Original Article

Influence of surface layer on mechanical and corrosion properties of nickel-titanium orthodontic wires

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ABSTRACT

Objective: To analyze the effect of various coating formulations on the mechanical and corrosion properties of nickel-titanium (NiTi) orthodontic wires.

Materials and Methods: Uncoated, rhodium-coated, and nitrified NiTi wires were observed with a three-point-bend test, surface roughness (*Ra*) measurement, scanning electron microscopy, energy dispersive spectroscopy, and electrochemical testing (open circuit potential, electrochemical impedance spectroscopy, and cyclic polarization scan). Differences in the properties of tested wire types were analyzed with analysis of variance and Tukey post hoc test.

Results: Uncoated and nitrified NiTi wires showed similar mechanical and anticorrosive properties, while rhodium-coated NiTi wires showed the highest *Ra* and significantly higher modulus of elasticity, yield strength, and delivery of forces during loading but not in unloading. Rhodium-coated NiTi wires also had the highest corrosion current density and corrosion potential, lowest impedance modulus, and two time constants on Bode plot, one related to the Rh/Au coating and the other to underlying NiTi.

Conclusion: Working properties of NiTi wires were unaffected by various coatings in unloading. Nitrification improved corrosion resistance. Rhodium coating reduced corrosion resistance and pronounced susceptibility to pitting corrosion in artificial saliva because of galvanic coupling between the noble coating and the base alloy. (*Angle Orthod.* 2014;84:1041–1048.)

KEY WORDS: NiTi; Surface coating; Three-point-bend test; Corrosion; Electrochemical testing

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INTRODUCTION

Nickel-titanium (NiTi) orthodontic archwires possess both shape memory and superelastic properties, and their low constant forces during a longer period of time are considered to be desirable for tooth movement.[1,2] Although there has been much research on basic wire types used in orthodontic therapy, every day we meet with novel modifications of those basic materials that are still insufficiently investigated.[3–7] Changes in surface composition (for improved esthetic or reduced friction) could affect wires' roughness parameters[8,9] and working and corrosive properties,[10–13] and as a result, archwires could behave differently within the oral cavity.[14,15] The surface roughness of archwires in orthodontics is still considered a controversial parameter as far as its relation to friction and corrosion are concerned as well as its relation to clinical use.[5,16–18]

Uncoated NiTi wires have titanium oxides (TiO_x) as anticorrosive components; rhodium-coated NiTi (Rh NiTi) has gold and rhodium in a 0.5-μm thin layer (as stated by the manufacturer). Both are noble metals and form a protective surface layer. Nitrified NiTi

Table 1. Chemical Composition of Uncoated, Rhodium-Coated, and Nitrified Nickel-Titanium Orthodontic Wires With Energy-Dispersive Spectroscopy Analysis in "As Received" and "After Corrosion Test" Conditions

	Chemical Composition, wt %				
Wire Type	Ni	Ti	Rh	Au	N
Uncoated, as received	53.0	47.0			
Uncoated, after test	51.2	48.8			
Rh coated, as received	11.3	10.4	33.7	44.6	
Rh coated, after test, bright spot	6.1	4.0	44.3	45.6	
Rh coated, after test, dark spot	43.4	48.4	8.2		
Nitrified, as received	52.3	46.8			0.9
Nitrified, after test	51.5	46.3			2.2

(N NiTi), through the process of nitrogen ion implantation, forms titanium nitride (TiN) on the surface, replacing allergenic nickel with nitrogen. Previous studies of TiN coating, produced experimentally in laboratory conditions, showed improvement of mechanical and biocompatible properties.[19,20] Exact data on the manufacturing process and conditions the coated wires investigated herein were not available from the manufacturer.

The corrosion of orthodontic wires in contact with saliva is indeed an electrochemical process. It is common to perform in vitro studies of corrosion processes using electrochemical methods[5–7,17,18,21,22] because the release of metal ions within the oral cavity occurs over a long period and is associated with discoloration of adjacent structures and teeth as well as allergic and toxic reactions.[14,15] Electrochemical impedance spectroscopy (EIS) testing determines the polarization resistance, a direct measure of corrosion resistance; cyclic polarization testing helps in the observation of the pitting tendencies of the alloy; and in vitro anodic polarization studies simulate the natural corrosion mechanism that usually happens over a protracted duration of time. With all of these reasons taken into account, clinicians should be informed about limitations and potential risks when using various materials.

