Scholars Journal of Dental Sciences

OPEN ACCESS

Abbreviated Key Title: Sch J Dent Sci
ISSN 2394-4951 (Print) | ISSN 2394-496X (Online)
Journal homepage: https://saspublishers.com

Study of the Corrosion of the Nickel-titanium Orthodontics Archwires in the Mouth

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DOI: 10.36347/sjds.2022.v09i02.002 | **Received:** 17.01.2022 | **Accepted:** 24.02.2022 | **Published:** 28.02.2022

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Abstract **Original Research Article**

Objective: The corrosion behavior of titanium alloy (NiTi) orthodontic wires shows a high resistance to corrosion in various solutions, such as Ringer's solution, artificial saliva, sodium chloride solution, and others. In these different liquids, NiTi corrosion resistance is higher than that of stainless steel or cobalt-based alloys. These research studies the behavior of the nickel-titanium Orthodontics archwires in the mouth by three methods. ***Methods***: The first testing was the SEM that can perform EPMA (Electro Probing Micro Analysis), microanalysis X, and local and quantitative elementary analysis, and the examination of NiTi are observed in two ways: cross sectional and surface examination. The second one was the electrochemical analysis performed by immersion in an artificial saliva solution consisting of lactic acid at 0.1 moles per liter and sodium chloride at 0.1 mole per liter: Two types of NiTi orthodontic arches were tested. The first ones are from the manufacturer Ortho Classic and were used in the mouth for 4-6 weeks. The second arches are from the manufacturer AZ Dent and were new. The last test was the TEM cartography that shows the atoms and their dispersion on the surface of the wire. ***Results***: The results showed that the presence of corrosion cells in the artificial saliva proved that the NiTi orthodontic archwires were able to initiate the process of corrosion in the mouth, particularly at the point of friction in contact with the braces (Ti6Al4V). ***Conclusion:*** The results obtained by the MET on thin sections of orthodontic arches prepared by the FIB method revealed the presence of double or multiple layers of titanium oxide and N-oxide plus aluminum.

Keywords: Corrosion, orthodontic, nickel-titanium, archwires.

I. INTRODUCTION

From an economic point of view, corrosion can be very expensive. Therefore, if a product is intended for prolonged use over time, it is advisable either to replace the parts or to provide regular maintenance (corrosion inhibitor, paint, etc.) [1]. The corrosion behavior of titanium alloy (NiTi) orthodontic wires shows a high resistance to corrosion in various solutions, such as Ringer's solution, artificial saliva, sodium chloride solution, and others. In these different liquids, NiTi corrosion resistance is higher than that of stainless steel or cobalt-based alloys. Stainless steel alloys are easily corroded in solutions containing chlorides, and titanium-based alloys readily corrode in acidic fluoride solutions. The degradation occurs after immersion of the NiTi in a fluoride solution and leads to embrittlement by hydrogen. To prevent the release of ions such as iron (Fe), chromium (Cr) and nickel (Ni) into the oral cavity, which leads to allergic, toxic or carcinogenic effects when absorbed by the human body, the corrosion resistance of orthodontic appliances is important [2]. In every routine orthodontic treatment, we can find nickel present in a large number of wires or brackets. Nickel-titanium archwires contain 47%-50% Ni and are consequently a high source of Ni in the intraoral environment cavity of orthodontic patients. Researchers have recognized and investigated the potential biological implications of Ni release in the human body, with a particular focus on corrosive products and alloy ions used in orthodontic treatment. However, most studies have shown in vitro approaches. To draw conclusions about the release of Ni in vivo is not only very difficult and methodically unreliable, but also impractical and clinically irrelevant due to the very different nature of the oral environment. Hence, in vitro results are inconclusive. Thus, this material and its many alloys used in dentistry and orthodontic appliances may be subject to this phenomenon, making it important to study in order to better understand and treat this problem [3]. In the present chapter, we will conduct various tests, such as SEM (Scanning Electron Microscopy) to understand the microstructure of NiTi arches. We will also conduct corrosion cell tests to see whether the corrosion process of the used NiTi arches has been initiated. Finally, we will observe the transmission electron microscopy (TEM) arc using an ion-focusing beam (FIB) at high magnification to observe the nanoparticles and the constitution of multiple oxidation layers.

1.1 Definitions

Corrosion is generally defined as a prolonged interaction process between a solid material (and biomaterial) and its chemical environment. This interaction leads, to a loss of material substance partially or totally, or even a complete loss of structural integrity [4]. In the corrosion process, the oxidation of the surface layer of the material can lead to the passivation of the alloy by the formation of a passive protective film composed of metal oxides. The metal or alloy is then said to be passivated. A passivated environment may continue to destroy itself, but at an extremely slow pace [4]. Even the oxide films will slowly dissolve to reform the protected passivation; it may also dissolve in the saliva present in the oral cavity [5]. The stability of NiTi corrosion resistance is assumed by the formation of this protective passive layer of TiO2 dioxide film. Fluoride-based products (e.g., toothpaste, rinse solution, mouthwash, etc.) contribute to the increase in oral acidity and cause sensitivity to corrosion of certain metals such as titanium. Furthermore, orthodontic NiTi from different manufacturers may have a different corrosion resistance, given that the composition of nickel and titanium likely differ from one company to another.

II. MATERIALS AND METHODS

2.1 The SEM (Scanning Electronic Microscope) Test for NiTi archwires

As it is equipped with special detectors, the SEM can perform EPMA (Electro Probing Micro Analysis), microanalysis X, and local and quantitative elementary analysis [6].

