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Elemental composition and structural characteristics of as-received TriTanium™ orthodontic archwire

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Abstract. Orthodontic archwires are among the most important devices of fixed orthodontic therapy. Many types of archwires are made available on the market by various manufacturers with different elemental composition and structural characteristics. Knowing this information is important when choosing a suitable archwire for a particular stage of orthodontic treatment. The aim of our study is to characterize a new type orthodontic archwires (TriTanium™, American Orthodontics) before their placement in the oral cavity. To achieve the aim, we used modern methods for determining their elemental composition and structural characteristics: laser-induced plasma spectroscopy (LIBS), X-ray diffraction analysis (XRD), scanning electronic microscopy (SEM), energy dispersive X-ray spectroscopy (EDX) and differential scanning calorimetry (DSC). The results obtained from the qualitative elemental analysis by LIBS and the quantitative elemental analysis by EDX showed that Ni and Ti are the main elements in the archwire studied. The room-temperature XRD patterns showed peaks typical for a Ni-Ti alloy with an austenite-type structure. Monitoring the phase transitions by means of DSC measurements in the temperature range from −50 °C to +50 °C, we showed that in TriTanium™ archwires, besides the austenite to martensite transition, there exists a rhombohedral intermediate phase (R phase). This study will be useful in assisting orthodontists in applying appropriate nickel-titanium orthodontic archwires in the clinical practice.

1. Introduction

Fixed orthodontic therapy is based on inducing tooth movement by generating light and continuous forces. Orthodontic archwires are key elements in producing such forces; when selecting them, clinicians should take into consideration both their surface/morphology characteristics and their properties. In the intraoral cavity there exist complex factors that may influence the biomechanical behavior of the archwires during treatment [1]. Nickel-titanium alloys are among the most widely used; their clinical advantages in orthodontics arise from the fact that they can exist in two different
crystal structures: martensite (low-temperature, stress-free phase, with a low-symmetry crystal structure, such as tetragonal, rhombohedral, orthorhombic, monoclinic, or triclinic), and austenite (high-temperature, stress-free phase, with a high-symmetry crystal structure usually based on a cubic lattice) [2]. The two different phases of nickel-titanium are responsible for two clinically significant properties of nickel-titanium: shape memory and super-elasticity [3]. Most of the researchers claim that the manufacturing process allows for more consistent values of the transition temperatures, thus providing controlled force delivery individualized for each patient [4]. The shape memory is resulting from the thermal behavior of the archwires and is closely related to the transition temperature range (TTR) of each alloy. The shape-memory (thermally-activated) Ni-Ti archwires present a great variability in the TTR; therefore, the elastic parameters of each Ni-Ti archwire should be provided by the manufacturers to allow the achievement of the best clinical performance possible [5].

A new Ni-Ti based archwire was recently marketed, namely TriTanium™ (American Orthodontics). It was first used in orthopedics as an outer layer for joint implants, because of its good osteo-compatibility due to its high porosity [6]. The valuable properties of this alloy in orthodontics are the three elasticity zones in the anterior, mid-region and posterior segments. The teeth in those segments have different root systems; thus, an archwire applying different forces to those segments is more effective, especially in the initial phase of the fixed appliance treatment. It is believed that multi-force orthodontic archwires minimize the incidence of root resorption and improve patient comfort [7].

The insufficient literature and manufacturing data on TriTanium™ archwires motivated us to investigate the structure, chemical composition, morphology and thermal transformation temperatures of as-received commercially-available TriTanium™ archwires in the three elasticity zones. This will enable us to compare our results to the results of studies of used TriTanium™ archwires and reveal any changes in their structure and chemical composition.

2. Materials and methods
An as-received orthodontic TriTanium™ archwire (dimension 0.016×0.022 in) was cut in three pieces corresponding to the anterior (A-B), mid-region (B-C) and posterior (C-D) segments (figure 1). The use of the methods such as LIBS, EDX, SEM, DSC and XRD for investigation of materials was a key point of the work performed that did not only deal with theoretical aspects, but also had a strong experimental contribution.

Laser-induced plasma spectroscopy was used for qualitative determination of the archwires’ elemental composition. LIBS allows express elemental analysis of a wide range of samples without preparation. The method is implemented by focusing a powerful laser beam on the sample (dental braces) by a lens of 25 cm focal length. The laser source was a pulsed nanosecond Nd:YAG laser (Quanta Ray GCR3) generating a wavelength of 1064 nm at a repetition rate of 1 Hz and pulse energy 30 mJ. The focused laser beam ablates the sample, yielding laser-induced plasma. The light emitted by the plasma is transmitted through an optical fiber to the registration system – a Mechelle Spectrograph 5000 equipped with an Andor iStar ICCD camera controlled by the Andor SOLIS 4.13 specialized software. Analyzing the plasma spectrum and identifying spectral lines specific to each element make it possible to obtain information about the elements in the target. To quantify the archwires’ elemental composition, we used EDS on a Bruker Esprit 1.82 system (accelerating voltage of 20 kV, relative error 0.5 – 1 wt %). The surface morphology and elemental composition were studied using a ZEISS FESEM Ultra 55 SEM (acceleration voltage 4.0 kV). The crystalline structure was assessed by a Bruker D8 Advance powder diffractometer with a Cu-Kα target, within the range from 5 – 80° 20 at a constant step 0.02° 20. The phase transformation characteristics of the archwires segments were studied using a Perkin-Elmer-
8000 DSC device (temperature range –170°C – +600 °C) with a model PE-TGA4000 TGA attachment. Before introducing the sample for each individual test, the DSC apparatus was calibrated with indium. The samples were scanned from –50 °C to +50 °C at a rate of 20 °C /min for heating and cooling.

