

# Transition temperature range of thermally activated nickel-titanium archwires

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## ABSTRACT

**O**bjectives: The shape memory resulting from the superelasticity and thermoelastic effect is the main characteristic of thermally activated NiTi archwires and is closely related to the transition temperature range (TTR). The aim of this study was to evaluate the TTR of thermally activated NiTi archwires commercially available. Material and Methods: Seven different brands of 0.019"x0.025" thermally activated nickel-titanium archwires were tested as received by differential scanning calorimetry (DSC) over the temperature range from -100°C to 150°C at 10°C/min. Results: All thermally activated NiTi archwires analyzed presented stage transformation during thermal scanning with final austenitic temperature (Af) ranging from 20.39°C to 45.42°C. Three brands of NiTi archwires presented Af close to the room temperature and, this way, do not present properties of shape memory and pseudoelasticity that are desirable in clinical applications. Conclusions: The thermally activated NiTi archwires present great variability in the TTR and the elastic parameters of each NiTi archwire should be provided by the manufacturers, to allow achievement of the best clinical performance possible.

**Keywords:** Differential thermal analysis. Orthodontic wires. Materials testing. Nickel. Titanium.

## INTRODUCTION

The NiTi archwires are widely employed in orthodontics because they present shape memory effect and superelasticity combined to good mechanical properties<sup>18,19</sup>.

The shape memory resulting from the pseudoelastic effect (superelasticity) and thermoelastic effect is the main characteristic of these archwires and is closely related to the TTR of each alloy<sup>4-7,22</sup>. The noticeable characteristics of thermally activated nickel-titanium orthodontic archwires are caused by a transformation in the crystalline structure of the alloy, called martensitic transition<sup>5,19,22</sup>.

The martensitic transition is initiated when the alloy passes through a critical temperature during cooling called Ms (Martensite start) and is completed at Mf (Martensite finish), when the material is totally martensitic, loses its structure and

shape and acquires maximum flexibility. With the increase in temperature, the martensitic transition in austenite is initiated at temperature As (Austenite start) and finalized at Af (Austenite finish), when the NiTi alloy is totally austenitic, recovering its shape and acquiring maximum rigidity<sup>6,14,22</sup>. As a response to the temperature variation, the crystalline structure presents deformations in the molecular arrangement, without changes in the atomic composition<sup>3</sup>.

Each Ni-Ti alloy has a temperature range, in which this phase transition occurs, called transition temperature range (TTR), which should correspond to the temperature variations in the oral environment to be beneficial for the orthodontic treatment<sup>5,6,22</sup>. This information is particularly important in orthodontics, because it determines the mechanical performance of the NiTi archwire during treatment<sup>5,19,22</sup>. However, not all manufacturers provide this information and,

when they do, it is not always based on reliable information on the TTR of archwires and their clinical performance<sup>22</sup>. Even though some studies have been conducted to evaluate the TTR of thermally activated NiTi archwires, most studies have been performed on smaller round and/or rectangular archwires<sup>4,6,7,20,21</sup>. Information on the TTR of larger rectangular archwires, such as 0.019"x0.025", is still scarce in the literature.

Therefore, the aims of this study were to evaluate the TTR of thermally activated NiTi archwires commercially available and mainly to investigate if the Af of these archwires agrees with that provided by the manufacturer. When it is not provided, to determine it and aid the orthodontist in selecting the thermally activated archwires with the greatest benefits for each stage of orthodontic treatment.

## MATERIAL AND METHODS

The study was conducted on seven different brands of 0.019"x0.025" thermally activated nickel-titanium archwires: Nitinol Termoativado (Aditek, Cravinhos, SP, Brazil); NeoSentalloy F200 (GAC, Bohemia, NY, USA); Thermo Plus (Morelli, Sorocaba, SP, Brazil); Copper Ni-Ti 35°C (Ormco, Glendora, CA, USA); Flexy Thermal 35°C (Orthometric, Marília, SP, Brazil); Superthermal Nickel Titanium Arches (Orthosource, Matão, SP, Brazil) and Heat Activated NiTi (Highland Metals, San Jose, CA, USA) (Figure 1). The wires mentioned above were randomly acquired in the dental market.

One specimen was obtained from the straightest portion of each brand (archwire) carefully trimmed with separating discs under cooling at low speed, connected to a pneumatic sectioning machine, in size of 5 mm or approximately 5±0.5 mg, weighed in a precision electronic scale with 10 µg accuracy.