Mechanical behavior, surface roughness, and anticorrosive potential in commercially available orthodontic NiTi wires with various coatings are investigated in this research. The hypotheses are that various coatings (1) do not affect mechanical properties, (2) change surface roughness parameters, and (3) improve anticorrosive properties of NiTi wires.

MATERIALS AND METHODS

Three types of preformed superelastic NiTi alloy orthodontic archwires (nickel, 50.4%; titanium, 49.6%), in dimensions of 0.020 × 0.020-inch BioForce Sentalloy (Dentsply GAC Int, Islandia, NY), were investigated:

- NiTi with untreated surface
- Rh NiTi (High Aesthetic)
- N NiTi (IonGuard)

Energy-dispersive spectroscopy was used to determine the chemical composition of the surface and near surface of the wires (QUANTAX EDS, Bruker, Karlsruhe, Germany) and to explore differences in bright and dark areas. The micromorphology of the surface of one as-received and one electrochemically tested wire of each wire type was observed with field-emission scanning electron microscopy (SEM) VEGA 3 (TESCAN, Brno, Czech Republic) at 2000× magnification.

Ten specimens, each 2.5-cm long, were cut from the straight ends of archwires for every wire type and used for a three-point bending test. The support span of Texture Analyser TA.HD.plus (Stable Micro Systems, Godalming, UK) was set to 12 mm and loaded with a low-force cell (5 kg, factory calibrated), according to current standards.[23,24] The temperature in a thermal chamber during measurements was set to 37°C with the help of a thermal coil and regulated automatically via thermal sensor. Each specimen was loaded to a deflection of 3.1 mm and then unloaded to zero deflection at a cross-head speed of 0.0167 mm/s. Load, in Newtons (N), and deflection, in millimeters (mm), were collected every 5 milliseconds for both loading and unloading of each specimen using the Texture Exponent software program (Stable Micro Systems). From the hysteresis loop, the following data were read off: loading and unloading elastic modulus (E) and yield strength (YS); analysis of forces in distances of 0.5, 1, 1.5, 2, 2.5, and 3 mm during loading and unloading; and unloading slope characteristics (average plateau force, average plateau length, and the percentage of useable constant force during unloading).

Measurement of surface roughness was performed using the Stylus instrument Perthometer S8P (Mahr GmbH, Göttingen, Germany). Traceability is assured by using certified calibration artifacts (Croatian national roughness standards). Determination of roughness parameter Ra was compliant with the geometric product specification standards (ISO 4287, 4288, and 3274). Traced profiles of the real surface were acquired with a diamond stylus of 5-μm radius. During the measurement, the stylus was moved at a constant speed across the samples with a measuring force of 1.3 mN. Five randomly selected specimens from each wire type were measured, and on each sample, Ra was measured on three profiles, using a Gaussian filter with a cutoff value of 0.8 mm and the evaluation length of 4 mm. The arithmetic mean of three repeated measurements was used for statistical analysis.

Specimens for corrosion testing were prepared from halved arch forms, each 50-mm long. Nail polish was

