

Analysis of elemental composition of a heat activated, multi-force, nickel titanium orthodontic archwire

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The heat activated, multi-force, nickel-titanium archwire have three distinct force regions. This archwire places the correct force to the appropriate region (anterior, bicuspid and posterior) of the arch to quickly torque, level and align, without sacrificing patient comfort. In this work, analysis of such archwire (cross section 0.41×0.56 mm.) with variable longitudinal elasticity is done. The elemental composition is obtained by different wire's regions with 5 mm separation by Laser-Induced Breakdown Spectroscopy (LIBS) and two other independent methods such as X-ray diffraction analysis (XRD) and Energy Dispersive X-ray method (EDX). The large area of application of LIBS is due to its ability to perform microprobe elemental analysis of wide variety of samples with no preliminary sample preparation. Pulsed Nd:YAG laser (λ 1,064 μm , pulse duration 8 ns) is focused on the sample surface, causing ablation and formation of laser-induced plasma plume. Collecting and processing the plasma emission gives us information of the sample elemental composition. The obtained results of unused wire provide useful knowledge needs for further investigations on the wire changes during the treatment.

Key words: thermo activated orthodontic archwire, XRD, SEM-EDX, elemental composition.

INTRODUCTION

Orthodontic arch wires are one of the main and most important components of the treatment with fixed appliances. They have evolved tremendously due to the major advances in materials science and metallurgy. During the different phases of treatment arch wires with specific qualities are used, which explains the vast choice of alloys and sizes. Arch wires are required to preserve their chemical and physical characteristics [1]. In the early phases of treatment, during teeth levelling, the most important characteristics of the selected wire are its elasticity and the amount of force, generated after the initial deflection. At first stainless steel arch wires were used, but because of their small elastic range [2, 3] they delivered very high forces. Soon they were

replaced by Ni-Ti archwires [4, 5] and superelastic Ni-Ti archwires [6, 7]. These wires deliver an almost constant force, regardless of the initial bending of the wire [8, 9]. The next step in the development of the aligning archwires was the heat activated wires. As a function of temperature these wires alter their austenitic structure to martensite and vice versa and thus the amount of force can be regulated [10, 11].

When an archwire with uniform qualities is deflected it generates an equal amount of force along its whole length although it acts on teeth with different root surface. This has led to one of the latest inventions in orthodontic material science – the invention of thermodynamic wires that deliver differential forces. The characteristics of these wires allow the ratio of delivered force/surface area to be almost equal for all teeth. This allows a shortening of the aligning phase of the treatment. Despite their improved elastic properties, it is interesting to analyze their chemical composition and structure in the different areas of elasticity.

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The aim of this work is to analyze the composition and structure of a heat-activated wire with differential force delivery.

MATERIAL AND METHODS

In this work, analysis of heat activated nickel-titanium orthodontic archwire (cross section 0.41×0.56 mm) with variable longitudinal elasticity is done. The archwire has 3 regions of elasticity – anterior (A), bicuspid (B) and posterior segment (C). According to the manufacturer the ratio of the delivered force in the three segments is approximately 1:1,5:2.

The studied wires were analyzed by X-ray powder diffraction method, scanning electron microscopy (SEM), X-ray dispersive analysis (EDX). The technique of Laser-Induced Breakdown Spectroscopy (LIBS), also was used as an independent method for elemental composition obtained by different wire’s regions with 5 mm separation.

Powder X-ray diffraction patterns were collected within the range from 5.3 to $80^\circ 2\theta$ with a constant step $0.02^\circ 2\theta$ on Bruker D8 Advance diffractometer with $\text{CuK}\alpha$ radiation and LynxEye position sensitive detector. Phase identification was performed by the program DiffracPlus EVA using ICDD PDF-2 (2009) database. The microstructure of the wires surface was studied by means of Zeiss EVO MA-15 scanning electron microscope (SEM) with LaB_6 cathode on the polished cross-section samples. The chemical composition was determined by the X-ray microanalysis using the energy dispersive spectroscopy (EDS) method and Oxford Instruments INCA Energy system. The qualitative and quantitative

analyses were carried out at an accelerating voltage 20 kV, an optimal condition for these samples.

