FUNCTIONALLY GRADED NiTi SHAPE MEMORY ALLOYS BY AGEING AND PARTIAL ANNEALING

MUHAMMAD NAQIB BIN NASHRUDIN

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by

MUHAMMAD NAQIB BIN NASHRUDIN

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LIST OF ABBREVIATIONS

SMA	Shape Memory Alloy
DSC	Differential Scanning Calorimeter
UTM	Universal Testing Machine
NiTi	Nickel Titanium
Ti_3Ni_4	Nickel Titanium precipitate
$TiNi_3$	Nickel Titanium precipitate
Ti_2Ni_3	Nickel Titanium precipitate
Au-Cd	Gold-Cadmium
Cu-Zn	Copper-Zinc
Cu-Zn-Al	Copper-Zinc-Aluminum
Fe-Mn-Si	Iron-Manganese-Silicon
Cu-Al-Ni	Copper-Aluminum-Nickel
CsCl	Caesium Chloride

LIST OF SYMBOLS

%	Percentage
T	Temperature
TC	Thermocouple
CO_2	Carbon dioxide
ε	Strain
M_s	Martensite start
M_f	Martensite finish
M_d	Martensite stabilization
A_s	Austenite start
A_f	Austenite finish
B2	Austenite phase
B19’	Martensite phase
R	R-phase
ΔH	Changes of enthalpy energy
ΔS	Changes of entropy energy
σ_f	Forward transformation stress
σ_r	Reverse transformation stress
$\Delta\sigma_f$	Forward transformation stress interval
$\Delta\sigma_r$	Reverse transformation stress interval

PENCERUNAN BERFUNGSI ALOI INGATAN BENTUK NiTi OLEH PENUAAN DAN PENYEPUHLINDAPAN SEPARA

ABSTRAK

Tingkah laku bentuk bentuk aloi NiTi menyampaikan daya tetap apabila beban digunakan di atasnya. Oleh itu, had untuk sebarang keadaan progresif atau perantaraan kawalan membuat aloi berfungsi hanya untuk aplikasi tindakan diskret. Untuk membolehkan kawalan yang lebih baik dalam aplikasi penggambaran, NiTi yang berfungsi secara berperingkat dicipta dengan memperkenalkan rawatan haba kecerunan di sepanjang aloi. Penyelidikan yang sebelum ini menunjukkan bahawa kecerunan rawatan penyepuhlindapan dan hamper equatomik aloi NiTi menghasilkan kemerosotan lereng dataran curam manakala rawatan kecerunan dalam aloi Ni-kaya menghasilkan ketengangan pemulihan yang baik pada transformasi terbalik. Kajian ini menerokai kesan penyepuhlindapan separa dan penuaan secara serentak menggunakan Ti-50.9at% Ni wayar yang digulung ke arah tingkah laku transformasi fasa martensit dan untuk mewujudkan wayar NiTi yang berfungsi secara berperingkat dengan meletakan rawatan haba kecerunan sepanjang wayar. Spesimen dipanaskan pada julat suhu 300 ℃ hingga 550 ℃ selama tempoh 10, 20, 30 dan 60 minit dalam argon. Tingkah laku transformasi fasa martensit termal dicirikan dengan menggunakan imbasan calorimeter berbeza dan tingkah laku ubah bentuk memori telah dikaji dengan menggunakan mesin tegangan sejagat.

Eksperimen ini adalah untuk meneroka konsep baru NiTi yang dinilai secara berfungsi. Profil suhu kecerunan dibuat secara tempatan oleh laser CO2 sepanjang wayar. Julat suhu yang berkesan untuk profil kecerunan untuk Ti-50.9at% Ni telah ditentukan pada 400 ℃ - 525 ℃ pada tempoh yang berbeza. Dua spesimen wayar diuji

dengan tempoh yang sama dan tempoh yang berlainan. Telah didapati bahawa spesimen dengan tempoh yang berbeza dalam wayar tunggal menghasilkan selang tekanan yang lebih tinggi. Satu spesimen dipanaskan secara tempatan pada suhu yang berbeza dan tempoh yang berbeza. Suhunya adalah 400 ℃ selama 10 minit, 475 ℃ selama 30 minit dan 525 ℃ selama 20 minit. Semasa ditarik bentuknya, wayar yang dipanaskan secara lokal dengan tempoh yang berlainan memperlihatkan selang tekanan ke hadapan yang lebih tinggi iaitu 301 MPa dan selang tekanan terbalik sebanyak 265 MPa. Pengubahan yang dihasilkan sekitar 10% dan sisa ketegangan adalah 1%. Kaedah rawatan haba ini menghasilkan hasil positif dalam mewujudkan pelbagai tekanan dataran untuk kecerunan aloi NiTi berfungsi.