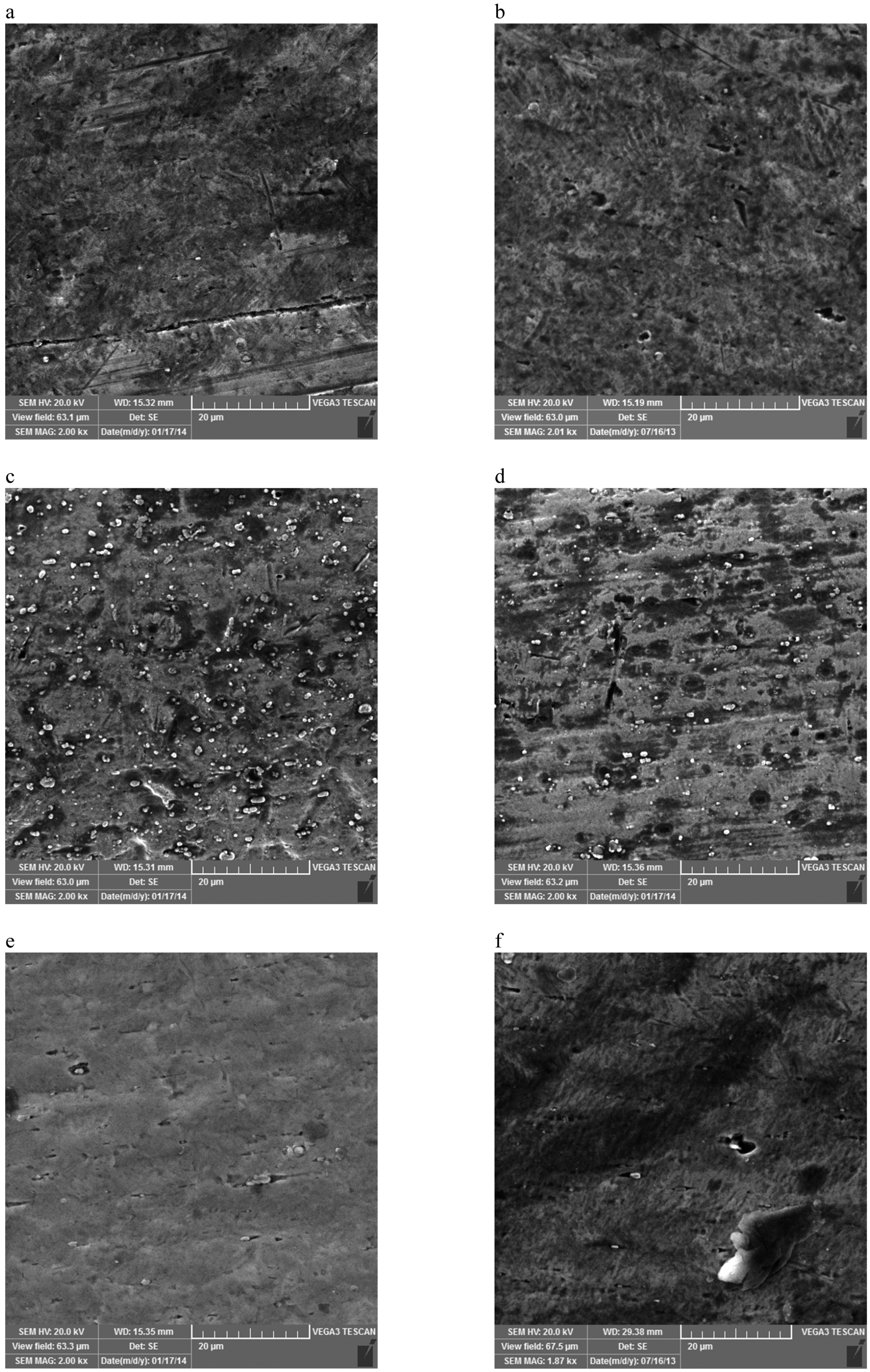

Figure 1. Scanning electron microscope images (2000× magnification) of as-received (left column) and after electrochemical testing (right column) of nickel-titanium (NiTi) wire samples. (a, b) Uncoated NiTi. (c, d) Rhodium-coated NiTi. (e, f) Nitrified NiTi.