Brand	Manufacturer	Type	Batch
Nitinol Termoativado	Aditek	Thermally activated	51.10.295091013
NeoSentalloy F200	GAC	Thermally activated	H580
Thermo Plus	Morelli	Thermally activated	1590627
Copper Ni-Ti 35°C	Ormco	Thermally activated	08M465M
Flexy Thermal 35°C	Orthometric	Thermally activated	2041108252
Superthermal Nickel Titanium Arches	Orthosource	Thermally activated	9004
Heat Activated NiTi	Highland Metals	Thermally activated	29350

Figure 1- Thermally activated NiTi archwires analyzed

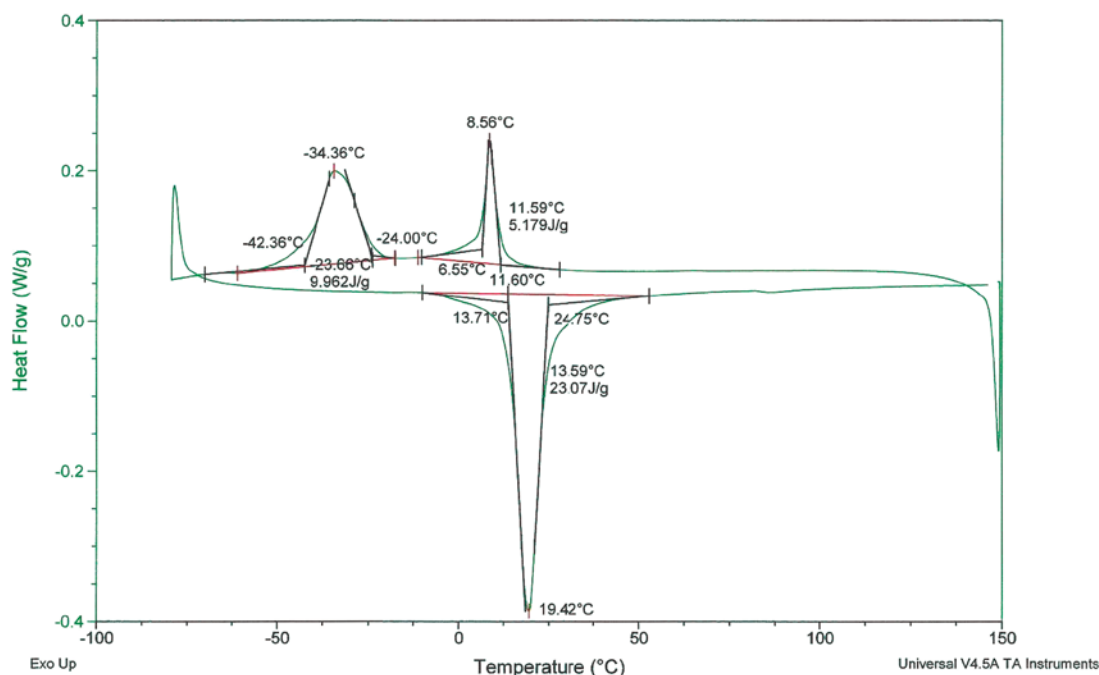


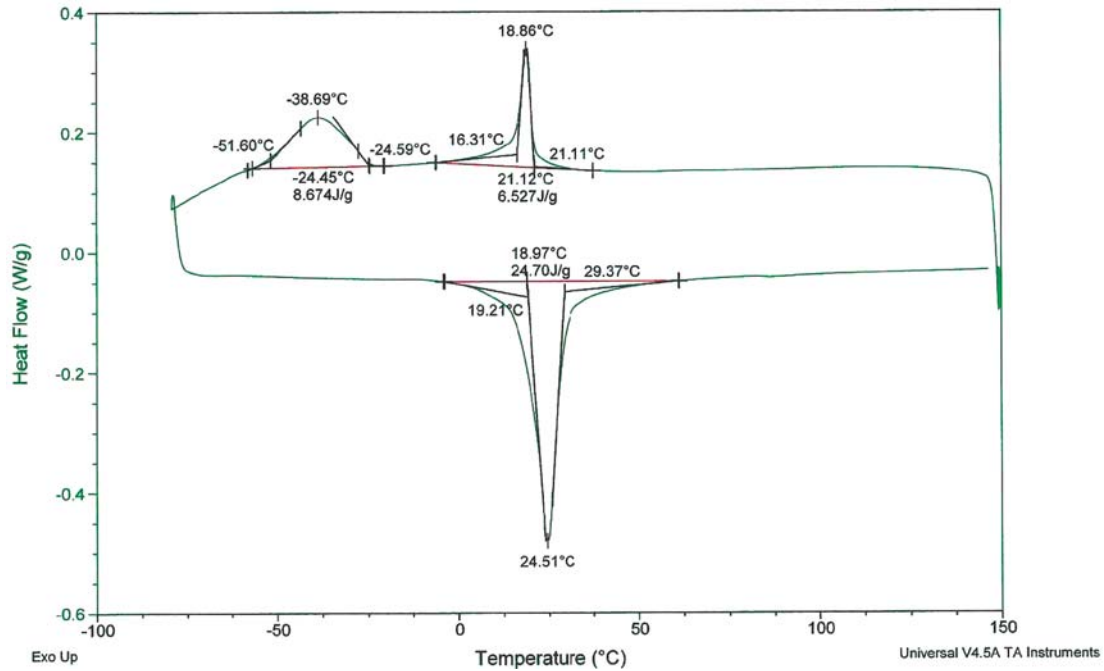
Figure 2- Differential scanning calorimetry (DSC) heating and cooling curves for as-received Nitinol Termoativado

