Original Article

Mechanical Properties and Surface Characterization of Beta Titanium and Stainless Steel Orthodontic Wire Following Topical Fluoride Treatment

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ABSTRACT

Objective: To study the effect of fluoride prophylactic agents on the loading and unloading mechanical properties and surface quality of beta titanium and stainless steel orthodontic wires.

Materials and Methods: Rectangular beta titanium and stainless steel wires were immersed in either an acidulated fluoride agent, a neutral fluoride agent, or distilled water (control) for 1.5 hours at 37°C. After immersion, the loading and unloading elastic modulus and yield strength of the wires were measured using a 3-point bend test in a water bath at 37°C. A one-way analysis of variance and Dunnett's post hoc, $\alpha = .05$, were used to analyze the mechanical testing data. Scanning electron microscopy was also used to qualitatively evaluate the wire topography as a function of the fluoride treatments.

Results: Unloading mechanical properties of beta titanium and stainless steel wires were significantly decreased ($P \leq .05$) after exposure to both fluoride agents. Corrosive changes in surface topography were also observed after exposure to both the neutral and the acidulated phosphate fluoride agents.

Conclusions: The results suggest that using topical fluoride agents with beta titanium and stainless steel wire could decrease the functional unloading mechanical properties of the wires and potentially contribute to prolonged orthodontic treatment.

KEY WORDS: Fluoride prophylactic agents; Corrosion

INTRODUCTION

A comprehensive orthodontic treatment is usually divided into three phases: (1) leveling and aligning, (2) space closure and anterior/posterior correction, and (3) detailing and finishing.[1] A variety of alloy wires are used to generate the biomechanical forces associated with tooth movement. Once the wire is activated or bent, it is the unloading or deactivating forces that produce orthodontic tooth movement.[2] With current orthodontic treatment, superelastic nickel-titanium wire is often used for phase 1, with beta titanium and stainless steel (SS) wires most frequently used for phases 2 and 3.[2,3] As a result, beta titanium and SS wires tend to be reused more often, leaving them exposed to the aqueous oral environment for a longer period of time. It is not uncommon during finishing to have the same wire in the mouth for up to 12 months.

Compromised oral hygiene, a frequent complication with orthodontic treatment, can lead to enamel demineralization and decay.[4,5] To address this potential treatment complication, orthodontists commonly prescribe a daily topical fluoride.[6] Although both beta titanium and SS alloys form corrosion-resistant passivation layers,[7–9] these protective oxide layers can be chemically disrupted, leading to corrosion susceptibility.[10,11] In addition to corrosion in artificial saliva and

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chloride media,[7,10–13] it has been recently reported that both beta titanium and SS have exhibited corrosion in the presence of experimental fluoride-based solutions.[9,12,14–18] In addition to corrosive surface changes, it has also been reported that experimental fluoride solutions degrade the tensile strength and microhardness of beta titanium and SS archwires.[14–16] Therefore, it is plausible that commercially available topical fluoride prophylactic agents may cause a similar corrosive interaction and associated mechanical property degradation of beta titanium and SS wires.

To date, the effects of prophylactic fluoride agents on the functional unloading mechanical properties of beta titanium and SS orthodontic wires have not been reported. Therefore, the purpose of this study was to evaluate the effects of prophylactic fluoride agents on the loading and unloading mechanical properties of beta titanium and SS orthodontic wire and also to characterize their effects on the topography of the wire surface.

MATERIALS AND METHODS

Preformed 0.019 × 0.025 inch beta III titanium (beta-Ti) archwires (77.55% titanium [Ti], 11.5% molybdenum, 6% zirconium, 4.5% tin, 0.35% iron [Fe], and 0.1% carbon [C]; lot G3105, Ref 4301-328, 3M Unitek, St Paul, Minn) and rectangular 0.019 × 0.025 inch SS wires, (68.85% Fe, 19% chromium [Cr], 9% nickel, <1% silicon, <2% manganese, <0.08% C, < 0.03% sulfur, and <0.045% phosphorus; lot 416, Ref 251-925, 3M Unitek). The chemical composition of the wires was provided by the manufacturers.

