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Influence of the treatment period on the morphology and the chemical composition of the thermally activated orthodontic archwires

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The thermally activated nickel-titanium archwires are widely employed in orthodontics because they present shape memory effect and super elasticity. The addition of copper to nickel-titanium alloy enhances the thermal properties of the wire while maintaining precise control of forces, comparing to "pure" nickel-titanium alloy. The purpose of this study is to investigate the chemical composition of the surface of thermally activated copper-nikel-titanium archwires with different cross sections (round – 0.33 mm and rectangular – 0.41 × 0.56 mm). For the purpose of comparison, the origin wires (unused and sterilized ones) are also investigated as a control group. All studied orthodontic wires have transition temperature range at 35 °C. The analyses are carried out by three independent methods as X-ray diffraction analysis (XRD), Scanning Electronic Microscope (SEM) and by an Energy Dispersive X-Ray method (EDX).

The obtained results show that the processes of sterilizations of the investigated orthodontic archwires have no adverse effects on their properties. 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 after treatment period up to 6 and after 8 weeks. This period covers the average time of residence of the orthodontic wires in the mouth of the patient.

Key words: thermally activated orthodontic wires, XRD, SEM-EDX, surface influence.

INTRODUCTION

The copper-nickel-titanium shape-memory alloys are applied in many areas, due to their unique thermal and mechanical properties [1]. One of the successful applications of the archwires made from these alloys is in orthodontics because of their characteristics, such as low hardness, flexibility, large stored energy, biocompatibility, the thermoelastic shape-memory effects and super-elasticity [2]. The orthodontic shape-memory archwires are also called martensite active, because they can be deformed in the martensitic phase, but when exposed in the oral cavity they are transformed into austenite, allowing the arch to regain its original shape [3].

During the initial placing of the orthodontic arch into the slot of the bracket it bends according to the dental arch when passing into austenitic phase orthodontic the orthodontic archwire is able to "remember" its pre-established form [4, 5]. Thus the orthodontic arch begins to influence and to move the teeth in the direction where they take the correct position, set by the shape of the arch. When lowering the temperature, the alloy is converted into martensite and becomes flexible and easily deformable.

Thermally activated arches were created in 1994 by Ormco Corporation. They are provided to the orthodontists in three temperature options: 27 °C, 40 °C and 35 °C, corresponding to the upper austenitic temperatures that finalize the transformation from martensite to austenite [6]. The element's analysis carried out by EDX shows that the variants of the three thermally activated archwires have similar composition of approximately 44 wt.% nickel, 51 wt.% titanium, small quantities of copper

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– 5 wt.%, and according [7] 0.2 wt.% to 0.3 wt.% chromium instead of titanium quantity. Kusy [8] reports that these arches contain at least 5–6% weight percentage of copper and 0.2% to 0.5% weight percentage of chromium. The archwire with transition of 27 °C contains 0.5% weight percentage of chromium in order to balance the influence of copper on the increase of the transition temperature over this in the mouth, and the 40 °C variant contains 0.2 wt.% chromium.

Regardless of the type of the alloys from which the arches are made, they are subject to the ongoing mouth chemical and electrochemical reactions resulting in degradation of the existing and formation of new chemical compounds. The conditions in the oral cavity are very aggressive, causing corrosion of metal alloys. All these factors suggest a rapid aging of the metal orthodontic materials, affecting the morphology, structure and their mechanical properties.

The objective of the present study is to trace the influence of the residence period in the patient's mouth on the morphology and chemical composition of the copper-nickel-titanium orthodontic arches with two different profiles (round – 0.33 mm and rectangular – 0.41 × 0.56 mm). The results are compared with those obtained for unused (factory) and autoclaved unused archwires in order to establish the changes occurring in their properties after a prolonged stay in the oral cavity.

MATERIAL AND METHODOLOGY

The present study is on the selected orthodontic archwires with two different cross sections used for the treatment that stayed in the patient's mouth, respectively up to 6 and after 8 weeks. This period covers the average time of residence of the orthodontic wires in the mouth of the patient in order to perform a certain stage of orthodontic treatment. The type D0 wires are with round cross section 0.33 mm, and the second D1 have a rectangular cross section – 0.41 × 0.56 mm, Table 1. Two wires are used of each type, and are compared with the corresponding starting wires, called as-received.

