

## Surface characteristics of retrieved coated and nickel-titanium orthodontic archwires

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### Abstract

**Objective:** The purpose of this study was to investigate the effects of oral fluids and archwire-bracket friction on the surface characteristics of NiTi alloy orthodontic archwires with/without aesthetic coating, *in vivo* for 2–3 months. **Materials and Methods:** Different cross-sections of NiTi Archwires (DENTSPLY GAC International) and Titanol Cosmetic Archwires (FORESTADENT® USA Inc.) were examined by electron microscopy with dual-beam and spectroscopy analysis, before and after a collecting protocol from patients with multi-technique. **Results:** Initially, the orthodontic archwires showed microscopic manufacturing and coating defects in the physiognomic layer. After intra-oral exposure, amorphous organic matter deposits were observed on the surface of the NiTi Archwires and the wire coating presented exfoliation on the oral areas of friction with brackets. X-ray microanalysis revealed changes in all atomic and mass percentages of chemical elements from the surface of all retrieved dental archwires, nickel and titanium ion depletion and the occurrence of additional elements due to interactions with saliva. **Conclusions:** Intra-oral exposure of NiTi Archwires and the archwire-bracket friction of coated wire altered the morphology and changed the elemental composition of the surface due to the process of corrosion, adhesion of organic matters and ionic exchange with oral fluids.

**Keywords:** nickel-titanium archwire, coated wire, oral fluids, archwire-bracket friction, electron microscopy with dual-beam, spectroscopy analysis.

### ☐ Introduction

In order to achieve dental movements, orthodontists use stainless steel,  $\beta$ -titanium, cobalt-chromium-nickel or nickel-titanium (NiTi) archwires [1]. Over the last years, dental mechanics were successfully improved using mini-implants [2, 3]. The NiTi alloy was introduced in orthodontics in the '70s, in recognition of its superiority in mechanical and chemical properties.

NiTi archwires gained popularity due to their elasticity of 20% higher than stainless steel alloys. The biocompatible nature of NiTi alloys was improved over the last decades, by the evolution of the alloys industry. However, doubts have been raised among orthodontists, due to the level of nickel over 50%, which, once released in the oral cavity can cause local and general side effects [4–6], but also a decrease in mechanical properties because of the corrosion processes [7–9]. The authors, who have studied the NiTi alloy cytotoxicity, have not yet reached a final and clear conclusion [10–12].

Many manufacturing companies have proposed various methods for obtaining and processing the NiTi archwires to decrease the negative effects and to meet patients' aesthetic needs. Thus, NiTi archwires were covered with Teflon based materials, composite resins, hydrogenated carbon or zirconium dioxide, which restricted corrosion, confined the release of Ni by 80% and did not alter the mechanical properties of the archwires [13–15].

In the specialty literature, there is few data on the wear of intra-orally exposed NiTi archwires and data of archwires with physiognomic layer is virtually non-existent.

The hypothesis tested in this experiment was that the NiTi alloy orthodontic archwires with/without physiognomic coating changes its structural conformation and surface properties under the action of oral fluids and archwire-bracket friction during dental movements. To this end, our study comparatively evaluated the morphology and elemental composition before and after 2–3 months of exposure *in vivo* and described the changes in the surface of these dental archwires.

### ☐ Materials and Methods

#### Materials

The orthodontic archwires used in this study were NiTi Archwires (DENTSPLY GAC International) and Titanol Cosmetic Archwire (FORESTADENT® USA Inc.). Both wires had cross-section dimensions of 0.014 inches and 0.016×0.022 inches. The archwires without macroscopically visible manufacturing defects were selected and were placed intra-orally for 2–3 months. The retrieved samples were washed in distilled water and were immersed in alcohol in an ultrasonic bath for 5 minutes at 46 KHz and then the specimens were dried.

