

**ANALYSIS OF PSEUDOELASTIC
DEFORMATION CYCLES OF GRADIENTLY
AGED NITI SHAPE MEMORY ALLOYS**

MUHAMMAD HANAFI BIN ISHAK

UNIVERSITI SAINS MALAYSIA

2021

ANALYSIS OF PSEUDOELASTIC DEFORMATION CYCLES OF GRADIENTLY AGED NITI SHAPE MEMORY ALLOYS

By:

MUHAMMAD HANAFI BIN ISHAK

(Matrix No.: 137844)

Supervisor:

Assoc. Prof. Ir. Dr. Abdus Samad Bin Mahmud

August 2021

This dissertation is submitted to

Universiti Sains Malaysia

As partial fulfilment of the requirement to graduate with honors degree in
BACHELOR OF ENGINEERING (MECHANICAL ENGINEERING)



School of Mechanical Engineering
Engineering Campus
Universiti Sains Malaysia

DECLARATION

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

Signed...*Hanafi*.....

(MUHAMMAD HANAFI BIN ISHAK)

Date: 12/7/2021

Statement 1

This thesis is the result of my own investigation, except where otherwise stated. Other sources are acknowledged by giving explicit references. Bibliography/references are appended.

Signed...*Hanafi*.....

(MUHAMMAD HANAFI BIN ISHAK)

Date: 12/7/2021

Statement 2

I hereby give consent for my thesis, if accepted, to be available for photocopying and for interlibrary loan, and for the title and summary to be made available outside organizations.

Signed...*Hanafi*.....

(MUHAMMAD HANAFI BIN ISHAK)

Date: 12/7/2021

ACKNOWLEDGEMENT

The completion of this thesis title “Analysis of Pseudoelastic Deformation Cycles of Gradiently Aged NiTi Shape Memory Alloys” would have been possible without the kinds of support and helps from many individuals. First and foremost, praises and thanks to Allah SWT for his shower of blessings on me throughout completing my final year projects successfully.

Next, I would like to express my deepest gratitude to my supervisor, Assoc. Prof. Ir. Dr. Abdus Samad Bin Mahmud for giving an opportunity to complete my final year project under his supervision and providing invaluable guidance and support.

In addition, my sincere thanks also go to the assistant engineers in the School of Mechanical Engineering especially Encik Abdul Latif Amzah and Encik Fakruruzi Fadzil who were always willing to help me in using the equipment and machines in the lab. Therefore, I would like to show my gratitude to Dr Muhammad Fauzinizam Razali and a PhD student from School of Mechanical Engineering, Ng Ching Wei for their guidance and knowledge about shape memory alloy.

I am indebted and thankful to my parents for their prayers and always support me throughout my studies. Last but not least, I would like to thank all my friends who have kept supporting me to finish this project. Thank you all for your encouragement.

TABLE OF CONTENTS

DECLARATION.....	iii
ACKNOWLEDGEMENT.....	iv
TABLE OF CONTENTS	v
LIST OF TABLES	vii
LIST OF FIGURES	viii
LIST OF SYMBOLS	x
LIST OF ABBREVIATIONS	xi
LIST OF APPENDICES	xii
ABSTRAK	xiii
ABSTRACT.....	xiv
CHAPTER 1 INTRODUCTION.....	1
1.1 Project Overview.....	1
1.2 Problem Statement	3
1.3 Objectives.....	3
1.4 Project Scopes	3
CHAPTER 2 LITERATURE REVIEW.....	4
2.1 Nickel Titanium (NiTi) Shape Memory Alloy.....	4
2.2 Crystal Structure of NiTi Shape Memory Alloy	5
2.3 Thermal Martensitic Phase Transformation of NiTi Shape Memory Alloy	6
2.4 Deformation behavior of NiTi Shape Memory Alloy	8
2.4.1 Psuedoelasticity.....	9
2.4.2 Shape Memory Effect	10
2.5 Functionally Graded NiTi Shape Memory Alloy.....	11
2.6 Cyclic Deformation.....	13

CHAPTER 3	METHODOLOGY.....	14
3.1	Preparation of Gradient Furnace	14
3.2	Ageing Treatment of NiTi Shape Memory Alloy	16
3.2.1	Isothermal Ageing Treatment	17
3.2.2	Gradient Temperature Ageing Treatment	18
3.3	Thermal Phase Transformation Behaviour Analysis	19
3.4	Deformation Behaviour Analysis.....	21
CHAPTER 4	RESULT AND DISCUSSION.....	22
4.1	Gradient Furnace Result.....	22
4.2	Thermal Transformation Behaviour Analysis.....	24
4.2.1	Thermal Transformation of Isothermal Ageing	24
4.2.2	Thermal Transformation of Gradient Ageing	27
4.3	Deformation Behaviour Analysis.....	29
4.3.1	Isothermal Ageing Deformation Behaviour.....	29
4.3.2	Gradient Ageing Deformation Behaviour	31
4.4	Cyclic Deformation Behaviour Analysis	33
4.4.1	Cyclic Deformation Behaviour of Isothermally Aged NiTi	33
4.4.2	Cyclic Deformation Behaviour of Gradiently Aged NiTi.....	36
CHAPTER 5	CONCLUSION AND FUTURE WORK	39
5.1	Conclusion.....	39
5.2	Future Work Recommendation	39
REFERENCES.....		40
APPENDICES		

LIST OF TABLES

	Page
Table 2.1 Result of previous research based on different condition and parameter ..	11
Table 4.1 Transformation temperatures on DSC curve for different ageing temperature	26
Table 4.2 Transformation temperatures on DSC curve at different section on the gradient specimen	28
Table 4.3 Deformation characteristic of gradient ageing specimen.....	32

LIST OF FIGURES

	Page
Figure 2.1 Austenite lattice structure	5
Figure 2.2 Martensite lattice structure	5
Figure 2.3 Thermal transformation behaviour of shape memory alloy [7].....	6
Figure 2.4 Two-stage phase transformation behaviour of NiTi shape memory alloys [10]	7
Figure 2.5 Thermomechanical behaviour of a near equiatomic NiTi. The red lattice represents austenite, the blue square-shaped lattice represent the self-accommodated martensite, and the blue parallelogram-shaped lattice represent the oriented martensite [13].	8
Figure 2.6 Stress-strain curve for pseudoelastic effect [14].....	9
Figure 2.7 Schematic diagram of the shape memory effect [15].....	10
Figure 2.8 Gradient pseudoelastic behaviour of gradient-aged Ti–50.8 at%Ni wire deformed under tension at 25 °C [2].	12
Figure 2.9 Stress-strain curve of pseudoelastic behaviour by cycling load of the Ti– 50.8 at% Ni alloy [18].....	13
Figure 3.1 Gradient temperature furnace setup.....	14
Figure 3.2 Schematic diagram of heating element loop of furnace	15
Figure 3.3 Schematic diagram of thermocouples and NiTi wire location	16
Figure.3.4 MTI tube furnace used for initial solution treatment and isothermal ageing treatment.....	17
Figure 3.5 TA Q20 differential calorimeter device.....	19
Figure 3.6 Sectioning of DSC specimen from the total length of gradient aged NiTi wire	20
Figure 3.7 Tensile Test Setup (Instron 3367 Universal Tensile Machine)	21
Figure 4.1 Real time plot of temperature recorded in gradient furnace.....	22

