ASSESSMENT OF MECHANICAL STRENGTH AND CORROSION RESISTANCE AT VARIABLE pH OF ORTHODONTIC WIRES

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Abstract

Orthodontic wires are components of fixed appliances used to perform the necessary tooth movements in the course of the orthodontic treatment. A variety of materials e.g. metals, alloys, polymers and composites are used to produce orthodontic wires. This study examined the mechanical strength and cracks resistance of three different types of wires, i.e. made of: austenitic steel grade AISI 303, NiTi alloy and Tiβ alloy. Corrosion processes are regarded to have a harmful effect on the properties of orthodontic wires, such as their strength, biocompatibility and aesthetic appearance. In this study, we investigated the corrosive behaviour of the wires in the artificial saliva solutions with varied pH simulating the natural oral cavity environment. It was demonstrated that the orthodontic rectangular wires made of austenitic steel grade AISI 303 exhibited the highest tensile strength. The NiTi alloy wires exhibited the best plastic properties of all the examined samples. In the case of electrochemical tests (changes in corrosion potential over a period of 24 h), the wire made of austenitic steel and the NiTi alloy wire reached a stable level of the stationary potential in the acidic environment. For the wires made of Tiβ, the highest stationary potential was observed in the alkaline environment. Additionally, the Tiβ alloy wire revealed the broadest passivation area in the specified potential scope.

Keywords: *tensile test, orthodontic wires, austenitic steel, nitinol (NiTi), Tiβ, corrosion resistance*

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Introduction

Malocclusion may result from abnormalities in the structure and position of the jaw bones in relation to each other or from a disturbed arrangement of the dental arches. It may be a hereditary or acquired condition, leading to distorted speech, difficulty breathing or eating. In such cases, orthodontic treatment is helpful for patients because of both health and aesthetics [1-3].

The orthodontic treatment is carried out for adolescents and adults, and concerns primarily in correcting crowded, rotated, buried or prominent front teeth. The group of 6000 children in Polish elementary schools was screened for dental caries by Wrigley Polska Company in 2014. The studies revealed that up to 60% of pupils required orthodontic treatment [4]. According to [5], 60% of people in Poland have a malocclusion.

The application of alloys with elastic memory dates back to the 1970s when Andreasen began their promotion in orthodontics [6]. In modern stomatology, the wires are made of alloys characterized by a one- and two-directional effect of elastic memory and superelasticity [7-9]. The most popular and frequently used orthodontic wires are arches made of the NiTi (Nitinol) alloy, the beta-type titanium alloy (Tiβ) and stainless austenitic steel [10-12]. The developments in nickel-titanium (NiTi) wire technology lowered the popularity of stainless steel wires for initial alignment. However, stainless steel archwires are still used by a small proportion of orthodontists [13]. During the treatment, the choice of the orthodontic wire is not random. The selection of the metallic material (wire) is dictated by a good corrosion resistance in the environment of the oral cavity, where the electrolyte is mostly constituted by the saliva and the food products consumed by the patient [14-16].

Nickel compounds have been well established as carcinogenic but their underlying mechanisms are still not fully understood. Yet not all nickel compounds are equally carcinogenic, because their carcinogenic potency is directly related to their ability to enter cells [17]. Nickel is the most common metal to cause contact dermatitis in orthodontics but nickel-titanium alloys and stainless steel are widely used in orthodontic appliances. Nickel-titanium alloys may contain nickel in excess of 50%, which can potentially result in an allergic reaction. Stainless steel has a lower nickel content (8%) and because the nickel is bound in a crystal lattice it is not available to react [18]. The good corrosion resistance of the NiTi alloy is mainly provided by titanium oxides which form a coherent passive layer on the surface of the material [14,19,20]. Austenitic corrosion-resistant steels were the first metals implanted in the human body. The content of austenite and ferrite-forming elements should be selected taking into account their impact so that the austenite is thermodynamically stable [21]. The presence of chromium in the amount above 13% ensures a positive corrosion potential and good corrosion resistance in oxidizing environments. Additionally, chromium reduces the passivation current density and increases the resistance to pitting corrosion [15,16,22]. As the nickel content increases, the resistance to stress corrosion cracking increases [23]. The operation time of austenitic steel should not exceed 2 years, due to the stress corrosion, material destruction and toxic elements penetrating the body tissue [22]. A very popular and commonly available biomedical material is titanium due to its high corrosion resistance, also in physiological fluids. The Tiβ alloy is mono-phase, therefore it possesses better anticorrosive and tribological properties than the dual-phase alloy $\alpha+\beta$ [15,16,24].

Materials and Methods

In the research three types of orthodontic wires were investigated: a rectangular wire made of austenitic steel grade AISI 303, with the dimensions 0.48x0.63x167 mm (FIG. 1a), a wire made of Nitinol alloy (NiTi), Ø = 0.5 mm in diameter and l = 160 mm long (FIG. 1b) and a rectangular wire made of beta-type titanium alloy (Tiβ), with the dimensions 0.4x0.55x160 mm (FIG. 1c).

