

## The Effect of Residence Time on the Tensile Properties of Superelastic and Thermal Activated Ni-Ti Orthodontic Wires

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Since the 1980s, different devices based on superelastic alloys have been developed to fulfill orthodontic applications. Particularly in the last decades several researches have been carried out to evaluate the mechanical behavior of Ni-Ti alloys, including their tensile, torsion and fatigue properties. However, studies regarding the dependence of elastic properties on residence time of Ni-Ti wires in the oral cavity are scarce. Such approach is essential since metallic alloys are submitted to mechanical stresses during orthodontic treatment as well as pH and temperature fluctuations. The goal of the present contribution is to provide elastic stress-strain results to guide the orthodontic choice between martensitic thermal activated and austenitic superelastic Ni-Ti alloys. From the point of view of an orthodontist, the selection of appropriate materials and the correct maintenance of the orthodontic apparatus are essential needs during clinical treatment. The present work evaluated the elastic behavior of Ni-Ti alloy wires with diameters varying from 0.014 to 0.020 inches, submitted to hysteresis tensile tests with 8% strain. Tensile tests were performed after periods of use of 1, 2 and 3 months in the oral cavity of patients submitted to orthodontic treatment. The results from the hysteresis tests allowed to exam the strain range covered by isostress lines upon loading and unloading, as well as the residual strain after unloading for both superelastic and thermal activated Ni-Ti wires. Superelastic Ni-Ti wires exhibited higher load isostress values compared to thermal activated wires. It was found that such differences in the load isostress values can increase with increasing residence time.

**Keywords:** Ni-Ti alloys, hysteresis, orthodontics wire, superelastic behavior

### 1. Introduction

Ni-Ti alloys are known as important shape memory alloys (SMA) and have many applications in the medical field. In particular, they are widely used as orthodontic wires and endodontic instruments in dentistry as a result of their pseudoelastic behavior<sup>1,2</sup>. The shape memory and superelasticity of such alloys allow the alignment and leveling of dental elements by the application of constant and smooth forces. Hence, each tooth and its root are progressively moved to the correct position with minimum discomfort to the patient, reducing the risks of tissue hyalinization and root resorption<sup>3,4</sup>.

This is especially due to detwinning of the microstructure that accompanies deformations on iso-stress conditions resulting from austenitic-to-martensitic transformations<sup>2</sup>. Shape memory alloys are generally defined as materials that have the unique ability to recover their shape after undergoing large deformations through either heating (known as shape memory effect) or removal of load (known as the superelastic effect). In superelasticity, austenitic wires undergo a stress-induced transformation, where martensite is formed upon mechanical loading and austenite on unloading<sup>5-17</sup>. Superelastic NiTi alloys can be strained several times more than ordinary metal alloys without being plastically deformed, which reflects its rubber-like behavior. In the case of martensite thermal activated Ni-Ti alloys, with increase of stress the SMA wire experiments a phase change from

twinned into detwinned martensite. If temperature is enough ( $>A_f$ : austenite finish temperature) then a phase transformation between detwinned martensite to austenite may happen. Typically, martensite is stable at low temperatures and high stress values, whereas austenite is stable at high temperatures and low stress values.

The hysteresis tensile test is a very useful tool to evaluate the superelastic behavior of metallic alloys such as the Ni-Ti ones. It is based on loading by uniaxial forces standardized samples to a pre-determined strain before the plastic regime is achieved. After that, the sample is completely unloaded. Es-Souni et al.<sup>18</sup> developed a comparative study of the properties of a binary Ni-42 wt. (%) Ti and a ternary Ni-42 wt. (%) Ti-7 wt. (%) Cu shape memory alloy using hysteresis tests. Whereas the binary alloy showed a R-phase transition due to cold working, the ternary alloy was characterized by a direct austenitic to martensitic transformation. R-phase is an intermediate phase, which depends on both solution treating temperature and cooling rate. The ternary alloy depicted superior mechanical properties. In this case, the superelastic hysteresis stress is lower resulting in higher unloading isostress plateau.