Figure 4.2 Temperature profile along recorded by the thermocouples.....	23
Figure 4.3 Thermal transformation behaviour of Ti-51at%Ni alloy after isothermally aged at 400°C for 10 minutes.....	24
Figure 4.4 Thermal transformation behaviour of Ti-51at%Ni alloy after isothermally aged at 430°C for 10 minutes.....	25
Figure 4.5 Thermal transformation behaviour of Ti-51at%Ni alloy after isothermally aged at 450°C for 10 minutes.....	26
Figure 4.6 Thermal transformation behaviour along the length of gradient ageing specimen	27
Figure 4.7 Pseudoelastic behaviour of 51at%Ni after isothermal ageing for 10 minutes at test temperature 27°C	29
Figure 4.8 Effect of ageing temperature on stress forward and reverse plateau of Ti-51at%Ni alloy aged for 10 minutes.....	30
Figure 4.9 Effect of ageing temperature on residual strain of Ti-51at%Ni alloy aged for 10 minutes	30
Figure 4.10 Gradient deformation behaviour of gradient ageing specimen	31
Figure 4.11 Cyclic deformation of 51at%Ni after isothermal ageing at a) 400°C, b) 430°C, c) 450°C for 10 minutes.....	34
Figure 4.12 Pseudoelastic behaviour of isothermally aged specimen after 5 th cycle of cyclic test	35
Figure 4.13 Pseudoelastic behaviour of gradiently aged NiTi under cyclic test.....	36
Figure 4.14 Effect of cycling loading to the critical stress for forward transformation	37
Figure 4.15 Effect of cyclic loading on the plateau strain and residual strain.....	38

LIST OF SYMBOLS

Ms	Martensite start temperature
M _f	Martensite finish temperature
A _s	Austenite start temperature
A _f	Austenite finish temperature
A	Austenite phase
M	Martensite phase
R	R-phase
T	Temperature

LIST OF ABBREVIATIONS

NiTi	Nickel Titanium
SMA	Shape Memory Alloy
DSC	Differential Scanning Calorimetry
SME	Shape Memory Effect

LIST OF APPENDICES

- Appendix A DSC Curve of Gradient Aged Specimen
- Appendix B Navigator DAQNav Data Logger
- Appendix C Temperature Controller Box (FP93-8P-90-0000)

ABSTRAK

Aloi memori bentuk NiTi dengan sifat tingkah laku ubah bentuk bercerun adalah pengubahsuaian aloi untuk mempunyai sifat termomekanik berterusan yang berbeza dengan memperluas selang transformasi seperti perubahan tekanan dan suhu. Dalam kajian ini, wayar NiTi dengan sifat tingkah laku ubah bentuk bercerun diciptakan dengan tingkah laku pseudoelastik yang optimum melalui rawatan penuaan bercerun. Tungku penuaan bercerun dibina untuk menua wayar dalam profil suhu kecerunan. Spesimen yang telah dirawat dalam keadaan bercerun diuji dengan Calorimetri Pengimbasan Berbeza (DSC) untuk memerhatikan dan menganalisis suhu transformasi wayar NiTi yang tambahbaik. Tingkah laku pseudoelastik NiTi dengan sifat tingkah laku ubah bentuk bercerun dianalisis dengan melakukan ujian tegangan dengan beban berulang pada suhu bilik. Hasilnya, specimen dengan sifat tingkah laku ubah bentuk bercerun menunjukkan kecerunan tekanan hadapan dan berbalik masing-masing 1.42GPa dan 2.52GPa dengan regangan sisa 2.7%. Untuk ubah bentuk kitaran, pengurangan tekanan transformasi maju dan mundur diperhatikan. Ketegangan dataran tinggi dan sisa terjejas akibat ubah bentuk pemuatan kitaran. Ketegangan residu cenderung stabil tanpa simptom pada kitaran kemudian berbanding dengan yang sebelumnya. Oleh itu, buat NiTi yang dinilai mempunyai tingkah laku psuedoelastik yang baik dibawah ujian bebanan berulang berbanding dengan wayar asal dan wayar yang menjalani pendekatan penyepuhlindungan.

ABSTRACT

Functionally graded NiTi shape memory alloy is a modification of the alloy to have a continuous varying thermomechanical property by expanding the transformation interval such as transformation stress and temperature window. In this study, a functionally graded NiTi wire was created with an optimum pseudoelastic behaviour through a gradient ageing treatment. Gradient ageing furnace was built to age the wire in a gradient temperature profile. The aged specimens were test with the Differential Scanning Calorimetry (DSC) to observe and analyse the transformation temperature of graded NiTi wire. The pseudoelastic behaviour of the functionally graded NiTi was analysed by conducting tensile test with cyclic load in room temperature. As the result, the gradient aged specimen exhibited forward and reverse stress gradient of 1.42GPa and 2.52GPa respectively with residual strain of 2.7%. For the cyclic deformation, reduction in the forward and reverse transformation stress was observed. Plateau and residual strain were affected from the cyclic loading deformation. Residual strain tends to stabilize asymptotically in the later cycles compared to the earlier one. Thus, make the graded NiTi have a good psuedoelastic behaviour upon cyclic deformation compared to the original wire and isothermal aged wire.

CHAPTER 1

INTRODUCTION

1.1 Project Overview

Shape memory alloys have the unique shape recovery ability after being deformed. Shape memory alloys exist in two different phases which are austenite and martensite while in three different crystal structure which are twinned martensite, detwinned martensite and austenite. Austenite is occurred in high temperature phase while martensite is occurred in low temperature phase. There are two unique deformation of the shape memory alloys that attract the researchers out there in shape memory alloys which are pseudoelasticity and shape memory effect.

Among the various shape memory alloys, NiTi is the most widely used in many applications such as medical field, innovative designs and smart structures. In order to further expand the capabilities of shape memory alloys, many developments are worthy of noting such as functionally graded NiTi, NiTi honeycomb or cellular structures and in-situ NiTi-Nb nanowire composites have been developed[1]. The improvement NiTi shape memory alloy such as functional graded NiTi alloys provide a significant benefit in creating wider transformation stress and temperature windows by providing a gradient microstructure property in the alloy. The widened transformation stress and temperature effected the deformation behaviour of the alloy.

There are several researches have been done to have a good functionally graded NiTi alloy. There are some methods to create the functionally graded NiTi alloy such as heat treatment by ageing or by lasering process. Ageing can be two types of ageing: isothermal ageing and gradiently ageing. In this project we are focusing on gradiently ageing to achieve the gradiently NiTi alloy. Isothermal ageing also is carried out to see the different of the deformation behaviour between these two ageing and the normal NiTi alloy. In order to achieve a good functionally graded NiTi alloy, we need to consider several parameters such as composition of the alloy, ageing temperature and ageing duration. These parameters are very crucial to achieve optimize gradiently NiTi alloy.

NiTi alloy with composition of 51% at %Ni is selected to be used in this project. Ageing treatment is carried out at the optimum ageing temperature in order to design near-perfect gradient pseudoelastic behaviour of NiTi alloy. Since the NiTi alloy is very sensitive to temperature and time, ageing time also needs to be considered to form the precipitate. The area encompassed by TiNi and Ti₃Ni₄ limits the temperature and time of ageing employed to improve the pseudoelastic behaviour of NiTi alloys. Larger temperature ranges and longer ageing times are available for alloys with a higher Ni content. Generally, Ni-rich NiTi alloys have a higher Ni content, making them more likely to precipitate Ti₃Ni₄. In this project, parameters from previous research is taken to have the optimize graded NiTi alloy[2].

Test is continued by undergo the functionally graded NiTi alloy by applying cyclic load to analyse the pseudoelasticity behaviour of the gradiently ageing alloy with isothermal ageing alloy and normal shape memory alloy. The present study is to observe and analyse the pseudoelastic behaviour deformation cycles of the gradient aged NiTi alloy.

1.2 Problem Statement

Shape memory alloys are unique alloy that have an ability to remember its true shape after undergoes large deformation under loading or thermal cycles. Graded NiTi alloy refers to NiTi alloy that have received heat treatment condition such as ageing process and it can improve the alloy's microstructural characteristics. Gradient microstructure properties can be achieved by having the graded NiTi and thus improve its pseudoelasticity. After a few cycles of cyclic deformation, normal NiTi will exhibit a distinct shape memory behaviour. Stress hysteresis between forward and reverse stress plateau is decreased prior to cyclic deformation. So, this research is carried out to analyse the pseudoelastic behaviour of functionally graded NiTi alloy under cyclic deformation.

