# INVESTIGATION ON THERMAL BEHAVIOR OF NITINOL BASED ACTUATING ELEMENTS FOR BIOMEDICAL APPLICATIONS

H. S. L. PERERA (148483 G)

MEng/PG Diploma in Manufacturing Systems Engineering Department of Mechanical Engineering University of Moratuwa

Sri Lanka

February 2020

# INVESTIGATION ON THERMAL BEHAVIOR OF NITINOL BASED ACTUATING ELEMENTS FOR BIOMEDICAL APPLICATIONS

Hettige Suranja Lakmal Perera

(148483 G)

Thesis/ Dissertation submitted in partial fulfillment of the requirements for the degree Master of Engineering

**Department of Mechanical Engineering** 

University of Moratuwa Sri Lanka

February 2020

#### **DECLARATION**

I declare that this is my own work and this thesis/dissertation does not incorporate without acknowledgement any material previously submitted for a Degree or Diploma in any other University or institute of higher learning and to the best of my knowledge and belief it does not contain any material previously published or written by another person except where the acknowledgement is made in the text.

Also, I hereby grant to University of Moratuwa the non-exclusive right to reproduce and distribute my thesis/dissertation, in whole or in part in print, electronic or other medium. I retain the right to use this content in whole or part in future works (such as articles or books).

Signature : Date :

Name of Student : H S Lakmal Perera Registration No : 148483G

The above candidate has carried out research for the Masters thesis/dissertation under my supervision.

Signature : Date :

Name of Supervisor : Dr. Y.W.R. Amarasinghe

#### Abstract

In modern material world, important consideration is given to the group of fascinating materials called shape memory materials (SMMs) which respond quickly to a definite change of heat, light and chemical. The shape memory materials that have been established to date are shape memory alloys (SMA), shape memory polymers (SMPs) and shape memory hybrids (SMH). SMA play a significant role in various applications such as sensors, actuators, clamping devices, etc. Nickel – titanium (NiTiNOL) alloys are heavily used in SMA due to their strain recovery, excellent thermal characteristics, reliability and commercial availability, in addition to being used in macro and micro electro mechanical systems based biomedical applications (BMA) due to high biocompatibility, resistance to corrosion and high fatigue limit.

Previous researches have focused on developing integration between thermal stability and SMA microstructure. But they don't have enough thermal behavior data with different heat treatment temperatures. Although phase transformation temperatures and microstructure patterns with different heat treatment temperatures are unique characteristics of NiTiNOL. The aim of this study is to investigate NiTiNOL characteristics and thermal behavior of SMA based actuating elements for biomedical applications. The overall objective of this research study is to investigate the phase transformation temperatures for NiTiNOL alloy during different heat treatment temperatures and to propose the appropriate geometric shape of the actuating element in BMAs.

Therefore, a number of experiments were done at the laboratory level to characterize the thermal related behavior of the NiTiNOL alloy. Differential scanning calorimetry test measurements are used in this study to analyze the dissimilarities in phase transformation temperatures and properties of NiTiNOL (Ni-54 and Ti-46, weight percentages) alloy due to the variation of heat treatment temperature ranging from 400 °C to 600 °C. Further, microstructure and Energy – dispersive X-ray are determined using Scanning Electron Microscopy. It is found that most critical phase transformations are taken place between heat treatment temperatures of 550 °C and 600 °C and extraordinary unique behavior of phase transformations are exhibited by the respective specimens subjected to these temperatures. Further it is found that thermal behavior of actuator elements is dominated by the changes incurred in the microstructure of the NiTiNOL alloy during heat treatment.

Keywords: NiTi materials, Mechanical behavior, Transformation temperature

### Acknowledgements

I would like to thank my project Supervisor Dr. Y.W.R. Amarasinghe, Senior Lecturer from Department of Mechanical Engineering, University of Moratuwa and his aspiring guidance, invaluably constructive criticism and friendly advice during the project work. I am sincerely grateful to him for sharing their truthful and illuminating views on a number of issues related to the project.