## Patients

The archwires were retrieved from patients during the regular orthodontic treatment visits from the Orthodontics Clinic at the Ambulatory of the “St. Spiridon” University Emergency Hospital, Iassy, Romania. The patients were selected randomly from a batch of participants established on the following criteria: no medication or other intra-orally administered substances, a bacterial plaque index of 0–1, crowding malocclusions and fixed orthodontic appliances (Straight Wire technique, brackets of 0.022-inches slot size and elastic ligations). The selection protocol consisted of the following variables: name and age of patients, dates of orthodontic archwires insertion and removal.

## Focused Ion Beam (FIB) with Scanning Electron Microscopy (SEM) observations

The microstructural morphology of the archwires surface was examined with focused ion beam combined with scanning electron microscope Quanta 200 3D (FEI COMPANY Holland) at the Research Laboratory of Discipline of Material Science and Surface Engineering, Department of Machine Elements and Mechatronics, Faculty of Mechanical Engineering, “Gheorghe Asachi” Technical University, Iassy, Romania. Each type of archwires were examined before and after using. All specimens were examined with a FIB/SEM microscope. It was operating at the high voltage (HV) of 30 kV, with a working distance (WD) of 15–13 mm, on a large field detector (LFD) with specific low vacuum (WDS), and secondary electrons (SE mode), at a horizontal field with the width (HFW) of 249–932  $\mu\text{m}$ , under the pressure of 40–60 Pa, at the temperature of 20°C.

## Energy Dispersive X-ray spectroscopy (EDX) analysis

The surface's qualitative elemental microanalysis of each type of wire was examined before and after application with the same electron microscope and under the same working conditions. The EDX spectrum, the mass (Wt) and atomic (At) percentage of chemical

composition was determined for the surfaces of all examined samples.

## Results

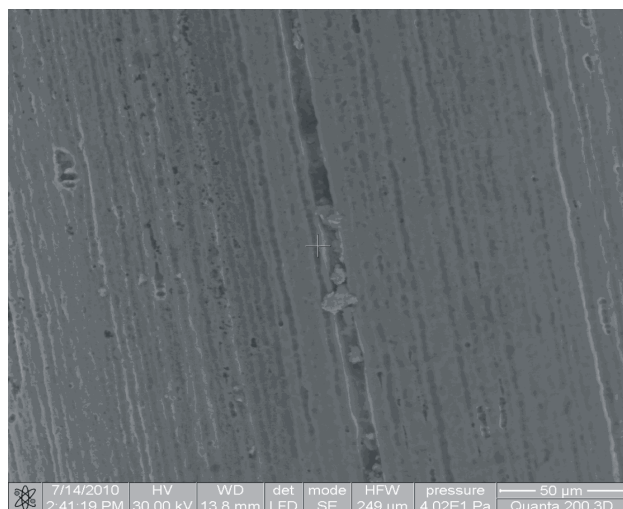
The FIB/SEM observation revealed that NiTi Archwires presented surface threads and internal blisters before insertion, resulted from the wiredrawing process. The surface flaws were scratches and micrometric sinkholes, sufficiently numerous to facilitate organic deposits (Figure 1).

Surfaces of the retrieved NiTi Archwires after intra-oral exposure displayed retention of organic matter in the oral cavity, as regional accumulations of amorphous material (Figure 2).

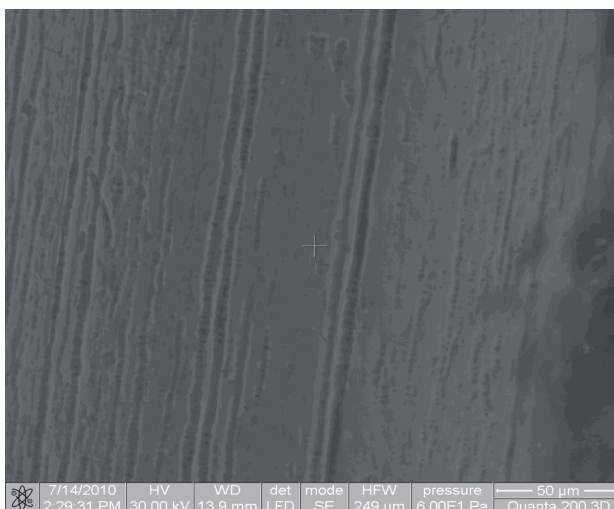
Figure 3 shows the inhomogeneous protective coating of Titanol Cosmetic Archwires before using that creates internal tensions between the layer and substrate, which is favorable for the exfoliation process.