The rectangular wire made of austenitic steel (FIG. 1a) is suitable for big malocclusions, where a high force is required and it is mostly used in the first stage of the orthodontic treatment. Rectangular arches made of austenitic steel are characterized by the best resistance to the operation of the tooth's movement forces [2,25]. They are applied for a sagittal shift of the teeth in the side segment, arrangement of the canines in I canine class, arrangement of the incisors in the proper angulation, correction of the median line. In the contraction phase, they are used to arrange the canine teeth in the proper sagittal position. The NiTi alloy orthodontic wire (FIG. 1b) is used at the beginning of the orthodontic treatment to level the position of the brackets in the horizontal and vertical plane [2,25]. Additionally, it prevents rotation and is responsible for the position of the molar teeth. Such an arch also makes sure that the molar teeth and the canine teeth assume the proper direction of levelling. The Tiβ orthodontic wire (FIG. 1c) has a smooth surface. It is used both in the course and the contraction of the treatment to provide: sagittal shifts of the teeth in the side segment, "artistic" arrangement of the incisors, correction of the incisors, correction of the median line, levelling of rotation [2,25]. The Tiβ wires are usually used in the case of patients allergic to nickel. The chemical compositions of the examined wires used in orthodontic treatment are presented in TABLE 1.

The strength tests of the analyzed materials were performed according to the PN-EN 10002-1 standard [27]. They were carried out with the use of a Zwick/Roell Z250 tester equipped with tensometric sensors and electronic extensometers. With the use of the Test Expert V5 program, it was possible to precisely steer the power feed and to record all the parameters in the scope of a static tensile test with the assumed deformation-stress cyclogram. In the case of the presented investigation results, a tensometric sensor with the scope of the measured force of up to 10 kN and an electronic extensometer with a varying measurement base (80 mm) was applied. The test parameters were the 50 N initial force and the 5 mm/min movement speed of the traverse. For each examined wire, three static tensile tests were performed and the results presented as averaged values.

The scanning electron microscope HITACHI S-3500N was used to examine the wires surface after the pull tests.

The oral cavity environment is highly moist and subjected to the temperature and pH changes depending on beverage and food consumed, thus it favours dental materials degradation [28]. The electrochemical tests were carried out in a physiological solution of artificial saliva, whose chemical composition is given in TABLE 2 [29]. Saliva plays a key role in lubrication, mastication, taste perception, prevention of oral infection and dental caries. Saliva is one of the most important factors in preventing dental caries, too [30]. The tests were performed to determine the effect of a varying pH of the artificial saliva solution on the corrosion resistance of the examined wires made of austenitic steel, alloy NiTi and alloy Tiβ. TABLE 2 shows the chemical composition of the artificial saliva treated as an environment with a physiological pH of 7.4. Additions of HCl (for pH = 4.0) and NaOH (for pH = 8.0) were applied to obtain various pH values of the artificial saliva.