1.3 Objectives

- 1) To produce a functionally graded Ni-rich Ti-51at%Ni alloy through gradient ageing treatment with the optimization of gradient deformation behaviour.
- 2) To study the effect of cyclic deformation towards pseudoelastic behaviour of the functionally graded Ni-rich Ti-51at%Ni alloy.

1.4 Project Scopes

This research focuses on creating a functionally graded Ni-rich NiTi alloy and observing the effect of cyclic deformation on the functionally graded Ti-51at%Ni alloy. NiTi alloy with a composition of 51at%Ni is used and undergo a heat treatment of gradient ageing in a gradient furnace to form the functionally graded Ti-51at%Ni alloy. The gradient furnace is rebuilt as the previous research. The optimum temperature range and ageing duration are selected based on previous research in order to obtain the optimum gradient deformation behaviour of the gradient aged alloy. Tensile test is conducted on the graded NiTi alloy by applying cyclic loads on the specimens in room temperature. Universal Testing Machine is used in this tensile test. Deformation of the graded NiTi alloy is observed in this project to analyse the pseudoelastic behaviour of the graded NiTi alloy.

CHAPTER 2

LITERATURE REVIEW

2.1 Nickel Titanium (NiTi) Shape Memory Alloy

Shape memory alloys have a unique ability that can memorize its original shape after being deformed beyond its elastic limit. Due to this unique ability, many researchers start to study and try to apply this special and unique deformation behaviour into various field such as medical and dental devices, sensor and actuator devices and civil structures. There are several numbers of alloy that belong to the shape memory alloy and the famous alloys are Ni-Ti, Ni-Ti-Cu, Cu-Al-Ni, Cu-Zn-Al, Fe-Mn-Si, Fe-Pt, Fe-Pd and Fe-Ni-Co-Ti[3]. Nickel Titanium (NiTi) alloy is a metal alloy based of nickel and titanium which belong to the class of shape memory alloy.

In NiTi alloy, two elements are existed in roughly equal atomic ratio (near-equiatomic) and it exists in different crystallographic structures. Although NiTi is an alloy with near-equiatomic composition, the percentage of nickel and titanium in the alloy is very crucial and the characteristic and behaviour act differently due to different composition of alloy. NiTi shape memory alloy have good mechanical properties such as good formability, high energy storage, high ductility, low stiffness, lower elastic modulus and its special characteristic which is pseudoelasticity and shape memory effect[4].

2.2 Crystal Structure of NiTi Shape Memory Alloy

Shape memory alloy exhibit the ability to return to its original shape after large deformation because It can go through a reversible martensitic phase change when subjected to external stress or heat. There are two forms of atomic lattice in NiTi alloy which are austenite (A) and martensite (M) phase. Transformation of the phase is influenced by the changes of temperature and stress[2]. Austenite phase is in form of body-centered cubic (B2) structure and exist at high temperature while martensite phase is in form of monoclinic (B19') structure that exist at lower temperature[5]. In austenite phase, single nickel atom located at the center cube structure with the titanium atom located at all corners of the cube structure as shown in Figure 2.1 but in martensite phase, there are different atoms located at every corner of the structure[6] as shown in Figure 2.2. In comparison, austenite is more stable crystal structure compared to martensite due to the symmetric of their structure.

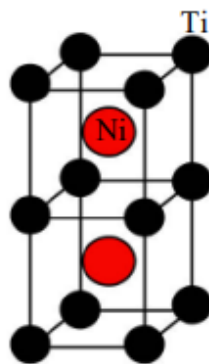


Figure 2.1 Austenite lattice structure

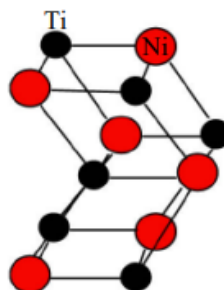


Figure 2.2 Martensite lattice structure

2.3 Thermal Martensitic Phase Transformation of NiTi Shape Memory Alloy

Shape memory alloy can undergo reversible phase from austenite to martensite and martensite to austenite. Martensitic phase transformation involves reversible phase transformation in between high-temperature phase which is austenite and low temperature phase which is martensite. There are four critical temperatures where define the thermal martensitic phase deformation; Martensite starts temperature for the forward $A \rightarrow M$ transformation (M_s), martensite finish temperature for the forward $A \rightarrow M$ transformation (M_f), austenite starts temperature for the reverse $M \rightarrow A$ transformation (A_s) and austenite finish temperature for the reverse $M \rightarrow A$ transformation (A_f). All these temperatures can be determined by using Differential Scanning Calorimeter (DSC) instrument. Figure 2.3 shows the typical thermal transformation behaviour of shape memory alloy[7].

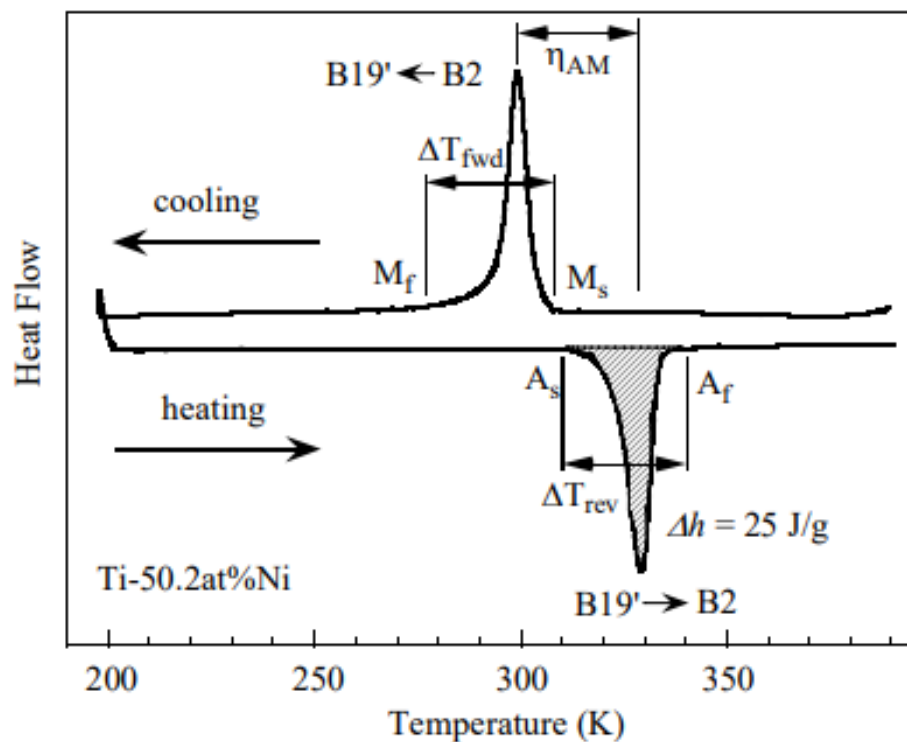


Figure 2.3 Thermal transformation behaviour of shape memory alloy [7]

Generally, NiTi shape memory alloys always show a single-stage $A \rightarrow M$ and $M \rightarrow A$ transformation as Figure 2.4 that is known as forward and reverse transformation, respectively. Two-stage phase transformation has been reported to occur when this type of alloy is frequently subjected to thermal cycling, annealing process after cold work and ageing of Ni-rich alloys at certain temperature[8]. The phase transformation will undergo two-stage of $A \leftrightarrow R \leftrightarrow M$ transformation as shown in Figure 2.4. For Ti-51at%Ni wire, it was observed that B2-R or $A \leftrightarrow R$ transformation peak appeared in the differential scanning calorimetry (DSC) measurement when the alloys were aged at the temperature between 400°C to 475°C for 30 minutes. Further ageing at 500°C to 550°C yielded two stage transformation, B2-R-B19' or $A \leftrightarrow R \leftrightarrow M$ in cooling [9].