Li et al.<sup>19</sup> performed thermo-mechanical cycling (TMC) tests on Ti49.8Ni50.2 shape memory alloy (SMA) wire. Recovery strain and fatigue life were determined. Different behaviors were observed

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concerning the recovery strain of SMA wire with several annealing heat treatments in comparison with the results seen for constant loads. These changes are significant in the initial 200 cycles and recovery strain tends to reach a steady state in the further cycling. The wire with shorter heat treatments exhibited lower fatigue life while higher recovery strain. Indeed, short-time annealing is commonly applied as shape setting or passivation treatment on Ni-Ti wire. Undisz et al.<sup>20</sup> investigated the influence of this heat treatment on the austenite finish ( $A_f$ ) temperature as well as the martensite-austenite transformation characteristics. It was found that short-time annealing at 450-600 °C can shift the  $A_f$  temperature from  $\Delta A_f = +25$  to  $-15$  K and can render the phase transformation either one-stage or two-stage.

Manufacturers have attempted to produce Ni-Ti superelastic and thermal elastic archwires by adjusting the temperature range where the transformation takes place aiming at isostress lines with the typical forces necessary for smooth dental movement. Since the development of these alloys is rather recent, most of the information available on their mechanical behavior relies on laboratory tests carried out on as-received archwires. Mechanical tests covering clinical situations are still scarce in the specialized literature. Mohlin et al.<sup>21</sup> strongly recommended a combination of mechanical tests simulating a clinical situation and surface examination for more accurate development of new wire materials.

NiTi shape memory alloys are suitable materials for medical applications due to their unique properties such as high corrosion resistance, biocompatibility, super elasticity and shape memory behavior. Beyond mechanical behavior, tribological and corrosion properties of Ni-Ti alloys are also required to be better comprehended in order to help dental practitioners in their everyday activities. Some recent researches have been developed with this purpose. Grosgeat et al.<sup>22</sup> examined the effects of both sterilization and surface treatment on the orthodontic arch wire friction coefficient. The overall results highlighted two types of tribological response: abrasive and adhesive behavior. Furthermore, Khalil-Allafi et al.<sup>23</sup> performed electrochemical tests with a Ni-Ti alloy into two physiological environments of Ringer solution and NaCl 0.9% solution. Results indicate that the breakdown potential of the NiTi alloy in NaCl 0.9% solution is higher than that in Ringer solution. The main identified corrosion mechanism was pitting corrosion.

The objective of the present study was to assess the mechanical behavior of Ni-Ti archwires as a function of the orthodontic treatment time. The hysteresis results were compiled in order to identify possible changes in the elastic behavior, which could affect the clinical performance of the wire.

## 2. Experimental Procedure

Austenitic superelastic (active in the transition temperature range  $A_s = -15 \pm 5$  °C) and martensitic thermal activated wires (active in the transition temperature range  $A_s = 30 \pm 5$  °C) made of Ni-Ti were examined.  $A_s$  is the austenitic start temperature. The average chemical composition of the alloys is listed in Table 1. The Ni-Ti wires were furnished by Dental Morelli Ltda (Sorocaba, São Paulo, Brazil).

Commercially available archwires of different diameters and intended for orthodontic use were purchased. Tensile tests were carried out on both as-received and used wires in order to compare the corresponding hysteresis profiles. Wires used for different periods of orthodontic treatment (1, 2 and 3 months) were tested. Three samples of each wire diameter were tested for both types of wires. A Shimadzu AG-I testing machine equipped with a 100 kN load cell was used. Individual wire samples were placed between two sandblasted aluminum plates positioned at each end of the sample to prevent sliding and deformation. The assembly was then gripped to