On the retrieved coated wires after intra-oral exposure there were observed two alternative protecting coating areas, depending on the archwire's wear. The coated areas that were not in contact with brackets did not suffer the exfoliation process. There were found areas with exfoliated coating on the oral side of the archwire that was in contact with slot brackets, that were destroyed due to its friction with the brackets during dental movement, therefore losing its initial properties (Figure 4).

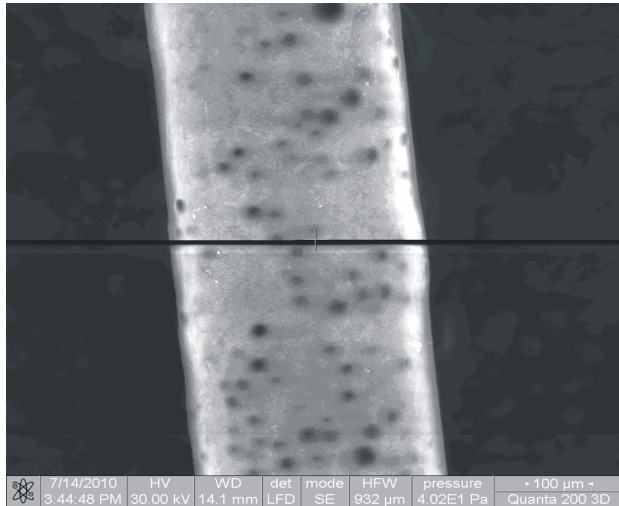
EDX Spectroscopy of a NiTi Archwires before using revealed Ni and titanium (Ti) as specific chemical elements in alloys with shape memory and uniform distribution throughout the analyzed surface, ensuring the same physical properties of the archwire at any point on its surface. There were also observed other chemicals that are nonspecific like carbon (C), oxygen (O), silicon (Si), chlorine (Cl) and calcium (Ca), in smaller percentages (Figure 5). On the retrieved NiTi Archwires surface there were observed additional other chemicals like aluminum (Al), phosphorus (P) and potassium (K), confirming the presence of organic adhesions after intra-oral exposure (Figure 6).



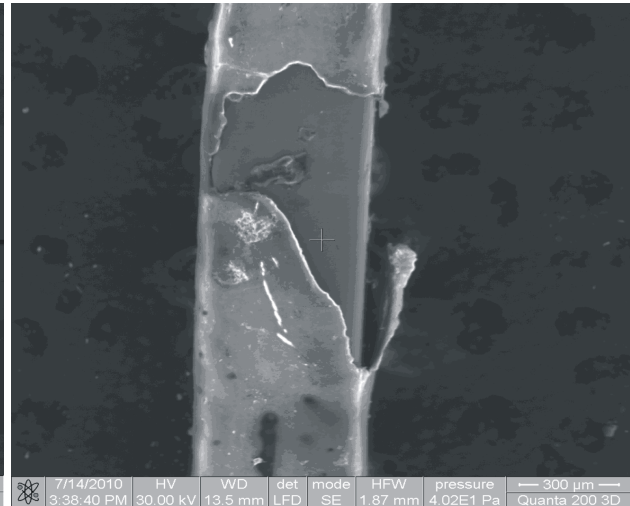
**Figure 1 – FIB/SEM photomicrograph of NiTi Archwire surface before insertion with small defects on the lamination direction (original magnification,  $\times 50$ ).**