The studied wires were analyzed by X-ray powder diffraction (XRD), X-ray dispersive analysis (EDX) and scanning electron microscopy (SEM). Powder X-ray diffraction patterns were collected within the range from 5.3 to 80° 2θ with a constant step 0.02° 2θ on Bruker D8 Advance diffractometer with CuKα 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 analyzes were carried out at an accelerating voltage 20 kV, an optimal condition for these samples.

RESULTS AND DISCUSSION

For the purpose of comparison, the origin wires (control group) were also analysed before and after avtoclave sterilization process. The specific requirements for the prevention and control of the healthcare associated infections require every orthodontist not to allow contaminated orthodontic archwires to be placed in the patient's mouth. Most of the leading companies offer their products in individual sealed packages suitable for autoclaving. The autoclaving was carried out in an autoclave type B at 121 °C for 21 min.

After the analysis done by XRD Scanning Electronic Microscope and by an Energy Dispersive X-Ray method (EDX) it is established that there are no significant changes in the chemical composition

Table 1. Characteristics of investigated cooper –nickel –titanium orthodontic wires

Type of orthodontic wires	Cross section [mm]	Time of residence in the mouth
D0 – round thermal activated cooper-nickel-titanium	0.33	D0_as-received
		D0_6 – up to 6 weeks
		D0_8 – after 8 weeks
D1 – rectangular thermal activated cooper-nickel-titanium	0.41 × 0.56	D1_as-received
		D1_6 – up to 6 weeks
		B1_8 – after 8 weeks

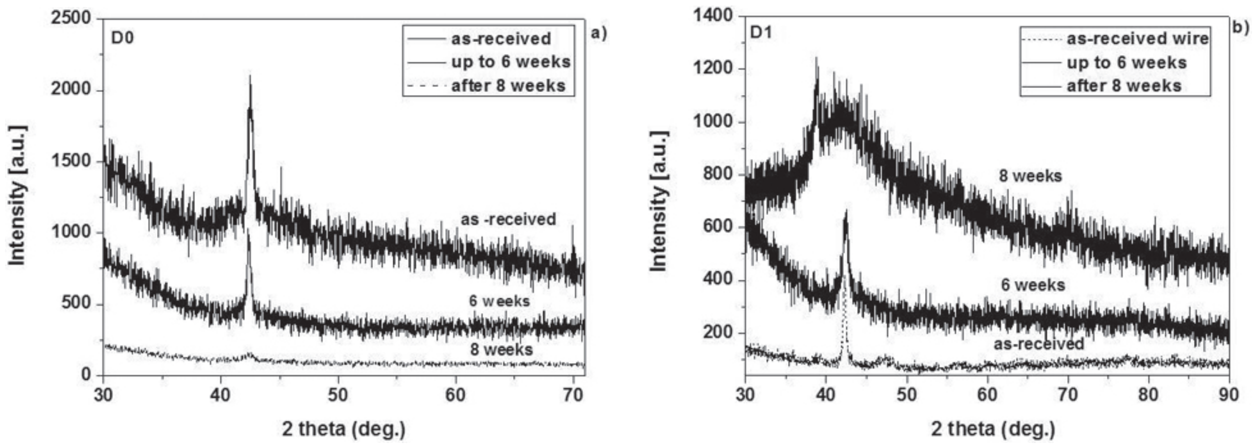

Fig. 1. a) XRD patterns of the orthodontic archwires type D0 at different stages of treatment; **b)** XRD patterns of the orthodontic archwires type D1 at different stages of treatment

of the surface of the autoclaved wires. There are also no changes in the surface characteristics of the archwires made of the investigated alloys. The obtained results show that the processes of autoclaving of the investigated orthodontic archwires have no adverse effects on their properties [9].

