Effect of Ni-Content on Mechanical and Transformation Behavior of NiTi Shape Memory Alloys for Orthodontics Applications

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Abstract

This study aims to investigate the effect of Ni-content on mechanical properties and transformation behavior of NiTi shape memory alloys for utilizing as orthodontic wires. NiTi binary alloys with Ni-content ranging from 50 to 51 at% were prepared. The specimens were cold-rolled with percentage reduction of 10, 20 and 30%, respectively. Then they were heat treated at 400°C and 600°C for 3,600s, respectively. The results show that transformation temperatures strongly depend on Ni-content, i.e., transformation temperatures rapidly decrease with the increase of Ni-content. Moreover transformation temperature decreases with the increase of cold-rolling reduction ratio. However, the higher is the reduction ratio, the superelastic properties become more evidently. Further heat treatment temperature 400°C provides specimens with better properties compared to those of 600°C. The results obtained can be use to determine optimum alloy composition of NiTi alloy to be used as orthodontic wires.

Keywords*:* orthodontic wires, Ni-content, Reduction ratio

1. Introduction

NiTi was introduced to be used in clinical orthodontic for leveling phase in 1971[1]. The physical properties of nickel-titanium alloy have several advantages over precious metals and stainless steel. NiTi alloys have extraordinary properties: shape memory effect and superelasticity with excellent corrosion resistance, as well as good mechanical properties and biocompatibility. NiTi alloy are wildly used in clinical orthodontics since their superelasticity property gives continuous and light forces transmitted to the dentition over a long activation period, resulting in a desirable biological response [2-4]. The relative alloy composition of martensite and austenite is a function of mechanical stress and ambient temperature. Some key characteristics of superelastic nickel-titanium may show exceptional temperature sensitivity [5-7]. Small chemical composition variations can produce significant modifications of such behavior, which can be analyzed considering variation of the start of martensitic transformation (Ms) temperature [8-9]. The properties of NiTi can be modified to a great extent by judicious choice of composition, cold work and heat treatment. This study will be a preliminary work to fabricate of NiTi alloy samples.

The purpose of this study is to evaluate the chemical composition, mechanical properties and phase transformation behavior of the fabricated near equiatomic NiTi alloy samples. The influence of degrees of cold-rolling and heat treatment temperatures will be discussed in order to further develop NiTi alloy used in orthodontics.

2. Experimental procedure

2.1 Materials

The raw materials used commercial grade with high purity; nickel 99.9% and titanium 99.8%. The targeted composition for each sample is equiatomic NiTi alloy (50-51 at.% Ni). Firstly, nickel and titanium were cleaned in the acid ($HF:HNO_3:H_2O$, 5:4:1) and then rinsed by acetone to remove surface grease and oxide before melting.

2.2 Melting method

A conventional Vacuum Arc Re-melting technique in argon atmosphere was employed. After charging the constituent element in crucible Fig.1(a), the furnace was purged with argon at pressure of 0.3-0.5 bar. Melting of the raw elements was performed with arc rotation torch created by tungsten electrode Fig.1(b). The ingot was turned over and re-melted five times to ensure chemical homogeneity. The examples of melted ingot is show in Fig.1(c). All melted ingots were then homogenized at 800°C for 3600s Fig.1(d).

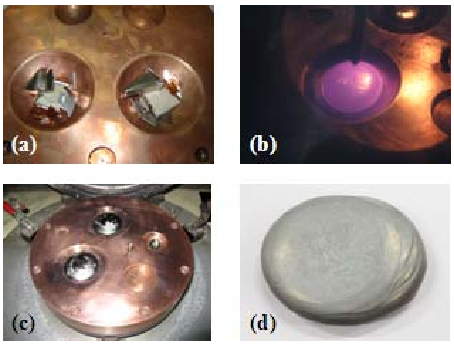

Fig. 1 Sequence of the VAR process: (a) pilling up raw materials, (b) rotating torch, (c) melted ingot on a copper crucible, (d) melted ingot after homogenized.