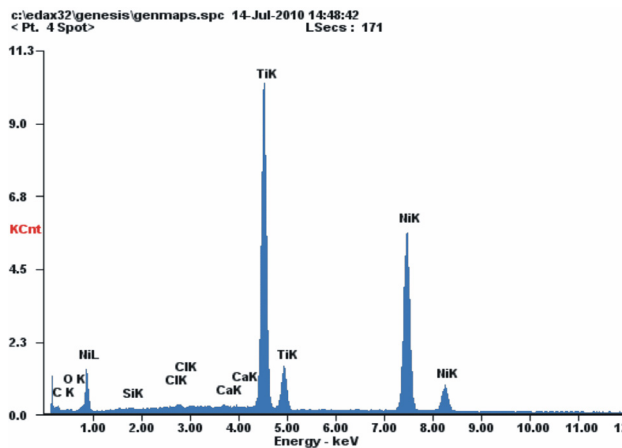
**Figure 2 – FIB/SEM photomicrograph of retrieved NiTi Archwire surface after insertion with different degrees of deposition of organic materials (original magnification,  $\times 50$ ).**



**Figure 3 – FIB/SEM photomicrograph of Titanol Cosmetic Archwire surface before insertion showing small coating flaws (original magnification, ×100).**

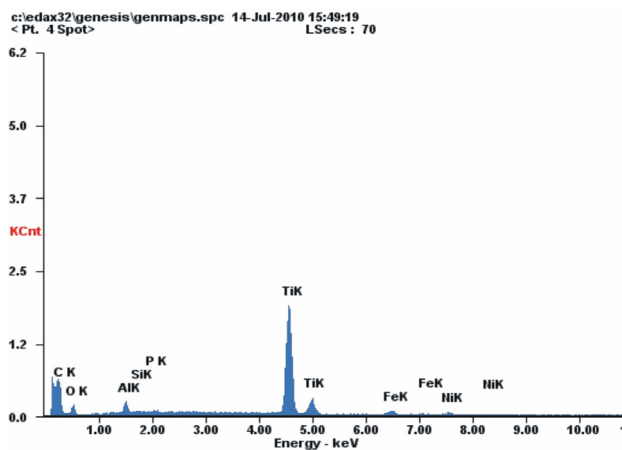


**Figure 4 – FIB/SEM photomicrograph of oral surface of retrieved Titanol Cosmetic Archwire with exfoliated coating (original magnification, ×300).**

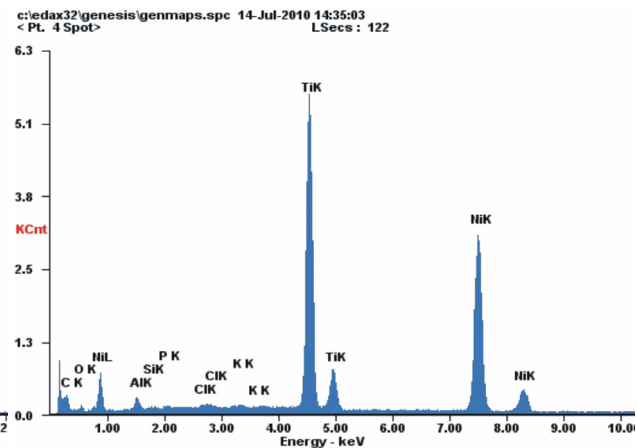


**Figure 5 – EDX spectrum of chemical elements on NiTi Archwire's surface before insertion (K and L represent the atomic layers where the X-ray was emitted).**

Figure 7 shows the composition of chemical elements in substrate and the protective layer of Titanol Cosmetic Archwires before usage. On the exfoliated area of retrieved coated archwires after intra-oral exposure, there were found higher peaks of Ti and Ni in substrate and Ca, an additional element (Figure 8).