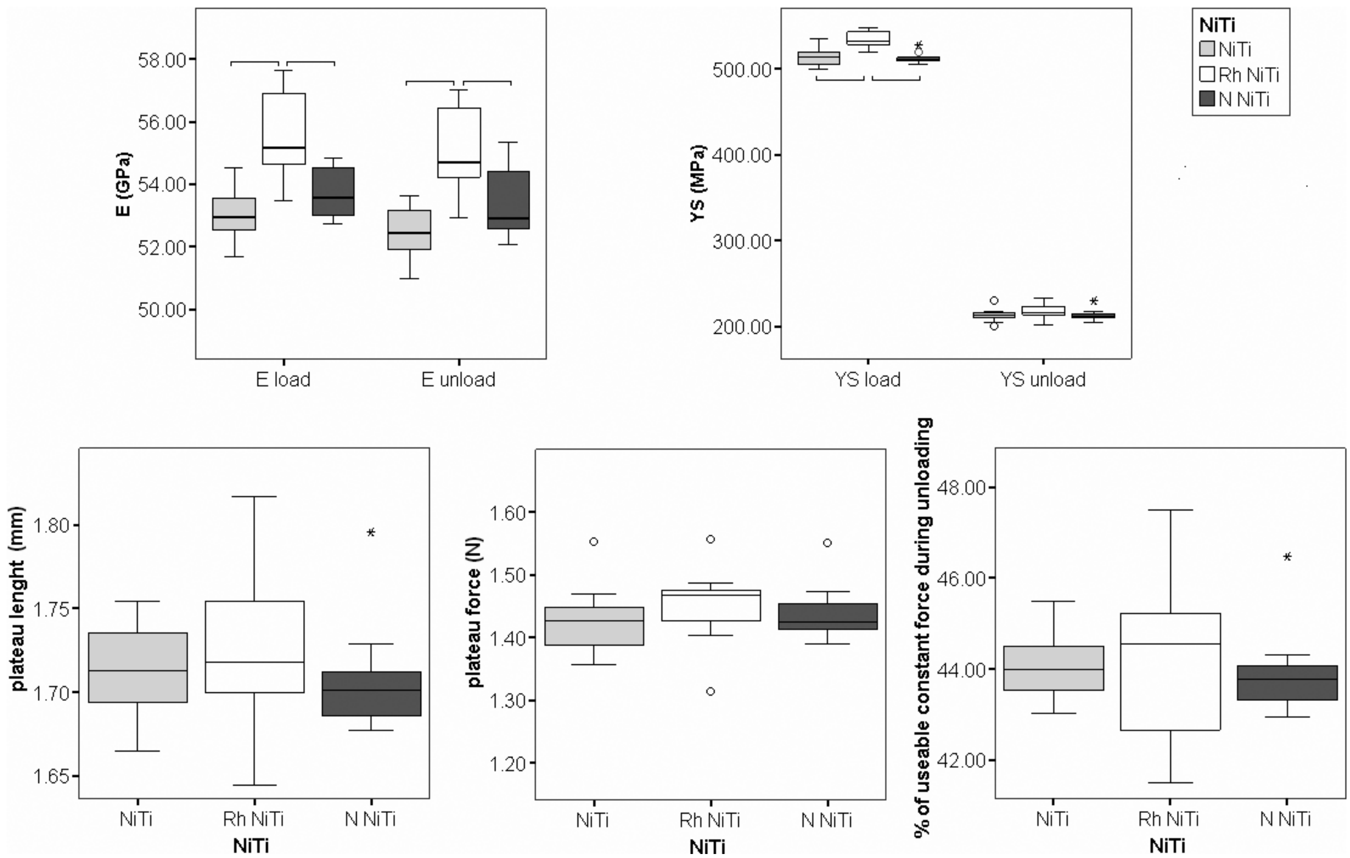

Figure 2. Loading and unloading elastic modulus (*E*), yield strength (YS), and parameters of unloading slope for nickel-titanium (NiTi) wires (uncoated [NiTi], nitrified [N NiTi], and rhodium-coated [Rh NiTi] wires). Statistically significant differences are marked with horizontal bars ($P < .05$).

used to isolate straight distal 30-mm-long segments of the specimens, with a total testing surface of 60.96 mm^2. Electrochemical testing was performed on triplicate specimens of each wire type in an artificial saliva solution (1.5 g/L KCl, 1.5 g/L $NaHCO_3$, 0.5 g/L $NaH_2PO_4xH_2O$, 0.5 g/L KSCN, 0.9 g/L lactic acid, pH 4.8)[25] at 37°C. The corrosion tests were performed using a three-electrode cell connected to a potentiostat PAR 263A and frequency response detector 1025. A saturated calomel electrode (SCE) and platinum wire were used as a reference and counter electrode, respectively. All potentials mentioned in this article refer to SCE. Electrochemical testing consisted of three steps. First, the open circuit potential (OCP) was monitored for 2 hours. This allowed stabilization of the OCP after initial instability when the wire was first introduced into the electrolyte. Next, EIS measurements were conducted at the OCP. The amplitude of the voltage perturbation was 10 mV_{rms}. Impedance measurements were conducted in the frequency range from 100 kHz to 10 mHz. Finally, a cyclic polarization scan was conducted starting from −300 mV vs OCP up to the potential where the current density reached 100 $\mu A\ cm^{-2}$, and at that point, scanning was reversed. The cyclic polarization was performed at a scan rate of 1 mV/s.

