

2014

# Factors influencing phase transformations of NiTi wires for civil structure applications

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## Publication details

Fang, H, Wong, MB & Bai, Y 2014, 'Factors influencing phase transformations of NiTi wires for civil structure applications' in ST Smith (ed.), *23rd Australasian Conference on the Mechanics of Structures and Materials (ACMSM23)*, vol. I, Byron Bay, NSW, 9-12 December, Southern Cross University, Lismore, NSW, pp. 205-210. ISBN: 9780994152008.

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## FACTORS INFLUENCING PHASE TRANSFORMATIONS OF NITI WIRES FOR CIVIL STRUCTURE APPLICATIONS

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### ABSTRACT

NiTi shape memory alloys are smart materials which can be used as actuators for controls and strengthening of civil structures. The application of NiTi shape memory alloys as actuators is based mainly on the shape memory effect that arises from the phase transformation between martensite and austenite. In the design of structural actuator using NiTi wires, it is important that the characteristics of phase transformation in NiTi are understood and measured accurately. In this study, the effects of heat treatment conditions and heating/cooling rates on the phase transformation behaviours of NiTi alloys were investigated through the use of differential scanning calorimetry (DSC) methods. The results show that heat treatment must be applied to NiTi shape memory alloys in order to achieve satisfactory shape memory effect. Besides, it was found that the phase transformation temperatures of NiTi alloys are dependent on the heating/cooling rate, a variable which is important in determining the temperatures of structural elements at high temperatures. Upon heating at a lower rate, NiTi alloys tend to transform from martensite to austenite at lower temperatures. Likewise, NiTi shape memory alloys cooled at a lower rate transform to martensite phase at higher temperatures. Therefore, the transformation temperatures under different heating rates used for actuator design should be obtained under the working conditions for the intended usage.

### KEYWORDS

NiTi shape memory alloys, heat treatment, heating/cooling rate, phase transformation behaviour.

### INTRODUCTION

Extensive research has been conducted on shape memory alloys for applications to civil structures. Shape memory alloys (SMAs) applications rely on the material's exhibition of shape memory effect and pseudoelasticity due to the crystallographic changes that SMAs transform between martensite and austenite. This phase transformation is characterized by four transformation temperatures: martensite start ( $M_s$ ), martensite finish ( $M_f$ ), austenite start ( $A_s$ ) and austenite finish ( $A_f$ ). An intermediate R phase appears in the cooling cycle between austenite and martensite phases if the SMAs are annealed at a temperature below the recrystallization temperature. Among all the available shape memory alloys, NiTi SMAs have the greatest potential for civil structure applications due to their advanced thermomechanical performances. Therefore, NiTi wires were adopted for the current study. In the



design and analysis of structures using NiTi SMA, it is necessary that the phase transformation temperatures are measured accurately.

The phase transformation temperatures were found to be affected by a number of factors such as annealing temperature and cold work (e.g. Miller and Lagoudas 2001; Sadiq et al. 2010; Abel et al. 2004). In those studies, the phase transformations temperatures of NiTi alloys annealed at temperatures between 300°C and 700°C were investigated. In this study, NiTi wires without heat treatment were tested together with those that are heat treated at 350°C and 800°C. A wider temperature range was used for a better understanding on the effect of heat treatment in order to determine the optimal heat treatment conditions for different applications.

In addition to the heat treatment effect, the influence of heating/cooling rate on the transformation temperatures of NiTi is also investigated in this study. The phase transformation temperatures of NiTi alloys are dependent on the heating/cooling rate according to the kinetics of solid state phase transformation as illustrated by Liu et al. 2004, 2007. In many civil engineering applications, the NiTi SMAs are subject to heating under varying rates (e.g. Li et al. 2006; Sadiq et al. 2013), a variable which is important in determining the phase transformation temperatures of SMAs at high temperatures. By knowing the exact transformation temperatures, the time to activate the SMAs and accurate structure behaviours can be predicted. No previous research of this kind has ever been carried out.

## EXPERIMENTAL PROCEDURE

### Material and Preliminary Heat Treatment

The experiments were performed on a commercial NiTi wires produced by Memry. The as-received wire was non-annealed with 40% cold drawn to a diameter of 0.95mm. The wires were first cut into specimens shown in Figure 1. Each specimen has a weight of approximately 8mg. Prior to the characterization by differential scanning calorimetry (DSC), the samples were annealed at 350°C and 800°C for one hour and then quenched into cold water.