The topical fluoride agents were high fluoride-ion concentration gels, Phos-flur gel (1.1% acidulated phosphate fluoride [APF], 0.5% w/v fluoride, pH = 5.1; lot 408030, Colgate Pharmaceuticals, Canton, Mass) and Prevident 5000 (1.1% sodium fluoride neutral agent [NaF], 0.5% w/v fluoride, pH = 7; lot 409021, Colgate Pharmaceuticals). These fluorides were chosen because of widespread use, identical method of application and fluoride ion concentration, and differences in pH.

Specimen Preparation

The wires were cut into 25-mm-long specimens; with beta-Ti wires, specimens were cut from the straight ends of the archwire. Wire specimens were incubated at 37°C in individual 20-ml plastic vials (Fisher Scientific, Hanover Park, Ill) with 5 mL of one of the fluoride solutions or distilled, deionized H_2O (dH_2O), control, for 1.5 hours, approximating 3 months of 1-minute daily topical fluoride treatments. Specimens were then removed from their respective solutions and rinsed with dH_2O and placed into a new, clean, and individually-labeled vial that was submerged in 37°C water prior to wire mechanical testing. Based on preliminary data and a power analysis, 15 wire specimens were determined to be adequate for each experimental condition (three conditions × 15 wires of each type) in order to meet the constraints of $\alpha = .05$ and power = .80.

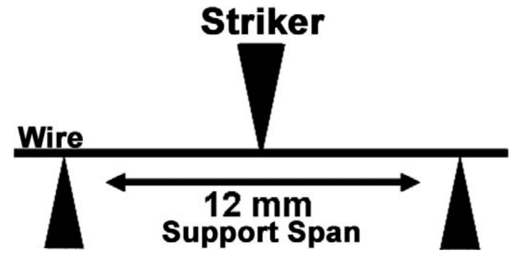

Figure 1. Diagram of three-point bend fixture. Radii of each support and the striker are 0.05–0.13 mm. (Adapted from ANS/ADA specification 32.[19])

Mechanical Testing

Mechanical testing was based on the current American National Standard/American Dental Association Specification No. 32 for orthodontic wires.[19] Randomly selected specimens were tested using the three-point bending test on a universal testing machine (Model 1125/5500, Instron Corp, Canton, Mass). By the use of a three-point bend test, the wires are subjected to flexural loading, which provides a collective measure of both compressive stress on the upper surface of the specimen and tensile stress on the lower surface. Currently, flexure tests are preferred for many dental materials such as orthodontic wires, cements, and dental composites, because the stress distribution more closely simulates what occurs under clinical function.[20] The configuration of the 3-point fixture (Figure 1) was a support span of 12 mm with radii of each support and the striker at 0.05–0.13 mm. Each specimen was loaded to a deflection of 3.1 mm, and then unloaded to zero deflection at a cross-head speed of 1 mm/min. In order to more closely relate to the clinical situation, the specimens were tested in a dH_2O bath at 37°C ± 1°C to simulate the aqueous oral environment. Load in newtons and deflection in millimeters for both loading and unloading was recorded for each specimen with the Merlin software program (v 5.43, Instron Corp).

Based upon the load-deflection curve and the dimensions of the specimen, the flexural stress as a function of flexural strain was determined for each specimen. The flexural stress, σf, was calculated as follows: $\sigma_f = 3PL/2bd^2$, where P is the load at a given point on the load-deflection curve, L is the support span, b is the width of the beam, and d is the depth