2.1.1 Examination of NiTi using SEM Samples are observed with the SEM in two ways: - cross sectional and - surface examination.

- Cross sectional examination the maximum magnification achieved during this examination is 2500 (Fig 1).

Fig-1: Cross-sectional image of orthodontic wires

We obtained the cartography of the section of nickel and titanium repartition (Fig 2) for NiTi 0.016-0.022 inch.

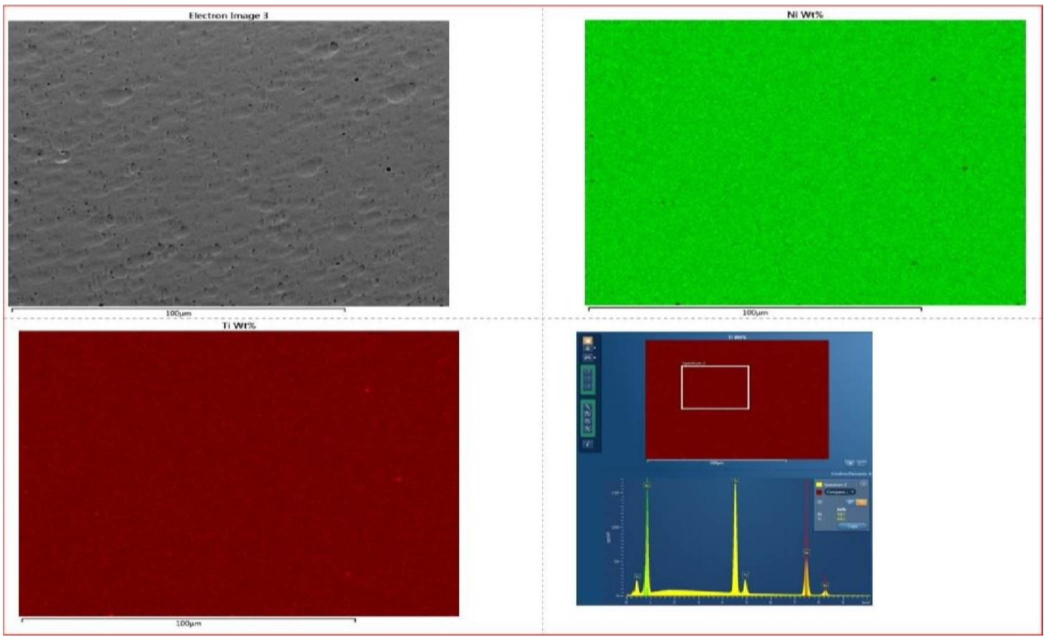


Fig-2: Regular repartition of all particles of nickel and titan for a new Ortho Classic 0.016-0.022 archwires under a high magnificent

Fig-3: Shows the highly regular repartition of the particles of Titanium for a new 0.016 inch archwires.

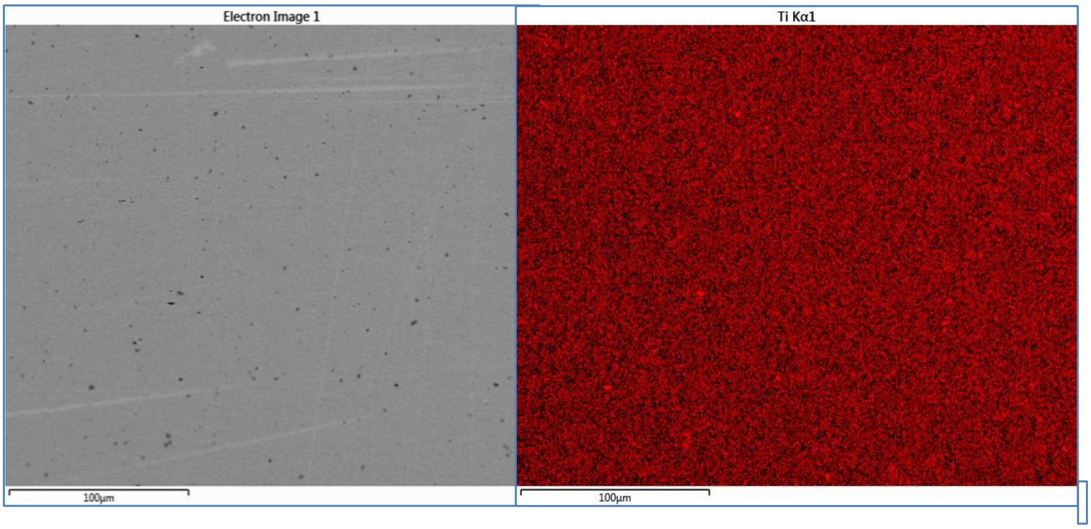


Fig-3: New 0.016-inch archwires using SEM and showing the smooth surface of the wires with good repartition of nickel

- **Surface examination by SEM**

The use of SEM allows us to observe the surface evolution of orthodontic arches before and after use, enabling us to pinpoint corroded or damaged areas.

