-Nb) and titanium-tantalum (Ti-Ta) to replace Ti-Ni. In the research, Ti-51at.%Ni, Ti-23at.%Nb and Ti-30at.%Ta SMAs were produced from elemental powders and fabricated by microwave sintering with addition of ternary elements namely, cerium, silver and tin. The microstructures of the sintered alloys were characterised by using differential scanning calorimetry (DSC) equipment, optical microscope, scanning electron microscope (SEM) and X-ray diffractometer (XRD). The mechanical and shape memory properties were determined using compressive test and specially designed equipment respectively, whereas corrosion and antibacterial behaviour were determined by using electrochemical test in simulated body fluid and agar disc diffusion technique with E.coli bacteria, respectively. Based on the experimental work on varying the sintering temperatures and times, it was found that 700˚C for 15 min gave the least porosity of 20% for Ti-51at.%Ni alloy, whereas 900˚C for 30 min gave the lowest porosity of 23% and 28.72% for Ti-23at.%Nb and Ti-30at.%Ta, respectively. It was observed that the microstructures of Ti-51at.%Ni alloys feature B2 austenite and B19´ martensite phases, while Ti-23at.%Nb alloys exhibit β austenite, α" martensite and α phases. The binary Ti-51at.%Ni alloy gives the highest hardness of 152 Hv, whereas the ternary Ti-Ta-3Ag gives the lowest hardness of 43 Hv. It was also found that Ti-Ni-1Ce alloy has the lowest elastic modulus of 5.2 GPa indicating good biocompatibility. The addition of Ce, Ag and Sn elements to Ti-23at.%Nb and Ti-30at.%Ta SMAs improved the total strain recovery (ƐT). The highest and lowest ƐT of 51.68% and 30.17% are shown by Ti-Ni-0.5Sn and Ti-Ni-3Ce alloys, respectively. The corrosion resistance was enhanced for all the ternary alloys due to the formation of passive layer on the surface and various phases within the material. The lowest corrosion rates observed in each type of the SMAs are 0.4076, 0.0155 and 0.0059 mm/year for Ti-Ni-0.5wt.%Sn, Ti-Nb-3wt.%Sn and Ti-Ta-0.5wt.%Sn alloys, respectively. Antibacterial property was improved after the addition of the alloying elements for all the ternary alloys indicated by the size of the inhibition zones against E. coli bacteria. It was found that Ti-Ta-3wt.%Ce alloy has the best anti-bacterial property with the largest inhibition zone of 7.75 mm compared with other SMAs. It can be concluded that the Ni-free SMAs, namely Ti-Nb and Ti-Ta alloys with the addition of alloying elements show promising candidates to be used as biomaterials due to their enhanced biocompatibility properties.