The care during sectioning of NiTi archwires was extremely important in the study, to avoid changes in the proportion of austenite and martensite in the NiTi alloy due to mechanical stress. After trimming and weighing, each specimen was cleaned with alcohol, dried and placed in a pan. This pan was covered and sealed and then placed in a DSC (Differential Scanning Calorimetry) machine for thermal analysis of thermally activated NiTi archwires.

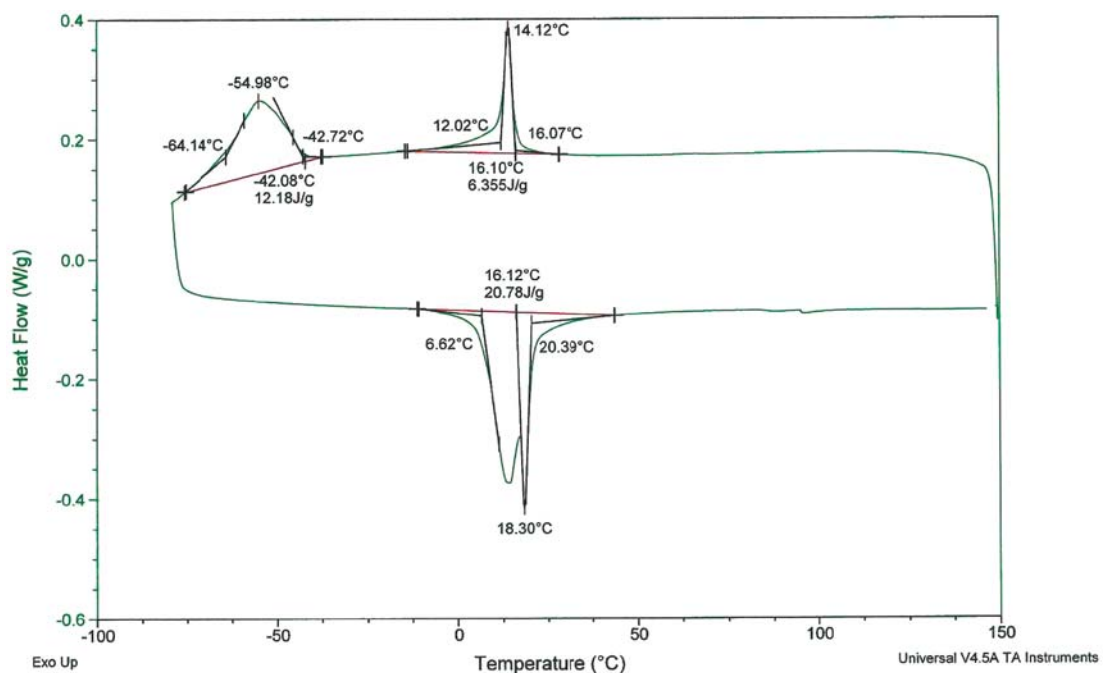
The tests were performed following the

guidelines of the International Organization for Standardization (ISO), ISO 15841 (ISO, 2006), and American Society for Testing and Materials (ASTM), ASTM D3418-08 (ASTM, 2008)<sup>2,15</sup>. A DSC machine (model Q20; TA Instruments, New Castle, DE, USA) was used for thermal scanning of thermally activated archwires.

The heating chamber was filled with one nitrogen atmosphere at 50 ml/min, to avoid water condensation and oxidation of the material, and another empty aluminum melting pot was used as