**Table 1.** Chemical composition of Ni-Ti alloys.

Element	Weight %
Nickel	55.79
Titanium	43.98
Oxygen	0.05 max
Carbon	0.05 max
Mn, Si, Cr, Co, Mo, W, V	< 0.01
Nb, Al, Zr, Cu, Ta, Hf	< 0.01
Ag, Pb, Bi, Ca, Mg, Sn, Cd	< 0.01
Zn, Sb, Sr, Na, As, Be, Ba	< 0.01
Fe	< 0.5
B	< 0.001

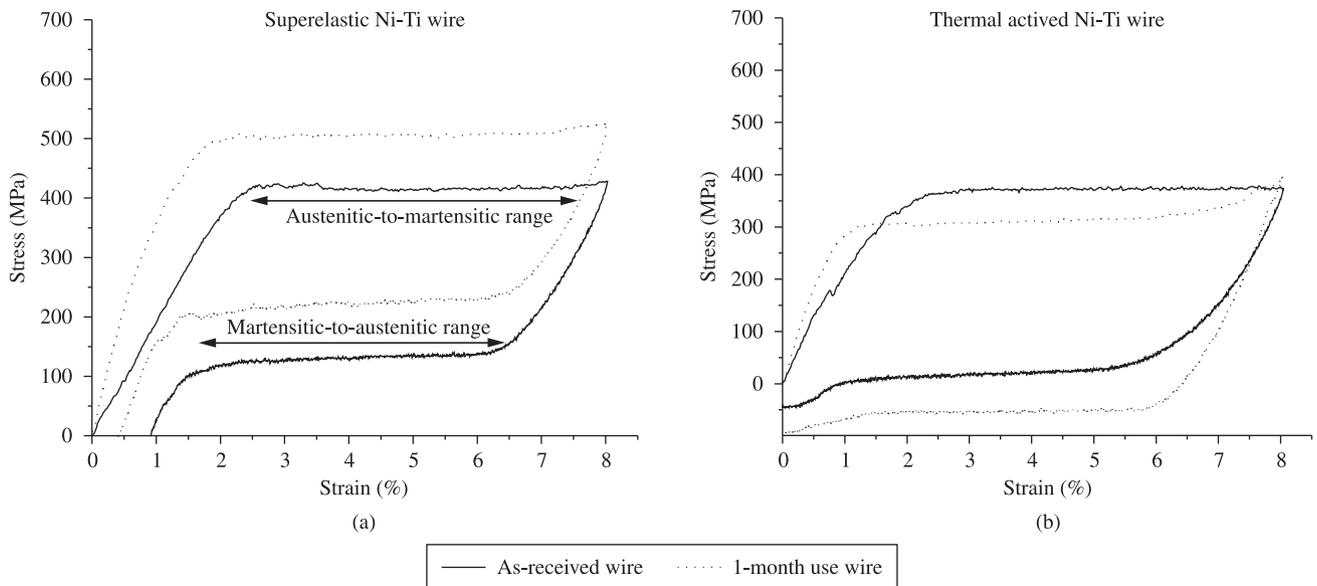
the machine allowing a free length of 50 mm. The samples were then loaded at a constant speed of 10 mm/min until the archwire reached 8% strain. After the test, the sample was unloaded at a constant speed of 3 mm/min.

Deformations due to an applied stress in SMA are recovered by heating the material above the austenite finish temperature ( $A_f$ ). Yoneyama et al.<sup>24</sup> conclude that the majority of Ni-Ti archwires exhibit  $A_f$  in a range from 17 to 32 °C. In addition, tests were carried out at  $35 \pm 5$  °C in order to simulate the average temperature of the oral cavity. The whole apparatus (sample and grips) was maintained inside a furnace at 35 °C during one hour for temperature homogenization before the test was started.