Again I am thankful Prof. R.A.R.C. Gopura, Head of the Department of Mechanical Engineering, University of Moratuwa and Dr. Manoj Ranaweera the course coordinator of this programme, they always encouraged us to continue this course module with past experiences.

I thank Mr. T.A.U. Roshan (Research Student, Department of Mechanical Engineering, and University of Moratuwa) for being very supportive, and for all the encouragements he has provided to me in these days.

Lastly, I would like to acknowledge all the people who provided me with the facilities being required and conductive conditions for my MEng research project

## Content

Abstract	ii
Acknowledgements	iii
List of Figures	vii
List of Tables	x
List of Abbreviations	xi
Chapter 01	1
1 Introduction	1
1.1. Motivation	1
1.2. Scope of Study	2
1.3. Aim and Organization of Thesis	2
1.3.1. Aim	2
1.3.2. Objectives	3
1.3.3. Chapter Organization	3
Chapter 02	4
2 Literature Review	4
2.1. Shape Memory Alloy	4
2.1.1. A brief history of SMA	4
2.1.2. The Phase of Shape Memory Alloy	6
2.1.3. Principles of Shape Memory Effect	15
2.1.4. Summarization of shape memory effect	21
2.1.5. Stress and Temperature	22
2.2. Transformation Temperatures measuring techn	niques23
2.2.1. Thermal Analysis	23
2.2.2. Types of thermo analytical methods	23
2.3. Measuring Transformation Temperatures in Na	iTi Alloys28
2.4. Mechanical Characteristics of NiTi Alloy	30
2.5. Thermodynamic Modeling of Shape Memory 2	Alloy31
2.5.1. Small deformation (strain) models of SMA	A elements32
2.5.2. Models available in the literature	33
2.6. SMA for Biomedical Applications	36
2.7. Historical development of biomedical actuator	s38

2.8. C	haracterization of SMA for Biomedical Applications	39
2.8.1.	SMA-Based Actuators	39
2.9. S	MA-Based Actuator Elements	40
2.9.1.	Importance of NiTi Elements	40
2.10.	Controlling of Actuator elements	41
2.10.1	. Thermal operated actuator elements	43
2.10.2	2. Electrical operated actuator elements	43
2.11.	SMA based Macro level Biomedical Applications	43
2.12.	SMA based Micro (MEMS) Actuator for Biomedical Applications	45
2.12.1	. MEMS applications of Biomedical Applications	46
2.12.2	2. Dimensions of Biomedical (BM) microgrippers	51
Chapter 03	;	53
3 Invest	tigation of Thermal and Topological Characteristics	53
3.1. S	pecifications of experimental apparatus	53
3.1.1.	Electric Muffle Furnace	53
3.1.2.	Differential Scanning Calorimetry (DSC)	53
3.1.3.	Scanning Electron Microscope (SEM)	54
3.2. E	xamine the thermal properties with DSC	55
3.2.1.	Sample preparation for DSC	56
3.2.2.	Sample preparation for OM &b SEM	56
Chapter 04	۱	58
4 Verifi	cation of Thermal Behavior and Topological Characteristics	58
4.1. A	nalysis of thermal behavior of NiTiNOL material	58
4.2. T	opological analysis of NiTiNOL material	64
Chapter 05	í	68
5 NiTiN	NOL actuating element for Biomedical Applications	68
5.1. W	Vorking principle of SMA helical spring based actuator	68
5.2. D	esign of SMA based Helical Spring	69
5.2.1.	Parameters of SMA based Helical Spring	69
5.2.2.	Design and Development of Spring Forming Fixture	70
5.2.3.	Forming of NiTiNOL Helical Spring	71
5.3. H	leat Treatment Process (Shape Setting) of NiTiNOL Spring	72