**Figure 3-** Differential scanning calorimetry (DSC) heating and cooling curves for as-received NeoSentalloy F200

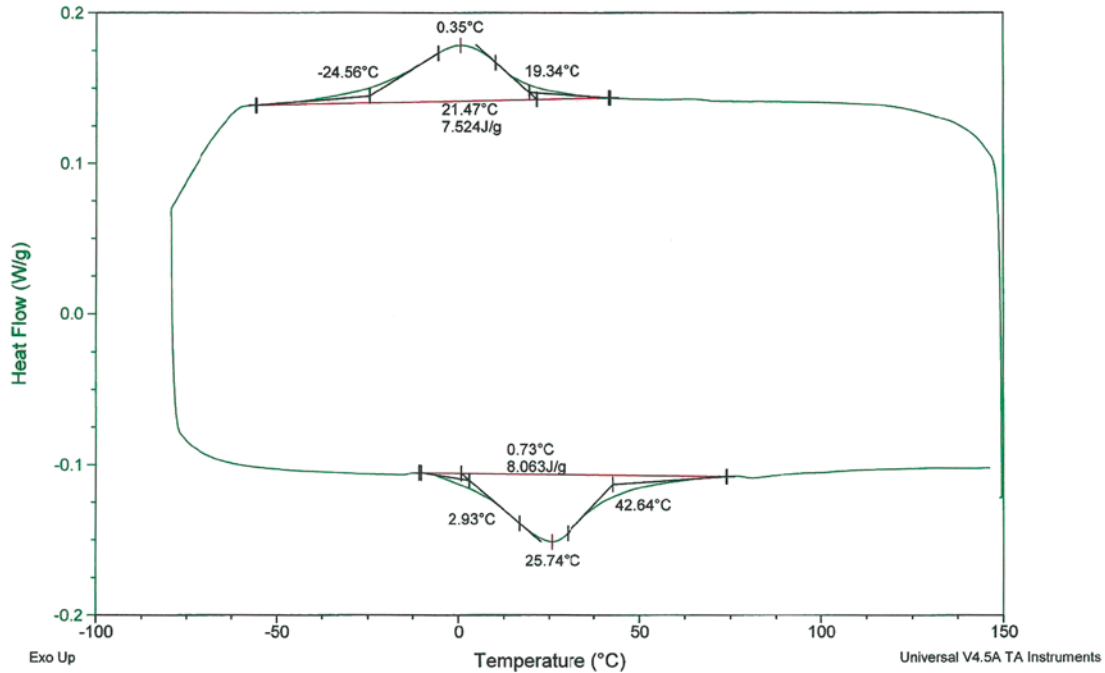


**Figure 4-** Differential scanning calorimetry (DSC) heating and cooling curves for as-received Thermo Plus

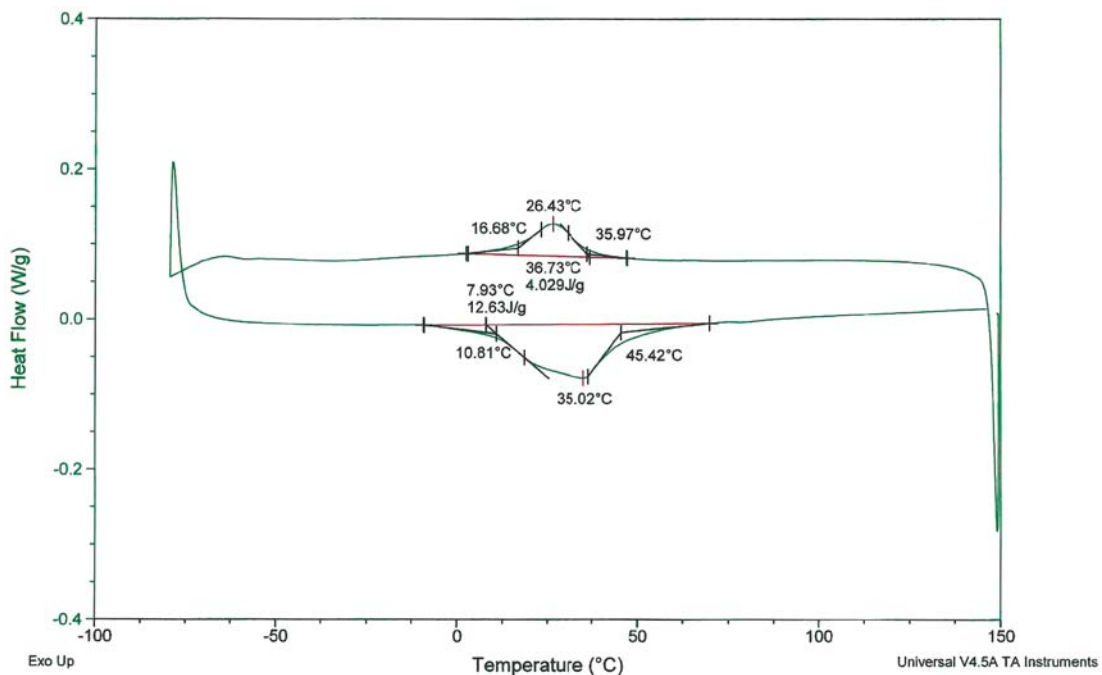
inert reference. The machine was calibrated right before testing, using mercury/indium as standard. The warming and cooling rates were set at 10°C/min, in a temperature range between -100°C and 150°C. The DSC machine was directly connected to a computer and the software Platinum (TA Instruments, New Castle, DE, USA) exhibited graphs of curves of the exothermic reaction in cooling and endothermic reaction in warming (Figures 2 to 8). These graphs (DSC thermograms) provided the enthalpy values and initial and final

temperatures of each reaction (endothermic and exothermic) (Figures 2 to 8).