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ABSTRACT

A shape memory behavior of NiTi alloy delivers constant forces when load is applied on it. Thus, the limitation for any progressive or intermediate state of control makes the alloy to function only for discrete action application. To enable a better control in actuation applications, a functionally graded NiTi is created by introducing a gradient heat treatment along the alloy. Recently, few works have been reported on gradient annealing treatment of cold-worked near-equatomic NiTi alloy to produce steeper stress plateau slope while gradient ageing treatment in Ni-rich alloy produces good recovery strain on the reverse transformation. This study explored the effect of partial annealing and ageing treatment of a cold-worked Ti-50.9at%Ni wire towards martensite phase transformation behavior and to create multiple steps of stress plateau NiTi wire by localizing the heat treatment along the wire. Specimen used in this study was a Ti-50.9at%Ni wire with a diameter of 2 mm. The specimens were heated at temperature range of 300℃ to 550℃ for duration of 10, 20, 30 and 60 minutes in argon atmosphere. The thermal martensite phase transformation behavior was characterized by using a differential scanning calorimeter (DSC) and the shape memory deformation behavior was studied by using a universal testing machine (UTM).

The experimental work explored a concept of functionally graded NiTi materials. The gradient temperature profile was locally created by CO_2 laser along its length. The effective temperature range for gradient profile for functionally graded cold-worked Ti-50.9at%Ni was determined at 400℃ - 525℃ at different duration.

Two specimens of wire were tested with same temperature and different duration. It was found that the specimen with different duration in a single wire produced higher stress interval. The temperatures are 400℃ for 10 min, 475℃ for 30 minutes and 525℃ for 20 minutes. During tension, the localized heat treated with different duration exhibited forward stress interval which is 301 MPa and reverse stress interval of 265 MPa. The deformation yielding around 10% transformation strain and the residual strain is 1%. The localized laser heat treatment method executes a positive result in creating multiple steps of stress plateau of functionally graded behavior of NiTi alloy.

CHAPTER ONE

INTRODUCTION

1.1 Research Background

Shape memory alloy (SMA) is a unique material in metallic alloys group. The alloy has the capability of memorizing its original shape after being deformed beyond elastic limit. This shape recovery can be achieved instantly upon the release of the load or heated to above its critical temperature. During tension, this alloy experiences huge strain and can return to its original length upon unloading due to its ability to undergo reversible thermoelastic martensite phase transformation where the phase changes between austenite and martensite that can be induced by temperature or stress input (Otsuka & Ren, 2005). Austenite phase is stable at high temperature also known as the parent phase and the martensite phase that is stable at low temperature (Ozbulut, Hurlebaus, & Desroches, 2011). In some SMAs, this transformation can also be stimulated by magnetic (Mohd Jani, Leary, Subic, & Gibson, 2014) and electrical (Cui, Song, & Li, 2010). This transformation phenomenon can be classified as shape memory effect and pesudoelasticity. Shape memory effect refers to recovery of a deformed alloy to its original shape and size when heated to beyond its transformation temperature. Pseudoelasticity refers to the instantaneous recovery of the deformed alloy upon the release of load (Elibol & Wagner, 2015).

Figure 1.1 shows the phenomenon of the shape memory behavior of SMA in real life. Initially, the alloy rod is in straight condition. When the rod is bend at below its martensite start temperature (martensite phase), the rod remains in deformed condition upon the removal of the load, demonstrating that the deformation is non-elastic, (refer (a)). When the bent rod is heated up to above its reverse transformation

temperature (austenite phase), it returns to its original shape instantaneously. This behavior is known as the shape memory effect. At the same situation, if the rod is deformed at high temperature which is above the transformation temperature for shape recovery, the rod will recover to its original position spontaneously upon unloading, as shown in (b). This is known as pseudoelasticity.

