

# Study of the Impact of the Treatment Period on the Surface Characteristics of Nitinol Heat-Activated Orthodontic Wires

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**Abstract:** Heat-activated orthodontic arches exhibit a shape memory property, namely, when the temperature in the mouth increases, a phase transition is induced to the austenitic phase with the corresponding restoration of the wire's shape to the original one. The clinical experience has shown that during orthodontic treatment a wire remains in the patient's mouth most often between 4 and 6 weeks, and less often, between 8 and 12 weeks. The different transition effects in the heat-activated nickel-titanium arch wires are determined by various processes and factors, such as the overall composition, the time for annealing and the formation of alloys. In the work presented, the influence was investigated of the treatment period on the chemical composition and the structure of heat-activated nickel-titanium wires (3M Unitek, Monrovia, Calif.) with two different profiles – round with a diameter of 0.013 in and rectangular 0.016×0.022 in. The analyses were carried out by three independent techniques, namely X-ray diffraction analysis (XRD), scanning electronic microscopy (SEM) and energy-dispersive X-ray analysis (EDX). The results indicated that the basic chemical composition of the orthodontic wires investigated did not change during a treatment period of up to 10 weeks. When the duration of treatment was increased, we observed a gradual amorphization of the material (in both arch profiles). The presence of chemical alloys and deposits is associated with the individual oral hygiene of each patient.

**Keywords:** Nitinol Heat Activated orthodontic wires, XRD, SEM-EDX

## 1. Introduction

In recent years and through the application of special metallurgical treatments of the nickel-titanium alloy, wires have been developed such that undergo phase transitions as the temperature is varied. [1] Two types of orthodontic heat-activated wires are known: one containing copper in view of reducing the amount of nickel [2]; and the other not containing copper, with a composition close to that of the so-called classic Ni-Ti wires developed by Andreasen and Hilleman [3]. Special additional metallurgical treatments are applied in order to reduce the transition temperature. The effect of these transitions within the clinical application should be known by orthodontists in order to take full advantage of the properties of these products. The heat-activated orthodontic wires exhibit the property of shape memory and are thus called martensite active. The shape memory effect takes place during a thermo elastic transition from martensite to austenite; sometimes there appears an intermediate metastable R-phase [4]. The austenite possesses a higher modulus of elasticity, which leads to a higher arch hardness. The austenite phase is formed and then disappears when the temperature is raised, while the martensitic phase is formed and disappears as the temperature is lowered; this is detected by the changes in the elasticity of the heat-activated

wire at room temperature and at the mouth temperature (34 °C) [5], the effect being particularly enhanced when one eats ice cream and drinks hot coffee. The common practice is to prescribe consumption of hot drinks in order to increase temporarily the pressure and to speed up the tooth's motion. Conversely, the cold rinses relieve the discomfort [6]. The different transition effects in the nickel-titanium arch are determined by various processes and factors, such as the overall composition, the time of annealing and the formation of alloys. The alloys' behavior can be controlled by the chemical composition and the thermo-mechanical treatment during the manufacturing process [7-9]. According to various studies, the differences between the thermal and the stress transformations of the orthodontic archwires are due to the chemical composition [10, 11].

The aim of the present study was to analyze the impact of the treatment period on the chemical composition and the structure of heat-activated nickel-titanium wires (Nitinol Heat Activated of 3M Unitek, Monrovia, Calif.) with two different profiles - round with a diameter of 0.013 in and rectangular 0.016×0.022 in.

## 2. Material and Methodology

The proper selection of the material to be tested is essential to the goal set and the respective tasks. The different stages of treatment by a fixed orthodontic technique require the use of arch wires of various types with different cross-sections and for different treatment times. The clinical experience has shown that during the orthodontic treatment a wire resides in the mouth of the patient most often between 4 and 6 weeks,

and less often, 8 to 12 weeks. The wires investigated in the present work were initially sterilized in a type B autoclave at 121 °C for 21 minutes. The samples were divided into two subgroups depending on the time of residence in the mouth (after 4 and after 8 weeks) and their cross-section – round with a diameter of 0.013 in and rectangular 0.016×0.022 in as shown in Table 1.