The amount of heat released or absorbed during phase transition was recorded according to the temperature (Figures 2 to 8). Simultaneously, the transition temperatures [initial austenitic (Ai), final austenitic (Af), initial martensitic (Mi), final martensitic (Mf), initial rhombohedral (Ri) and final rhombohedral (Rf)] were determined by the Platinum Software®. The initial and final temperatures of each phase transition were



**Figure 5-** Differential scanning calorimetry (DSC) heating and cooling curves for as-received Copper Ni-Ti 35°C



**Figure 6-** Differential scanning calorimetry (DSC) heating and cooling curves for as-received Flexy Thermal 35°C

determined from lines tangent to the DSC curve, in which there is deviation of adjacent base lines. The rhombohedral phase (R) indicates that the NiTi alloy presents shape memory effect in the two directions.

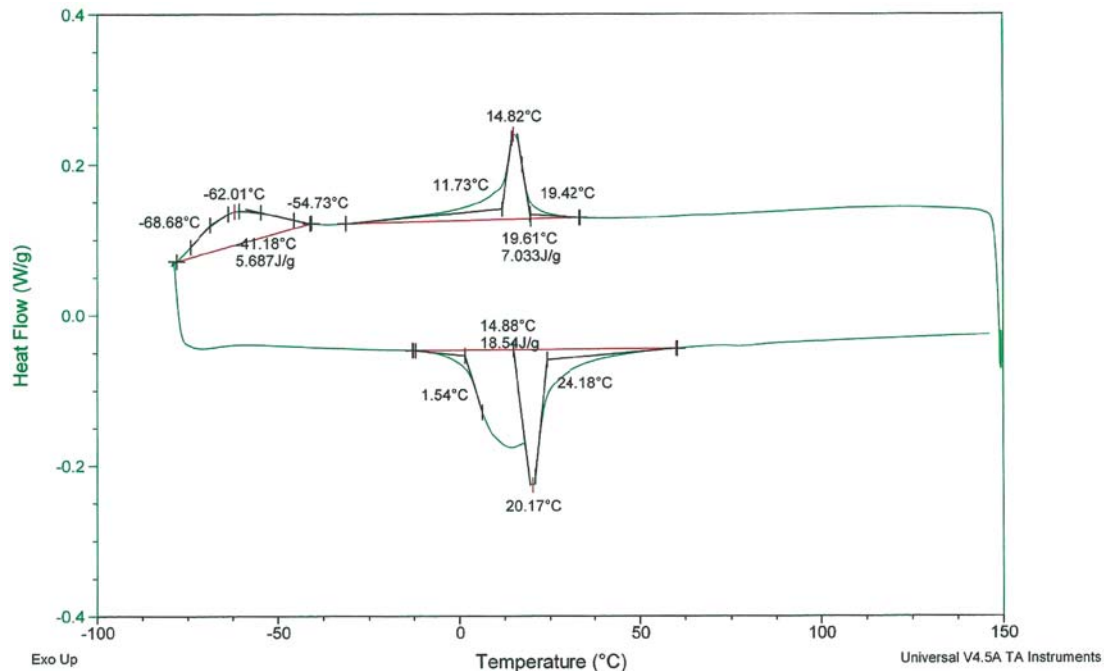
## RESULTS

All thermally activated NiTi archwires analyzed presented stage transformation during thermal scanning in the range between  $-100^{\circ}\text{C}$  and  $150^{\circ}\text{C}$