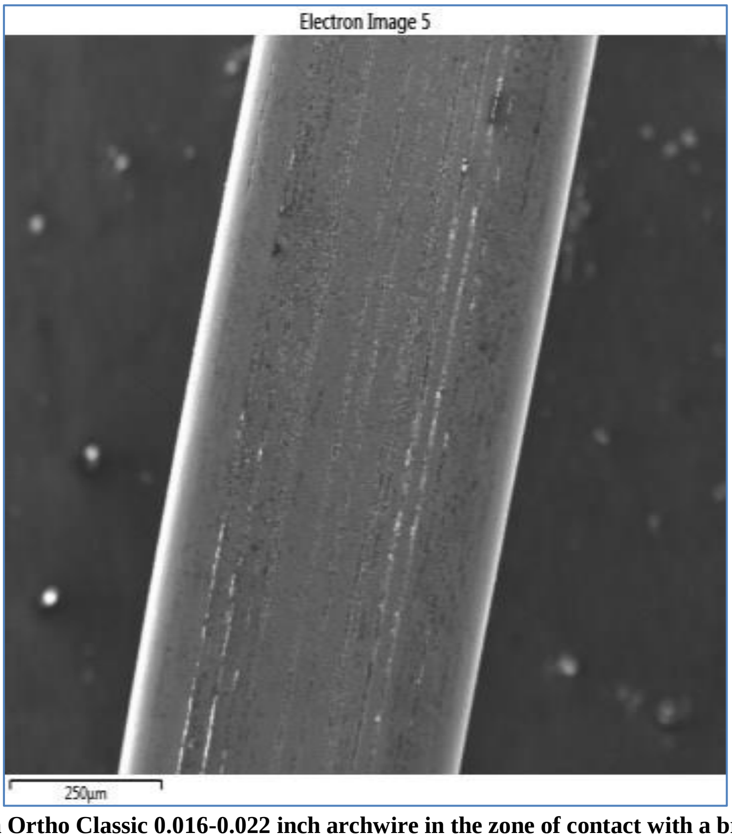


Fig-4: Spot of corrosion on Ortho Classic 0.016-0.022 inch archwire in the zone of contact with a brace (left) and a new Ortho Classic 0.016-0.022 inch

Analysis of the area of the arc used in the region in contact with the braces shows the presence of Ti and oxygen-rich particles for this used Ortho Classic 0.016-0.022 inch archwire. (Fig 4).

For used arches, we observe a rough surface and numerous striations (Fig 5).

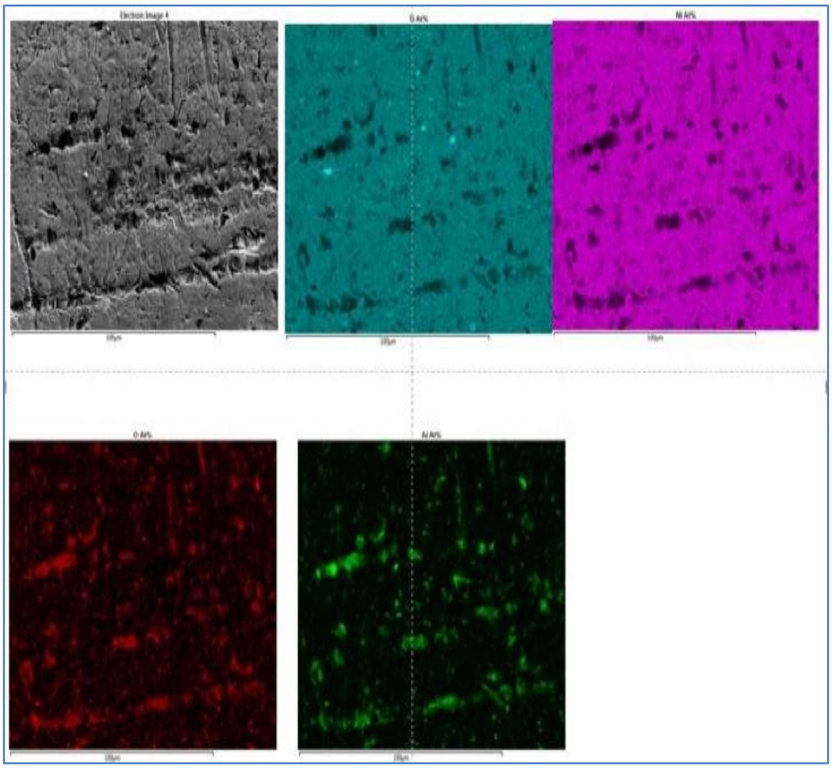


Fig-5: Retrieved Ortho Classic archwire 0.016 inch with Porosity in the whole surface.

2.2 Microanalysis using SEM microprobe

EPMA is a microscopy tester used primarily for non-chemical in situ and microstructural analysis of solid samples to investigate a very small area of the sample. The significant of EPMA is its ability to acquire small and accurate quantitative elemental analyses at very small sizes (e.g., 1 to 2 microns) [7].

2.2.1 Analysis of NiTi archwires constitution

The first step is to carry out a series of qualitative analysis on the different areas of our examination to specify the elements presented in the measurement area. For this, an electronic signal is sent to the sample to be studied and the X-ray spectrum derived during the interaction of the electrons with the target atoms is measured by the spectrometer. Analysis of this X-ray identify which elements are present in the sample by determining their characteristic energy. Once all the different elements have been identified, a quantitative analysis was done to determine the mass concentration of the elements present in the volume of the sample [8].

2.2.2 Results and evolution of the of NiTi orthodontic archwires composition by microprobe Analysis

This experimental study focuses on conventional orthodontic arches with new and worn arches from the American Orthodontic Company. Figures 6 and 7 show the different compositions of our NiTi archwires for 0.016 and 0.016-0.022 inch. Microprobe analysis of the friction zone at the level of the braces shows an important evolution of the Ni and Ti content with an evolution of oxidation significant. The composition of NiTi archwires in cited in table 1.