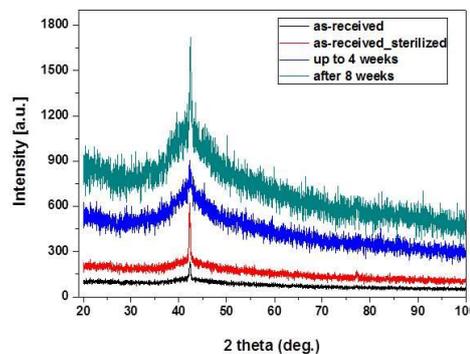
**Table 1:** Characteristics of the investigated Nitinol heat-activated orthodontic wires

Type of orthodontic wires	Profile [in]	Time of residence in the mouth
Th – round Nitinol heat-activated	diameter 0.013	Th0_as-received
		Th0_4-up to 4 weeks
		Th0_8-after 8 weeks
Th1 – rectangular Nitinol heat-activated	0.016×0.022	Th1_as-received
		Th1_4-up to 4 weeks
		Th1_8-after 8 weeks

The wires studied were analyzed by X-ray powder diffraction (XRD), X-ray dispersive analysis (EDX) and scanning electron microscopy (SEM). The XRD patterns were acquired within the range from 5.3 to 80° 2θ at a constant step of 0.02° 2θ by a Bruker D8 Advance diffractometer with CuKα radiation and a LynxEye position-sensitive detector. The phase identification was performed by means of the DiffracPlus EVA software using the ICDD-PDF-2 (2009) database. The microstructure of the wires' surface was studied by a Zeiss EVO MA-15 SEM with a LaB6 cathode on the polished cross-section of the samples. The chemical composition was determined by X-ray microanalysis using energy-dispersive spectroscopy (EDX) and an Oxford Instruments INCA Energy system. The qualitative and quantitative analyses were carried out at an accelerating voltage of 20 kV, an optimal condition for these samples.

## 3. Result and Discussions

The changes in the structure morphology and the surface's chemical composition of the heat-activated nickel-titanium (Nitinol) wires were studied as a function of the treatment period. The powder diffraction patterns obtained at room temperature for the two profiles of wires studied (round or rectangular) showed peaks typical for the cubic austenite phase. Figure 1 presents the XRD results of the Nitinol heat-activated orthodontic arch wires with a round cross-section. When raising the duration of treatment, we observed a gradual amorphization of the material (in both arch wire profiles), which was probably due to the increase of the defectiveness of the structure, on the one hand, and as a result of the chemical attack during treatment on the material's surface, on the other.



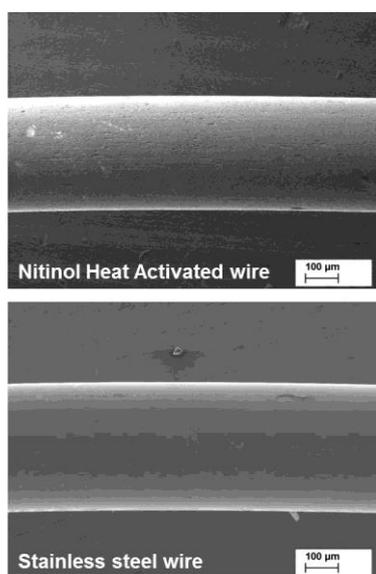
**Figure 1:** XRD patterns of the Nitinol heat-activated orthodontic arch wires at different stages of treatment.