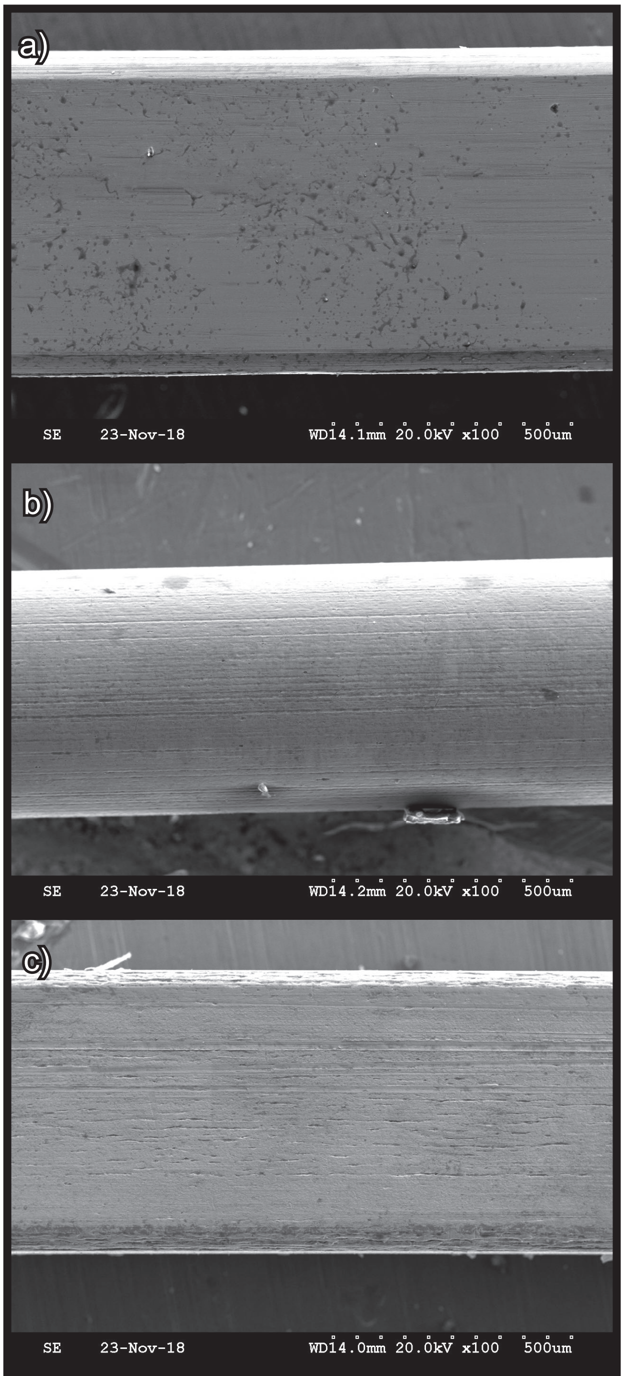

FIG. 1. SEM images of the surface morphology of orthodontic wires made of AISI 303 austenitic steel (a), NiTi alloy (b), Tiβ alloy (c).

TABLE 1. Chemical composition of orthodontic wires made of austenitic steel AISI 303 grade (a) [19], NiTi (Nitinol) (b) [18,19] and Tiβ alloy (c) [25,26].

a) Chemical composition, % mass.				
Cr	Ni	Mn	Co	Fe
18	9	2	0.75	rest
b) Chemical composition, % mass.				
Ni	Ti			
55.82	rest			
c) Chemical composition, % mass.				
Zr	Mo	Sn	Ti	
4-8	8-12	2-5	rest	

The examinations of the corrosion resistance of the orthodontic wires were conducted by means of stationary potential meters OCP – Keithley 2000 Multimeter and potentiostat Autolab PGSTAT302N. The electrochemical tests for each of the tested wires, and for each of the pH values lasted 24 h.

The electrochemical tests on a global scale were performed by means of a standard electrolytic vessel with 3 electrodes (FIG. 2) [24]. The vessel contained a working electrode (the examined sample of an orthodontic arch made of: austenitic steel grade AISI 303 or alloy NiTi or Tiβ), a platinum counter-electrode and a reference electrode (chlorosilver electrode).

The tests were carried out in the artificial saliva solution with free access to air. The measurements were made in a water bath with a constant temperature of 37°C where the electrochemical vessel was placed. By means of the potentiostat, the accelerated tests were performed using electroless techniques (a stationary Open Circuit Potential for spontaneous processes) and direct current techniques (Linear Sweep Voltammetry potentiodynamic polarization with a constant potential change rate 1 mV/s).

TABLE 2. Chemical composition of a simulated saliva solution per 1 litre of distilled water [29].

MAS - Mayer Artificial Saliva Solution	The content of component [g], calculated per 1 litre of distilled water
NaCl	0.7
KCl	1.2
$NaHCO_3$	1.5
Na_2HPO_4	0.26
KSCN	0.3
$Na_2S \cdot 9H_2O$	0.005
Urea	1.0
pH of the solution	**7.4**

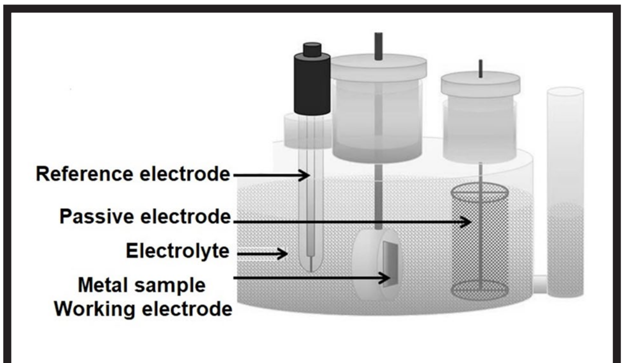

FIG. 2. Electrochemical vessel for research on a global scale [24].

Results and Discussion

The purpose of the static tensile tests was to determine the basic mechanical properties of the examined orthodontic wires, i.e. the Young modulus (E), the yield point $R_{p0,2}$, the tensile strength R_m, the plasticity reserve coefficient, the ratio $(R_{p0,2})/(R_m)$ and the rupture stress R_B, as well as the elongation with the highest force A_g. FIG. 3 and TABLE 3 present the averaged values of the three static tensile tests for each of the examined orthodontic wires.

Among the tested orthodontic wires, the NiTi alloy one achieved the highest recorded elongation value at the highest force, $\bar{A}_g$ = 10.5% a few times higher result than the other examined wires. This alloy exhibited the lowest values of proof stress ($\bar{R}_{p0,2}$ = 346 MPa) and Young modulus ($\bar{E}$ = 37.6 GPa) (TABLE 3).