In this work, we perform LIBS analysis on the studied orthodontic archwire for verifying the elemental composition obtained by XRD analysis. Laser-induced breakdown spectroscopy (LIBS) is widely applied for elemental composition determination because it can perform fast, preparation-free, microprobe analysis of solid samples [12, 13]. In LIBS technique, powerful pulsed laser is focused on the sample surface, causing ablation and formation of laser-induced plasma plume. Collecting and processing the plasma emission gives us information of the sample elemental composition. LIBS is capable of qualitative and in some conditions of quantitative analysis. The setup we use consists of pulsed Nd:YAG Quanta Ray GCR3 laser ($\lambda 1,064 \mu\text{m}$, pulse duration 8 ns), focused with a 15 cm lens on the archwire surface. The emission is collected with optical fiber (50 μm core) coupled to Mechelle 5000 spectrograph equipped with iStar DH734 iCCD camera which is controlled by PC. Each spectrum is accumulated from 30 laser pulses and registered in 300–800 nm spectral region. Under these experimental conditions the spectra of the pure Ti and pure Ni samples also are measured.

EXPERIMENTAL RESULTS AND DISCUSSION

The results obtained by XRD X-ray diffraction analysis (XRD analysis done at room temperature) show peaks typical for cubic austenite-type phase, Fig. 1. The position of the peaks of samples taken from different part of the archwire does not show

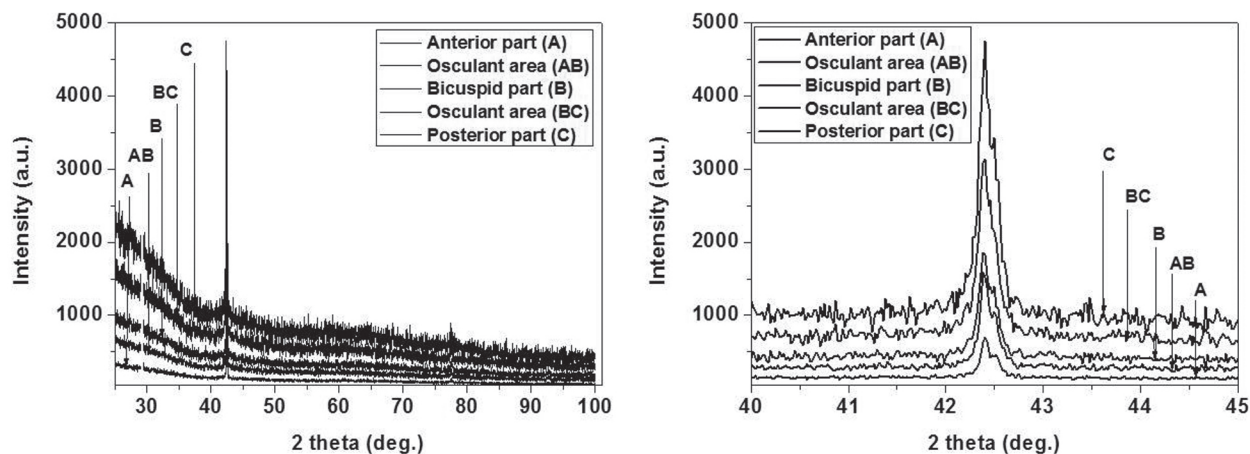


Fig. 1. XRD patterns of the main regions of orthodontic Variable Force 3 Archwire