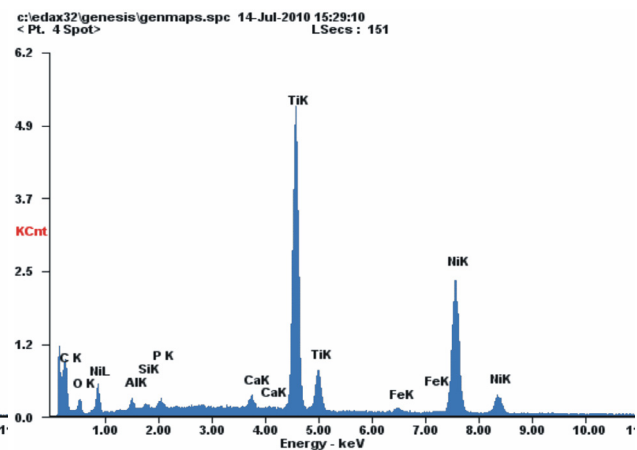


**Figure 7 – EDX spectrum of chemical elements on Titanol Cosmetic Archwire's surface before insertion (K and L represent the atomic layers where the X-ray was emitted).**



**Figure 6 – EDX spectrum of chemical elements on retrieved NiTi Archwire's surface (K and L represent the atomic layers where the X-ray was emitted).**

Table 1 shows the changes that occurred in the elemental composition of the surface after intra-oral exposure of dental archwires. On NiTi Archwires there was observed a decrease of Ni and Ti, and on the Titanol Cosmetic Archwires a decrease of Ti and an increase of Ni.



**Figure 8 – EDX spectrum of chemical elements on a retrieved Titanol Cosmetic Archwire's surface (K and L represent the atomic layers where the X-ray was emitted).**

**Table 1 – Comparative results of EDX analysis**

Elements	NiTi Archwire				Titanol Cosmetic Archwire			
	Before using		After using		Before using		After using	
	Wt%	At%	Wt%	At%	Wt%	At%	Wt%	At%
Ni	53.27	41.02	44.38	24.81	01.29	00.39	28.28	11.09
Ti	40.32	38.05	33.49	22.95	30.24	11.10	27.40	13.17
C	04.04	15.20	14.85	40.59	45.50	66.61	31.82	61.00
O	01.71	04.82	03.65	07.49	17.05	18.74	08.01	11.53
Al	–	–	02.38	02.89	02.87	01.87	01.58	01.35
Si	00.28	00.45	00.29	00.34	00.67	00.42	00.66	00.54
P	–	–	00.39	00.42	00.51	00.29	00.78	00.58
Cl	00.16	00.20	00.31	00.29	–	–	–	–
Ca	00.23	00.26	–	–	–	–	00.82	00.47
K	–	–	00.25	00.21	–	–	–	–
Fe	–	–	–	–	01.87	00.59	00.65	00.27

## Discussion

Biomaterials used in orthodontics must be inert, enabling only the desired interactions, without changes in the original properties of the material used and in the environment of introduction. Complex analysis of the NiTi alloy physiognomic coated archwires' surface, compared with the uncoated ones represents a particular interest of the performance of *in vivo* dental biomaterials investigated in this study. Literature provides information about properties of orthodontic polymers [16–18], of brackets [19, 20], of stainless steel archwires and NiTi uncoated alloys [12, 21] and the polymeric elastics [22].

In this experiment, there were selected patients with good oral hygiene, to avoid introducing additional variables, such as plaque bacteria effect on the archwires. The effects desired by us were focused on the action of oral fluids and friction of the archwires with brackets during the orthodontic treatment.

To avoid measuring errors, the samples were prepared before the examination because the investigation methods used were sensitive to surface contamination. We used a combination of SEM, FIB and EDX, to have a full picture of the surface's state and the chemical composition of the analyzed archwires. Convergence of these methods and short working distance allows sectioning "slice-and-view" with precision and a high-resolution chemical analysis, which is an enhancement technique used in similar studies [21, 23].