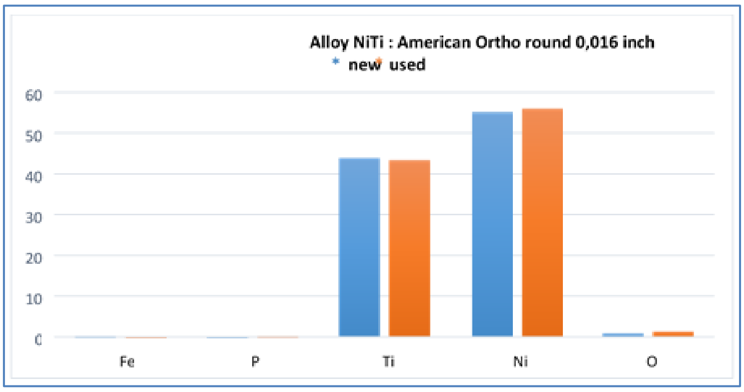


Fig-6: Composition of iron, phosphorus, titanium, nickel and oxygen of round used and new 0.016-inch wires.

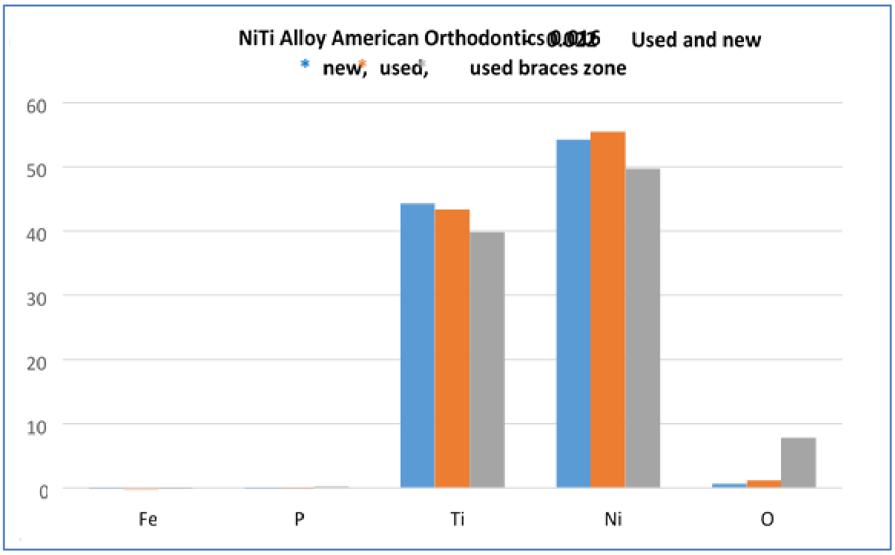
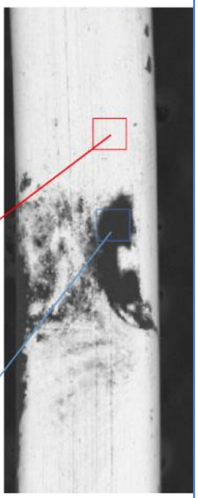


Fig-7: Composition of iron, phosphorus, titanium, nickel and oxygen of round used and new 0.016-inch wires with the zones of contact with the braces.

Table-1: Composition and image of NiTi archwire 0.016-0.022 inch in different positions

	F	*P*	*Ti*	*Ni*	*O*	*Total*
A new	0,04	0,00	44,05	54,75	0,68	99,52
A new	-0,12	0,01	43,99	55,32	0,70	99,91
A new	-0,02	-0,01	43,97	55,38	0,72	100,03
A new	0,00	0,00	43,63	55,90	0,52	100,06
A new	0,13	-0,02	44,47	53,86	0,85	99,29
A new	-0,01	0,01	44,73	53,49	0,67	98,89
A new	0,10	0,03	44,48	53,46	0,70	98,77
A new	0,00	0,00	44,73	53,48	0,69	98,89
A new	0,04	-0,02	44,21	53,29	0,65	98,17
A new	0,19	0,01	44,60	52,75	0,71	98,26
Mean	0,03	0,00	44,29	54,17	0,69	
Std dev.	0,09	0,01	0,37	1,08	0,08	
B used	0,12	0,02	43,42	55,30	1,32	100,18
B used	0,01	0,03	43,10	56,04	1,58	100,76
B used	-0,06	0,05	43,69	55,56	1,47	100,72
B used	0,08	0,01	43,54	55,88	1,08	100,59
B used	-0,05	0,02	42,47	56,57	1,70	100,71
B used	-0,08	0,02	43,94	55,00	1,29	100,16
B used	-0,18	0,01	43,59	54,90	0,82	99,15
B used	-0,13	0,01	43,56	54,57	1,02	99,04
B used	-0,08	0,01	42,68	55,75	0,52	98,88
B used	0,05	0,01	43,55	55,09	1,20	99,91
Mean	-0,03	0,02	43,35	55,47	1,20	
Std dev.	0,09	0,01	0,46	0,61	0,36	
B used black	-0,07	0,23	38,52	48,64	16,59	103,91
B used black	0,06	0,34	37,97	48,35	6,59	93,31
B used black	-0,01	0,03	43,11	54,42	1,48	99,03
B used black	0,12	0,12	41,12	52,09	7,14	100,59
B used black	0,12	0,38	38,48	45,15	7,50	91,63
Mean	0,04	0,22	39,84	49,73	7,86	
Std dev.	0,08	0,15	2,20	3,59	5,46	



2.3 Complex Impedance of corrosion behavior of intra-oral NiTi orthodontic arches

The oral cavity, due to its temperature variations, changing pH levels, high humidity, biomechanical forces and presence of microorganisms, is a favorable place for degradation of dental biomaterials. This section presents a comparative assessment of the intensity of NiTi alloy orthodontic archwire corrosion under laboratory conditions. Corrosion resistance examinations are studied using the impedance index and the potentiodynamic methods.