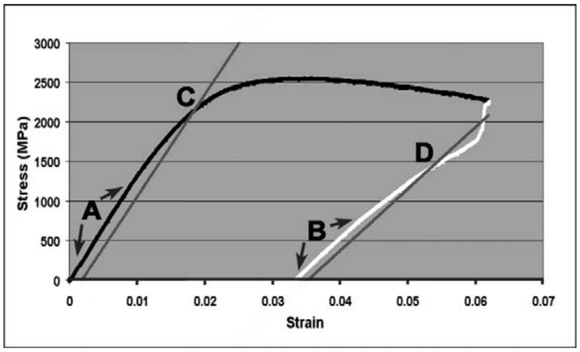

Figure 2. Representative stress-strain curve; loading segment is black and unloading segment is white. Loading and unloading modulus of elasticity are determined from the slope of linear segments, indicated by A and B, respectively. Loading and unloading yield strength are determined at 0.2% offset, denoted by the intersect point with lines C and D, respectively.

of the beam. The flexural strain, ϵf, was calculated as follows: $\epsilon_f = 6Dd/L^2$, where D is the deflection at the center of the beam at a given point on the load-deflection curve, d is the depth of the beam, and L is the support span. The stress and corresponding strain were then plotted to produce a stress-strain curve for each specimen with a representative curve presented in Figure 2. It should be noted that all specimens produced stress-strain curves with the unloading curve deviating from the loading curve, the result of mechanical hysteresis. Mechanical hysteresis is exhibited by both superelastic alloy wires, such as nickel titanium (NiTi), and nonsuperelastic wires, such as SS and beta-Ti, when the alloys are tested beyond the elastic range.[21,22] Because of these differences in loading and unloading properties and the fact that unloading properties are responsible for tooth movement,[2] the most recent specification for orthodontic wires[19] indicates that unloading mechanical testing data shall be reported.

Using engineering beam theory,[23] loading and unloading flexural modulus and yield strength were calculated based on the generated stress-strain curves. The resulting stress-strain curves consist of two linear sections connected by a curvilinear section. The two linear sections correspond to the elastic deformation that occurs during loading and unloading. The loading and unloading flexural modulus of elasticity (E) of each specimen was calculated from the slope of the two linear portions of the generated stress-strain curves (Figure 2, slope of segments A and B, respectively).

Yield strength (YS) at 0.2% strain offset was also determined from each loading and unloading portion of the curve. This was accomplished by using a strain offset of 0.2% to construct a linear offset stress-strain curve parallel to the linear portion of the actual stress-strain curve for both the loading and the unloading segments. The intersection of the linear offset stress-strain curve with the actual loading and unloading segments of the stress-strain curve is the loading and unloading YS (Figure 2, intersects at C and D, respectively). Note that a strain offset of 0.2% is typically used for metals that exhibit a gradual elastic-plastic transition.[24]

The mechanical property data were analyzed by a one-way analysis of variance, $\alpha = .05$, for both the loading and unloading E and YS for each wire type. If there was significant difference between groups, a Dunnett's post hoc test was used to determine which groups were significantly different from the dH_2O control.

Wire Surface Characterization

Following mechanical testing, three representative specimens from each wire/experimental condition group were examined with scanning electron microscopy (SEM) analysis (Philips XL30 field emission SEM, Phillips Electron Optics, Hillsboro, Ore) at 15.0 kV to qualitatively characterize the topography of the wire surface. This qualitative analysis was included to determine whether there was a potential link between the fluoride agent's effect on the wire surface topography and any mechanical property degradation.

RESULTS

The mechanical testing results are presented in Table 1. The statistical analysis indicated no significant effect ($P > .05$) of fluoride treatment on loading E or YS of either wire. In contrast, the fluoride treatments appeared to produce a significant effect ($P \leq .05$) on the unloading E and YS of both wires. A one-sided Dunnett's post hoc indicated that both the Phos-flur and Prevident fluoride gels significantly ($P \leq .05$) decreased the unloading E and unloading YS as compared to the dH_2O control.

Representative SEM images of beta-Ti wires exposed to dH_2O, Prevident, and Phos-flur gel are shown in Figure 3. As compared to the dH_2O control wire (Figure 3A), following exposure to both Prevident and Phos-flur gel, the overall beta-Ti wire surface appeared rougher and the cracks along the wrought structure were more prevalent and more accentuated/deeper, indicating some surface corrosive effects (Figure 3B,C).