Already during the tensile tests, the NiTi wire underwent significant elongation and deformation (FIG. 3).

The alloy was probably reinforced already during the tests due to the plastic deformation and the formation of martensite, which strongly strengthened the alloy [31-33]. The plasticity reserve coefficient $\bar{R}_{p0,2}/\bar{R}_m$ equalled 0.29, the result two or three times higher than the other materials (the lower the value of this parameter, the higher the alloy's plasticity). The tensile strength ($\bar{R}_m$) of the NiTi wires was 1200 MPa.

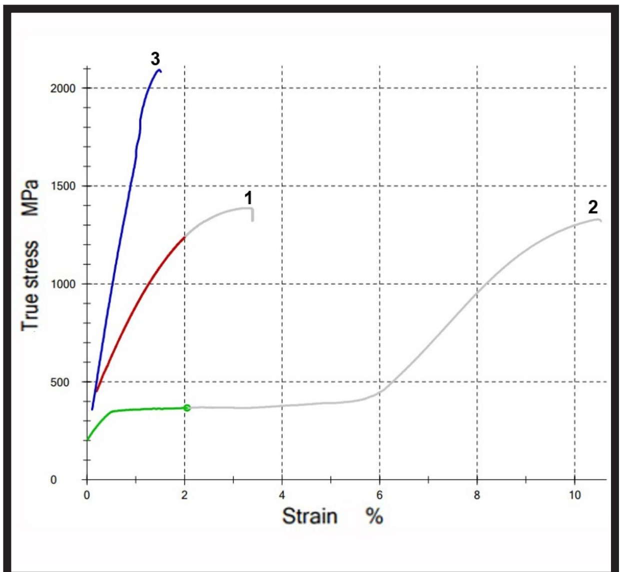

FIG. 3. Averaged waveforms of the static tensile test of the tested orthodontic wires. Curve 1 - Tiβ alloy wire, curve 2 - NiTi alloy, curve 3 - austenitic stainless steel of AISI 303 grade.

TABLE 3. Averaged values of the properties obtained from the tensile tests of the tested orthodontic wires.

Tested orthodontic wire	a_0, mm	b_0, mm	S_0, mm²	$\bar{E}$, GPa	$\bar{R}_{p0,2}$, MPa	$\bar{R}_m$, MPa	$\bar{R}_{p0,2}/R_m$	$\bar{R}_B$, MPa	$\bar{A}_g$, %
Tiβ (1)	0.40	0.55	0.22	62.8	965	1340	0.72	1270	3.2
NiTi (2)	0.50	0.50	0.25	36.7	346	1200	0.29	1190	10.5
Austenitic steel (3)	0.48	0.63	0.30	158.0	2020	2060	0.99	2050	1.5

The determined value $\overline{R_m}$ exceeded the upper limit of the values given by the producer (i.e. 800-1000 MPa) [25]. The NiTi rupture stress $\overline{R_B}$ was the lowest of all the tested materials and equalled ($\overline{R_B}$) = 1190 MPa (TABLE 3).

The highest tensile strength was exhibited by the rectangular wires made of austenitic steel grade AISI 303, whose value ($\overline{R_m}$) equalled 2060 MPa. This result is close to the lower limit guaranteed by the producer, i.e. 2190-2345 MPa [26]. For this material, the highest values of Young modulus were obtained ($\overline{E}$ = 158 GPa), significantly exceeding the values for the NiTi and Tiβ wires. The austenitic steel samples underwent the fastest rupture with the highest rupture stress ($\overline{R_B}$) = 2050 MPa and reached the lowest elongation of all the tested materials ($\overline{A_g}$ = 1.5%) (TABLE 3).

The Tiβ alloy, similarly to the austenitic steel, demonstrated a much lower elongation than of the nickel alloy with titanium $\overline{A_g}$ = 3.2%. For this alloy, the recorded tensile strength value was ($\overline{R_m}$) = 1340 MPa, which confirms the values declared by the producer, i.e. 1120-1320 MPa [25]. The significantly lower Young modulus values were also recorded ($\overline{E}$) = 62.8 GPa in comparison to austenitic steel which were still higher than those for NiTi. The mean value of the rupture stress for Tiβ equalled ($\overline{R_B}$) = 1270 MPa (TABLE 3).

The SEM observations of the orthodontic wire fractures were performed to evaluate the obtained surfaces and determine the morphology and topography of the analyzed materials in the area of their rupture. FIG. 4 shows SEM images of the fractography of the sample (wire) surfaces after the performed strength tests.

The fractography of the samples made of austenitic steel grade AISI 303 and Nitinol demonstrated a dimpled ductile fracture (FIG. 4a-d). Both the rectangular wire made of austenitic steel (FIG. 4a, b) and the NiTi one (FIG. 4c, d) formed necks which prove the material rupture (local reduction of the sample cross-section). The topography of the plastic fracture is characterized by a large set of dimples (craters) of different sizes and shapes.