Following the analysis FIB/SEM of NiTi Archwires we have noticed flaws on the material's surface due to the manufacturing technology, which favored plaque bacteria accumulation and calculus deposits, with undesirable effects on the periodontal structures. These irregularities of calculus deposits can cause limitations of the archwire's action, by affecting its physical properties (elasticity, memory capacity and mechanical strength).

Eliades T *et al.* (2000) reported surface composition alteration of NiTi Archwires after intra-oral exposure for 1–6 months due to the occurrence of amorphous precipitates and microcrystalline particles in proteinaceous biofilm [21].

Materials used as coating for the orthodontic archwires must meet certain qualities: to be biocompatible,

to provide pleasant aesthetics to have translucence similar to the aesthetic brackets, to be easily applied, to provide increased electrical resistance, higher toughness, lower friction and proper thermal conductivity [24, 25]. Recent studies on the effect of slip resistance of physiognomic coated archwires have noticed superior surface properties and have reported the influence of friction on the effectiveness of teeth movement [26, 27].

We have observed some discontinuities of the protective layer in Titanol Cosmetic Archwire before using, which causes the appearance of intern tensions and exfoliation of this archwire after using in the oral friction with the brackets zones. The layer's discontinuities can affect the properties and mechanic efficiency of the archwire, because of the growth in friction and uniformity of the information transferred to the brackets that can cause unwanted and uncontrolled dental movements. The accumulation of the layer's material from the tubes and the brackets' slots can limit or cancel the dental movement. The phenomena of friction between the metallic surface of the exfoliated archwire and the bracket's slots of composite material can cause shape modifications or the fracture of the brackets. The quality and difference of elasticity between the protective layer and substrate, high variations of temperature or acidity in the oral cavity and the undue tooth brushing can also cause the exfoliation of the protective layer, which can represent future research topics.

From the EDX analysis, we have observed three categories of significant changes in the chemical composition of the studied archwire's surface:

- The ionic depletion, with the release of Ni and Ti ions in the oral cavity is followed by processes of corrosion in the oral sphere. The corrosion phenomena were more striking at NiTi Archwires.
- The emergence of other chemical elements in the initial composition of the surface was due to the organically adherences and dental calculus.
- The change in the percentages of the other chemical components was due to the ionic changes with saliva. For the retrieved NiTi Archwires, the percentages have increased, but decreased for the retrieved Titanol Cosmetic Archwires, with the exception of P, which are high.

The aesthetic coating protects the subjacent archwire against corrosion processes, but the archwire's corrosion is possible after a prolonged utilization in the oral cavity, due to the complex action of the oral fluids [28]. Cai F *et al.*, studying the resistance to corrosion and to fracture of different types of archwires with protective coatings compared to conventional archwires, have found three categories: archwires without modifications, archwires with crystallographic changes and archwires with distanced Teflon coating [9]. We can assume that the processes of corrosion have negative consequences over biocomparability, aesthetics and frictional behavior at the brackets level and guidance of the dental archwire over the duration of the orthodontic treatments [28].

## Conclusions

The results of our study can be summarized:

▪ The NiTi Archwires show defects of fabrication on the surface, where calculus deposits appear after being intra-orally exposed.

▪ The protective layer of the physiognomic archwires show imperfections of depositing, and the exfoliation takes place in the mechanical strain of the archwires, during the orthodontic treatment.

▪ The composition of the chemical elements of the orthodontic archwires' surface changes in the oral fluid.

▪ The microstructural and chemical changes in described surface show that the retrieved orthodontic archwires alter during the treatment, with effects over the mechanical dental movements and the biocomparability in the oral sphere.

The following research *in vivo* are necessary to resolve the described implications of the exfoliation of the protective layer in the friction with the brackets zones, the adherence of organic materials, corrosion processes and ionic changes with oral fluids.

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