2.3 Characterization

Ingots were sliced into small plates (1.5 mm. in thickness) using a CNC wire cutting machine and then cold-rolled at determined reduction ratio, i.e., 10%, 20% and 30 %, respectively. The lubricant used for the rolling is ISO cut 570A in combination with sodium strearate soap. After removing oxide layers and surface contaminants on the specimen surfaces by mechanical polishing, they were annealed at 400°C and 600°C in heat treatment furnace for 3,600s. The specimens were then cut into specific by a CNC wire cutting machine. Specimens used for investigation phase-transformation behavior were test by using. Differential Scanning Calorimeter (DSC). During the test temperature was varied in the range of -50°C to 100°C with cooling and heating rate of 10°C/min. The hardness of the specimens was determined by Vickers Microhardness tester with a Vickers diamond tip at room temperature under a maximum load of 500 g_f. To examine load-deflection characteristics of melted NiTi specimens, a three-point bending tests using the Instron Universal Testing Machine (load cell 100N) were performed. The span for bending test was 10 mm. Specimens were loaded to till a maximum deflection of 1.5 mm and deflection rate is 5 mm/min. The influences of Ni-content for NiTi on the mechanical properties and transformation behavior of the alloys were then discussed.

3. Results and Discussion

3.1 Transformation temperature behavior

The transformation temperatures of NiTi. Austenetic finish (Af) and Martensitic start (Ms) are critical factors of their transformation behavior. The results of Af and Ms values obtained are shown in Table 1. Actually, we intended to make a superelastic NiTi alloy having transitional temperature lower than oral temperature. It is generally known this can be achieved by increasing Ni content over 50 at.%. From Table 1, the NiTi having nominal composition of $Ni_{50.4}Ti_{49.6}$ at.% and $Ni_{50.6}Ti_{49.4}$ at.% provides Austenite finished temperature (A_f) set as 42.5°C and 32°C, which are closed to oral temperature.

Table 1 Transformation temperature of the specimens obtained by DSC

Nominal Composition (at.%)	Transformation temperature (°C)			
	Ms	**Mf**	**As**	**Af**
$Ni_{50}Ti_{50}$	51.5	20	50.5	79
$Ni_{50.2}Ti_{49.8}$	27	7	42	62.5
$Ni_{50.4}Ti_{49.6}$	12	-12	16.5	42.5
$Ni_{50.6}Ti_{49.4}$	4.5	-31	-8	32
$Ni_{51}Ti_{49}$	-37	-	-41	-4

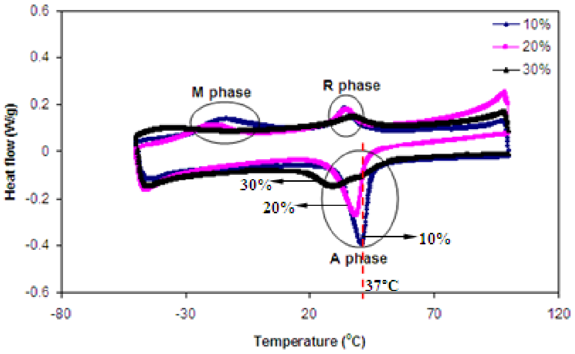

Fig. 2 Thermographs of $Ni_{50.4}Ti_{49.6}$ at.% with 10%, 20% and 30% reductions followed by heat-treatment at 400°C for 3,600s

Fig. 2 shows the results of DSC for $Ni_{50.4}Ti_{49.6}$ at.% with 10, 20 and 30% reductions followed by 400°C heat treatment for 3,600s. It can be found from the result that the peak on cooling curve reveals the R-phase transformation or the intermediate phase occurs. This R-phase transformation often occurs when the alloys are work-hardened, which also can occur in nickel-rich NiTi alloys. Further $Ni_{50.4}Ti_{49.6}$ at.% does not reveals superelasticity properties at the oral temperature because its A_f is higher than 37°C.