Analysis of variance and Tukey post hoc test were used for analysis of effect of wire coating on the mechanical and corrosion properties of NiTi wires.

RESULTS

Uncoated NiTi and N NiTi wires had similar surface composition prior to and after corrosion tests (Table 1), while considerable change was noted after electro corrosion of Rh NiTi, with differences among bright and dark areas (Figure 1d), indicating localized loss of noble metal coating.

SEM images of as-received samples showed surface irregularities in the form of dark spots, with the largest dark areas visible on Rh NiTi (Figure 1a,c,e). After corrosion testing, surface irregularities on Rh NiTi wires were more prominent and larger in number when compared with smoother surfaces of NiTi and N NiTi (Figure 1b,d,f).

Rh NiTi showed significantly higher loading and unloading *E* (Figure 2; $P < .001$), exhibiting the highest stiffness among tested wires. It needed the

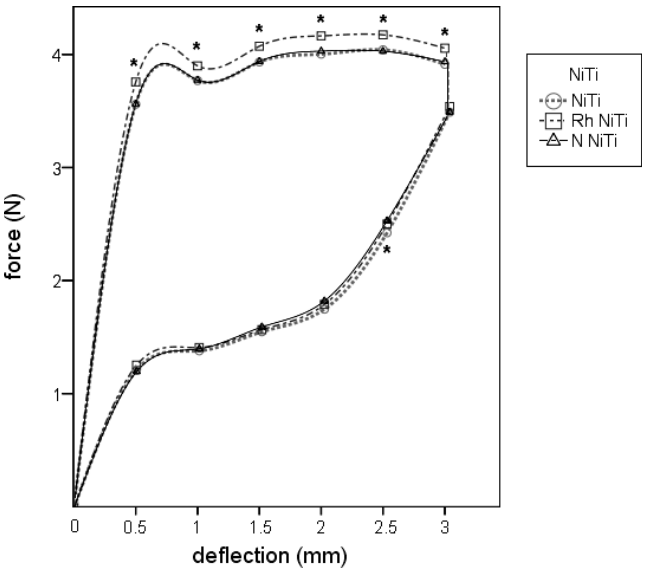

Figure 3. Comparison of loading and unloading forces in distances of 0.5, 1, 1.5, 2, 2.5, and 3 mm on representative load-deflection curves for nickel-titanium (NiTi) wires (uncoated [NiTi], nitrified [N NiTi], and rhodium-coated [Rh NiTi] wires). Statistically significant differences are marked with an asterisk (*$P < .05$).

highest stress for plastic deformation, visible in higher loading YS (Figure 2; $P < .001$).

Rh NiTi demonstrated significantly higher forces for every observed distance during loading ($P < .05$) when compared with uncoated and N NiTi wires, and uncoated NiTi differed from N NiTi only in 2.5 mm during unloading ($P < .05$; Figure 3).

Parameters of unloading slope demonstrated similar working properties for all three wire types (Figure 2).

Rh NiTi wires had the highest and uncoated NiTi wires the lowest surface roughness parameter (Table 2), but differences were not significant.

For uncoated NiTi and N NiTi wires, polarization curves appeared to be quite similar, with a huge passive region and breakdown potentials (E_{bd}) occurring at potentials as high as 1300 mV_{SCE} (Figure 4). At reverse scan, samples easily repassivate; therefore, pitting tendency is negligible. On the contrary, the polarization curve for Rh NiTi showed quite a short passive region and exhibited positive hysteresis at reverse scan. Thus, Rh NiTi passive film was unable to repair breakdown at localized areas.