The use of fixed appliances in orthodontics therapy for the treatment of malocclusion is widespread in modern orthodontics dentistry. Orthodontic brackets can be made of metal alloys, ceramics, or composite materials. To ensure better biocompatibility performance, manufacturers and scientific researchers use brackets with minimum content of nickel. Instead, they prefer products made from titanium alloys to benefit from the mechanical properties of titanium [9].

Saliva is an electrolyte and the different kinds of material used makes the orthodontic appliance a kind of a cell electrochemical connection in the intra-oral environment, subjected to continuous electrochemical processes of corrosion [9, 10].

A typical oral cavity of an individual with an orthodontic appliance features temperature variations from 55 to 0 °C, changing pH, high humidity, impedance of mechanical dental forces, and various microorganisms [7, 11]. All of these factors lead to the aging of dental biomaterials. Which can lead to corrosion, since the materials have different electrochemical reactions with the oral fluid [12, 13] Corrosion, when left unchecked, may cause serious degradation of the structure of restorative materials, prostheses, orthodontic archwires and brackets. . Components containing nickel, cobalt and chromium are generally recognized as biologically incompatible [14, 15].

Hence, it is important to explore the biological and cellular-medical characteristics of commercially available biomaterials in the context of the biocompatibility of their use in our body [16].

2.3.1 Experimental protocol

The aim of this section is to make a comparative in vitro assessment of the corrosion susceptibility of three different types of orthodontic archwires made of NiTi alloy and manufactured by the American Orthodontics Company, Ortho Classic, and AZ Dent. Tests will be conducted on the alloys employing an artificial saliva solution and using both impedance and potentiodynamic methods.

This testing process allows us to show any differences in cell corrosion between new and used arches after immersion of the wires in artificial saliva and during the production of a current circuit. Two types of NiTi orthodontic arches were tested. The first ones are from the manufacturer Ortho Classic and were used in the mouth for 4-6 weeks (samples 9 and 11). The second arches are from the manufacturer AZ Dent and were new (samples 32 and 35). Electrochemical analysis was performed by immersion in an artificial saliva solution consisting of lactic acid at 0.1 moles per liter and sodium chloride at 0.1 mole per liter (DIN 13927).

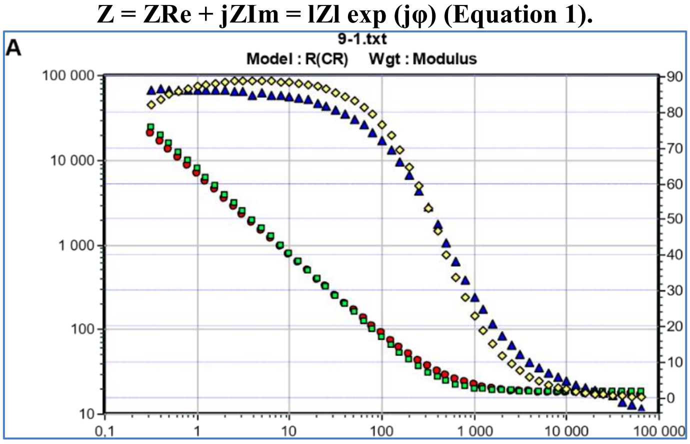
The electrochemical measurements were made at room temperature using a PGZ 100 potentiostat controlled by the voltmeter 4 software [17]. The results of complex impedance spectroscopy were analyzed using ZSimpWin software. Corrosion potentials were measured throughout the immersion, which lasted 10 days, and measurements were made at immersion times of 1h, 1 day, 2 days and 10 days using complex impedance spectroscopy. The measuring cell consisted of the orthodontic arches to be analyzed, a platinum counter electrode, and a saturated calomel reference electrode (ECS). The measured potentials were taken with respect to this reference.

The corrosion potential for the different samples evolved in a very similar way, with an initial immersion value close to that of the reference used and moving towards a similar value at the end of the experiment of close to 0.2 V compared with the ECS. This shows that the measure does not differentiate a new arch from a used one, and nor does it differentiate manufacturers.

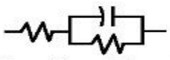
2.3.2 Results of Immersion of archwires NiTi in artificial saliva

Using spectroscopy, the results of complex impedance are shown in Figure 11 as ZRe = f (-ZIm) and in Figure 11b as lZl = f (ω) and φ = f (ω), where ZRe and ZIm represent the real part and the imaginary part of the electrochemical impedance of the studied electrode (i.e., orthodontic arches), respectively, and where lZl and φ represent the modulus and the phase of this same impedance Z, respectively.

Z = ZRe + jZIm = lZl exp (jφ) (Equation 1).



|Z|, Msd.
|Z|, Calc.
Angle, Msd.
Angle, Calc.