The fractography of the Tiβ wire shows two kinds of fractures on the formed neck (FIG. 4e). A plastic fracture was formed on the external walls of the neck (FIG. 4f, g), while inside it, a brittle transcrystalline fracture was visible (FIG. 4f, h). The formed microcracks can propagate through the grains simultaneously in a few parallel and proximate planes of cleavage, followed by a formation of jogs (edges) visible on the fracture surface. Based on the observations, it can be concluded that the sample ruptured first on the internal side of the neck and next on the external sides.

In the further part of the study, electrochemical tests were performed with changing pH values. Three different corrosion environments were simulated. An artificial saliva solution was treated as an environment with the physiological 7.4 pH; the same solution with additions of HCl (for pH = 4.0, acid reaction), and of NaOH (for pH = 8.0, alkaline reaction) was used. For each of the tested orthodontic wires, the stationary potential was determined in the function of time (OCP) along with the polarization curves in the examined artificial saliva solution for three different solution pH values: 4.0, 7.4 and 8.0 (FIG. 5).

For the austenitic steel wire, the stationary potential was reached in each artificial saliva solution (FIG. 5a). The highest potential was recorded in the acidic environment (pH = 4.0), which means that in this solution austenitic steel has the highest corrosion resistance. A very similar potential value was reached by the sample in the environment with pH = 7.4. The lowest potential was reached by the steel sample in the environment with the highest alkaline pH (pH = 8.0).

For the NiTi arch, the change in the stationary potential in time is illustrated in FIG. 5b. The highest potential was reached in the artificial saliva solution with pH = 4.0 and it was maintained at this level (about 0.05 V vs Ag/AgCl) from the beginning of the measurement. In the case of pH = 7.4 and pH = 8.0, the stationary potential stabilized only after about 12 h. For pH = 7.4, the stationary potential values in the first 3 h were very unstable and varied in the scope of -0.15 to -0.5 V vs Ag/AgCl, after which the OCP values began to stabilize in the following hours. In the solution with pH = 8.0, the potential rapidly dropped after about 7 h from -0.5 V vs Ag/AgCl to -0.25 V vs Ag/AgCl, reaching a stable value of about -0.3 V vs Ag/AgCl after approx. 24 h.

The Tiβ wire (FIG. 5c) in each of the tested artificial saliva solutions was able to reach a stable stationary potential fast. The highest stationary potential was reached in the artificial saliva solution with pH = 7.4, which equalled about 0.01 V vs Ag/AgCl. The highest potential was assumed in pH = 4.0, it stabilized after about 4 h and equalled -0.35 V vs Ag/AgCl. FIG. 6a shows the polarization curve obtained for austenitic steel in the solution with free access to oxygen in the artificial saliva solution with three different pH values. One should note the curve obtained in the solution with pH = 4.0, in which both the cathodic and anodic current densities were higher as compared to the other solutions. There was also no distinguishable passivation area of the sample (FIG. 6a). This proved the lowest corrosion resistance of the austenitic steel wire in the solution with pH = 4.0. The largest passivation area from -0.15 to 0.7 V vs Ag/AgCl was exhibited by the steel sample in the artificial saliva solution with pH = 8.0. In turn, in the solution with pH = 7.4, a rapid current increase (a breakdown of the passive layer) took place with the potential 0.15 V vs Ag/AgCl (FIG. 6a).

In the case of alloy NiTi, the polarization curves (FIG. 6b) showed lower current values on the anodic side for each of the three artificial saliva solutions. The longest passivation area from about -0.3 V vs Ag/AgCl to 0.6 V vs Ag/AgCl was demonstrated in the artificial saliva solution with pH = 8.0. Nevertheless, there were no significant differences in the alloy corrosive behaviour depending on the solution pH, as was the case with austenitic steel. This was illustrated by the very similar curves in terms of shape.

The Tiβ alloy polarization curves (FIG. 6c), proved the highest corrosion resistance of all the examined materials.

The current densities on the anodic side were not much different from the cathodic side. Compared to the previous samples, the Tiβ alloy behaved similarly in each artificial saliva solution, regardless of its pH. The slightly lower current densities on the anodic side were obtained in the solution with pH = 4.0. The curves did not show a characteristic breakdown, a rupture of the corrosion resistance (rupture of the passive layer continuity). It was seen that the passivation area was large for titanium, i.e. from the value of 2.5 V vs Ag/AgCl, where only at about 1.75 V vs Ag/AgCl a characteristic increase in the anodic current density took place. This phenomenon occurred due to the titanium oxide TiO_2 transformation into Ti_2O_3 on the sample surface.