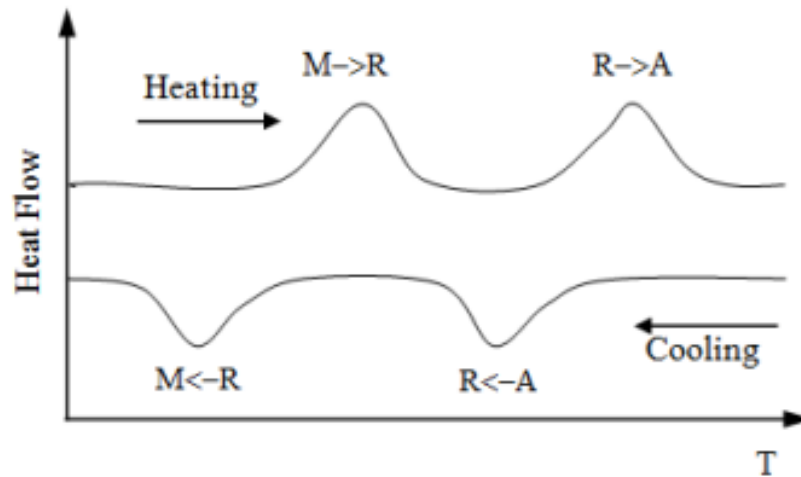


Figure 2.4 Two-stage phase transformation behaviour of NiTi shape memory alloys [10]

As a result of a generated internal stress field related with either distortion or precipitates formation, M_s were discouraged below R-phase transformation temperature, resulting in R phase transition or as known as the rhombohedral phase. [10]. R-phase transition is examined as another martensitic phase transformation as it also exhibits shape memory behaviour. The $A \leftrightarrow R$ transformation exhibits a very small strain while the $A \leftrightarrow M$ and $R \leftrightarrow M$ transformations are characterised by larger transformation strain [11].

2.4 Deformation behavior of NiTi Shape Memory Alloy

Shape memory alloys are unique because of the special deformation behaviours compare to the conventional alloys which are pseudoelasticity and shape memory effect [12]. Depending on the ambient temperature, the martensitic transition in SMAs may exhibit varied mechanical behaviours. Figure 2.5 shows different deformation behaviours of near-equiatomic NiTi with DSC measurement curve at varied temperatures which are (a) pseudoelastic behaviour (PE), (b) shape memory effect (SME) and martensite reorientation (MR).

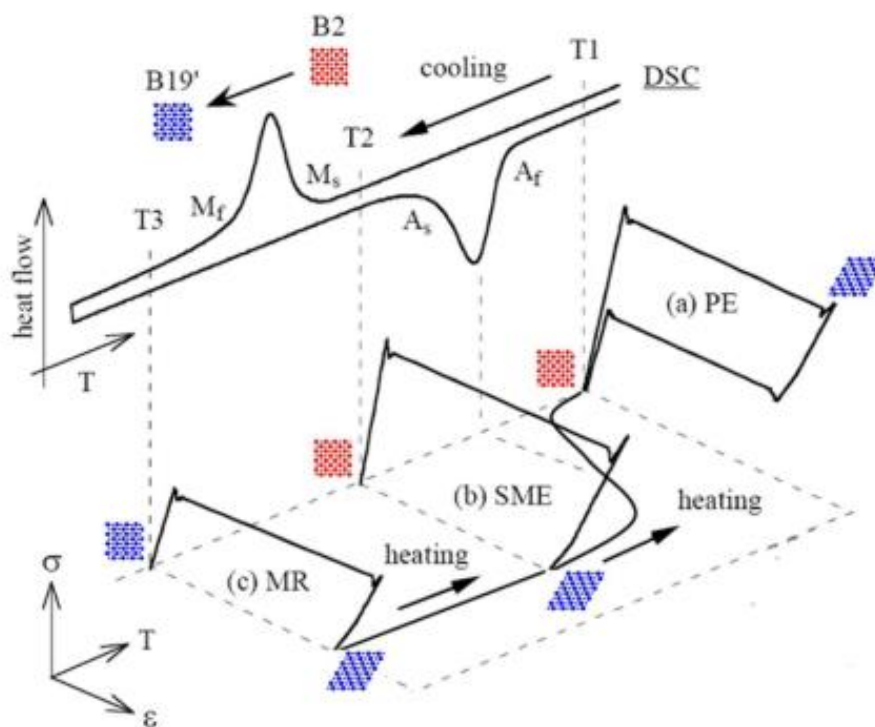


Figure 2.5 Thermomechanical behaviour of a near equiatomic NiTi. The red lattice represents austenite, the blue square-shaped lattice represent the self-accommodated martensite, and the blue parallelogram-shaped lattice represent the oriented martensite [13].

2.4.1 Pseudoelasticity

Pseudoelasticity is a characteristic where the alloys are instantly recovery back to its original shape after the applied load is removed. This behaviour is observed when a thermoelastic martensitic transformation is induced mechanically at above A_f . The stress-induced $A \rightarrow M$ transformation happens at a critical stress over a stress plateau in a “Lüders-like” deformation behaviour with a finite strain in the direction of the load corresponding to the lattice distortion of the martensite[13]. Martensite with variants orientation is produced due to the stress-induced transformation. Then, the oriented martensite will reverse back to austenite when unloading the load because the martensite is thermodynamically unstable at $T > A_f$. [7], [13]. Figure 2.6 show the stress-strain curve for pseudoelastic effect.

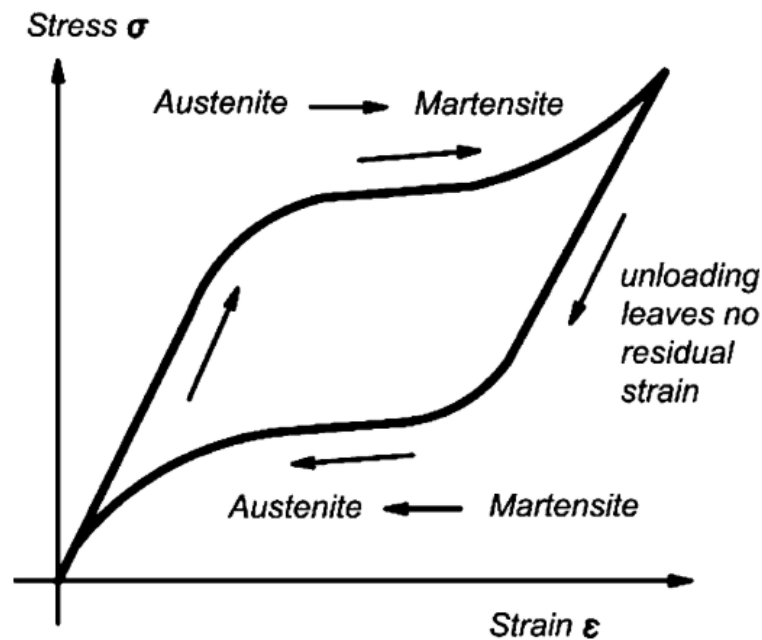


Figure 2.6 Stress-strain curve for pseudoelastic effect [14]

2.4.2 Shape Memory Effect

Shape memory effect refers to an alloy's ability to reshape after being deformed while in the twinned martensitic phase by heating over A_f . At temperatures below M_f , shape memory alloy seems to be completely twinned martensite. If we deformed the alloy at temperature below A_f , it would not reverse back to the original shape instantaneously when unloading the load. When the twinned martensite is deformed by applying load, the twin boundaries will be moved and eliminated then the detwinned martensite can accommodate. So, heat is needed to induce the transformation back and the recovery of the residual strain[7]. Stress-strain-temperature curve of shape memory effect of shape memory alloys is shown in Figure 2.7.