The nitrified NiTi showed the lowest corrosion current densities, while Rh NiTi showed three times higher values when compared with the uncoated NiTi (Table 2), indicating that much more of the material in Rh NiTi would be lost because of the instability of the surface induced by corrosion.

From Bode plots (the EIS measurements; Figure 5), it can be seen that the N NiTi exhibited the highest impedance modulus at low frequencies, while the Rh NiTi exhibited the lowest impedance modulus. For uncoated and N NiTi, only one time constant can be observed corresponding to the protective oxide (nitride) surface layer. On the contrary, for Rh NiTi, two time constants appeared on the Bode plot, one probably related to the Rh/Au coating and the second corresponding to NiTi.

DISCUSSION

Analysis of data showed that various coatings affect mechanical properties of NiTi wires while preserving their working properties in unload; rhodium and nitride surface coatings tend to slightly increase the surface roughness of the NiTi wires, and nitride decreases while rhodium increases the susceptibility to corrosion in comparison with uncoated NiTi wires. The change in the corrosion rates of NiTi wires could affect their biocompatibility, despite having similar nominal chemical composition (NiTi).

The rhodium coating of as-received wires contained gold in amounts similar to previous findings,[9] but after corrosion tests, the chemical composition of the surface coating altered considerably (Figure 1; Table 1) because of uneven and nonhomogenous initial coating of noble metals. Previous study of composition and thickness of coating for N NiTi was not found.

Results from the three-point-bending test showed that coatings tend to increase E, making the wires stiffer. A small increase in E was observed for N NiTi and a higher increased was found for Rh NiTi when compared with uncoated NiTi.

Rh NiTi demonstrated the highest YS value in loading in comparison with uncoated and N NiTi, which is in accordance with higher stiffness values of Rh NiTi. Values for the unloading YS of all wires were rather uniform.

Table 2. Surface Roughness Parameter *Ra* and Electrochemical Parameters (Corrosion Current Density [j_{corr}], Corrosion Potential [E_{corr}], Breakdown Potential [E_{bd}], and Repassivation Potential [E_{rp}]; Mean [SD]) Determined From the Cyclic Polarization Curves for Uncoated (NiTi), Rhodium-Coated (Rh NiTi), and Nitrified (N NiTi) Nickel-Titanium Wires*

Wire Type	*Ra*, µm	j_{corr}, nAcm^{-2}	E_{kor}, mV$_{SCE}$	E_{bd}, mV$_{SCE}$	E_{rp}, mV$_{SCE}$
NiTi	0.169 (0.002)	31 (16)[a]	76 (20)[ab]	1318 (36)	1205 (12)
Rh NiTi	0.182 (0.007)	101 (40)[b]	162 (34)[b]	—	—
N NiTi	0.176 (0.009)	24 (20)[a]	−32 (65)[a]	1329 (82)	1193 (30)

* Different letters denote significant differences ($P < .05$) within each parameter and wire type.