Figure 1. DSC test wire specimens

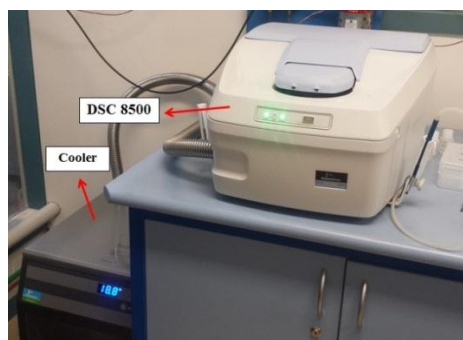


Figure 2. Equipment system of DSC tests

### DSC Test Procedures

The DSC tests were conducted on Perkin Elmer DSC 8500 shown in Figure 2, between -50°C and 180°C to measure sample heat flow. Different heating and cooling rates were used to examine the

heating/cooling rate effect on the phase transformation of NiTi. Three groups of samples which are identified by different annealing conditions were tested and heat flows of samples were recorded. The detail of the sample groups and temperature rates is summarized in Table 1. According to ASTM standard E1269 for material testing by DSC, the test procedures were determined as below:

- (1) The specimens were cooled to  $-50^{\circ}\text{C}$  and kept for 4 min to achieve thermal equilibrium;
- (2) The specimens were then heated to  $180^{\circ}\text{C}$  at a specified rate listed in Table 1;
- (3) The temperatures of specimens were held at  $180^{\circ}\text{C}$  for 4 min; and
- (4) The specimens were cooled down to  $-50^{\circ}\text{C}$  at a rate according to Table 1.

Table 1. Test specimens and heating/cooling rates

Sample group	Heat treatment conditions	Heating/cooling rate ( $^{\circ}\text{C}/\text{min}$ )
No.1	No heat treatment	20, 15
No.2	$350^{\circ}\text{C}$	5, 10, 15, 20, 25
No.3	$800^{\circ}\text{C}$	5, 10, 15, 20, 25

## RESULTS AND DISCUSSIONS

Figures 3 and 4 display the DSC curves of sample groups 1, 2 and 3 obtained under the heating/cooling rates of  $15^{\circ}\text{C}/\text{min}$  and  $20^{\circ}\text{C}/\text{min}$  respectively. From Figures 3 and 4, it can be seen that cold drawn samples without annealing don't exhibit any phase transformation in the heating and cooling cycles. With heat treatment at low temperature of  $350^{\circ}\text{C}$ , a small build-up of heat flow was identified as a result of the phase transformation from martensite (M) to austenite (A). As the annealing temperature increases to  $800^{\circ}\text{C}$ , peaks of heat flow curves due to phase transformation are more pronounced than those of curves for a sample annealed at  $350^{\circ}\text{C}$ . In the heating cycle, the NiTi alloys annealed at  $350^{\circ}\text{C}$  transform into austenite approximately from  $46^{\circ}\text{C}$  to  $86^{\circ}\text{C}$  while the phase change in NiTi heat treated at  $800^{\circ}\text{C}$  starts at  $77^{\circ}\text{C}$  and finishes at  $107^{\circ}\text{C}$ . At the cooling stage, the material heat treated at  $350^{\circ}\text{C}$  transforms to R phase first. At this heat treatment temperature, the R-phase is not obvious from observing the heat flow curve in Figure 3(b) and 4(b). In another heat treatment temperature of  $450^{\circ}\text{C}$  (the results are not included in this paper) the R-phase has been shown to be quite significant. This is consistent with the study by Sadiq et al. 2010. The R-phase appears in SMAs annealed at  $350^{\circ}\text{C}$  at around  $70^{\circ}\text{C}$  and the material completely transforms into martensite phase at  $-17^{\circ}\text{C}$ . The transformation from austenite to martensite in samples annealed at  $800^{\circ}\text{C}$  occurs between  $39.5^{\circ}\text{C}$  and  $66^{\circ}\text{C}$  which are a lot higher than those obtained in samples annealed at lower temperature. Therefore, the four phase transformation temperatures as  $M_s$ ,  $M_f$ ,  $A_s$  and  $A_f$  tend to increase upon the increment of annealing temperatures. This agrees with the results in literature (e.g. Sadiq et al. 2010; Miller and Lagoudas 2001).