Thermally active orthodontic archewires have shape-memory property and are called martensite active. They can be deformed in their martensitic phase, as when increasing the temperature in the mouth a phase transformation to the austenitic phase is induced, and the corresponding restoring of the arch's to the original. The copper-containing thermally activating archwires have constant and weak forces when activating and deactivating in comparison with Ni-Ti arches. Adding copper to the nickel-titanium alloy is effective to stabilize the thermal transformation and to narrow the temperature hysteresis between the formation of the austenite during heating and martensite during cooling. Furthermore, the copper affects the mechanical properties of nickel-titanium alloy. Powder diffraction patterns obtained at room temperature for the two profiles of arches studied (round or rectangular) show peaks typical for cubic austenite phase (Fig. 1a and Fig. 1b). With the gradual increase of the residence's period in the mouth, it is noticed the formation of amorphous "humps" in the diffraction patterns of both wire profiles which probably is a result of two occurring phenomena: first – an increase in defectiveness of the structure (micro-deformations and strains) as a result of deformation effects and second increase in defectiveness of the structure due to the corrosive processes starting from the surface of the material as a result of the chemical interaction with environment during treatment. The latter is more visible in bends of rectangular section (SEM Fig. 3).

The information about the change in the surface morphology of the orthodontic wires is obtained by scanning electron microscopy (SEM), Fig. 2. The presented scanning microscopical pictures are of unused wire and after treatment period up to 6 and after 8 weeks correspondingly.

The results indicate that the wire's surface does not change for the examined period of treatment. On

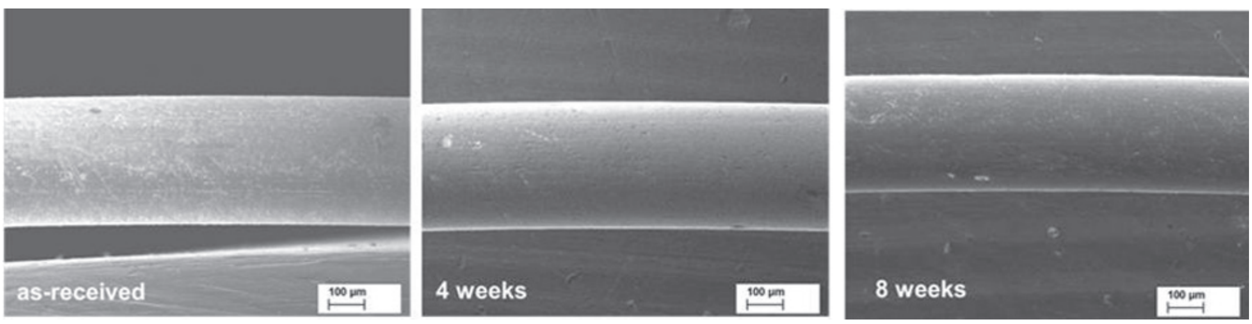

Fig. 2. Scanning electron microscopy images of cooper –nickel –titanium with round cross-section 0.33 mm

Table 2. Elements content of investigated D0-cooper–nickel–titanium orthodontic wires with round cross-section 0.33 mm

Round thermal activated cooper-nickel-titanium (0.33 mm)	Elements, weight %			
	Cu	Ni	Ti	Cr
Industrial data (information)	6.5	50	43	0.50
D0_as-received	*5.51*	*48.75*	*45.18*	*0.23*
D0_6 – up to 6 weeks	5.49	48.60	45.66	0.25
D0_8 – after 8 weeks	5.47	48.58	45.67	0.28

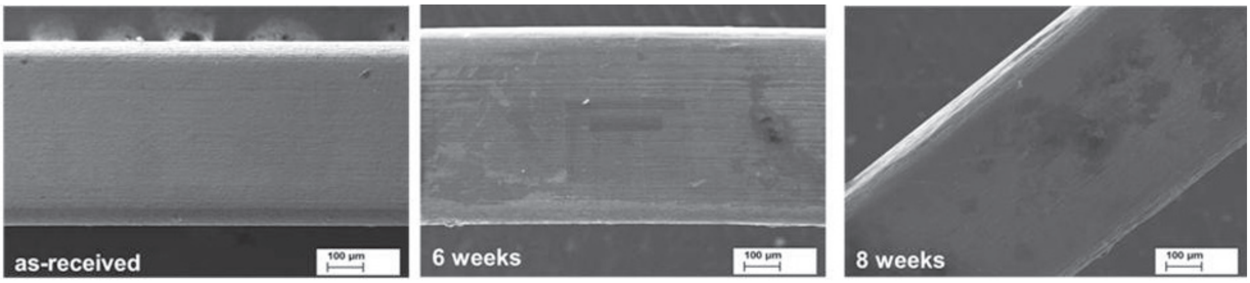

Fig. 3. Scanning electron microscopy images of cooper–nickel–titanium with cross section 0.41 × 0.56 mm