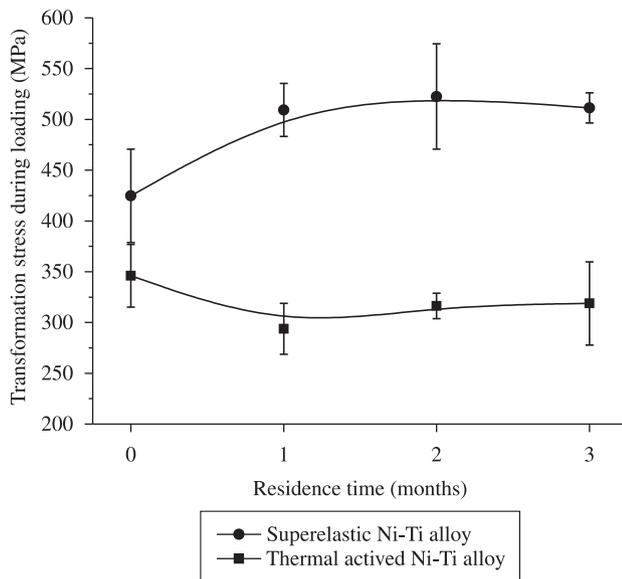
## 3. Results and Discussion

In the orthodontic treatment using conventional alloys (stainless steel and Co-Cr alloys), wires are usually replaced every 30 days to account for losses in stiffness and resilience which decreases the forces applied to the teeth. The new wires can either have the same diameter as before, if the magnitude of the force applied must be maintained, or be thicker, and therefore used to apply higher forces. The same approach is commonly used with Ni-Ti wires. However, the results presented herein revealed a significant difference comparing the load/unload isostresses of as-received and 1-month used wires. Figure 1 shows typical hysteresis diagrams obtained for as-received and 1-month used Ni-Ti wires 0.016" thick. The behaviors of superelastic and thermal activated wires are shown in Figure 1a and b, respectively. The stress curves upon loading depicted a linear elastic region followed by an isostress plateau for both superelastic and thermal activated wires. In the plateau, the elastic strain increased under constant tensile stress, resulting in smooth and constant forces acting on the dental element.

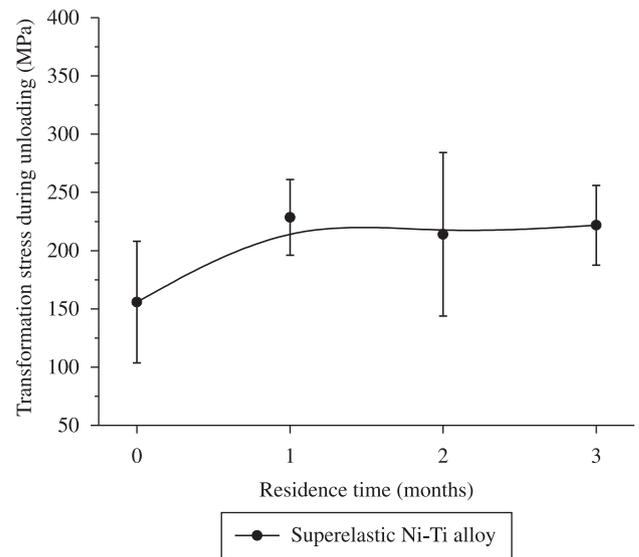
The complete (covering all strain range) isostress plateau found for the thermal activated wire (Figure 1b) during unloading cycling suggests that austenite to martensite transformation may be exclusively thermal-driven due to the fact that test temperature lay exactly within the temperature range previewed to the structural transition of such alloy. As mentioned before, each Ni-Ti alloy depicts a specific range of temperature for the phase transformation. The superelastic wires used in the scope of this study were austenitic active with  $A_s = -15 \pm 5$  °C whereas the thermal activated wire was martensitic active with  $A_s = +30 \pm 5$  °C. Based on Figure 1a, the superelastic wire was originally austenitic. In this case, it can be seen that the transformation to martensite is stress-induced and reversed upon unloading.



**Figure 1.** Stress-strain plots of as received and 1-month used 0.016 inch Ni-Ti wires. a) superelastic ( $A_s = -15 \pm 5^\circ\text{C}$ ); and b) thermal activated wires ( $A_s = 30 \pm 5^\circ\text{C}$ ).