Randles Circuit

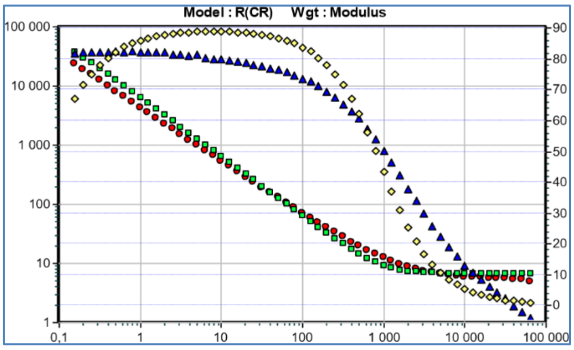
le, deg

Iter #: 1
Chsq: 1 , 46E-02

Of pars with rel. std. errors
>20%: 1 / 3
>1000%: 0 / 3

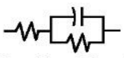
Frequency, Hz

If we look at the results obtained and modeled with this system (Fig 8), we can see that the experimental values (represented in red and blue) are not in good agreement with those determined by modeling (represented in green and yellow), and this is only at the beginning of immersion. Figures 8A and 8B show data pertaining to the beginning of the immersion, while Figures 8C and 8D show data pertaining to 10 days later.



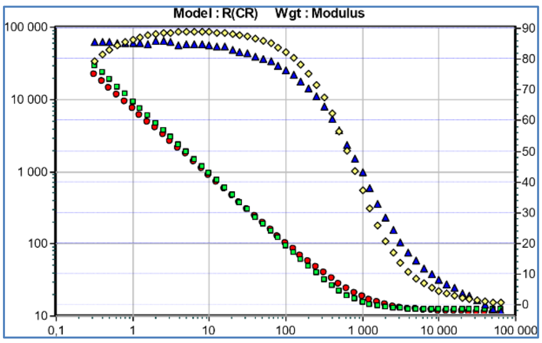
B

|Z|, Msd.
|Z|, Calc.
Angle, Msd.
Angle, Calc.

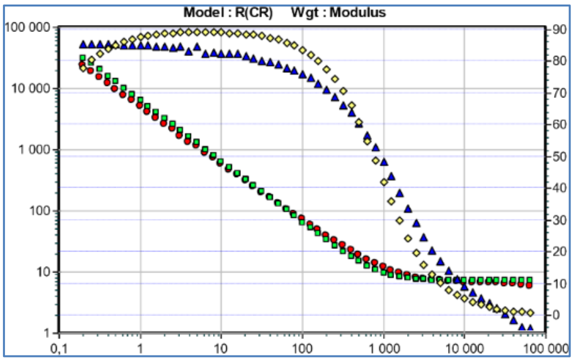

Randles Circuit

le, deg

Iter #: 1
Chsq: 4,11E-02



C



D

35-4 .txt

Fig-8: A, B, C, and D show Bode diagrams for different times: Diagrams of samples 9, 35, 8 A and 8 B are after 1 hour immersion in solution DIN 13927, while 8C and 8D are after 10 days. Modeling is done using equivalent circuit of Randles.

Many other models were done but the best modeling results were obtained by using the equivalent circuit represented below (Fig. 9).

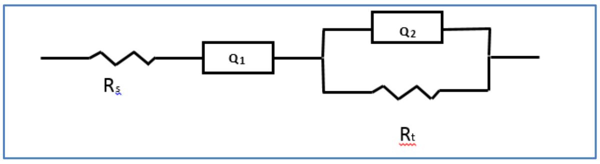


Fig 9: R (Q) équivalent circuit (RQ)

Where Q represents CPE (constant phase element), which models the behavior of a double layer (i.e., an imperfect capacitor).

It is important to note that modeling with C1 and C2 capabilities in place of Q1 and Q2 leads to the same imperfections as those obtained with previous models.

The Calculated parameters from the different models done for different samples after 1h and 10 days were represented in table 2

Table-2: Calculated parameters from equivalent circuits for different samples after 1h and 10 days

samples	**Rs /Ω**	**$Y01/S.s^{n1}$**	**n1**	**$Y0/S.s^{n2}$**	**n2**	**R / Ω**
9 after 1 hour	17.2	$2.5.10^{-5}$	0.96	$9.1.10^{-4}$	0.69	7.3
9 after 10 days	11.2	$2.3.10^{-5}$	0.94	$6.8.10^{-4}$	0.74	4.6
11 after 1 hour	13.4	$1.6.10^{-5}$	0.97	$3.6.10^{-4}$	0.71	35.3
11 after 10 days	6.8	$1.9.10^{-5}$	0.95	$5.2.10^{-4}$	0.76	10.0
32 after 1 hour	6.5	$6.3.10^{-5}$	0.89	$4.7.10^{-4}$	0.73	24.0
32 after 10 days	6.5	$4.4.10^{-5}$	0.91	$3.9.10^{-4}$	0.78	15.5
35 after 1 hour	5.4	$4.3.10^{-5}$	0.92	11.10-4	0.67	19.4
35 after 10 days	6.4	$3.5.10^{-5}$	0.94	$5.9.10^{-4}$	0.78	11.5

2.4 Testing of NiTi archwires by Transmission Electron Microscopy (TEM)

The TEM approach consists of the interaction of electrons emitted from a material and the detection of the electrons that have passed through the sample. For this technique, the sample must be extremely fine, requiring any specimen to be cut in extra fine sections or slices no more than a few tens of nanometers thick. The TEM allows observation of the microstructural organization and the internal structure composition of these ultra-fine slices [18]. Additionally, the TEM determines a chemical Nano-analysis of the NiTi arches as well as cartography of the distribution of the main elements (Ni, Ti, Al, and O).