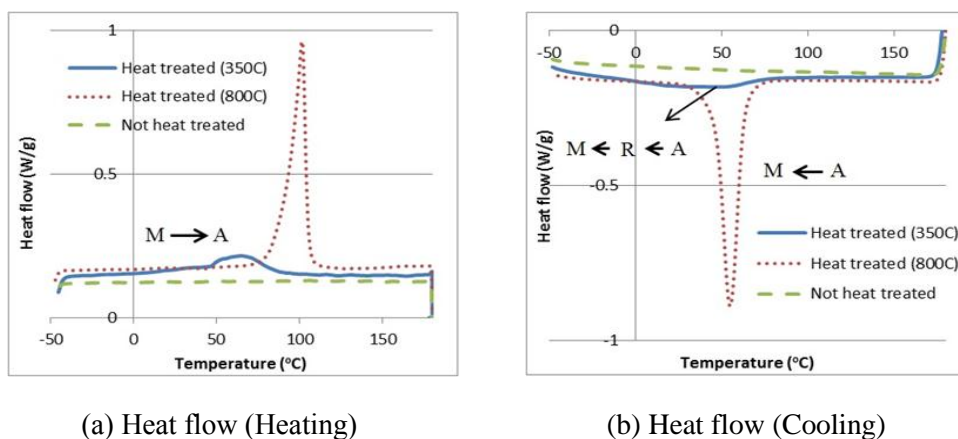
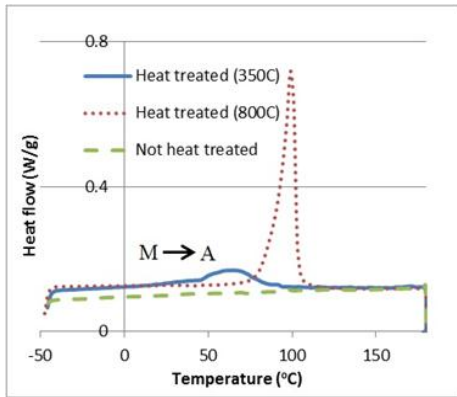
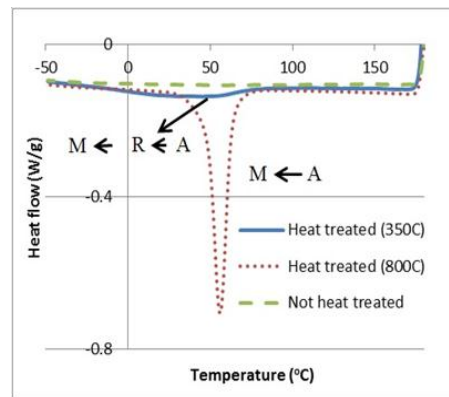


Figure 3. DSC curves ( $20^{\circ}\text{C}/\text{min}$ ) of samples annealed at different temperatures



(a) Heat flow (Heating)

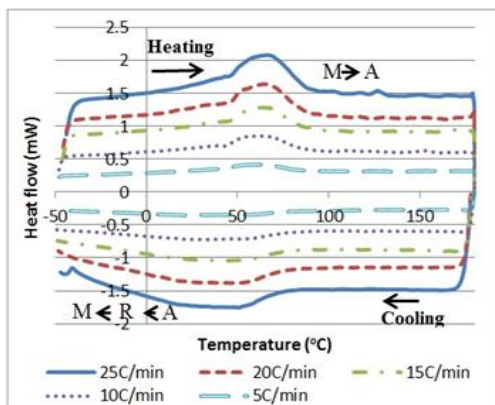


(b) Heat flow (Cooling)