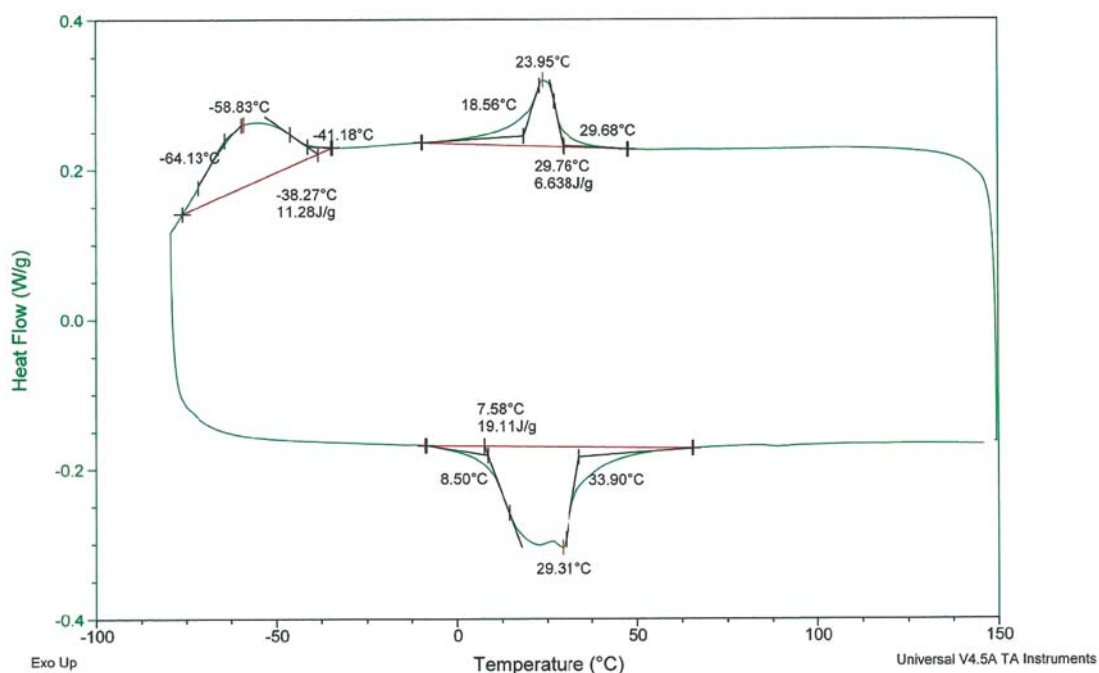
(Figures 2 to 8).

For the thermally activated NiTi archwires Nitinol Termoativado, NeoSentalloy F200, Thermo Plus, Superthermal Nickel Titanium Arches and Heat Activated NiTi there was presence of rhombohedral phase (R) during cooling. Conversely, none of the archwires presented rhombohedral phase (R) during warming (Table 1).

The transition temperatures of archwires analyzed are described in Table 1.



**Figure 7-** Differential scanning calorimetry (DSC) heating and cooling curves for as-received Superthermal Nickel Titanium Arches



**Figure 8-** Differential scanning calorimetry (DSC) heating and cooling curves for as-received Heat Activated NiTi

**Table 1-** Transition temperatures (°C) of thermally activated NiTi archwires analyzed

Archwires analyzed	Cooling				Warming			
	Ri	Rf	Mi	Mf	Ri	Rf	Ai	Af
Nitinol Termoativado	11.60	6.55	-24.00	-42.36	---	---	13.71	24.75
NeoSentalloy F200	21.11	16.31	-24.59	-51.60	---	---	19.21	29.37
Thermo Plus	16.07	12.02	-42.72	-64.14	---	---	6.62	20.39
Copper Ni-Ti 35°C	---	---	19.34	-24.56	---	---	2.93	42.64
Flexy Thermal 35°C	---	---	35.97	16.68	---	---	10.81	45.42
Superthermal Nickel Titanium Arches	19.42	11.73	-54.73	-68.68	---	---	1.54	24.18
Heat Activated NiTi	29.68	18.56	-41.18	-64.13	---	---	8.50	33.90

The Af of archwires Nitinol Termoativado, Thermo Plus and Superthermal Nickel Titanium Arches was lower or close to the room temperature, and thus there was no phase transition at body temperature (Table 1).

The Af of archwires NeoSentalloy F200 and Heat Activated NiTi was close to the body temperature and thus, at room temperature, there is a combination of austenite and martensite in the NiTi alloy, which presents full transition to austenite at the oral temperature (Table 1).

The Af of archwires Copper Ni-Ti 35°C and Flexy Thermal 35°C was higher than the body temperature; thus, at the oral temperature, there is a combination of austenite and martensite in the NiTi alloy (Table 1).

## DISCUSSION

Analysis of the TTR of thermally activated NiTi archwires is relevant in orthodontics to allow identification of archwires with the best clinical performance. The Af is particularly important, since it indicates the temperature in which the material will entirely return to the original shape and consequently acquire greater rigidity.

The oral temperature presents wide variation during the day because of ingestion of hot and cold foods and drinks, and different areas in the oral cavity present different temperatures. However, experimental studies adopt temperatures between 35°C to 37°C to reproduce the oral environment<sup>1,17,25</sup>, considering a room temperature around 25°C<sup>1</sup>.

The shape memory property of NiTi alloys does not necessarily imply the application of light and continuous forces in orthodontics<sup>22</sup>. In fact, this property may only be clinically beneficial in orthodontic practice if the Af of NiTi is slightly below the oral temperature, so as the archwire will be mainly in austenitic phase (more rigid) in the oral cavity and almost completely in martensitic phase (more flexible) at room temperature<sup>20-22</sup>. When

the alloy is completely transformed into austenite (temperature higher than Af), the load/deflection curve follows the typical performance of other metallic alloys, such as stainless steel, as a direct proportion between force and deformation, without the typical superelastic plateau<sup>22</sup>. That is to say, the NiTi alloy completely transformed into austenite is definitely more elastic compared to other metallic alloys, yet it does not present superelasticity in the absence of tension<sup>22</sup>.