Representative SEM images of SS wires exposed to dH_2O, Prevident, and Phos-flur gel are shown in Figure 4. There was also an apparent difference in surface topography between wires exposed to dH_2O (control) and the fluoride agents. With the SS control wire (Figure 4A), the vertical wrought structure and a semicircular pattern were minimally apparent. Follow-

Table 1. Mean Values and Standard Deviations of the Mechanical Properties of Stainless Steel and Beta Titanium Wires Following Exposure to dH_2O, Phos-Flur Gel, or Prevident

Wire	Treatment (n = 15)	Loading Elastic Modulus (GPa)	Unloading Elastic Modulus (GPa)	Loading Yield Strength (MPa)	Unloading Yield Strength (MPa)
Stainless steel	dH_2O (control)	132.1 ± 2.5	77.5 ± 3.2	2145 ± 41	1458 ± 100
	Phos-flur gel	132.1 ± 4.4	70.7 ± 2.7*	2176 ± 109	1299 ± 67*
	Prevident	133.4 ± 3.2	70.0 ± 2.8*	2196 ± 70	1345 ± 111*
Beta titanium	dH^2O (control)	66.7 ± 2.9	42.0 ± 3.3	1440 ± 75	615 ± 37
	Phos-flur gel	66.8 ± 1.0	39.2 ± 0.9*	1472 ± 29	572 ± 19*
	Prevident	66.3 ± 0.9	39.0 ± 1.2*	1444 ± 43	582 ± 27*

* Significantly different from control, $P \leq .05$.

ing Prevident or Phos-flur gel treatment (Figure 4B,C, respectively), the linear wrought structure and the semicircular pattern were more pronounced, suggesting a loss of surface material.

DISCUSSION

The corrosion resistance of beta-Ti and SS wires depends on the formation of a passivation layer, an oxide film.[10,16,25] Titanium-based alloys form a passive film of primarily titanium oxides, with TiO_2 being the most prevalent.[9,15] Although the SS passivation layer is very complex,[26] the protective character is due primarily to chromium oxide, Cr_2O_3.[8,27] Surface passivation prevents further oxygen diffusion, resulting in corrosion resistance[9]; however, if the passivation layer is disrupted, the wires become susceptible to corrosion.[10]

Both Prevident and Phos-flur gel produced qualitative surface topography changes on beta-Ti and SS wires. Following fluoride exposure, beta-Ti wire exhibited an overall rougher surface, and the cracks along the wrought structure were deeper and more accentuated (Figure 3B,C). With SS wires, the linear wrought structure and an associated semicircular pattern were more apparent following fluoride treatment (Figure 4B,C). These results correspond with other research in which surface morphology of beta-Ti wire was also affected by an APF solution,[14,15,17] a commercially available APF agent,[9] and a neutral fluoride solution.[15] Although Watanabe and Watanabe[9] did not report beta-Ti surface changes with a commercially available neutral fluoride agent, the product used in that study had a lower fluoride concentration than the NaF product used in this study. Results similar to the current SS wire surface changes have also been reported following APF exposure[14]; however, to date, there are no previous investigations using SS wire with neutral fluoride. Collectively, the current investigation's surface topography changes following fluoride exposure suggest that the protective oxide layer has been disrupted, leading to possible corrosive surface changes. However, to confirm the presence of fluoride-related corrosion products, future studies would need to be done using technology such as x-ray diffraction or spectroscopy.

In this investigation, there was also a statistically significant decrease in the unloading mechanical properties of both beta-Ti and SS wires following exposure to both the APF and the neutral fluoride agent. These results are in agreement with a reported decrease in unloading mechanical properties of NiTi wire following exposure to the same topical fluoride agents.[28] Potentially, the mechanical property degradation can be linked to the demonstrated qualitative surface topography changes that might be the result of fluoride-related disruption of the passive layer.