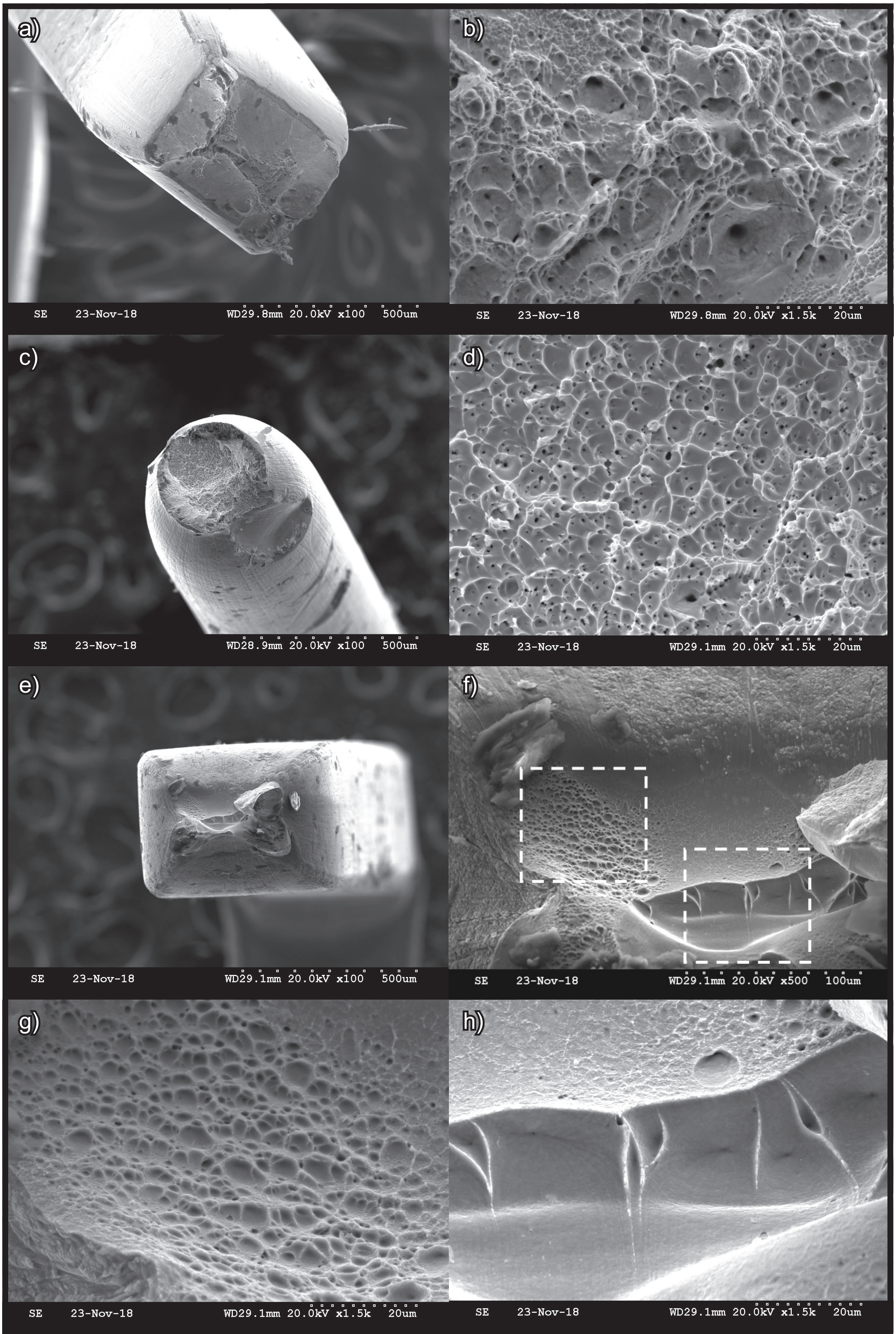

FIG. 4. SEM images of fractures of orthodontic wires: edge wire made of austenitic steel of AISI 303 grade (a and b), NiTi alloy (c and d), Tiβ alloy (e-h).