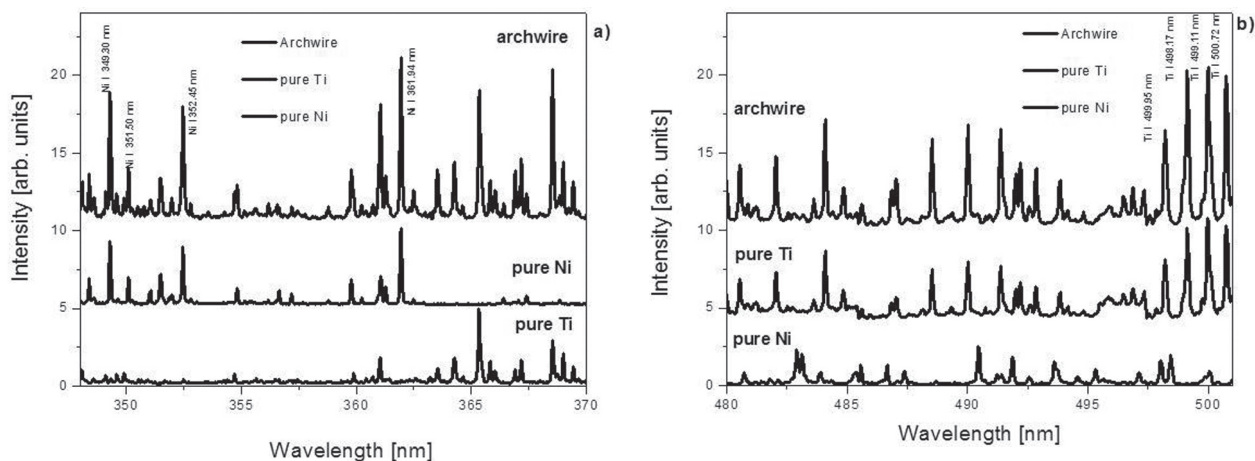


Fig. 2. Laser-induced plasma emission spectra from orthodontic archwire, pure Ti and pure Ni samples

differences of unit cell parameters which lead us to the conclusion that the phase composition of arc is constant all along the wire.

The measured orthodontic archwire spectrum by LIBS is plotted against the spectra of pure Ti and pure Ni. In Fig. 2a is shown part of the spectra (348–370 nm) in which analytical Ni I lines (361.94 nm, 352.45 nm, 351.50 nm and 349.30 nm) [14, 15] are present. Similarly, Fig. 2b displays portion of the spectra (480–501 nm) with analytical Ti I lines (500.72 nm, 499.95 nm, 499.11 nm, 498.17 nm) [14, 15]. It is seen in the figures that the analytical lines of both pure elements are clearly observable in the spectrum of the orthodontic archwire. This verifies that only Ni and Ti are present in the elemental composition of the archwire.

The presented scanning microscopically pictures (Fig. 3) are of main areas of the wires’ surface, where a point element analysis is made as well.

Table 1. Elements content of selected points of orthodontic archwire

Spectrum №	1	2	3	4
Elements				
Titanium, Ti (wt.%)	63.24	64.20	46.37	46.35
Nickel, Ni (wt.%)	36.76	35.80	53.63	53.65
Total, %	100	100	100	100

Wire is not coated with any layer and the average values of the element composition is approximately Ti 46.28 wt.% and Ni 53.72 wt.%. The surface microstructure is identical for all different parts of wires, which were confirmed by (Energy Dispersive X-Ray method (EDX), Table 1.

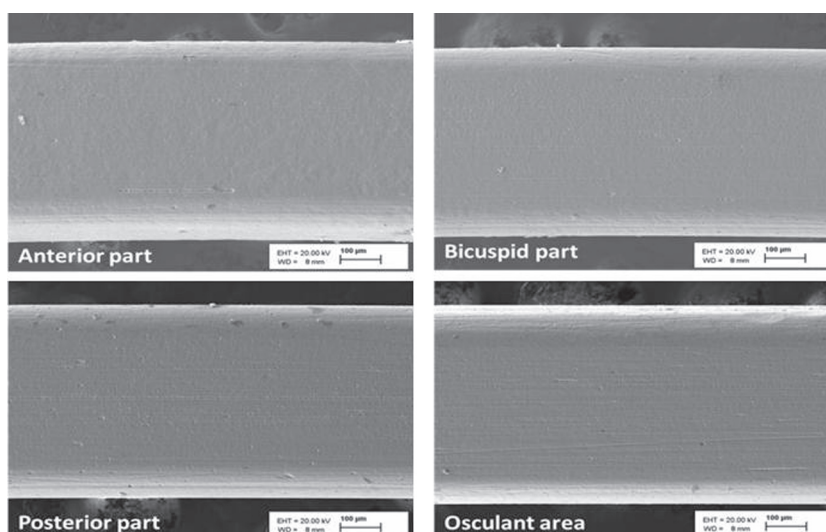


Fig. 3. Scanning electron microscopy images of the main regions surface of orthodontic archwire