The quantitative identification of the material's chemical composition was carried out by EDX. The average values of the elements' content are shown in Table 2. The results were compared with those obtained for as-received and sterilized wires, in order to ascertain the influence of the autoclave sterilization process. The EDX analysis confirmed that the processes of sterilization did not affect the main elements' content in the Nitinol heat-activated orthodontic archwires. Thus, having eliminated all external factors, we focused our research on acquiring statistically significant results. The latter demonstrated that the period of residence in the mouth had no significant effect on the proportion of elements in the orthodontic wires tested.

**Table 2:** Elemental content of the investigated Nitinol heat-activated orthodontic wires

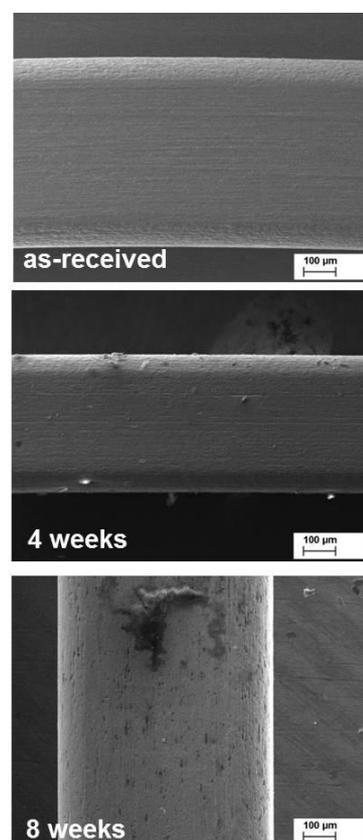
Ni - Ti heat-activated orthodontic wires	Elements, weight %				
	Ni	Ti	Cr	P, S, Cl, K, Ca	Total
Th0_ as-received	54.56	45.44	-	-	100
Th_1 as-received (sterilized)	54.54	45.46	-	-	100
Th_4 up to 4 weeks (0.013 in)	54.54	45.46	-	-	100
Th1_4 up to 4 weeks (0.016 ×0.022 in)	54.57	45.38	0.05	-	100
Th_8 after 8 weeks (0.013 in)	54.67	45.16	-	~ 0.17	100
Th1_8 after 8 weeks (0.016 ×0.022 in)	54.46	45.54	-	-	100

The SEM analyses established that the Nitinol heat-activated orthodontic archwires were characterized by a rough surface, in contrast to the smooth surface observed in stainless steel (SS) wires (Figure 2) [12]. This is due to the higher content of titanium, which adheres to the matrix during the production process and scratches the surface of the archwires.



**Figure 2:** SEM images of the surface of as-received orthodontic arch wires: Nitinol heat-activated (top) and stainless steel (bottom).

However, all types of orthodontic wires are subjected to the complex processes taking place in the oral cavity, such as the temperature differences, the saliva composition, and the accumulation of plaque and the establishment of a biofilm on surfaces.



**Figure 3:** SEM images of Nitinol heat-activated orthodontic wires: as-received; up to 4 weeks of treatment; after 8 weeks of treatment.

All these factors suggest a rapid aging of the metal orthodontic materials, affecting the morphology, structure and their mechanical properties.

Irrespective of the factory imperfections observed on the surface of the Nitinol heat-activated arch wires, the SEM analysis showed that no significant changes had occurred on the used wire's surface following the different treatment

periods (Figure 3). The small changes seen on the surface of the material could be the result of the chemical environment during treatment. This fact was confirmed by the EDX analysis, as shown in Table 2.

#### 4. Summary

Heat-activated orthodontic wires possess a shape memory property consisting in restoration of the arch's shape to the original one due to a phase transition to austenite when the temperature in the mouth is increased. The clinical experience has shown that during the orthodontic treatment an arch resides in the patient's mouth most often between 4 and 6 weeks, and less often, between 8 to 12 weeks.

The results obtained in this work demonstrate that the basic chemical composition of the surface of the investigated orthodontic arch wires does not change within a treatment period of up to 10 weeks. The small changes observed on the surface of the material could have arisen due to action of the chemical environment during treatment.

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