ABSTRAK

Aloi memori bentuk, Titanium-Nikel (Ti-Ni) telah digunakan secara meluas untuk aplikasi bioperubatan. Walau bahgaimanpun baru-baru ini Ni diketahui sebagai unsur toksik yang boleh menyebabkan hipersensitiviti pada tubuh manusia. Oleh itu, pembangunan aloi memori bentuk bebas Ni untuk aplikasi bioperubatan adalah penting. Calon terbaik untuk menggantikan aloi Ti-Ni adalah aloi jenis β Ti yang terdiri daripada unsur-unsur yang tidak bertoksik. Tujuan penyelidikan ini adalah untuk mengkaji kemungkinan Ti aloi tanpa Ni, iaitu, titanium-niobium (Ti-Nb) dan titanium-tantalum (Ti-Ta) sebagai pengganti Ti-Ni. Dalam kajian ini, aloi memori bentuk Ti-51at.% Ni, Ti-23at.%Nb dan Ti-30at.%Ta dihasilkan daripada unsur serbuk dan diperbuat dengan menggunakan alat gelombang mikro pensinteran dengan penambahan unsur pertigaan iaitu serium, perak dan timah. Mikrostruktur aloi yang telah melalui pensinteran dicirikan dengan menggunakan peralatan kalorimetri pengimbasan pembezaan (DSC), mikroskop optik, mikroskop elektron pengimbasan (SEM) dan meter pembelauan sinar-X (XRD). Sifat mekanikal dan memori bentuk masing-masing telah ditentukan dengan menggunakan ujian mampatan dan peralatan yang direka khas, manakala kelakuan kakisan dan antibakteria masing-masing ditentukan dengan menggunakan ujian elektrokimia di dalam cecair badan tiruan dan teknik serapan cakera agar dengan bakteria E.coli. Berdasarkan hasil eksperimen ke atas suhu dan masa pensinteran yang berbeza-beza, didapati bahawa suhu 700˚C selama 15 minit memberikan keliangan paling kurang iaitu 20% bagi aloi Ti-51at.%Ni, manakala suhu 900˚C selama 30 minit memberikan keliangan yang paling rendah, masing-masing 23% dan 28.72% bagi aloi Ti-23at.%Nb dan Ti-30at.Ta%. Hasil kajian menunjukkan bahawa mikrostruktur aloi Ti-51at.% Ni mempunyai fasa B2 austenit dan B19' martensit, manakala aloi Ti-23at.% Nb mempamerkan fasa β austenit, α" martensit dan fasa α . Aloi binary Ti-51at.%Ni memberikan kekerasan tertinggi iaitu 152 Hv, manakala aloi pertigaan Ti-Ta-3Ag memberikan kekerasan terendah iaitu 43 Hv. Hasil kajian juga mendapati bahawa aloi Ti-Ni-1Ce mempunyai modulus kenyal paling rendah iaitu 5.2 GPa yang menunjukkan bioserasi yang baik. Penambahan unsur Ce, Ag dan Sn kepada aloi Ti-23at.%Nb dan Ti-30at.%Ta telah menambah baik jumlah pemulihan terikan (ƐT). Nilai tertinggi ƐT iaitu 51.68% dan terendah iaitu 30.17% masing-masing bagi aloi Ti-Ni-0.5Sn dan Ti-Ni-3Ce. Rintangan kakisan dipertingkatkan untuk semua aloi pertigaan disebabkan pembentukan lapisan pasif pada permukaan dan pelbagai fasa dalam bahan. Kadar kakisan yang paling rendah yang diperhatikan dalam setiap jenis aloi memori bentuk ialah 0.4076, 0.0155 dan 0.0059 mm/tahun masing-masing bagi aloi Ti-Ni-0.5wt.%Sn, Ti-Nb-3wt.%Sn dan Ti-Ta-0.5wt.Sn. Sifat antibakteria telah bertambah baik selepas penambahan unsur-unsur pengaloian bagi semua aloi pertigaan yang ditunjukkan dengan saiz zon perencatan terhadap bakteria E.coli. Kajian ini menunjukan bahawa aloi Ti-Ta-3wt.%Ce mempunyai sifat anti-bakteria terbaik dengan zon perencatan terbesar iaitu 7.75mm berbanding dengan aloi memori bentuk yang lain. Ia dapat disimpulkan bahawa aloi memori bentuk bebas Ni, iaitu aloi Ti-Nb dan Ti-Ta dan dengan penambahan unsur pengaloian menunjukkan calon yang terbaik untuk digunakan sebagai bahan-bio kerana sifat bioserasi yang dipertingkatkan.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Ti – Ni	-	Titanium Nickel
Ti – Nb	-	Titanium Niobium
Ti – Ta	-	Titanium Tantalum
Ce	-	Cerium
Sn	-	Tin
Ag	-	Silver
SMAS	-	Shape Memory Alloys
SME	-	Shape Memory Effect
SE	-	Superelasticity
bcc	-	Body Centered Cubic
FCC	-	Face Centered Cubic
SEM	-	Scanning Electron Microscope
OM	-	Optical Microscope
SEM	-	Scanning Electron Microscope
XRD	-	X-ray Diffraction
EDS	-	Energy Dispersive Spectroscopy
DSC	-	Differential Scanning Calorimetry
ASTM	-	American Society for Testing and Materials
EDM	-	Electro-discharged Machining
SPS	-	Spark plasma sintering
GA	-	Gas atomization