The DSC method is often employed to evaluate the TTR of thermally activated NiTi archwires, because it allows identification of phase transition temperatures and the quantity of energy released or absorbed during cooling and/or warming. This method, adopted in this study, allows comparison with results of other studies with similar methodology<sup>3,4,6,7,13,14,16,20</sup>.

The present results demonstrate that not all NiTi archwires present thermal performance that may provide clinical benefits (Table 1) (Figures 2 to 8). Yoneyama, et al.<sup>26</sup> (1993) employed the DSC method and demonstrated that most orthodontic archwires present TTR between 17°C and 32°C. From a clinical standpoint, archwires with lower Af do not present pseudoelastic behavior in clinical applications. Santoro, Nicolay and Cangialosi<sup>22</sup> (2001) reported that most superelastic archwires commercially available present Af ranging from 22°C to 28°C, while thermally activated archwires present Af with temperatures ranging from 35°C to 40°C.

The archwires Nitinol Termoativado, Thermo Plus and Superthermal Nickel Titanium Arches analyzed in this study presented TTR ranging from -42.36°C (Mf) to 24.75°C (Af), from -64.14°C (Mf) to 20.39°C (Af) and from -68.68°C (Mf) to 24.18°C (Af), respectively (Table 1). For these archwires, only the austenitic phase is present during orthodontic treatment. The low Af of these archwires indicate that they are in the austenitic phase both at room and oral temperature. Thus, these NiTi archwires do not present the properties of shape memory



and pseudoelasticity that are desirable in clinical applications. Conversely, Figueiredo, et al.<sup>11</sup> (2012) compared the mechanical behavior of the same archwires, except for the Heat Activated NiTi, and revealed that these archwires present pseudoelasticity. Evans and Durning<sup>10</sup> (1996) found Af of 24°C for the archwire Thermomemoria (Leone, Oxnard, CA, USA), suggesting similar clinical performance as the aforementioned archwires.

The archwires NeoSentalloy F200 and Heat Activated NiTi presented TTR in this study ranging from -51.60°C (Mf) to 29.37°C (Af) and from -64.13°C (Mf) to 33.90°C (Af), respectively (Table 1). These results agree with the studies of Bishara, et al.<sup>5</sup> (1995) who analyzed 10 specimens of archwire NeoSentalloy size 0.018"x0.025" and found a TTR of 21°C to 28.8°C. Santoro, Nicolay and Cangialosi<sup>22</sup> (2001) reported Af of 28°C for archwire NeoSentalloy in the absence of deflection and Af of 36°C with load application. Brauchli, et al.<sup>8</sup> (2011) found Af of 29.2°C for archwire NeoSentalloy size 0.016"x0.022". Conversely, Evans and Durning<sup>10</sup> (1996) observed Af of 68°C and Mf of 24°C for archwire Heat Activated NiTi size 0.016"x0.022", which disagrees with this study. The present results suggest that these two archwires present the best clinical performance considering the shape memory effect and pseudoelasticity property, since the Af of these archwires is closer to the oral temperature.

The archwires Copper Ni-Ti 35°C and Flexy Thermal 35°C in this study presented TTR ranging from -24.56°C (Mf) to 42.64°C (Af) and from 16.68°C (Mf) to 45.42°C (Af), respectively (Table 1). These archwires may be considered truly superelastic at the oral temperature, at which they exhibit a mixture of martensite/austenite and do not present shape memory during clinical applications. Brantley, Iijima and Grentzer<sup>7</sup> (2003), using the modulated DSC, found Af of 35°C for archwire Copper Ni-Ti 35°C size 0.016"x0.022". Biermann, Berzins and Bradley<sup>4</sup> (2007) evaluated the Af of archwires Copper NiTi 35°C by the DSC and concluded that the Af of this archwire presents variability of  $\pm 2^\circ\text{C}$  compared to values informed by the manufacturer. Conversely, Laino, et al.<sup>16</sup> (2012), in a sample of five archwires Copper Ni-Ti 35°C size 0.016"x0.022", also used the DSC method for thermal scanning of these archwires and found Af of 34.4°C (SD $\pm 2.1$ ). Brauchli, et al.<sup>8</sup> (2011) analyzed a sample of 10 specimens of archwires Copper NiTi 35°C size 0.016"x0.022" and found Af of 27.4°C (SD $\pm 0.51$ ). In this study, the Af of archwires Copper Ni-Ti 35°C and Flexy Thermal 35°C was substantially higher than informed by the manufacturers, namely 35°C. These results reveal that the transition of martensite in austenite only occurs in these archwires when the oral temperature is above the normal, suggesting that

the elasticity of these archwires may not reach the best performance in clinical applications.