The dissolution of the passive layer of titanium-based orthodontic wires exposed to APF solutions could potentially be explained by previous reports that hydrofluoric acid (HF), produced according to equation 1, could dissolve the protective oxide layer on the surface of titanium and its alloys according to equations 2 and 3.[17,29,30]

$$H_3PO_4 + 3NaF \rightarrow Na_3PO_4 + 3HF \quad (1)$$

$$TiO_2 + 4HF \rightarrow TiF_4 + 2H_2O \quad (2)$$

$$TiO_2 + 2HF \rightarrow TiOF_2 + H_2O \quad (3)$$

The beta-Ti mechanical property changes in this study may be related to hydrogen absorption that has been reported to occur once the protective layer is degraded.[14] Hydrogen absorption, ie, hydrogen embrittlement, of beta-Ti treated in APF solutions has been linked to decreased tensile strength and increased brittle fracture.[14–17]

As already indicated, SS orthodontic wires have been previously shown to be susceptible to corrosion in experimental fluoride solutions.[14] Just as with tita-

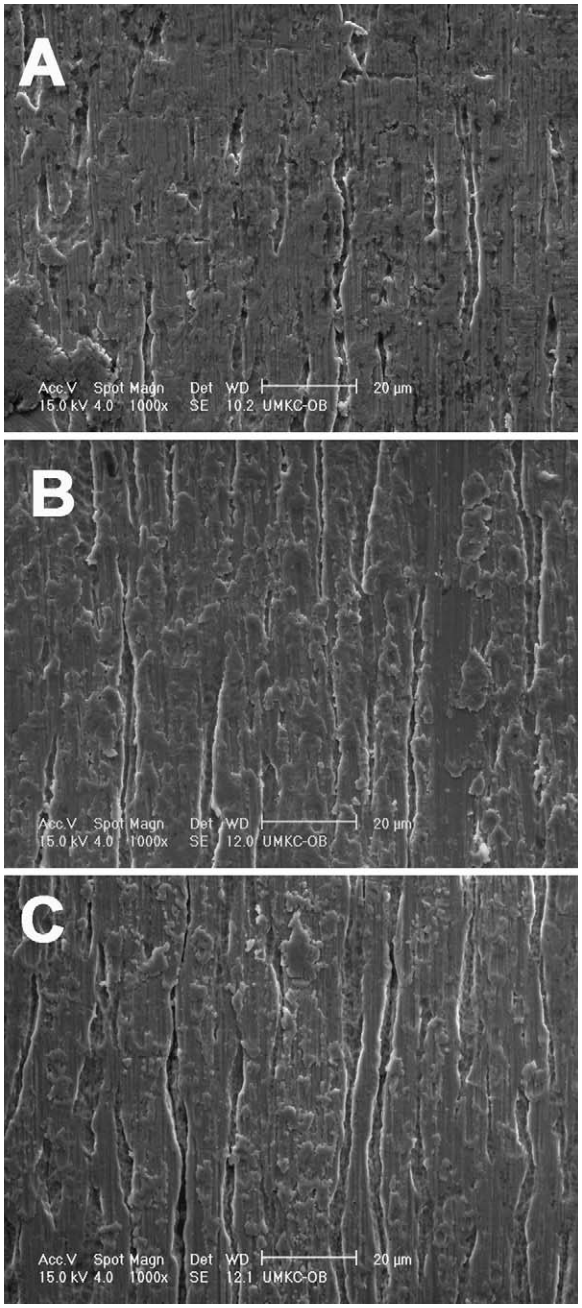

Figure 3. Representative SEM images of beta titanium wires. (A) Exposed to dH_2O (control); (B) Prevident-treated; (C) Phos-flur gel–treated (1000× magnification).