Chapter 06	74
6 Results and Discussion	74
6.1. Representation of DSC thermograms	74
6.2. Validation of the SMA spring actuator element	79
7 Conclusions	80
References	82
Appendix A	85
Shape Memory Alloy material manufacturer	85
Appendix B	86
SMA helical spring setting fixture design	86
Appendix C	89
DSC Thermograms with more details	89

# List of Figures

Figure 2.1: Crystal structure of martensite phase [9]	7
Figure 2.2: Crystalline structure of martensite phase [9]	7
Figure 2.3: The stress and strain curve of martensite phase [12]	8
Figure 2.4: The crystal structure of austenite phase (Nenno, 1982; Funakubo, 1987)	9
Figure 2.5: Crystalline structure of austenite phase [4]	9
Figure 2.6: The stress and strain curve [7]	10
Figure 2.7: Microscopy view of phase transformation [7]	11
Figure 2.8: Thermomechanical behavior of NiTi wire [15]	12
Figure 2.9: Stress/ strain figure Vs austenite and martensite (twinned, detwenned) [3]	12
Figure 2.10: One Way Shape Memory Effect: [11]	16
Figure 2.11: The phase changes in OWSME [7]	17
Figure 2.12: The curve of stress, strain and temperature in [7]	17
Figure 2.13: Two Way Shape Memory Effect [11]	18
Figure 2.14: The phase changes in TWSME [7]	19
Figure 2.15: The curve of stress, strain and temperature in TWSME [7]	19
Figure 2.16: The phase changes in pseudoelasticity [7]	20
Figure 2.17: The curve of stress, strain and temperature in pseudoelasticity [7]	21
Figure 2.18: Shape memory alloy stress-temperature phase diagram [13]	22
Figure 2.19: Indicating of SE and SME in stress Vs temperature [13]	23
Figure 2.20: Disk-type DSC [20]	25
Figure 2.21: Cylinder-type DSC [20]	25
Figure 2.22: Power compensating DSC [20]	26
Figure 2.23: Sample Length Vs Temperature (constant load) [13]	28
Figure 2.24: DSC experimental thermogram for common NiTi alloy [13]	29
Figure 2.25: Curve of Active Af for common NiTi alloy [13]	30
Figure 2.26: Power to weight ratio vs. weight diagram of different actuators [18]	39
Figure 2.27: Constant force for opposite direction	41
Figure 2.28: Spring to opposite direction	42
Figure 2.29: SMA in two one-way direction	42

Figure 2.30 Biomedical applications of SMA [33]	44
Figure 2.31: Shemetic view micrvalve and pump [27]	46
Figure 2.32: The micro-wrapper made of TiNi films [28]	47
Figure 2.33: The 100µm wire for microelectrode clipping [28]	47
Figure 2.34: Types of microgrippers and grasping mechanisms [27]	48
Figure 2.35: Geometry of the microgripper [29]	49
Figure 2.36: New microgripper (fabricated in superelastic alloy) [29]	49
Figure 2.37: Microgripper with two NiTi-Si cantilever beams bonded together [30]	49
Figure 2.38: Working method of the above microgripper (left) and a SMA based cant system microgripper (Right) [31]	ilever 50
Figure 2.39: The SMA microgripper in open (left) and closed (right) state [31]	50
Figure 2.40: 3D view (left) and the drawing (right) of the design [31]	51
Figure 3.1: Electric Muffle furnace	53
Figure 3.2: DSC analyzer placement of specimen	54
Figure 3.3: Scanning Electron Microscope (SEM)	54
Figure 3.4: Schematization of DSC analysis machine	55
Figure 3.5: NiTINOL specimen and holder	56
Figure 3.6 All specimens for OM and SEM	57
Figure 4.1: DSC Thermogram for Raw material	59
Figure 4.2: DSC Thermogram for Specimen 01	60
Figure 4.3: DSC Thermogram for Specimen 02	61
Figure 4.4: DSC Thermogram for Specimen 03	61
Figure 4.5: DSC Thermogram for Specimen 04	62
Figure 4.6: DSC Thermogram for Specimen 05	63
Figure 4.7: SEM image of NiTiNOL raw material	64
Figure 4.8: EDS diagram of NiTiNOL raw material	65
Figure 4.9: SEM image of NiTiNOL at 400°C	65
Figure 4.10: EDS diagram of NiTiNOL at 400°C	65
Figure 4.11: SEM image of NiTiNOL at 500°C	66
Figure 4.12: EDS diagram of NiTiNOL at 500°C	66
Figure 4.13: SEM image of NiTiNOL at 550°C	66
Figure 4.14: EDS diagram of NiTiNOL at 550°C	67
Figure 5.1: Top view of developed gripper assembly [32]	68