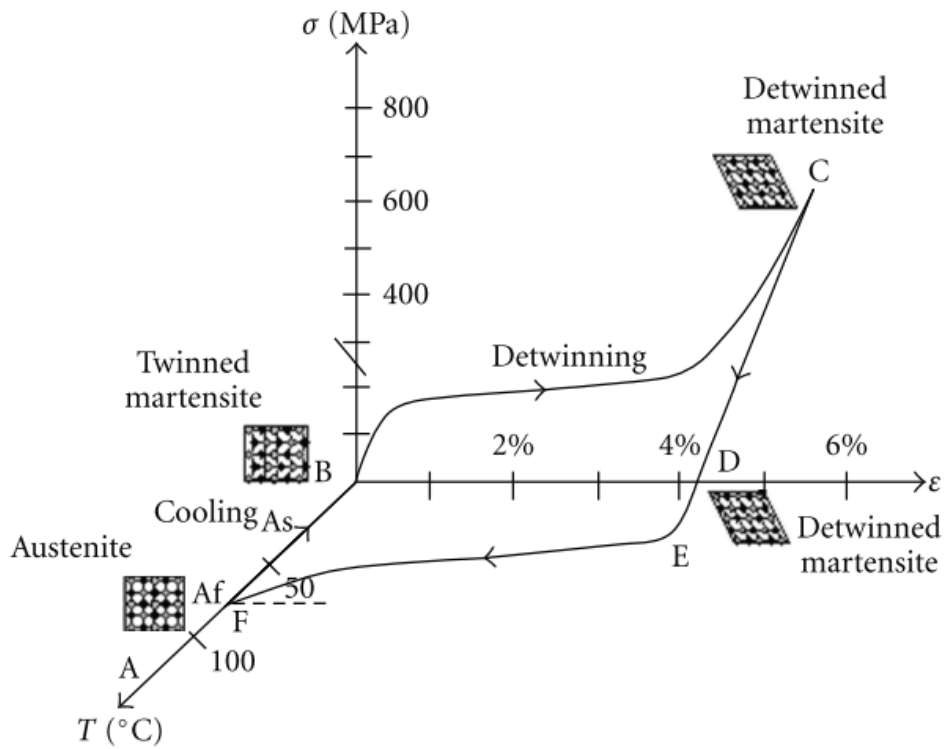


Figure 2.7 Schematic diagram of the shape memory effect [15]

2.5 Functionally Graded NiTi Shape Memory Alloy

Functionally graded NiTi shape memory alloy is a modification of the alloy to have a continuous varying thermomechanical properties by expanding the transformation interval such as transformation stress and temperature window[1]. Three types of graded NiTi alloy were produced so far, such as microstructurally graded, compositionally graded and geometrically graded. Meanwhile, electrical resistance heating, contactless radio frequency heating, gradient furnace heating, laser heating, gradient Ni diffusion, gradient geometry, and gradient pre-straining have all been described in the production of graded shape memory alloy material in order to age the specimen at varying temperatures and durations to induce a progressive microstructural change over its length. Study in the literature reported that gradient annealing and gradient ageing method successfully produced a graded NiTi alloy [2],[12]. Thus, to create the functionally graded of NiTi alloy, ageing temperature, ageing time and the composition of Ni in the alloy need to be considered[9].

From the previous research, there is a gradient temperature furnace that have been introduced. The change in heating element loops throughout the tube length was used to manage the formation of a gradient temperature profile inside the tube furnace. From one end to the other, the space between each heating element loop was gradually increased. The temperature gradient in the heating area was measured with thermocouples placed at certain intervals. Below shows some parameters and results of the previous research that have been studied[2], [16].

Table 2.1 Result of previous research based on different condition and parameter

Parameter/Condition			Result	
Composition of Alloy	Ageing Temperature	Ageing Time	Forward/Reverse Stress Interval	Residual Strain
Ti-50.8% at %Ni	573-773 (K)	2 hours	Not stated	Not Stated
Ti-50.8% at %Ni	400-460 (C)	10 minutes	130 MPa	0.5-1 %

Gradient pseudoelastic behaviour of gradient-aged Ti–50.8at%Ni wire deformed under tension at 25 °C with ageing temperature 400-460°C is shown in Figure 2.8 below. As we can see, the wire initially deformed elastically up to 1% of total measured strain. Then, the stress-induce martensite transformation started at 350MPa and finished at 480Mpa and yielded about 5% transformation strain. The estimated stress gradient for the forward transformation was 2.6GPa while the reverse stress-induced transformation was estimated to be 3.1GPa which is steeper than the forward one. The recovery of strain upon unloading was about 0.6% residual strain.

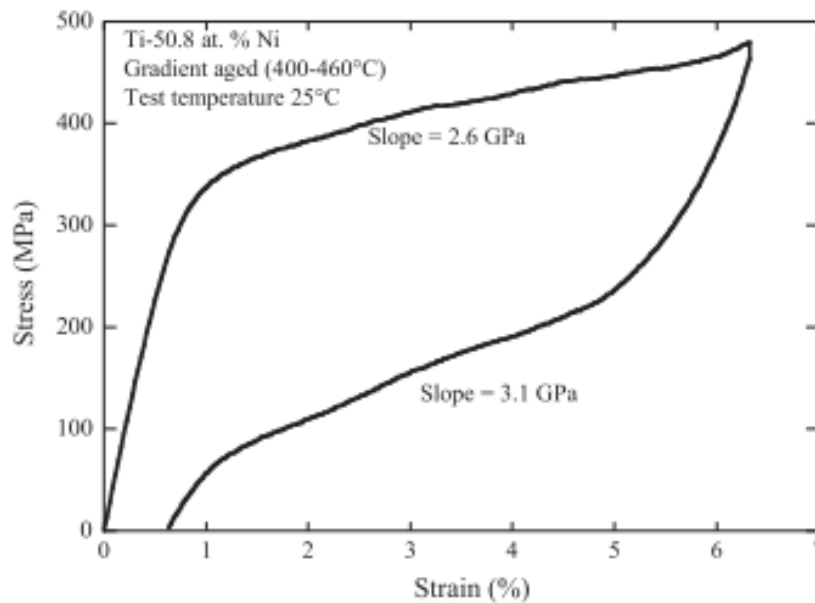


Figure 2.8 Gradient pseudoelastic behaviour of gradient-aged Ti–50.8 at%Ni wire deformed under tension at 25 °C [2].

2.6 Cyclic Deformation

During cyclic loading, the NiTi shape memory alloy exhibited different shape memory behaviour after being deformed based on the number of cycles. The increasing the number of cycles can reduce the stress hysteresis between forward and reverse plateau. The energy dissipated during martensitic transformation, given by loop's area, decreases with the increase of the number of cycles [17]. The forward stress plateau gradually reduced prior to the number of cyclic deformations as shown in Figure 2.9.

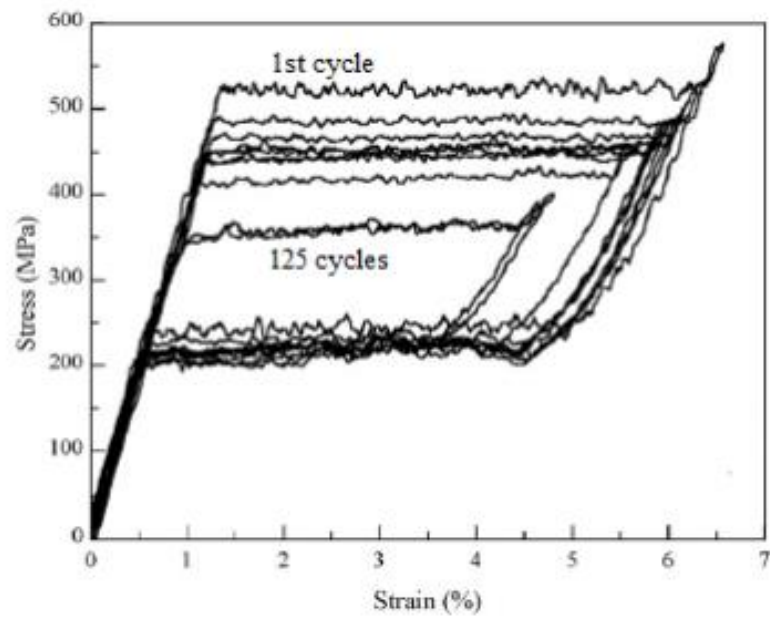


Figure 2.9 Stress-strain curve of pseudoelastic behaviour by cycling load of the Ti-50.8 at% Ni alloy [18]

CHAPTER 3

METHODOLOGY

3.1 Preparation of Gradient Furnace

The idea of the gradient furnace was obtained from the previous study as shown in Figure 3.1 and it was rebuilt to use for gradient temperature ageing treatment. Kanthal heating element was wound on the surface of the ceramic tube to heat up the furnace. The creation of the gradient temperature profile inside the furnace was controlled by the loop of the heating element. The number of loops was increased from one end to the other end. When the number of loops increase, it has many heating elements per length compared to the other end. Figure 3.2 shown the schematic diagram of heating element loop on the ceramic tube.