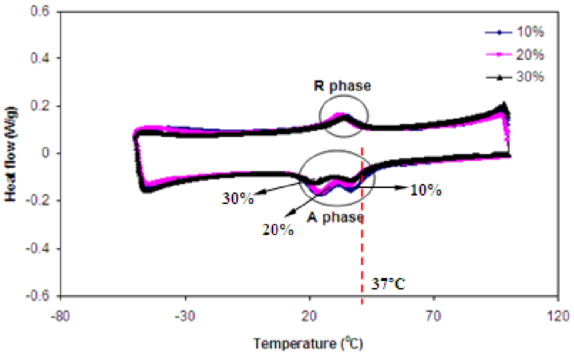

Fig. 3 Thermographs of $Ni_{50.6}Ti_{49.4}$ at.% with 10%, 20% and 30% reductions followed by heat-treatment at 400°C for 3,600s

Fig. 3 shows the DSC result for $Ni_{50.6}Ti_{49.4}$ at.% with 10, 20 and 30% reductions followed by 400°C heat treatment for 3,600s. The alloy has Af temperature very closed to oral temperature as shown previously. From Fig. 2, the higher of the percent reduction is the lower and shorter of transformation temperature of both heating and cooling paths are obtained. This can be implied that percent reduction has an impact on phase transformation, and can be explained that transformation was suppressed by internal stress due to cold work. In other words, the internal structure of the work-hardened material is composed of multiple dislocations that hinder the phase transformation. Some works reported that cold-worked NiTi alloys had wide transformation temperature range and the peak height was small. The broadening of the peak was enhanced by increasing the amount of cold-working reduction percent [10].

For the alloy heat treated at 600°C, influence of reduction ratio can not be observed, since this temperature (600°C) is higher than the alloy recrystalization temperature which is about 500-600°C [11]. This result confirms that the dislocation obstructing the phase transformation. Moreover, the Af temperature of the alloys obtained from all conditions are summarized and shown in Table 2.

Table 2 Transformation temperature of the specimens with heat-treatment at 400°C for 3,600s obtained by DSC

Nominal Composition (at.%)	**% Reduction**	**Transformation temperature (°C)**	
		Af	**Rs**
$Ti_{49.6}Ni_{50.4}$	10	49.8	48.8
	20	47	41.3
	30	45.7	39.1
$Ti_{49.4}Ni_{50.6}$	10	47	40
	20	40	39
	30	37	39

3.2 Vickers hardness test.

The micro-indentation hardness is measured at the cross-sectional areas of each alloy specimen. Fig. 4 and Fig. 5 shows the relation between the hardness value (HV) and the cold-rolled reduction ratio, for heat treatment temperature of 400 and 600°C, respectively.

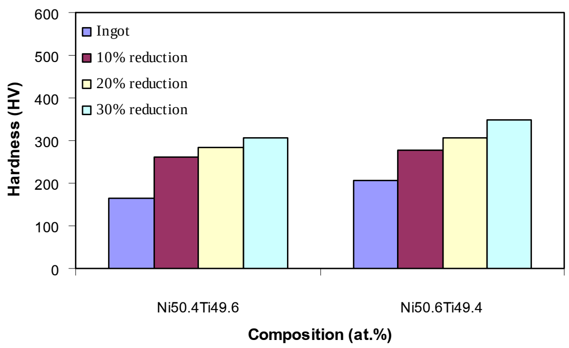

Fig. 4 Hardness values of NiTi with 10%, 20% and 30% reductions followed by heat-treatment at 400°C for 3,600s

