RESEARCH ARTICLE

SEM/EDS ANALYSES ON SHAPE MEMORY ALLOY SUBJECTED TO ELECTROCHEMICAL CORROSION

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ARTICLE DETAILS

ABSTRACT

The aim of the present paper was to investigate the modifications occurred in the Ti- based shape memory alloy subject to electrochemical corrosion in artificial saliva. By 2D and 3D microscopy and by qualitative determinations of the luminous variation we could notice the effects of electrochemical corrosion tests on the surface of the metallic material, and by EDS determinations (Line and Mapping modes) of the surface chemical composition we could determine the chemical modifications produced following the corrosion tests.

KEYWORDS

1. INTRODUCTION

Wire-bracket systems are commonly used in orthodontic techniques. Practitioners can choose from a wide range of wires and brackets made from different alloys: stainless steel, titanium, cobalt- chromium, and niobium. Ni-Ti alloys exhibit the shape memory effect (SME – Shape Memory Effect). The behavior of Ni- Ti alloy was studied for the first time by Buehler and Wiley in the U.S. Marine Artillery Lab. This equiatomic alloy of Nitinol type has a remarkable SME at temperatures close to those in the environment. Plastically deformed initially at temperatures lower than the room temperature, Ni-Ti alloy wires will return to their initial shape once the temperature increases (Bentahar et al., 2005; Rodriguez and Brown, 1975; Henderson et al., 2011).

In recent years it has been reported that in an acidic environment (Buccal cavity) and in the presence of mouthwashes, the corrosion resistance of certain materials, particularly Ti and Ti alloys, can deteriorate (Bentahar et al., 2005; Henderson et al., 2011; Baciu et al., 2017). The goal of this study consists in the determination of the modifications occurred in the shape memory Ni-Ti alloy used for the orthodontic fixed appliances following the electrochemical corrosion in Fusayama–Meyer artificial saliva.

2. MATERIALS AND METHODS

We used orthodontic wires from Ni-Ti-based alloy, SentalloyGaC Pak-Dentsply Sirona.

Table 1: Chemical composition of Fusayama–Meyer artificial saliva used for electrochemical corrosion tests.

Artificial saliva	NaCl g/l	KCl g/l	NaHCO3 g/l	Ure e g/l	CaCl2 g/l	NaH2PO4. H2O g/l	Na2S g/l
Fusayama –Meyer	0.4	0.4	0.69	1	0.65	0.69	0.05

The electro- corrosion tests were carried out in a Fusayama–Meyer artificial saliva solution having pH= 5.3, and a chemical composition very close to the natural saliva (Table 1). The behavior to corrosion was evaluated by fast electrochemical tests specific to dynamic potentiometry. After having finished the corrosion tests, the specimens made of Ni-Ti wires were chemically analyzed by means of an EDS detector and structurally analyzed by SEM electronic microscopy.

3. RESULTS AND DISCUSSIONS

The EDS energy specter of Ni-Ti alloy resulted after having conducted the resistance tests to electrochemical corrosion is given in Figure 1.
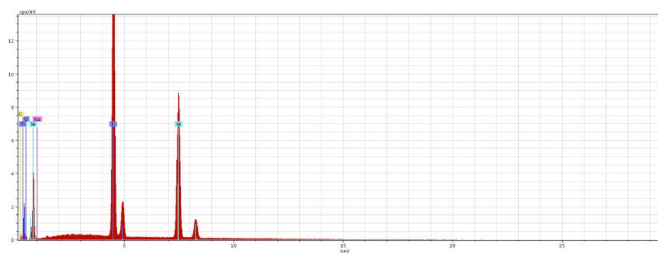
Figure 1: EDS energy specter of Ni-Ti alloy.

We identified five chemical elements:

- Ni and Ti proper to the smart alloy under study;

- Na, C and O formed on the alloy surface following the reactions with the electrolyte solution. Their presence is marked by a single energy value.

The chemical composition of the material surface after the electro-corrosion test in Fusayama– Meyer artificial saliva, as determined on a surface of 0.04 mm^2, is given in Table 2.

Table 2. Chemical composition of the Ni-Ti alloy surface after the electrochemical corrosion test in Fusayama–Meyer artificial saliva (S=0.04 mm²).

| Element	[wt.%]	[norm. wt.%]	[norm. at.%]	Error in %
Titanium	56.12	41.55	31.38	0.57
Nickel	52.15	38.61	23.79	0.33
Oxygen	25.10	18.58	42.01	2.19
Sodium	0.90	0.67	1.05	0.13
Carbon	0.79	0.59	1.77	0.21

From the registered data, we may see that the oxidized surface has a high oxygen percentage (18.6 mass %). Besides oxygen, we also identified Na and C, chemical elements that diffused from the electrolyte solution on the surface of the shape memory metal material. For this alloy, the nickel determined some corrosion behavior inferior to that of titanium (value decreased to 38.61 mass %) thus forming numerous oxides. Light elements (such as oxygen, nitrogen, etc.) frequently have lower measurement errors than those of metal chemical elements. From the general distribution, Figure 2, we may notice the tendency of agglomeration of the oxygen-based and sodium-based compounds in the areas of the superficial layer affected by the corrosion process.
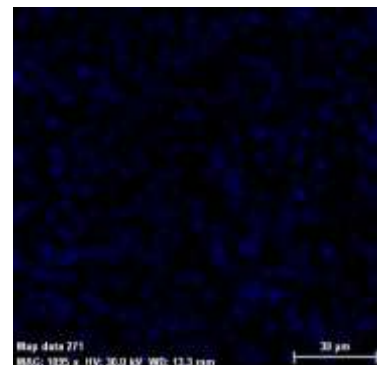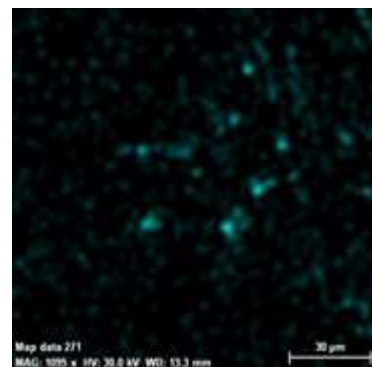