**Figure 2.** Load isostress of superelastic and thermal activated Ni-Ti wires as a function of clinical residence time.



**Figure 3.** Unload isostress of superelastic Ni-Ti wire as a function of clinical residence time.

The results of all hysteresis tests carried out were compiled and graphically shown in Figures 2 to 6. Each point represents the experimental mean value along with its standard deviation. Experimental fits were inserted to represent the experimental points in Figures 4 to 6.

Figure 2 shows the load transformation stress of superelastic and thermal activated Ni-Ti wires as function of the residence time of the wire in the oral cavity. Superelastic wires developed higher levels of isostress compared to the thermal activated NiTi alloy. The isostress line of thermal activated wires decreased as the residence time increased while the opposite behavior can be observed in superelastic wires. The difference between load isostress values of both types of wire increased from 1-month clinical use and maintained nearly constant up to 3 months.

Figure 3 shows the evolution of unload transformation stress of superelastic wire with residence time. The loading and unloading evolution for this kind of wire (Figures 2 and 3) showed similar behaviors, despite the lower stresses generated upon unloading. This means that dental elements may be exposed to larger forces during a period of up to three months using the same wire. Smaller wire diameters, such as 0.014", develop smaller forces which are applied during clinical treatment. Some studies<sup>25,26</sup> state that slight and continuous forces can be more acceptable in order to propitiate better physiological conditions. On the other hand, Acar et al.<sup>27</sup> observed that the application of non-continuous forces may enable the periodontal tissue regeneration in between of each discontinuity.

Figure 4 shows the effect of the strain corresponding to isostress onset on residence time. The strain increased with increasing residence

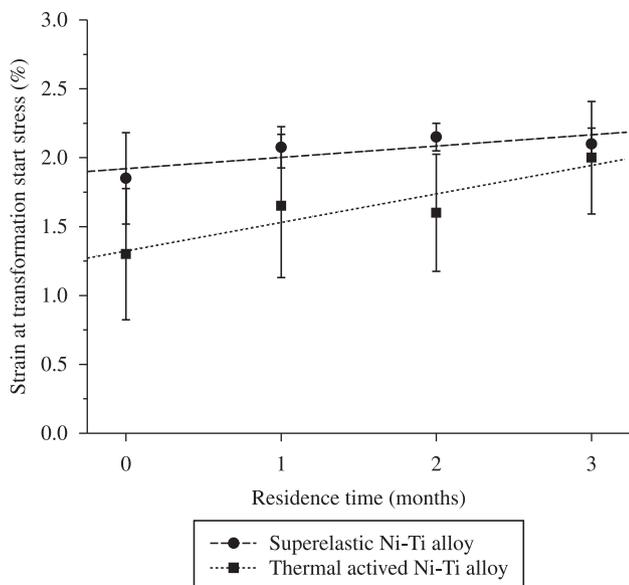


Figure 4. Strain at the beginning of isostress line vs. residence time for both Ni-Ti wires.

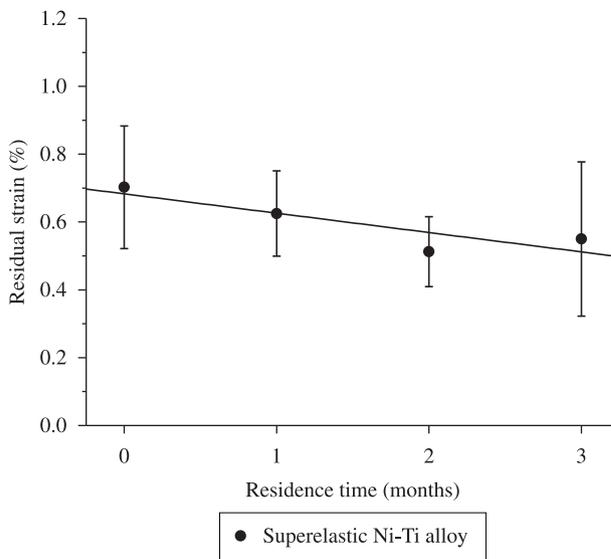


Figure 6. Residual stress of superelastic wire as a function of clinical residence time.