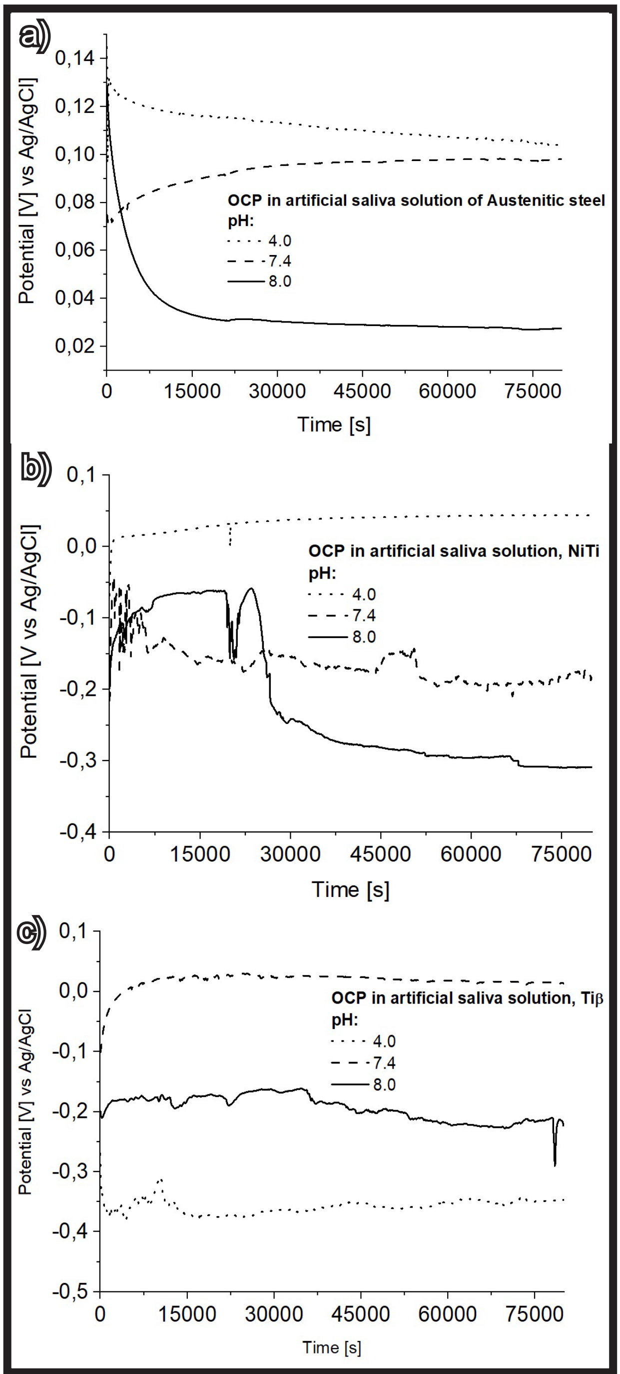

FIG. 5. Evolution of the corrosion potential vs. time determined for samples of austenitic steel of AISI 303 grade (a); Nitinol (b); Tiβ alloy, (c) in artificial saliva solution (different pH: 4.0, 7.4 and 8.0).

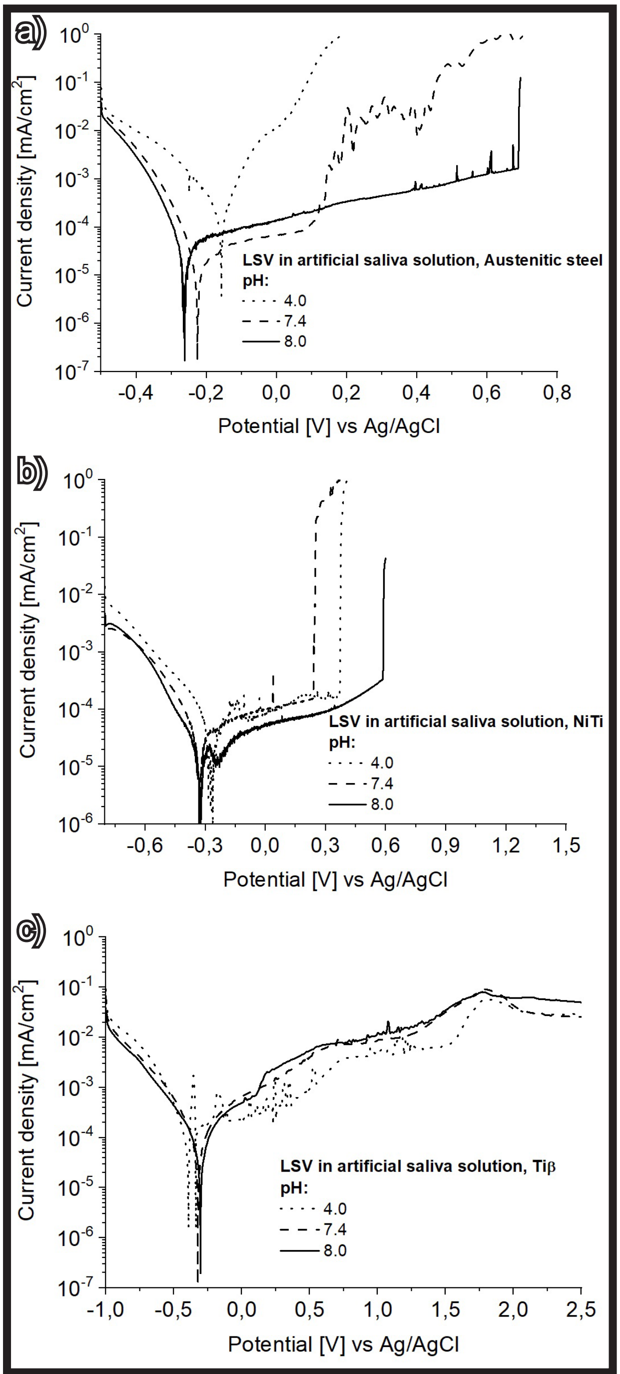

FIG. 6. Polarization curves obtained for samples of austenitic steel of AISI 303 grade (a), NiTi alloy (b) and Tiβ alloy, alloy (c) in artificial saliva solution (variable pH: 4.0, 7.4 and 8.0).