(a)
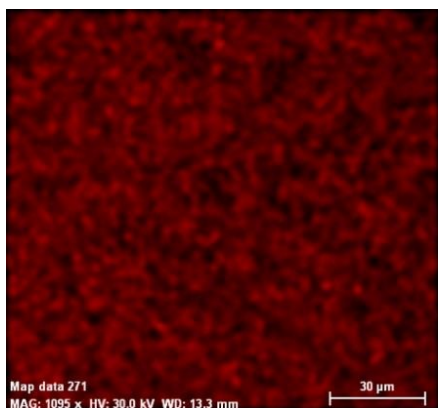
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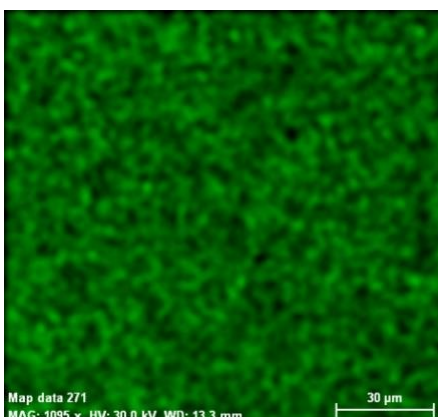
(c)

(d)

(e)

Figure 2: Distribution of main elements on the alloy surface after having conducted the corrosion tests: a) distribution of elements; b) titanium distribution; c) nickel distribution; d) oxygen distribution; e) sodium distribution.

Nickel and titanium chemical elements have a homogenous distribution into the alloy whereas sodium is mainly distributed in the superficial areas where chemical or inter-metallic compounds are present. Besides the formed oxides, the existence of sodium indicates the passage of some salts from the electrolyte solution (artificial saliva) on the surface of the metal material following the corrosion process. The shape and size of cavities resulted through pitting were evaluated on the 3-D micrographies of the surface of the material under testing, Figure 3. SEM images were made at a 10 µm scale. Figure 3d presents the light profilogram of the surface tested for corrosion. We may see that a diminution by 60 d.u. (dimensionless units) of the light intensity takes place on the corroded area as compared to the areas unaffected by this process (Sun et al., 2012). It is possible that the material "pits" seen on the surface might be the result of the chemical reactions between the metal material and the synthetic corrosion environment.
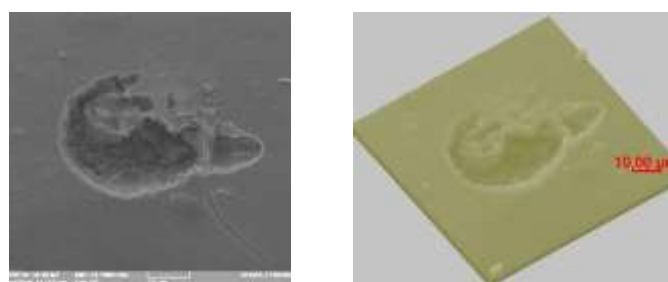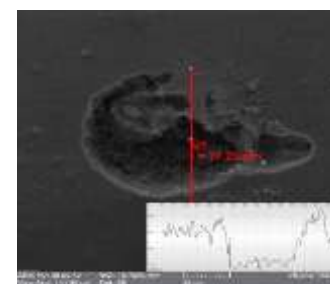

a)

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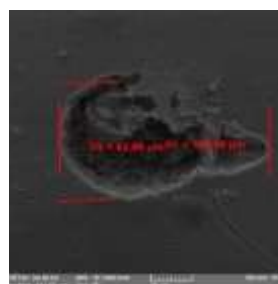
c)

d)

Figure 3: Formation of some micro cavities on the surface of the metal material subject to corrosion tests (SEM images): a) micro cavity of pitting type (2500:1); b) 3-D surface morphology; c) cavity linear sizing; d) cavity profilogram formed by pitting.

The chemical composition and concentration of the components of artificial saliva will influence the number and sizes of micro cavities formed as well as the dissolution speed of the metal subject to corrosion tests.

4. CONCLUSION

The analysis of experimental results shows that the SME alloy of Ni-Ti type for orthodontic fixed appliances is characterized by a good resistance to electrochemical corrosion in special synthetic environments.

The chemical composition and microscopic analyses conducted on the metal surface showed:

- the formation of reaction products (oxides, salts etc.) between the chemical elements of the alloy and the components of artificial saliva;

- modifications of the concentration of chemical elements in Ni-Ti alloy composition due to the process of dissolution in the corrosive environment;

- the appearance of "pits" on the metal surface as a consequence of the pitting phenomenon characterizing the electrochemical corrosion under analysis.

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