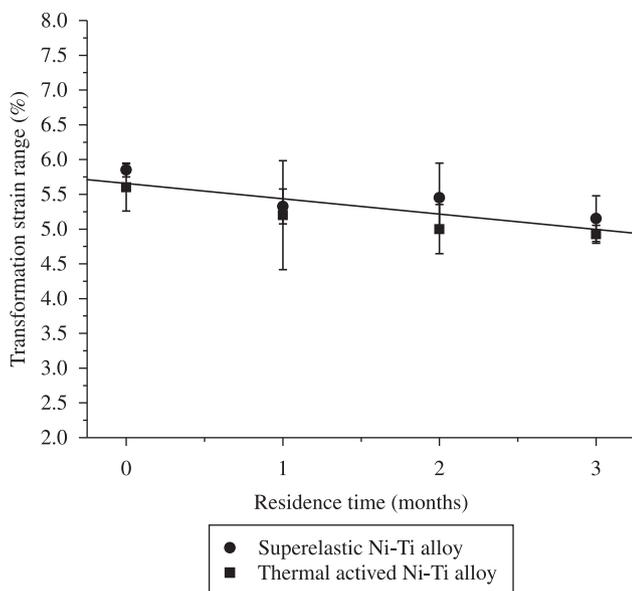


Figure 5. Evolution of deformation ability of superelastic and thermal activated wires with residence time.

time for both wires evaluated. As the period of clinical use increased the difference between the strain of superelastic and thermal activated wires decreased. Indeed, strains in the third month of treatment were quite close. In addition, as the time increased, the superelastic effect under hysteresis was postponed for any examined wire.

The measured strain values for both wires were fairly close. However, opposite trends with considerable differences between the isostress lines may be observed in Figure 2. From these results it can be inferred that thermal activated wires should be used only in the beginning of the treatment and be replaced earlier than usual, probably before 1 month due to significant stiffness loss under conventional treatment conditions. Orthodontic wires with low stiffness are suitable in the first stages of treatment whereas larger stiffness are required for the intermediate and final stages<sup>28</sup>.

The deformation ability of Ni-Ti wires are shown in Figure 5. It seems that a single fit is able to represent all experimental points. The transformation strain range slightly decreased as a function of residence time for both superelastic and thermal activated wires. The experimental results are very close for any time investigated. It seems that the type of wire did not affect the measured range of transformation strain. This can be observed not only for the as-received condition but also after clinical use (1 to 3 months). The deformation ability of Ni-Ti wires with superelastic effect are less affected for smaller residence times as those examined in the present study. However, larger residence times may be a concern due to the increase in the superelastic effect. This suggests that further investigations must be performed for long term orthodontic treatment.

After completely unloading the superelastic specimens, the residual strain was recorded (Figure 6). As it can be seen, the residual strain decreased as the residence time increased. Apart from a comparatively small amount of remaining deformation, it is clear that the superelastic Ni-Ti wires recovered strains of 8% with some amnesia. On the other hand, thermal activated wires recovered the same level of strain without amnesia.

#### 4. Conclusions

The following conclusions can be drawn from this study:

- Superelastic Ni-Ti wires exhibited higher load stress values than thermal activated wires. Opposite experimental trends were observed as a function of clinical residence time. Superelastic wires submitted to clinical treatment presented isostress values 60% larger than those found for thermal activated wires;
- Important stiffness losses characterized the thermal activated wires subjected to clinical conditions, whereas superelastic wires seem to be not affected during the period of treatment. Smoother forces can be applied to dental elements in the early stages of treatment using superelastic wires thinner than 0.014"; and
- A single experimental fit was enough to represent the experimental deformation capabilities of both evaluated wires. The transformation extent slightly decreased as residence time increased.

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