Therefore, it may be noticed that there is wide variability of Af in NiTi archwires according to the manufacturer or even between batches of the same manufacturer. Thompson<sup>23</sup> (2000) reported some factors that may contribute to differences in the TTR of NiTi archwires: differences between manufacturers, differences between batches of the same manufacturer, thermal treatment applied on the alloy during the metallurgical process, extent of cold working and ratios of nickel and titanium in the alloy.

The pseudoelasticity property is a localized phenomenon related to stress application on the NiTi alloy. However, the formation of martensite in the NiTi alloy requires Af of the alloy slightly lower than the oral temperature<sup>4-6,21,22</sup>. Conversely, if the Af of the archwire is considerably lower than the oral temperature, the crystalline structure will always have a tendency to remain in the austenitic phase, and application of a very high stress would be necessary to neutralize the effect of the temperature and maintain the presence of martensite induced by tension<sup>22</sup>. Thus, the austenitic alloy would present a superelastic behavior only in cases with very severe crowding<sup>22</sup>.

The present results should be carefully analyzed, since the TTR of all archwires were calculated in the absence of deflection ("as-received" condition), which does not perfectly represent the clinical conditions of the oral cavity. It is known that stress application in thermally activated NiTi alloys increases the Af of these archwires<sup>22</sup>. Coluzzi, et al.<sup>9</sup> (1996) evaluated the effect of stress application in the Af of NeoSentalloy F200 and Thermomemoria (Leone, Oxnard, CA, USA). The authors performed a thermal scanning on these alloys with and without deflection. In the absence of deflection, NeoSentalloy exhibited an Af of 28°C. Af increased proportionally with loading to a maximum of 34°C. Thermomemoria wire had an Af in unloaded conditions set at 20°C and increased to a maximum of 35°C with stress application.

For nearly all archwires analyzed, except for Copper NiTi 35°C and Flexy Thermal 35°C, there was presence of the R phase (rhombohedral) during cooling. This result corroborates the findings of Iijima, et al.<sup>13</sup> (2002) and Fisher-Brandies, et al.<sup>12</sup> (2003), who also did not observe rhombohedral phase (R) in phase transition for archwire Copper NiTi 35°C. However, Brantley, Iijima and Grentzer<sup>7</sup> (2003) observed the rhombohedral phase both on warming and cooling for the archwire Copper NiTi 35°C, which is similar to the reports of Bradley, Brantley and Culbertson<sup>6</sup> (1996) and Todoroki and Tamura<sup>24</sup> (1987).

## Clinical implications

One of the main properties of thermally activated NiTi archwires is the release of light and continuous forces, even when submitted to great deflections. This force characteristic is highly desirable in the initial stages of orthodontic treatment, favoring tooth movement with low risk of root resorption and damage to the supporting tissues and less pain by the patient.

In some clinical situations, thermally activated rectangular NiTi archwires may be inserted in brackets in the initial stages of treatment, delivering light and continuous forces and allowing three-dimensional control of tooth movement.

Considering the archwires analyzed in this study, only NeoSentalloy F200 and Heat Activated NiTi presented  $A_f$  corresponding to the temperature variations in the oral cavity. At room temperature, these archwires are flexible and easily deformable, which allows for easy insertion in slots of brackets bonded on misaligned teeth. With the increased temperature in the oral cavity, there will be phase transformation to austenite, leading these archwires to acquire greater rigidity and releasing light and continuous forces, allowing tooth alignment.

The archwires Nitinol Termoativado, Thermo Plus and Superthermal Nickel-Titanium Arches presented  $A_f$  close to room temperature and lower than the oral temperature, being completely in austenitic phase (greater rigidity) for clinical applications. When these archwires are used, the orthodontist may hardly insert rectangular archwires in the bracket slot of misaligned teeth in initial treatment stages. These archwires are marketed as thermally activated, yet they do not present the shape memory and pseudoelasticity properties required for clinical applications in orthodontics, behaving as first-generation or cold-worked nickel-titanium archwires. These archwires present a superelastic behavior only in cases with very severe crowding.

The archwires Copper Ni-Ti 35°C and Flexy Thermal 35°C presented higher  $A_f$  than the oral temperature, with great quantity of martensite both at room and oral temperatures, being extremely flexible and malleable and delivering very low forces on the dentoalveolar structures. Therefore, these archwires may be used in patients with periodontal involvement and low pain threshold. In these cases, the orthodontist should guide the patient to ingest warm drinks to temporarily increase the force delivered by these archwires and stimulate tooth movement. Conversely, cold drinks reduce the force delivered by these archwires.

## CONCLUSIONS

The thermally activated NiTi archwires present great variability in the TTR with  $A_f$  ranging from

20.39°C to 45.42°C. Thus, the elastic parameters of each NiTi archwire should be provided by the manufacturers, to allow achievement of the best clinical performance possible.

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