LIST OF SYMBOLS

Ms	-	Martensitic start temperature
Mf	-	Martensitic finish temperature
As	-	Austenite start temperature
Af	-	Austenite finish temperature
σ_S	-	Slip deformation
ε_y	-	Yield strain
ε_T	-	Total strain recovery
ε_R	-	Residual strain

APPENDIX

CHAPTER 1

INTRODUCTION

1.1 Background of the Research

Among the various classes of smart materials, there is a unique class of these materials known as Shape memory alloys (SMAs). SMAs have a unique characteristic in that when temperature is applied on them, they recover their shapes. Additionally, the application of cyclic mechanical load on SMA makes them undergo a reversible hysteresis change of form and thus absorbing or dissipating mechanical energy. Due to these unique characteristics, SMAs have found broad application in vibration and damping, sensing and actuation and impact absorption. Furthermore, the functional characteristics of SMA are attributed to thermoelastic martensitic transformation which usually takes place at a temperature of between -100 and 200 ˚C depending on the applied heat treatment and the composition of the alloy [1].

Intermetallic compounds, Such as Ti-Ni, with shape memory effects (SMEs), are an interesting group of materials. They are used in a wide range of industries, namely electronics, robotics, telecommunications, as well as in medicine and optics. For commercial alloys made from Ti and Ni, their martensitic transformation start temperature (Ms) falls below a temperature of 373K [1], and for this reason, the alloys of Ti-Ni are used in applications requiring temperatures below 373K. Due to this limitation of Ti-Ni based alloys, scientists have extensively pursued the development of high-temperature SMAs (HTSMAs) such as Ni–Al, Ni–Mn, and Ti–Ni–X (X = Zr, Hf, Pd, Pt, Au) based HTSMAs [2]. However, the actual application of the existing HTSMAs is hindered by their poor cold workability which makes it difficult for thin plates and fine wires to be fabricated from them. Nonetheless, there has been a lot of investigation recently on the β-type Ti-based SMAs which have better cold workability. For example, a lot of findings on shape memory behaviour of β-type Ti-base alloys have been reported recently. The excellent cold workability of β-type Ti-based SMAs has increased the interest of researchers and users in these SMAs, and they are currently being explored for practical applications due to their ease of fabrication into wires and plates that can be used for various purposes.

Nickel-free Ti-based alloys, such as Ti-Nb and Ti-Ta, are good candidate materials to replace Ti-Ni alloys for biomedical applications because it was recently demonstrated that nickel is toxic to human beings. Additionally, the application of these alloys has increased with the advancement of other methods of processing such as powder metallurgy (PM) and mechanical alloying (MA) which have allowed the control of composition and grain size of alloys [2, 3]. These processes make use of solid-state powder techniques which are widely applied in the production of dispersion-strengthen alloys, refractory metals, nanocrystalline materials, and amorphous composite materials [4-6].

The transformation characteristics of Ti-based alloys made using PM have been investigated by several researchers followed by application of sintering using different techniques [7-10]. Despite the advantages of these alloys, studies have shown that a majority of them consume a lot of time during fabrication, and secondly, they develop cracks which reduce their mechanical properties. In sintering, a new technique for sintering referred to as microwave sintering is applied to heat the green compacts until they attain a temperature close to that suitable for sintering to allow densifying and alloying of ceramics, metals, and composites. Microwave sintering integrates pre-alloyed elements and absorbs electromagnetic energy using volumetric means and leading to transformation into heat [11-13]. Microwave sintering is different from other sintering technologies and provides the following benefits; it has a rapid rate of heating, uses less amount of energy, reduces the time used for sintering, enhances mechanical and physical properties of the materials being sintered and results in the improvement of element diffusion process [11].