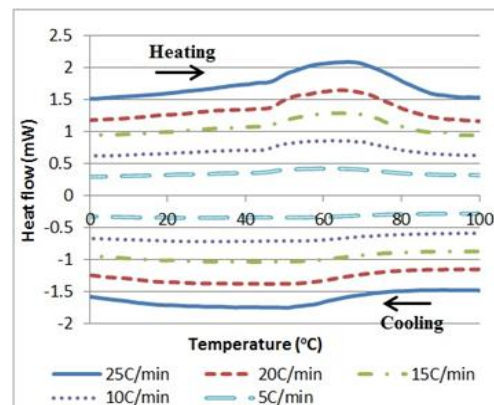
Figure 4. DSC curves (15°C/min) of samples annealed at different temperatures

In addition to the DSC results shown in Figures 3 and 4, heat flows of samples annealed at 350°C and 800°C measured at different heating/cooling rates are summarized in Figures 5(a) and 6(a) respectively. In order to reveal the heating rate dependency of phase transformation temperatures, the heat flow curves within the phase transformation temperature ranges were presented in Figures 5(b) and 6(b). It is obvious in Figures 5(b) and 6(b) that the temperatures at which the maximum heat flow occurs due to phase transformation are dependent on the heating/cooling rates.

The phase transformation temperatures are the starting and finishing temperatures of the peaks in heat flow curves representing the energy absorbed or released due to transformation propagation. The resultant phase transformation temperatures for samples annealed at different temperatures were presented in Figures 7 and 8. It shows that the austenite starting and finishing temperature for NiTi shape memory alloys heat treated at 350°C and 800°C increases with increasing heating rates. In contrast to the heating cycle, the transformation upon cooling starts and finishes at higher temperature when the cooling rate is lower. This phenomenon agrees with the kinetic theory of solid state phase transformation studied by Rai et al. 2009 and Liu et al. 2007. Based on the mechanisms of solid state phase transformation, the time for the NiTi alloys to absorb or release energy is longer when the shape memory alloys are heated or cooled at a lower rate. Thus, the energy contained in the material accumulates faster and reaches the activation energy to generate phase transformation earlier than those heated or cooled at a faster rate.

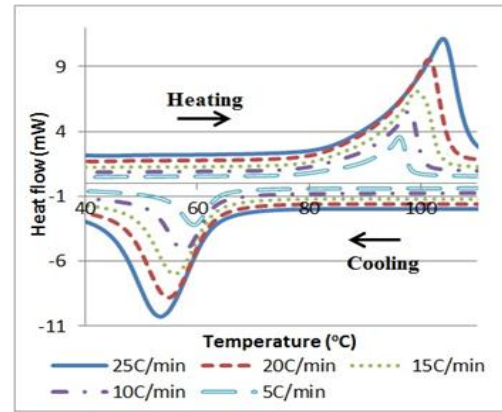
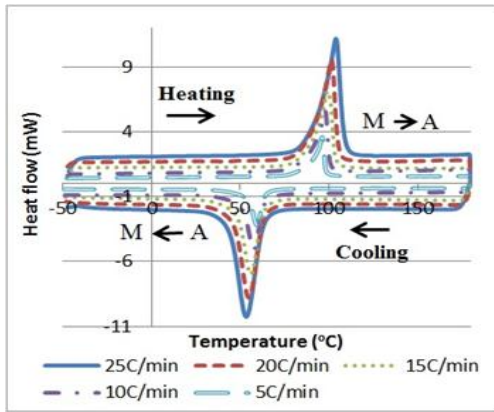


(a) Heat flow (-50°C~180°C)



(b) Heat flow (0°C~100°C)

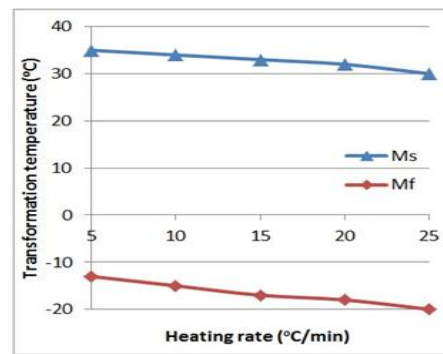
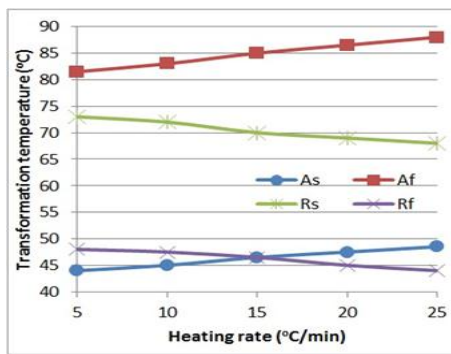
Figure 5. DSC curves of samples annealed at 350°C