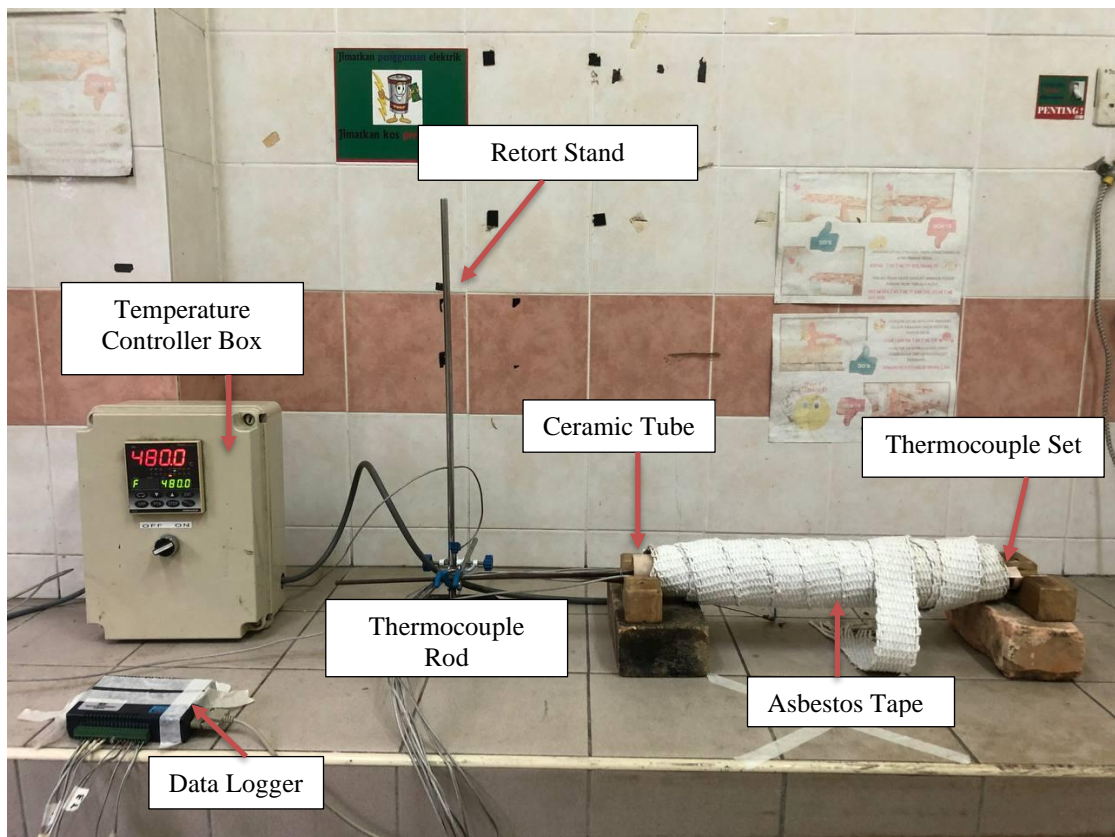


Figure 3.1 Gradient temperature furnace setup

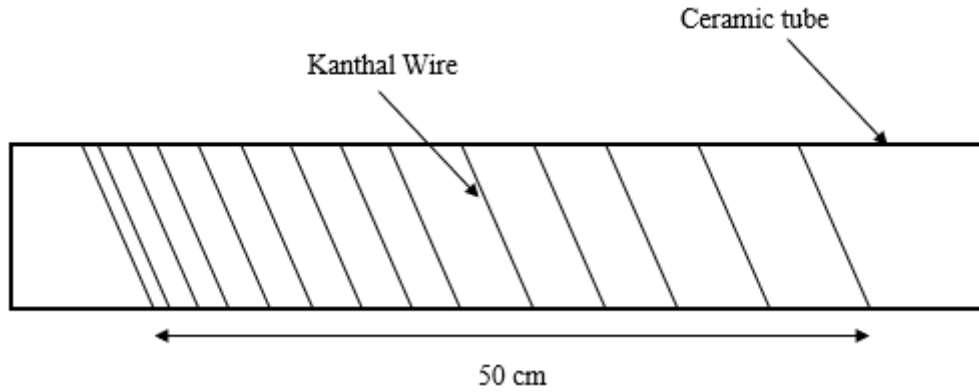


Figure 3.2 Schematic diagram of heating element loop of furnace

Heating element was connected to the output wire from the temperature controller box. When the heating process started, the heat from the heating element heated up the ceramic tube continuously. The temperature at the end with the large number of loops is higher compared to the other end. The temperature throughout the tube is reducing proportionally with the number of the heating loops and introducing a linear gradient temperature area. One end of the tube with the few loops was opened to ambient end to have an efficient heat transfer along the tube furnace.

At the high temperature end of the tube furnace, one thermocouple directly from the temperature controller box was attached. It is used to control and monitor the highest temperature of the furnace to be as the same as the temperature set from the temperature controller. The end with the high temperature was purposely closed to avoid rapid fluctuation of controlled temperature due to the ambient air. In addition, asbestos insulator tape was used to wrap the ceramic tube with the heating element to avoid the heat loss to the surrounding and improve the accuracy of the temperature reading.

The gradient temperature profile of the furnace was measured and recorded by eight thermocouples type-k. The thermocouples were mounted on a metal rod and set to be 10mm equal interval of the length as illustrated in Figure 3.3. The metal rod was clamped by a retort stand and positioned it at the center of the furnace tube diameter. So, the best gradient temperature profile of the furnace can be determined by moving the fixture to adjust the location of the rod position inside the tube furnace. Each thermocouple was connected to the data logger channel and “Navigator” software with Data Logger Utility from Advantech was used to monitor and record the temperature profile.

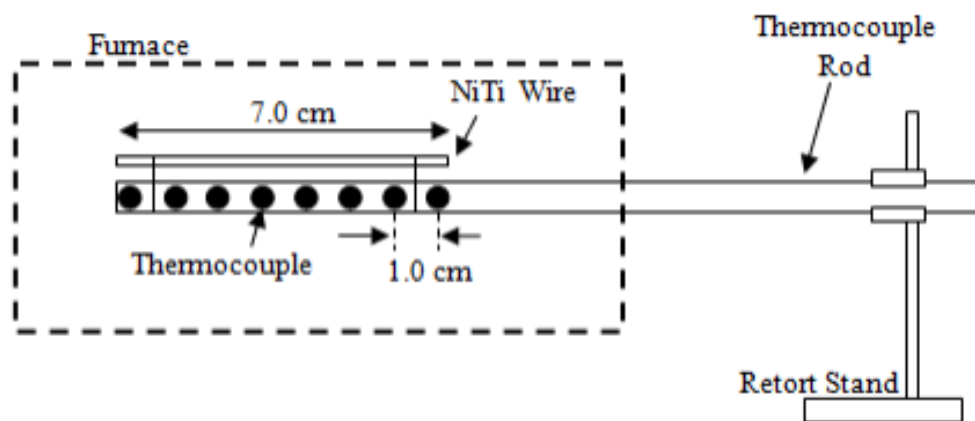


Figure 3.3 Schematic diagram of thermocouples and NiTi wire location

3.2 Ageing Treatment of NiTi Shape Memory Alloy

Ni-rich Ti-51at%Ni wire with 0.5mm diameter was used to undergo ageing treatment. There are two types of ageing treatment that were proceeded in this section which is isothermal ageing treatment and gradient temperature ageing treatment. The purpose of this study is to observe the effect of the ageing treatment to the NiTi alloy behaviour in two different ageing treatment condition. In both methods, the two group of specimens were prepared which are 70mm length for tensile test and 3mm length for thermal phase transformation test using DSC instrument.