Figure 5.2: NiTiNOL Helical Spring Parameters [32]	70
Figure 5.3: Shape setting fixture dimensions [32]	70
Figure 5.4: Important parts of spring forming fixture [32]	70
Figure 5.5: Forming of NiTiNOL based helical spring [34]	71
Figure 5.6: Complete unit for heat treatment [32]	72
Figure 5.7: Heat treated NiTiNOL based helical spring [32]	73
Figure 6.1: DSC Thermogram of all specimens for 30 minutes	74
Figure 6.2: Variation of phase transformation temperatures	76
Figure 6.3: Variation of Peak temperature values	77
Figure 6.4: Variations of Enthalpy values	78
Figure A.1: Manufacturer Invoice	84
Figure B.1: Complete assembly of fixture	85
Figure B.2: Dimensions of fixture	86
Figure B.3: Illustrated dimensions of holder and screw	86
Figure B.4: Illustrated dimensions of positioner	
Figure B.5: Illustrated dimensions of sleeve	
Figure C.1: DSC heat-treated Thermogram at 400 ° C for 30min	88
Figure C.2: DSC heat-treated Thermogram at 450 ° C for 30min	
Figure C.3: DSC heat-treated Thermogram at 500 ° C for 30min	
Figure C.4: DSC heat-treated Thermogram at 550 ° C for 30min	91
Figure C.5: DSC heat-treated Thermogram at 600 ° C for 30min	92

### List of Tables

Table 2:1: Shape memory alloy and composition [6, 9] 5
Table 2:2: Material systems with shape memory properties [7]
Table 2:3: Properties of NiTi under different phase [16, 17]      14
Table 2:4: Summary of SME in the different situations. 21
Table 2:5: Historical Development of NiTiNOL elements for biomedical actuators [3] 38
Table 2:6: Types of conventional macroscopic SMA actuator elements [07]
Table 2:7: Listed NiTiNOL based biomedical applications [33]
Table 2:8: Variation of microactuators capabilities [27]
Table 2:9: Geometrical dimensions of exiting macro and micro grippers [31]
Table 3:1: Labeling of heat treated of specimens. 55
Table 4:1: Variation of chemical composition of NiTiNOL alloy    67
Table 5:1: Detail description of gripper components 68
Table 5:2: Parameters of Helical Spring [32] 69
Table 6:1: Transformation temperatures of specimens
Table 6:2: Peak temperatures of specimens
Table 6:3: Enthalpy values of specimens
Table 6:4: Average enthalpy values of each phases    79
Table 6:5: Variation of composition with HTT 79

## List of Abbreviations

$M_{\rm f}$	: Martensite start
$M_s$	: Martensite finish
As	: Austenite starts
A <sub>f</sub>	: Austenite finish
Rs	: R – Phase start
R <sub>f</sub>	R – Phase finish
B2	: Austenite (cubic)
B2'	: R – Phase (rhomohedral)
B19	: Orthorhombic
B19'	: Martensite (monoclinic)
HTT	: Heat Treatment Temperature
TT	: Transition Temperature
DSC	: Differential Scanning Calorimetry
OM	: Optical Microscopy
SEM	: Scanning Electron Microscopy
EDX/EDS	: Energy – dispersive X-ray
BMA	: Biomedical Applications
MIS	: Minimally Invasive Surgery