Fig. 4. Scanning electron microscopy image of the cross-section of Posterior part of orthodontic archwire

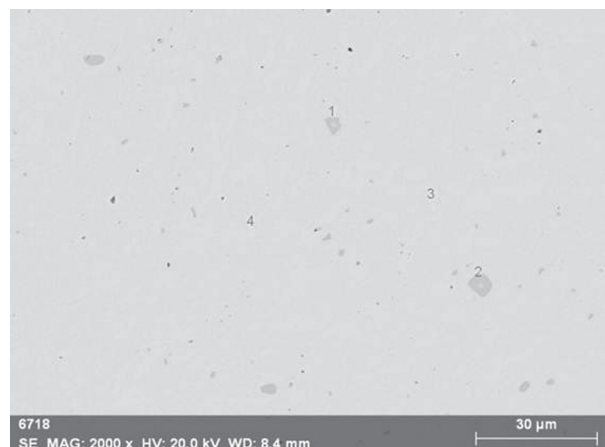


Fig. 5. SEM image on the surface of orthodontic archwire

Table 2. Elements content of investigated points of orthodontic archwire

Spectrum №	1	2	3	4	5	6	7	8
Elements								
Titanium, Ti (wt. %)	46.44	46.48	46.57	46.04	46.65	46.69	46.05	46.50
Nickel, Ni (wt. %)	53.66	53.52	53.43	53.96	53.35	53.31	53.95	53.50
Total, %	100	100	100	100	100	100	100	100

The analyzed cross-section of the end part (Posterior part) wire by Energy Dispersive X-Ray method (EDX) shows good homogeneity of the proportion of elements (Fig. 4, Table 2). There are small inclusions (2–5 μm) inside the compound, which composition is similar to Ti₂Ni (Fig. 5, Table 2), which could be result of producing process (exp. different annealing process).

CONCLUSION

From the carried out tests it is established that there are no significant changes in the chemical composition of the surface of the thermally activated wires with variable longitudinal elasticity. The obtained results of unused wire provide useful knowledge needs for further investigations on the mechanical properties of the wire as well as for structure changes during the treatment period.

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ИЗСЛЕДВАНЕ НА ТЕРМОАКТИВИРАЩА НИКЕЛ-ТИТАНОВА ОРТОДОНТСКА ДЪГА С ДИФЕРЕНЦИРАНО ОСВОБОЖДАВАНЕ НА СИЛА

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(Резюме)

Термоактивиращата никел-титанова дъга е с диференцирано освобождаване на сила в три различни зони. Дъгата освобождава подходяща сила в различните сегменти (фронтален, премоларен и дистален) на зъбната дъга, което позволява бърза изява на торка, вертикално и хоризонтално нивелиране, без да се нарушава комфорта на пациента. В настоящото изследване е анализирана такава дъга с променлива по дължината си еластичност и сечение $0,41 \times 0,56$ mm. Проследен е елементният състав в различните участъци на дъгата през интервал от 5 mm с помощта на Laser-Induced Breakdown Spectroscopy (LIBS) и два други независими метода като X-ray diffraction analysis (XRD) and Energy Dispersive X-ray method (EDX). Широката област на приложение на LIBS се дължи на възможността му да извършва елементен анализ на микропроби в широк спектър на образци без предварителната им подготовка. Импулс от Nd:YAG laser (λ 1,064 μ m, импулс с продължителност 8 ns) се фокусира върху повърхността на образеца, предизвиквайки аблация и образуване на лазерно-индуциран облак. Улавянето и обработката на плазмените емисии дават информация за елементния състав на образеца. Получените резултати от анализа на неизползвана дъга са необходими като основа за бъдещи изследвания на промените в дъгите при използването им в лечебния процес.