3.2.1 Isothermal Ageing Treatment

For the isothermal specimens, Ni-rich Ti-51at%Ni wire with 0.5mm diameter was undergo initial solution treated using single zone MTI tube furnace (GSL-1100X) as shown in Figure 3.4 at 900°C for 30 minutes in argon environment. The argon condition was used to avoid oxidation and eliminate the prior effect of heat treatment. Then the wire was quenched into cold water to avoid the oxidation effect. The specimens were aged at 3 different temperatures (400°C,430°C,450°C) at 10 minutes in single zone tube furnace.



Figure.3.4 MTI tube furnace used for initial solution treatment and isothermal ageing treatment

3.2.2 Gradient Temperature Ageing Treatment

The gradient ageing specimens were cut into 70mm length of Ni-rich Ti-51at%Ni wire with 0.5mm diameter. The specimens were initially solution treated same as isothermal specimens at 900°C for 30 minutes in argon environment and followed by quenching. Then, the specimens were inserted through the tube furnace within the specified temperature range for ageing. The temperature gradient was set to be within 400°C-460°C and 8 thermocouples were used to monitor the heating location positioned at 10mm intervals. “Navigator” software with Data Logger Utility from Advantech was used to monitor and record the temperature profile.

The real time plot of the temperature inside the furnace was recorded. The furnace took about 4 minutes to ramp up from the room temperature to the set value before the temperatures are stabilized roughly $\pm 5^{\circ}\text{C}$. Once the temperature profile fully stabilized, the specimen was loaded into the gradient furnace and heat up for 10 minutes.

3.3 Thermal Phase Transformation Behaviour Analysis

The objective of this experiment is to observe and analyse the transformation temperature of Ni-rich Ti-51at%Ni wire by means of Differential Scanning Calorimeter (DSC). The DSC analysis of the transformation behaviour was conducted by using a TA Q20 differential calorimeter device as shown in Figure 3.5. Samples were cut into 3-5mm length and weight around 6-10 mg and placed in the DSC device. Samples were heated up to 120°C and cooled until -120°C with the constant heating and cooling rate of 10°C /min. Liquid nitrogen was used as a coolant to cool down the specimens until the required temperature. Then, the specimens were reheated until 120°C with the same heating rate of 10°C /min. Critical temperature of the specimens of isothermal ageing and gradient ageing are analysed.



Figure 3.5 TA Q20 differential calorimeter device

For the gradient sample, it was cut into 3mm at specific position based on the temperature profile of the gradient sample. Figure 3.6 schematically shows the specific position of the gradient sample that have been cut for DSC measurement.

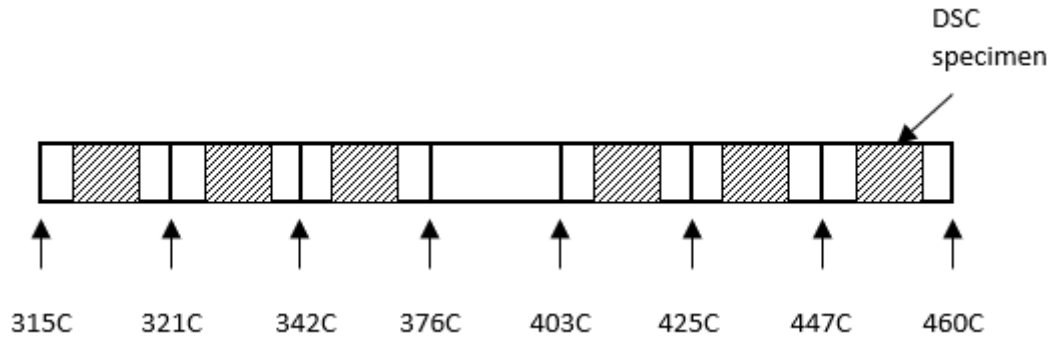


Figure 3.6 Sectioning of DSC specimen from the total length of gradient aged NiTi wire

3.4 Deformation Behaviour Analysis

The specimens from initial solution treatment, isothermal ageing and gradient ageing were tested by conducting a tensile test. Tensile test was carried out to evaluate the pseudoelastic behaviour of the aged NiTi alloy in an open air at room temperature. Tensile tests were carried out by using a universal testing machine (UTM) model 3367 from Instron equipped with 30kN load cell. Setup of the experiment was shown in Figure 3.6. The gauge length of the wire specimen was set to be 40mm length with 0.5mm wire diameter. During the tensile test, the specimen was elongated up to 4.0mm (10% strain) and unloaded back to zero strain with loading rate 1mm per minutes.

When the wire was initially strained, it will undergo the forward plateau and unload it back to undergo reverse plateau. Each of the stress-strain of the specimens are recorded and their pseudoelastic behaviour are analysed. Then, the test was continued with the cyclic tensile test by load and unload the loading repeatedly. The effect of cyclic loading-unloading was observed and analysed in cyclic stress-strain of the specimens. All the specimens were tested under the same condition in an open air at room temperature of 27°C.



Figure 3.7 Tensile Test Setup (Instron 3367 Universal Tensile Machine)

CHAPTER 4

RESULT AND DISCUSSION

4.1 Gradient Furnace Result

Figure 4.1 shows the real time plot of the gradient furnace that have been achieved. It took around 4 minutes for the furnace to ramp up from room temperature to the specified setting, and then the temperature stabilized around $\pm 5^{\circ}\text{C}$.