(a) Heat flow (-50°C~180°C)

(b) Heat flow (40°C~110°C)

Figure 6. DSC curves of samples annealed at 800°C

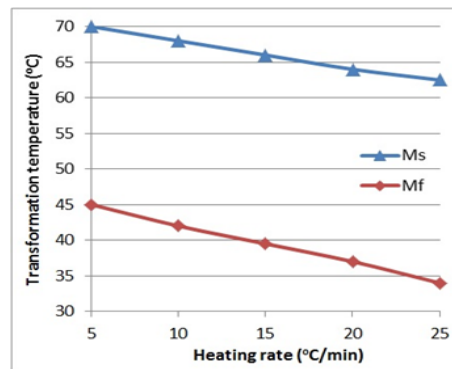
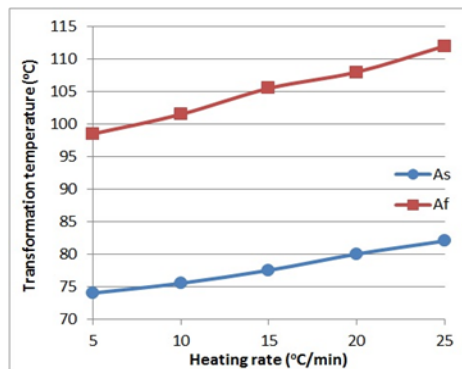


(a)  $A_s$ ,  $A_f$ ,  $R_s$  and  $R_f$

(b)  $M_s$  and  $M_f$

Figure 7. Transformation temperatures of samples annealed at 350°C

By comparing Figure 7 with Figure 8, the heating/cooling rate effect on phase changes in samples heat treated at 800°C are much more significant than that on samples annealed at 350°C. For samples heat treated at 800°C, differences of  $A_s$ ,  $A_f$ ,  $M_s$  and  $M_f$  obtained at 5°C/min and 25°C/min are 8°C, 13.5°C, 7.5°C and 11°C respectively. However, for the samples annealed at 350°C, the differences for the  $A_s$ ,  $A_f$ ,  $M_s$ ,  $R_s$ ,  $R_f$  and  $M_f$  measured at 5°C/min and 25°C/min are only 4.5°C, 6.5°C, 5°C, 4°C, 5°C and 7°C respectively. Thus, the influence of heating/cooling rates on the phase transformation is more appreciable for shape memory alloys annealed at higher temperatures. In order to achieve an accurate structural design of using NiTi wires, the heating and cooling rates based on the application working conditions should be used when measuring phase transformation temperatures.



(a)  $A_s$  and  $A_f$

(b)  $M_s$  and  $M_f$

Figure 8. Transformation temperatures of samples annealed at 800°C

## CONCLUSIONS

The effects of annealing temperatures and heating/cooling rates on the transformational behaviour of NiTi shape memory alloys were investigated. A differential scanning calorimetry method was used to measure phase transformation temperatures. The results show that shape memory alloys without heat treatment do not exhibit transformational behaviour and the phase transformation temperatures increase with increasing annealing temperatures. Proper heat treatment needs to be applied in order to obtain shape memory effect prior to the application of NiTi shape memory alloys.

Another factor influencing phase transformation temperatures is the heating/cooling rate. The austenite start and finish temperatures ( $A_s$  and  $A_f$ ) increase upon heating at a higher rate while the martensite start and finish temperatures ( $M_s$  and  $M_f$ ) decrease when the cooling rate is higher. Besides, the dependency on the heating/cooling rate is more significant in materials heat treated at 800°C than those heat treated at 350°C. The phase transformation temperatures can increase for up to 8-14°C when the heating rate rises from 5°C/min to 25°C/min. Upon cooling, the phase transformation temperatures decrease for up to 7-11°C with increasing cooling rates. Such differences of phase change temperatures due to heating/cooling rate effect should be taken into consideration in the design of structural actuator using NiTi shape memory alloys especially when the heating rate of the working conditions varies.

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