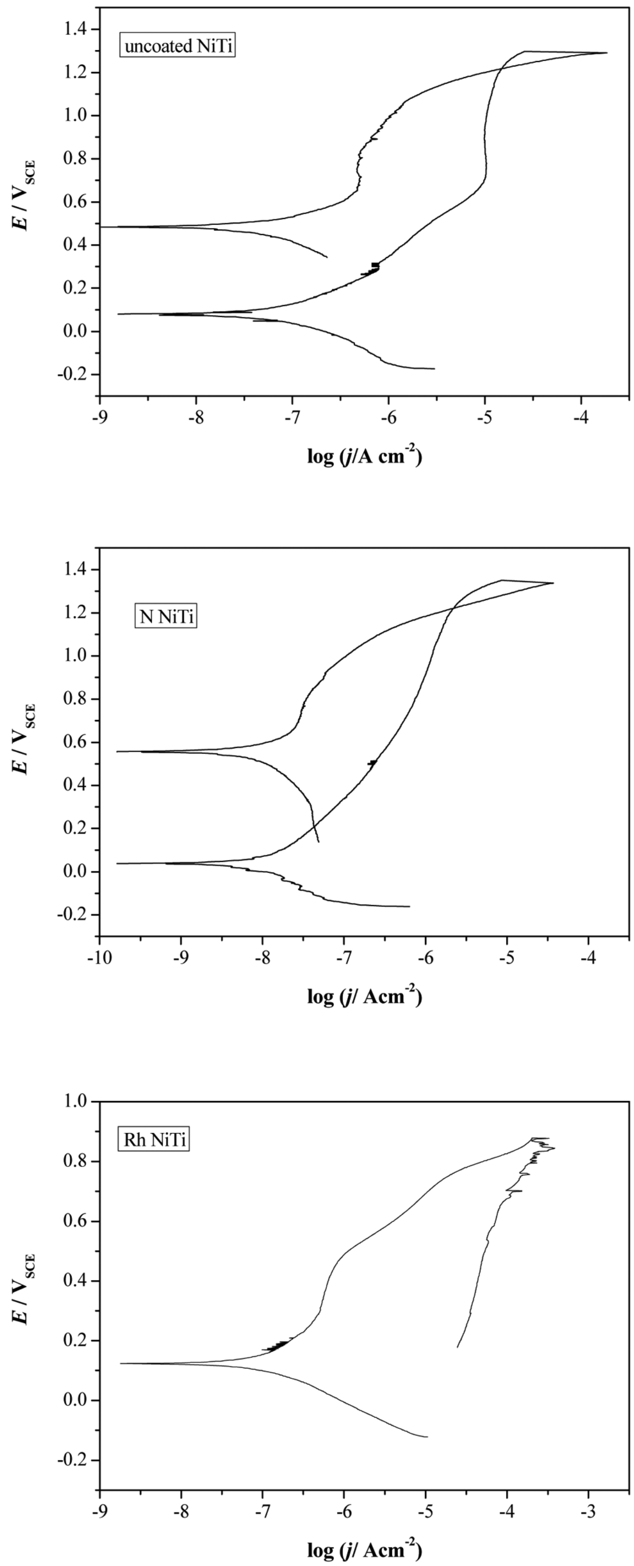

Figure 4. Cyclic polarization curves for nickel-titanium (NiTi) wires in artificial saliva at 37°C (uncoated [NiTi], nitrified [N NiTi], and rhodium-coated [Rh NiTi] wires).

The load-deflection curve presents phase transformation in crystal structure for wires with low-phase transformation temperatures, such as the types of Neo Sentalloy wires observed here.[26–28] Steep parts of curvature indicate stress transformation from the austenitic to martensitic phase, and the nearly horizontal part of the curve reverses the martensitic transformation toward the austenitic phase while generating a continuous useful force.[26] Analysis of forces during loading and unloading was used for comparison of the mechanical properties.[10] For Rh NiTi wires, we observed a significant increase in forces during loading, which was in accordance with earlier observed increased stiffness of Rh NiTi. N NiTi wires showed a significant increase in force in 2.5 mm of unloading curve, in the first steep part of the curvature during unloading. Other forces during curve showed no discrepancies among wires.

Unloading slope characteristics present the elastic characteristics of the wire during the period in which it moves the teeth. The proportion of useful constant spring-back action in total emitted force was rather uniform for all wires. Analysis of the unloading plateau showed that all wires had a similar average plateau force and length, as well as plateau bending action, with slightly higher values for Rh NiTi, thus having similar unloading working properties.

Although we hypothesized considerable change of surface roughness due to coating, a tendency toward only a slight increase was noted. A previous study of the effect of coatings[9] measured *Ra* for uncoated and Rh NiTi and showed data similar to ours for Rh NiTi but lower values for uncoated wires. Another study[16] measured the *Ra* of uncoated and N NiTi, and values for both were considerably higher. Rhodium coating showed the highest value in *Ra* among tested wires. Nowadays, there are researchers who suggest the atomic force microscopy (AFM) is the preferred noninvasive optical method for determination of surface roughness in nanoscale instead of the stylus profilometry, which we used.[29] However, our results were in concordance with those found in the AFM testing of surface roughness.[9]

Corrosion tests showed that Rh NiTi wires were susceptible to localized corrosion; their corrosion rate was higher than for the other two wire types. The appearance of two time constants in impedance diagrams also indicates that part of the base NiTi is exposed to artificial saliva because of the presence of the defects in coating, as explained in detail previously.[30]