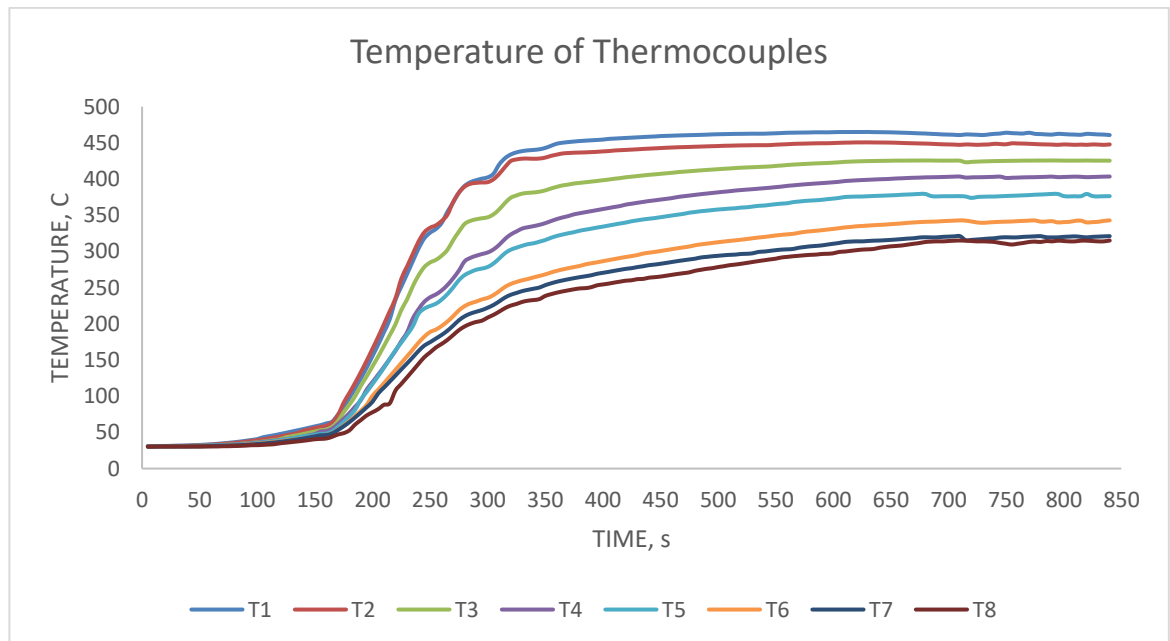


Figure 4.1 Real time plot of temperature recorded in gradient furnace

Figure 4.2 shows the temperature profile across the gradient heating space that have been recorded by eight thermocouples. Based on the temperature profile, the temperature interval between the thermocouples is varied. The interval that are required for the optimum gradient ageing is from 400°C to 460°C . But this gradient furnace has larger interval which is starting at the highest temperature 460°C at one end and temperature drop until 315°C at the other end. So, this larger range of temperature gradient can affect the pseudoelastic behaviour of the functionally aged NiTi wire.

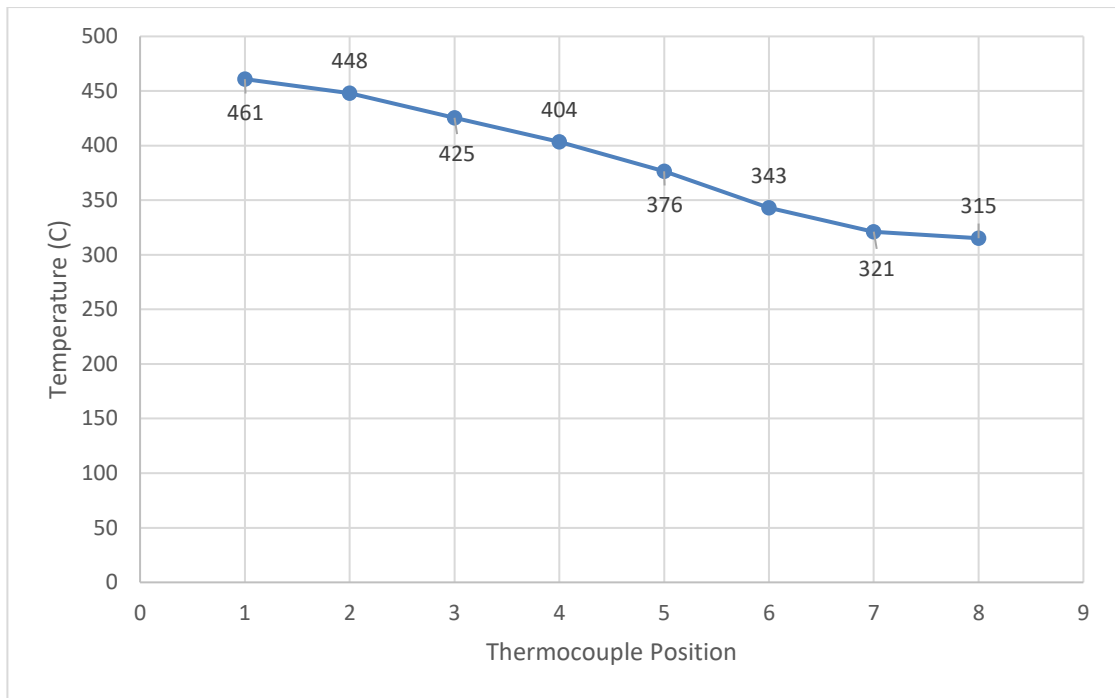


Figure 4.2 Temperature profile along recorded by the thermocouples

4.2 Thermal Transformation Behaviour Analysis

4.2.1 Thermal Transformation of Isothermal Ageing

Figures 4.3, 4.4 and 4.5 show the variation of transformation peak from the DSC test at ageing temperature 400°C, 430°C and 450°C for 10 minutes, respectively. The transformation peaks temperature appeared on both heating and cooling curve. On the cooling curve, the transformation peak represented as a transformation of austenite to martensite structure. However, there are two peaks occurred on cooling curve when the specimen was aged isothermally 430°C. The first peak indicated the transformation of austenite to R-phase structure and followed by second peak that indicated the transformation of R-phase to martensite structure. This was noticed that the transformation peaks temperature changed as the ageing temperature increased. This refers to the precipitates of Ti_3Ni_3 started to form and induce by various ageing condition.

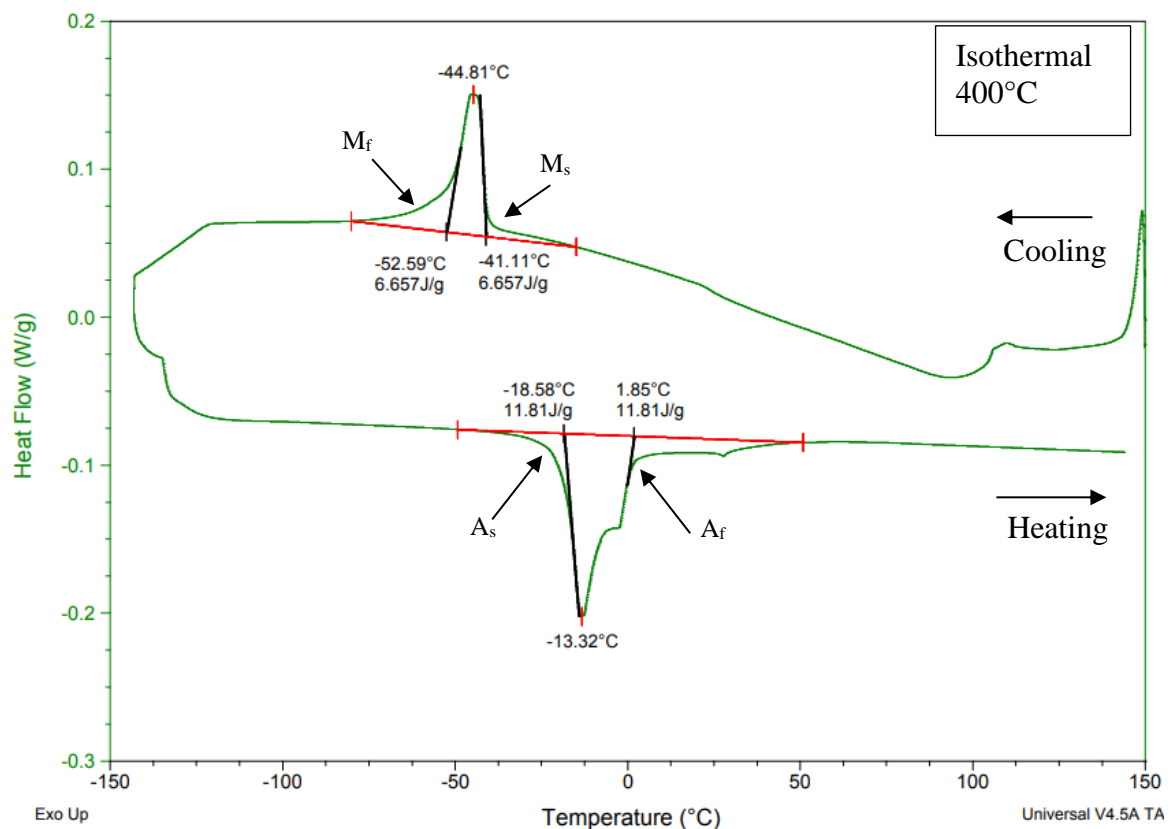


Figure 4.3 Thermal transformation behaviour of Ti-51at%Ni alloy after isothermally aged at 400°C for 10 minutes