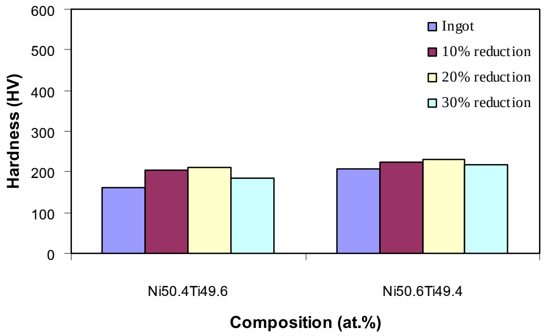

Fig. 5 Hardness values of NiTi with 10%, 20% and 30% reductions followed by heat-treatment at 600^{o}C for 3,600s

From Fig. 5, at 600°C heat treatment temperature, which is higher than alloy recrystalization temperature, dislocations are eliminated, hence there is no difference between the hardness value of the specimens undergone rolling at different reduction ratio.

3.3 Three-point bending test.

Three-point bending tests of the specimens are conducted at oral temperature or at 37^{o}C. The results are shown in Fig. 5 and Fig. 6 for the specimen with different. Composition for the $Ni_{50.6}Ti_{49.4}$ at.% with 10%, 20% and 30% reductions followed by heat treatment at 400°C for 3600s (Fig. 6), the completely reverse stress-strain curve is obtained only for the reduction ratio of 30%. From Fig. 7 for $Ni_{50.4}Ti_{49.6}$ at.% alloy, the completely reverse transformation cannot be obtained from any conditions.

This can be explained by the transformation temperature (Af) of the alloy. Since there is only $Ni_{50.6}Ti_{49.4}$ at.% undergone rolling 30% having Af lower than 37^{o}C, it becomes only one condition that gives superior superelastic behavior without permanent strain left after unloading.

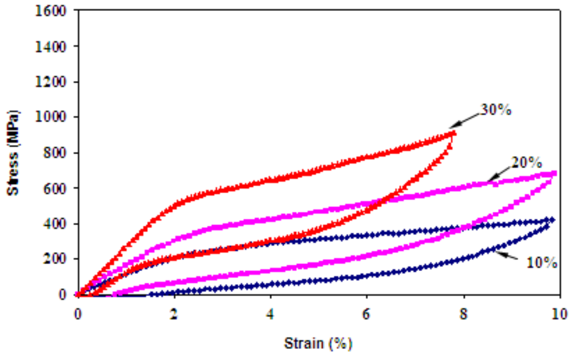

Fig. 6 Stress-strain curves for $Ni_{50.6}Ti_{49.4}$ at.% with 10%, 20% and 30% reductions follow by heat-treatment at 400^{o}C for 3,600s (tested at 37^{o}C)

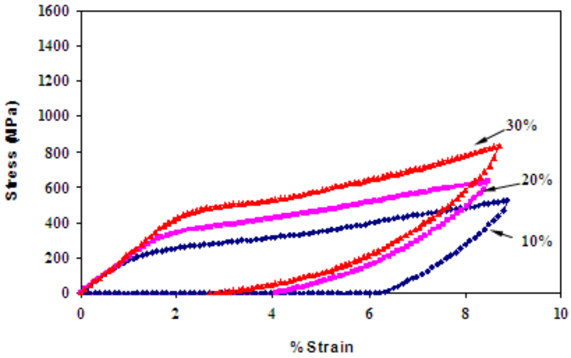

Fig. 7 Stress-strain curves for $Ni_{50.4}Ti_{49.6}$ at.% with 10%, 20% and 30% reductions follow by heat-treatment at 400^{o}C for 3,600s (tested at 37^{o}C)

4. Conclusions

In order to fabricate the NiTi shape memory alloy used in orthodontics, three principle factors, i.e., alloy composition, work hardening and heat treatment temperature, affecting the transformation behavior and mechanical properties of NiTi should be effectively determined. The cold work reduction higher than 30% tends to improve the superelastic property of the alloys. The heat treatment temperature higher than 600^{o}C remove all dislocation resulted in unsatisfied properties of the alloys. The fraction of Ni at 50.6% in the alloy provides the best mechanical properties as well as superelastic behavior to be used as orthodontic wires.

5. Acknowledgement

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6. References

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