Galvanic coupling of NiTi to noble metals or alloy leads to an increased corrosion rate of NiTi[31] because of differences in corrosion potential between noble materials gold and rhodium (representing the cathode site) and underlying nickel and titanium (representing

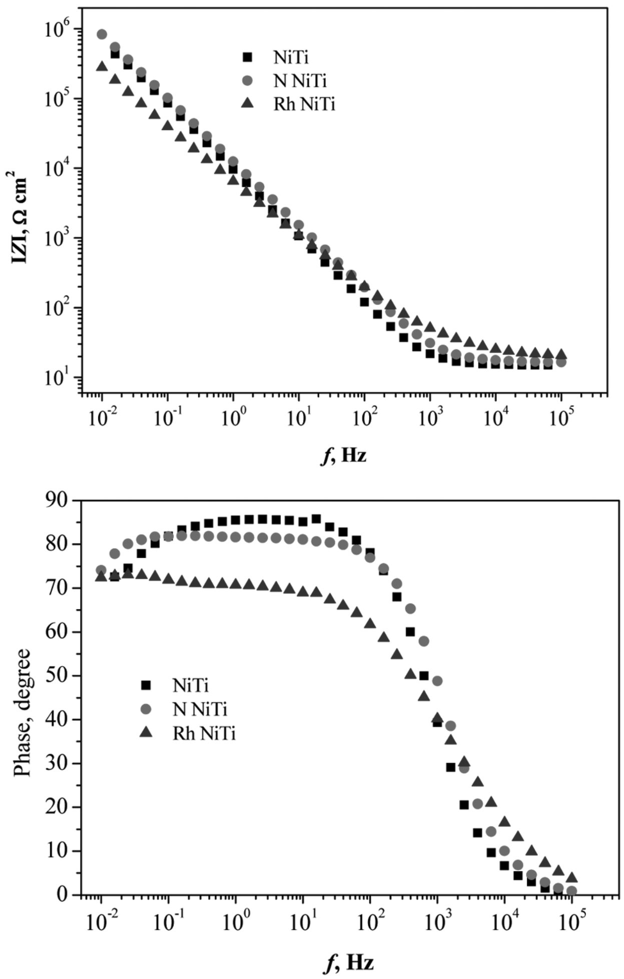

Figure 5. Bode plots for nickel-titanium (NiTi) wires in artificial saliva at 37°C (uncoated [NiTi], nitrified [N NiTi], and rhodium-coated [Rh NiTi] wires).

the anodic site). In our case, the anodic area (ie, bare alloy on spots where small pores and cracks occurred in the noble coating) was quite smaller than the cathode area, which may lead to a significant galvanic effect. In clinical use, friction on brackets could also cause damage to the coating. Metallic components (brackets, ligatures, metal rings, and fillings) could help galvanic corrosion within the oral cavity and loss of esthetic appearance.[12,13]

On the other hand, the TiN layer is known as an extremely hard ceramic material with low solubility and is often used for improvement of surface properties for various types of underlying materials. Its areas of application stretch from biomedical and bioelectronics to steelmaking and aerospace.[32]

Various research has found a variety of results for surface roughness parameters, and simple conclusions cannot be made regarding friction, because *Ra* depends on both wires' and brackets' design and material.[9,16,18,33] Also, previous research suggests that the type of material and the manufacturing process influence corrosion properties more than surface roughness parameters alone.[17,28]

Furthermore, additional investigation is necessary to clarify whether the above observed differences are the sole results of the coating or if they are influenced by the coating's manufacturing process.

Future research should also investigate allergenic potential in wires with various coatings, and their relation to friction on brackets should be investigated.

CONCLUSIONS

- The type of coating did not affect the working properties of the wires in unloading, but rhodium coating increases stiffness and produces higher forces during loading.
- Coatings slightly increase surface roughness, rhodium coating more than nitrification.
- Nitrification of wire surface improves corrosion resistance.
- Rhodium coating results in the lowest corrosion resistance and pronounced susceptibility to pitting corrosion because of galvanic coupling between the noble coating and the base alloy.

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