the wires' surface that has stayed in for 8 weeks surface irregularities are observed, which we assume to appear as a result of mechanical deformation effects. Additionally, the surface of the archwires was studied in details by means of energy dispersion technique (EDX).

The method used allows quantitative identification of the chemical composition by elements and focuses on the corrosion changes of the alloy rather than commenting on the complex oral flora. The average values of the element composition are shown in Table 2. The period of residence in the mouth has no significant effect on the proportion of elements in the tested orthodontic wires.

The obtained results of the second type studied orthodontic wires (rectangular cross-section) show similar behaviour. As a result of the carried out analyses by SEM was established that small changes have occurred in the structure on the surface of the rectangular wires after treatment process, Fig. 3. The determined chemical composition by EDX analysis is presented in Table 3.

The analysis confirms the presents only of the main components composition. The observed changes of the surface of the material could be as a result of the chemical environment during treatment. It is difficult to trace and investigate the corrosion according to the treatment's time stage and

Table 3. Elements content of investigated cooper–nickel–titanium orthodontic wires with cross section 0.41 × 0.56 mm

Rectangular thermal activated cooper-nickel-titanium (0.41 × 0.56 mm)	Elements, weight %			
	Cu	Ni	Ti	Cr
Industrial data (information)	6.5	50	43	0.50
D1_as-received	5.47	48.29	45.58	0.31
D1_6 – up to 6 weeks	5.41	48.55	46.05	–
D1_8 – after 8 weeks	5.75	48.11	45.82	0.27

the wires' residence in the mouth due to the individuality of each patient (type of food, hygiene, age, gender, etc.).

CONCLUSION

Thermally active orthodontic archewires have shape-memory property and are called martensite active. Regardless of the type of the alloys from which the arches are made, they are subject to the ongoing mouth chemical and electrochemical reactions resulting in degradation of the existing and formation of new chemical compounds. 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 (both – round and rectangular cross section) after treatment period up to 6 and after 8 weeks. The period covers the average time of residence of the orthodontic wires in the patient's mouth.

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ВЛИЯНИЕ НА ВРЕМЕТО НА ПРЕСТОЙ В УСТАТА НА ПАЦИЕНТА ВЪРХУ МОРФОЛОГИЯТА И ХИМИЧНИЯ СЪСТАВ НА МЕД-НИКЕЛ-ТИТАНОВИТЕ ОРТОДОНТСКИ ДЪГИ

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(Резюме)

Топлоактивиращите никел-титанови дъги са щироко използвани в ортодонтията заради свойството памет на формата, което притежават и голямата им еластичност. Добавянето на мед към никел-титановата сплав подобрява температурните трансформации в дъгата и поддържа по-постоянни и по-слаби връзки при активиране и деактивиране в сравнение с дъгите от „чиста“ никел-титанова сплав. Целта на настоящата работа е да изследва химичният състав по повърхността на топлоактивиращи мед-никел-титанови ортодонтски дъги с две различни сечения (кръгли – 0,33 mm и правоъгълни 0,41 × 0,56 mm) използвани за лечение. Получените резултати са сравнени с изследвания направени и на неизползвани и неизползвани автоклавирани дъги, споменати като контролна група. Всички изследвани топлоактивиращи дъги се характеризират с температура на преход 35 °C. За проведените анализи са използвани три независими метода: Рентгеноструктурен анализ (XRD), Сканиращ електронен микроскоп (SEM) и Енергийно-дисперсионен анализ (EDX).

Резултатите показват, че процесите на стерилизация не променят свойствата на дъгите. От проведените структурни изследвания е установено, че основният химичен състав на топлоактивиращите дъги не се променя в периода на лечение от до 6 и над 8 седмици.