UNIVERSITI TEKNOLOGI MARA

TiH_2 MICROPOROUS REPLICATION PSEUDO-ELASTIC NiTi ALLOY BY METAL INJECTION MOULDING

ROS ATIKAH BT ABDUL KADIR

Thesis submitted in fulfillment
of requirements for the degree of
Master of Science

Faculty of Mechanical Engineering

August 2017

AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student	:	Ros Atikah Bt Abdul Kadir @ Che Ismail
Student I.D. No	:	2014473235
Programme	:	Master of Mechanical Engineering
Faculty	:	Mechanical Engineering (EM 750)
Thesis Title	:	TiH_2 Microporous Replication Pseudo-elastic NiTi by Metal Injection Moulding
Signature of Student	:	....
Date	:	August 2017

ABSTRACT

Numerous studies have been done on implementing MIM to manufacture products for biomedical applications by employing NiTi alloy as a main subject. NiTi alloy is renowned materials with their good mechanical properties and pseudo-elastic behaviour. This study aims to study the effect of TiH_2 on thermal and behaviour of the feedstock, impurity contents and reversible phase transformation temperature (PTT) of NiTi alloy and to determine the mechanical properties of the samples. Two different compositions (50 at% Ni and 50.4 at% Ni) with three different powder loadings (65.5 vol%, 67.5 vol% and 69.5 vol%) were used in this research. The powder mixtures were blended in ball milling and mixed with two different binders known as palm stearin (PS) and polyethylene (PE) using brabender mixer. The flow behaviour of the feedstock was determined using rheology test. It shows that all feedstocks exhibit pseudoplastic behaviour which is suitable for MIM. The samples were injected into staple shape and tensile shape; then went through solvent debinding at temperature 60°C to remove palm stearin, thermal debinding at temperature 500°C in argon environment and finally sintered at temperature 1100°C. The characterisation of thermal, physical and mechanical properties and microstructure of the as-sintered samples was performed. The samples were tested for constituent phase morphologies, pore analysis, phase transformation temperatures and load-unload tensile test. During the sintering process, NiTi (B2) and other secondary phases ($NiTi_2$, Ni_3Ti and Ni_4Ti_3) with interconnected pores were formed due to the formation of Kirkendall effects. The use of TiH_2 was able to reduce the formation of intermetallic phases with the help of calcium hydride (CaH_2) as a reducing agent during the sintering process. It is possible that NiTi can be sintered using argon environment despite high vacuum condition; thus reducing the production cost of fabricating final samples. The pore size values for all samples were within the range of 20-100μm which was suitable for implant with minimum requirement of 50μm. Besides that, the carbon and oxygen content decreased significantly at highest powder loading because of the better inter-diffusion due to the existence of transient liquid formation which lead to the phase homogenization. The austenite temperature for the samples was within the range of 21°C to 28°C which proved the existence of pseudo-elastic behaviour and shape memory effect that correlated with the load-unload tensile test where the elastic deformation of all samples was around 2% to 6% strain. All related data demonstrated that NiTi was suitable as biomedical implants. Furthermore, the Young's Modulus value calculated from stress-strain curves was around 1.1 to 1.4 GPa which was very close to the cancellous bone (< 3 GPa); thus, it made these alloys suitable for bone implant.

TABLE OF CONTENTS

CHAPTER ONE
INTRODUCTION

1.1 RESEARCH BACKGROUND

Nickel Titanium (NiTi) has many potential applications in the engineering industry. Due to its unique shape memory effect (SME) and pseudo-elasticity (PE), it can be used as an active, adaptive or a smart structure for biomedical applications (Bansiddhi et al., 2008). In biomedical implant application, formation of pores in the nickel titanium alloy helps in allowing bone ingrowth and improve fixation. Besides, the variation of porosity amount can allow implant matching and bone stiffness to limit stress shielding effects. Low value of elastic modulus or connectivity in the form of foam is needed in biomedical applications for metal implants (Ismail, 2012).

It has been reported that most biomedical materials for bone implant research have been produced by powder metallurgy routes such as self-propagating high temperature synthesis (SHS) (Wisutmethangoon et al., 2009), hot isostatic pressing (HIP) (Samarov & Seliverstov, 2015) and conventional press and sinter method (Chen et al., 2013). Rather than the listed conventional method above, metal injection moulding (MIM) is the best method to produce complex shapes and small parts as it can create a variety of components from metal powders. Thus, MIM products are often superior to those produced by other fabrication methods (German & Bose, 1997).

Nowadays, many techniques have been developed to produce intricate parts with superior properties at low cost. It is believed that metal injection moulding (MIM) route can produce metal implants with excellent properties. Table 1.1 shows the overall advantages of MIM as compared to the other methods.