2.4.1 Preparation of Samples by FIB on TEM

Preparing high-quality sample specimens for examination in transmission electron microscopy (TEM) is critical for the success of TEM experimentation. In this section, some conventional FIB/TEM high quality preparation techniques for specimens are explained and their advantages and disadvantage compared. As well, a technique suitable for preparing TEM samples from ultra-fine specimens is clarified and demonstrated [19].

Focused ion beam (FIB) microscopes have become powerful important technical devices that contribute to the development of TEM specimen preparation. The technique has become very well-established over the past ten years (see Fig. 10). [19].

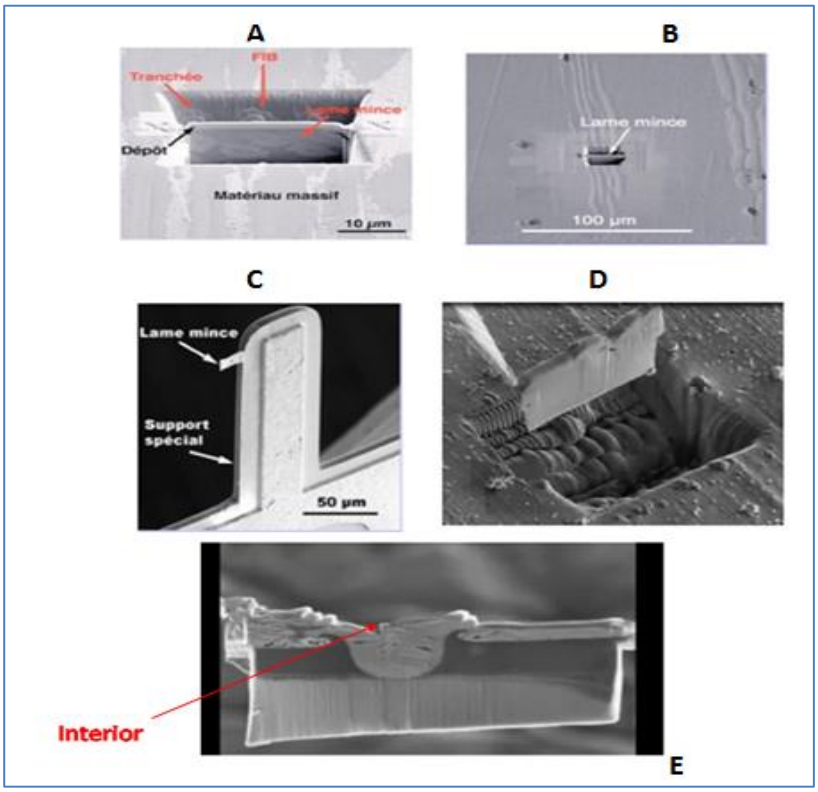


Fig-10: Preparation steps for thin blade in FIB testing, A, B: cutting the blade; C, D: placing the blade on the support, E: amincissement ionic process Decoupage orthodontic wire in this method of FIB.

In the present study, we conducted a TEM examination on FIB (Focused Ion Beam) cross-sectional lamella to characterize the surface oxide extra-fine layer and sub-surface NiTi matrix layer in greater detail [20].

2.4.2 Sample Preparation using Focused Ion Beam (FIB)

We used a new FIB technique to characterize the extreme surface of the orthodontic arches. We cut a small portion from the wire (Fig 15) in the zone of friction on the braces to find the highest place of deterioration. We then removed a very thin layer for FIB examination.

The samples were rinsed with distilled water and then sterilized using the steam method (autoclave). We can also see a small grain at different angulations, e.g., BF and DF (Fig. 11), and the grain size can be up to 100 nm.

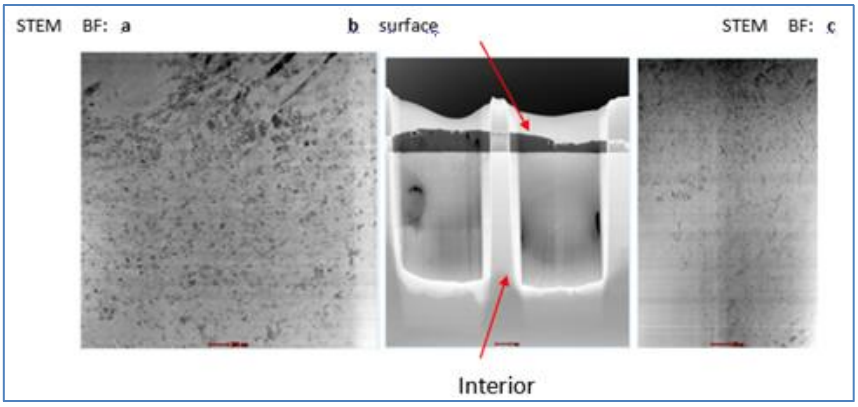


Fig-11: Position of the specimen in TEM using FIB: the STEM DF and BF are the angle of detector proportional to the specimen (a, b, c).

III. TEM RESULTS

- The TEM cartography shows the atoms and their dispersion on the surface of the wire.
- This cartography of MET/FIB preparation of Ortho Classic samples of 0.0160.022 inch rectangular archwires illustrate the presence of a double layer of **Ti-oxide** and **Ni-oxide.**

- It also can be seen that there is a presence of alumina nanoparticles, possibly coming from the orthodontic bracket and its environment (e.g., toothpaste).
- It also be observed that there is a presence of carbon nanoparticles (Fig 12).