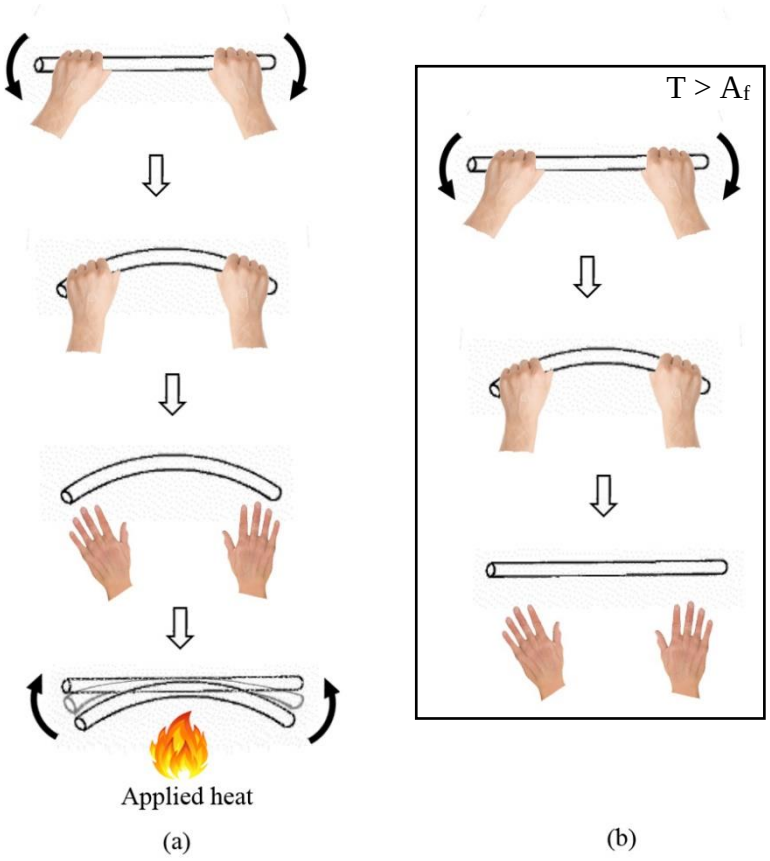

Figure 1.1. A graphic illustration of (a) shape memory effect and (b) pseudoelasticity exhibited by shape memory alloys.

There are few numbers of alloys that exhibit shape memory behavior, such as the binary Ni-Ti, Au-Cd, Cu-Zn and others, the tertiary iron-based and copper-based which are Cu-Zn-Al, Fe-Mn-Si and Cu-Al-Ni. Among all, NiTi is the most widely used for its superior shape memory behavior and much more significantly best for most applications. This is because others are impracticability and poor thermos-mechanic

performance compared to NiTi-based SMAs (Ezaz, Wang, Sehitoglu, & Maier, 2013; Lobo, Almeida, & Guerreiro, 2015).

Tensile deformation behavior of a shape memory alloy is illustrated in Figure 1.2. Thermoelastic martensitic transformation is also a mechanical deformation process. Therefore, it is possible to induce the transformation by the application of load, as is by the variation of temperature. During tensile, initially the alloy at austenite phase exhibits the linear elastic, stress plateau and hysteresis on the recovery. The upper level stress plateau resembles the forward phase transformation from austenite to stress-induced martensite, while the low level stress plateau indicates the subsequent reverse phase transformation (Seo, Kim, & Hu, 2015).