1.2 Problem Statement

Ti-based alloys are good candidates for biomedical applications compared to other biocompatible metals due to their enhanced biocompatibility and superior mechanical properties. However, it is found that Ti-Ni is detrimental to human health due to the presence of nickel element. Therefore it is important to investigate and search for new nickel-free Ti-based alloys, such as Ti-Nb and Ti-Ta. Both alloys are known to have great potential to replace Ti-Ni alloys for biomedical applications.

Recently many researches have been done to develop ternary Ti-based alloys in order to obtain better properties. Selection of alloying elements are important because they affect the microstructures and properties of the alloys. There are many alloying elements which can be added to binary Ti-based alloys, however, very few will give the required properties such as excellent biocompatibility and enhanced mechanical properties. The elements which have these attributes include cerium (Ce), silver (Ag) and tin (Sn).

Powder metallurgy (PM) is a method of production used for the production of components with a near-net shape and is aimed at reducing the cost of machining and finishing of parts as in the case of casting method. The PM method is simple, versatile and inexpensive, and the sintering process occurs at much lower temperatures than the melting point of the constituent elements. As compared to other sintering techniques, microwave sintering has several advantages that include rapid rate of heating, use less energy, reduced times for sintering and produce porous alloys which are suitable for biomedical purpose. Hence, variations in sintering time and temperature with the PM process are essential for producing porous alloys. On the other hand, the pore size, shape, distribution and density during sintering is also important because it affects the stiffness of the materials and reduce the density as well as the elastic modulus. Thus, the main purpose of this research was to develop Ni-free Ti-based shape memory alloys for biomedical applications in order to prevent the toxicity of Ni.

1.3 Objectives of this Research

The objectives of this research are as follows:

1. To determine the effect of sintering temperature and time on density, microstructure and transformation temperatures of Ti-Ni, Ti-Nb and Ti-Ta shape memory alloys fabricated by microwave sintering;

2. To examine the microstructure and phase variations of Ti-Ni, Ti-Nb and Ti-Ta shape-memory alloys produced by the powder metallurgy method;

3. To investigate the effect of a third element addition Ce, Ag and Sn on the microstructure, mechanical properties and shape memory behaviour as well as biocompatibility of Ti-Ni, Ti-Nb and Ti-Ta based shape memory alloys.

1.4 Scope of this Research

The scopes of this research are as follows:

1. Preparation of samples by powder metallurgy method which includes mechanical mixing of Ti, Ni, Ta, Nb and alloying element powders. This was followed by compaction using hydraulic press. The final fabricating method was sintering process using microwave sintering technique.
2. Characterization of the sintered materials by optical microscopy, scanning electron microscopy, energy dispersive spectrometry, and x-ray diffractometry to determine the porosity content, microstructures and phases formed.
3. Determination of phase transformation temperatures of the sintered materials using differential scanning calorimetry equipment.
4. Performing the compression test of pre-alloyed powder samples in order to establish the compressive stress and strain with an Instron 600 DX-type universal testing machine.
5. Conduct shape-memory test on the fabricated samples using specially designed compression test machine.
6. Perform electrochemical polarization test to evaluate the bio-corrosion properties on all fabricated samples.
7. Perform the antibacterial test using ager disc diffusion technique on the fabricated sample.

1.5 Significance of Research

The main aim of this research was to improve the mechanical properties and shape memory behaviour of Ti-51%Ni, Ti-23%Nb and Ti-30%Ta SMAs produced by powder metallurgy methods. This work provided significant information on the behaviour of these binary shape-memory alloys and on the influence of third elemental additions on their properties. Detailed investigation into phase transformation and microstructural variation is significant for determining the optimum powder metallurgy parameters that can yield Ti-based alloys with the desired properties. The findings of this research will benefit the biomedical field in terms of applications such as dental implants, joint replacement systems, mechanical heart valves and stents based on the materials improve mechanical properties, shape memory behaviour and bio-corrosion properties.

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