IV. DISCUSSION

As can be seen, the concentration of nickel has decreased, which means that a layer of NiTi surface has been removed and the corrosion process has begun. Furthermore, a layer of titanium has formed on the surface, which means that we now have a double layer of oxide. The layer of Titanium oxide can be clearly observed in fig 13 below.

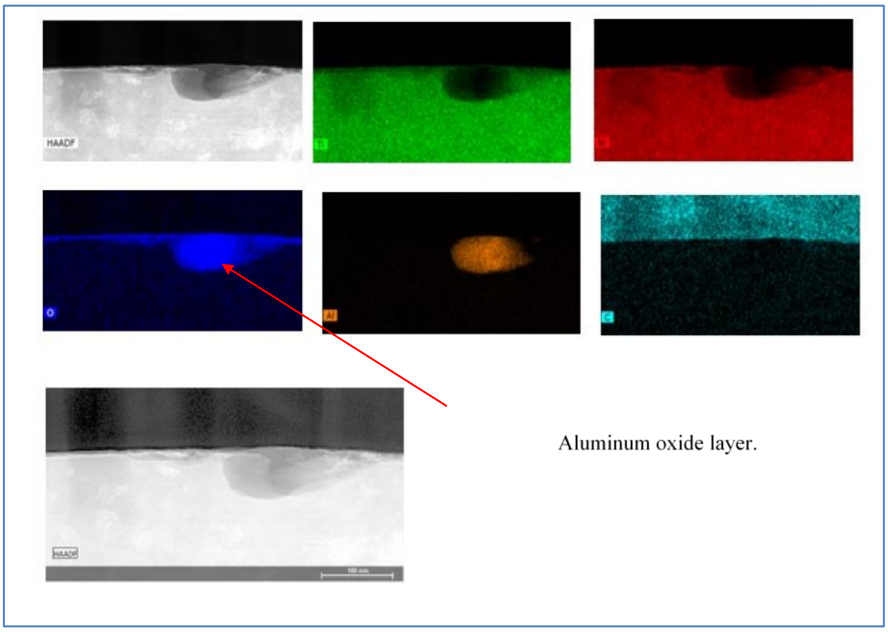


Fig-12: Presence of Aluminum Particles in the Surface of NiTi Archwire

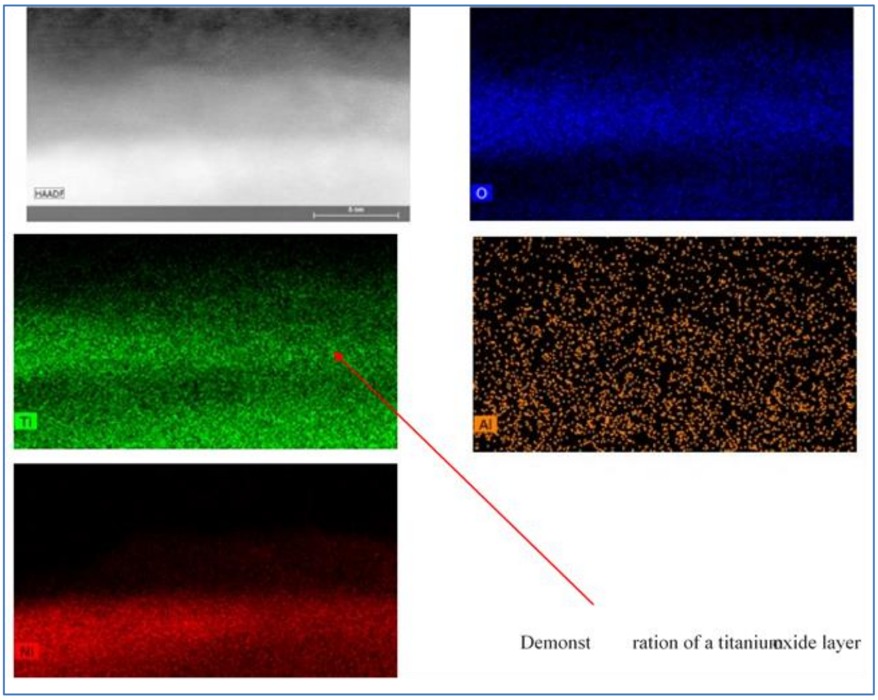


Fig-13: Layer of titanium oxide

V. CONCLUSIONS

1. The results obtained by the MET on thin sections of orthodontic arches prepared by the FIB method revealed the presence of double or multiple layers of titanium oxide and N-oxide plus aluminum. The Al layer might possibly have resulted from the friction of the braces (Ti6Al4V) with the wires (NiTi).

2. The FIB technique provided an accurate measurement of Ni and Ti particles and detected whether or not there was a lack or decrease in them.
3. SEM testing proved useful for differentiating the surface status of the archwires, especially with large magnification.
4. The presence of corrosion cells in the artificial saliva proved that the NiTi orthodontic archwires were able to initiate the process of corrosion in the mouth, particularly at the point of friction in contact with the braces (Ti6Al4V).
5. Ortho Classic archwires were affected by the corrosion behavior in artificial saliva, as demonstrated using FIB/TEM testing.
6. American Orthodontics Company archwires featured spots of corrosion, as revealed by examining the SEM microanalysis.

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