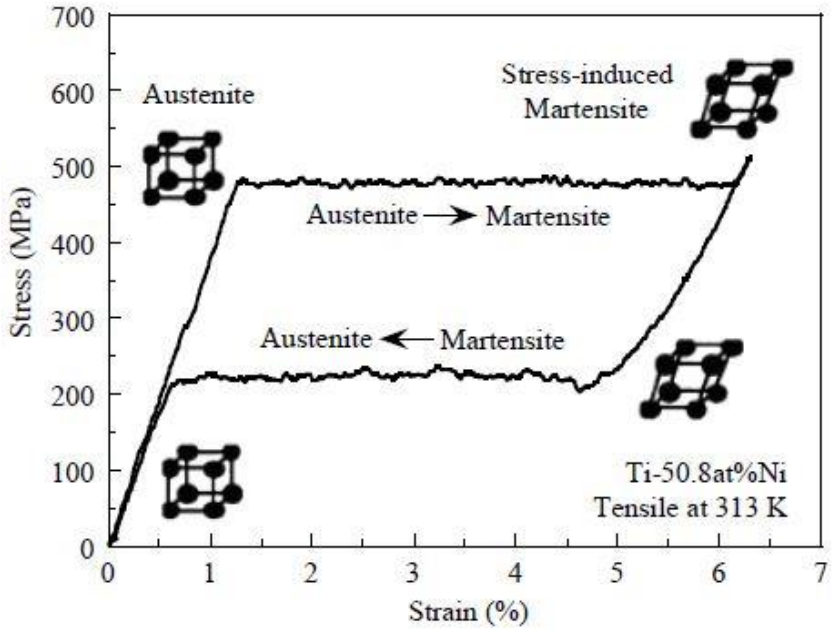

Figure 1.2. Stress-strain curve showing the pseudoelastic deformation behavior.

SMAs have the ability to recover from large deformation well beyond the normal elastic strain limits of metals. This behavior provides the material the ability to produce a mechanical work output without complex mechanical designs. Owing to the

special feature, shape memory alloys have been used widely around the world in various engineering applications and technical applications due to its unique characteristics such as orthodontic wire (Fernandes et al., 2011), actuators (Guo et al., 2015; Leary et al., 2013; Mohd Jani et al., 2014), sensors (Nespoli et al., 2010), aerospace (Barbarino et al., 2014), and biomedical applications (Morgan, 2004; Petrini & Migliavacca, 2011).

Although SMAs are widely used in many applications nowadays. A SMA can be used as or in actuators or sensors, based on the principle work output that responses to an external stimulation (Choudhary & Kaur, 2016). SMA actuators are triggered by application either stress or temperature, thus the window size of the control parameter is an important design parameter for SMA actuator applications. However, the alloys exhibit small temperature changes between the transformation phases where the bulk of the phase transition occurs within a narrow range of ~5℃ (Miller & Lagoudas, 2001). The stress interval of a stress-induced martensite transformation is fundamentally determined by the temperature changes interval between the start and finish transformation temperatures. Due to the small transformation temperature interval, this transformation occurs with a similar stress value that leading to zero transformation stress interval (Mahmud, Liu, & Nam, 2007). In stress-strain curve, this phenomenon is identified by a flat stress plateau over a large deformation strain of 5-8% which explains the instantaneous change of alloy over the single threshold value of stress and temperature (Shariat, Liu, & Rio, 2014). The sudden responds during deformation and recovery limits the applicability of shape memory application, especially for a case which requires intermediate or continuous control of the output. Industries found difficulties in controlling the motion of the shape memory apparatus as it requires a precise control in external input signals, either temperature or stress.

To solve this issue, it is necessary to expand the stress interval needed for stress-induced martensitic transformation along the length of the alloy to meet with the application requirement. There are three major ways that have been reported in literature to create functionally graded for the SMA such as microstructurally graded, compositionally graded and geometrically graded (Shariat, Meng, Mahmud, et al., 2017; Shariat, Meng, & Mahmud, 2017). Functionally graded and stepped alloy will be explored in this thesis by local heat treatment along an alloy wire. It would be a great advantage if step series of gradient microstructure properties can be programmed along the shape memory alloy.

1.2 Problem Statement

Modifying the stress-induced martensite transformation stress plateau was possible via partial anneal treatment and ageing (Mahmud et al., 2007; Razali & Mahmud, 2015). Figure 1.3 illustrates the stress-strain curve of deformation behavior of gradient annealed of Ti-50.5at%Ni alloy. The gradient behavior yielded a good gradient of 4.7 GPa stress slope on the forward martensite phase transformation and 8.6 GPa stress slope on the reverse. The stresses and strains were calculated based on engineering strain. The stress interval achieved by the functionally graded NiTi alloy for forward stress-induced martensite transformation was 280MPa, while for the reverse transformation was 300MPa. However, the recovery of strain on the reverse transformation was not fully achieved.