3. Results and discussions
The LIBS spectra in the range of wavelengths 360 – 370 nm of the as-received TriTanium™ orthodontic archwire are presented in figure 2.

To determine the presence of each element in the sample, the registered spectra are compared to the spectra of pure elements. As seen in figure 2, the sample is made of a Ni-Ti alloy.

The results are qualitative and indicate the presence or absence of an element in the segments examined. The data of the EDS quantitative elemental analysis of the as-received TriTanium™ archwire in all segments are presented in table 1. As seen, the as-received archwire contains pure nickel and titanium with no significant difference in the concentration in the three regions of the archwire. The SEM images (figure 3) also show no significant differences on the surface of the segments. The morphology is quite similar for the three segments; one can also notice the presence of porosity, demonstrating an irregular surface that can influence the archwires properties during treatment.

Table 1. Elemental content of the TriTanium™ archwire investigated.

| TriTanium™ Region       | Anterior (A-B) | mid-region B-C | Posterior C-D | Error |
|-------------------------|----------------|----------------|--------------|-------|
| Ti                      | 45.50 wt %     | 45.60 wt %     | 46.30 wt %   | ±0.8 %|
| Ni                      | 54.50 wt %     | 54.40 wt %     | 53.70 wt %   | ±0.8 %|

The crystalline structure of the TriTanium™ archwire assessed by XRD made at room temperature (figure 4) showed a cubic structure with parameters a = b = c = 3.1323, β = 90°, for all segments. Besides the austenite crystal structure, a small degree of amorphization of the material was seen, which can be due to contamination.

We further performed DSC analyses to determine the thermal behavior of the archwire’s three segments in the temperature range of –50 °C to +50 °C, to detect shifts in the transformation
temperatures of finished orthodontic archwires that are strained to different extent. This may serve as a marker of the changes and the applicability period of the archwires used by patients. Table 2 shows the measured Austenite, R-phase, Martensite temperatures and the enthalpy changes for phase transformation during cooling and heating within the three segments of the TriTanium™ archwire.

The anterior and mid-region segments exhibited higher austenitic temperatures as compared to the posterior segments with final austenitic temperature (Af) ranging from 25.55 °C to 20.82 °C, which is beneficial in the early stages of orthodontic treatment.

Table 2. DSC results within the three segments of the TriTanium™ archwire.

| Wire Brand | Wire Segment | Heating process | Cooling process |
|------------|--------------|-----------------|----------------|
|            |              | As temp (°C)    | Peak temp (°C) | Af temp (°C) | Enthalpy (J/g) | Rs (°C) | Peak (°C) | Rf (°C) | Enthalpy (J/g) | Ms (°C) | Peak (°C) | Mf (°C) | Enthalpy (J/g) |
| TriTaniumTM| anterior (A-B)| 16.16           | 21.83          | 25.55        | 8.5680        | 12.99   | 9.29      | 6.93    | -1.5011       | -21.07   | -28.97      | -36.80  | -3.9354       |
|           | mid-region (B-C)| 11.92           | 18.18          | 21.23        | 8.5100        | 12.76   | 9.78      | 6.57    | -12.89        | -30.93   | -36.88      | -46.17  | -2.8194       |
|           | posterior (C-D) | 8.26            | 14.71          | 20.82        | 6.4765        | 12.28   | 8.05      | 3.17    | -0.8698       | -38.95   | -43.61      | -35.11  | -0.3484       |

4. Conclusions

The results obtained by LIBS provided qualitative information on the chemical composition and showed that the sample investigated is made of a Ni-Ti alloy. The EDS quantitative elemental analysis showed that the TriTanium™ orthodontic archwire contain pure nickel and titanium with no significant difference in the concentration in the three regions of the archwire. The room temperature XRD patterns showed peaks typical for a Ni-Ti alloy with austenite-type structure. The phase transitions were studied by means of DSC in the temperature range of −50°C to +50°C. The different force values obtained depend on the archwire segment (anterior, mid-region, posterior).

The anterior and mid-region segments show higher austenitic temperatures as compared to the posterior segments; with final austenitic temperature (Af) ranging from 25.55 °C to 20.82 °C, thus presenting benefits for the early stages of orthodontic treatment. We plan comparative analyses of the above results and future results obtained on used samples, in order to provide guidelines for a more precise application of TriTanium™ orthodontic archwires in the clinical practice.

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