FIG. 7 compares the polarization curves for the three examined materials in the artificial saliva solution with pH = 7.4. We can see that Tiβ has a much broader scope of passivation potential than the other materials.

TABLE 4 shows a comparison of the tested materials in respect of the anodic current value with the potential 0 V vs Ag/AgCl and the width of the passivation potential scope. With the assumed potential, the highest resistance was exhibited by the austenitic steel grade AISI 303 wire (the lowest current density).

The obtained results were compared with other results described in the literature [34-36]. In [34], electrochemical tests showed that in the acidic environment (pH = 3.0) stainless steel wire had better corrosion resistance than nickel-titanium arc. In the present work, the austenitic steel and NiTi alloy wires reached a stable level of the stationary potential in the acidic environment (pH = 4.0), and for the Tiβ orthodontic wire, the highest stationary potential was observed for the alkaline environment (pH = 8.0). Future studies could concern microbiologically induced corrosion, similar to the one presented in [35,36].

TABLE 4. Anodic current density for the tested orthodontic wires at the potential of 0 V vs Ag/AgCl in artificial saliva solution at pH = 7.4.

Orthodontic wires	Current density·10^{-5} [mA/cm^2]	The area passivation [V] vs Ag/AgCl
Austenitic steel	6.2	-0.15 - 0.07
NiTi	9.9	-0.24 - 0.23
Tiβ alloy	61.9	-0.06 - 2.5 (in the range tested)

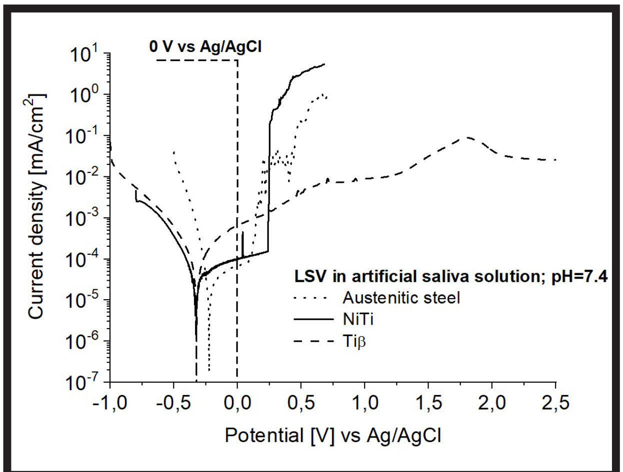

FIG. 7. Comparison of LSV of austenitic steel of AISI 303 grade, NiTi and titanium alloy β in artificial saliva solution with pH = 7.4.

Conclusions

The performed static tensile tests, observations of the fractures and electrochemical examinations of three different wires used in the orthodontic treatment made it possible to draw the following conclusions:

The performed strength tests showed that the highest tensile strength ($\overline{R}_m$ = 2060 MPa) and Young modulus ($\overline{E}$ = 158 GPa) was exhibited by the rectangular orthodontic wire made of austenitic steel grade AISI 303. The best plastic properties were demonstrated by the NiTi alloy wire ($\overline{A}_g$ = 10.2 %).

After a rupture, the SEM examinations of the sample fractures revealed a dimpled ductile fracture both for the austenitic steel wire and the NiTi wire. In the case of alloy Tiβ, a mixed fracture was revealed (ductile, formed on the external sides of the neck, and brittle transcrystalline, formed inside).

The electrochemical tests of the corrosion stationary potential change in time revealed the austenitic steel and NiTi wires stable stationary potential in the acidic environment. The Tiβ samples exhibited the highest stationary potential in the alkaline environment.

The performed electrochemical tests of the stationary potential and the polarization curves confirmed that all the materials in the examined artificial saliva solution were passivated in the scope of the potential they operated in freely when submerged in the solution.

The highest current density was observed for the Tiβ wire. Nevertheless, of the three examined biomedical materials it was also the Tiβ wire, which demonstrated the highest corrosion resistance, due to the widest passivation scope.

The corrosion polarization investigations helped determine the corrosion rate. For austenitic steel and NiTi, the broadest passivation area occurred in the solution with pH = 8.0.

The comparison of the polarization curves of the examined samples obtained during the tests in the artificial saliva solution with pH = 7.4 showed that the Tiβ wire had the broadest passivation area in the specified potential scope.

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