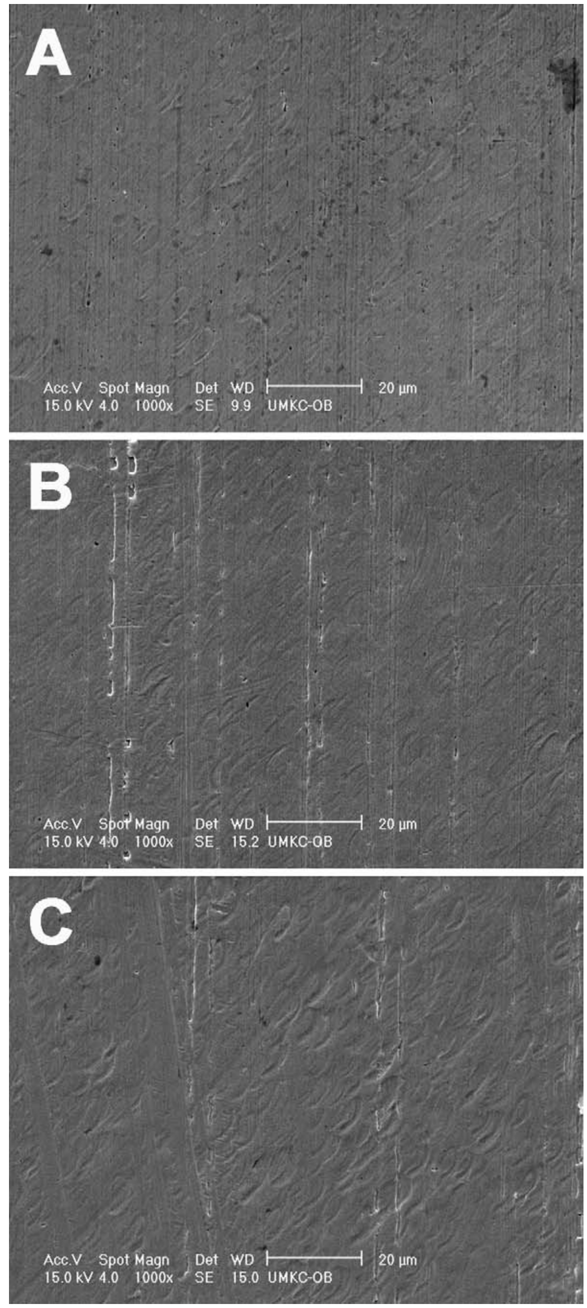

Figure 4. Representative SEM images of stainless steel wires. (A) Exposed to dH_2O (control); (B) Prevident-treated; (C) Phos-flur gel–treated (1000× magnification).

nium-based alloys, SS may interact with the HF acid that could be produced in the presence of acidulated fluoride (equation 1). However, because of wire compositional differences, the HF-SS alloy interaction would not be identical to the titanium-based alloy. Instead, potential degradation reactions with the SS alloy passive layer component might occur according to the following equation: $Cr_2O_3 + 6HF \rightarrow 2CrF_3 + 3H_2O$. Similar to titanium-based alloys, once passive layer degradation occurs, SS has a propensity for hydrogen absorption leading to embrittlement and stress corrosion cracking.[31,32] Stress corrosion cracking of SS in the presence of fluoride has also been reported,[33,34] with crack growth accompanied by anodic dissolution and cathodic hydrogen generation at the crack tip.[14] An associated decrease in SS tensile strength following exposure to an acidulated fluoride solution was also reported.[14]

In the current study, in addition to beta-Ti and SS unloading mechanical property degradation after APF exposure, comparable mechanical property degradation followed exposure to neutral fluoride. Although the acidic pH of fluoride agents is considered an important factor in the breakdown of the titanium-based alloy protective oxide layers leading to fluoride-related corrosion and hydrogen embrittlement,[30,35,36] corrosion resistance reduction and mechanical property degradation has also been reported with titanium-based alloys at a neutral pH when fluoride concentrations are 0.5% or greater.[28,37] Thus, in addition to pH, the fluoride concentration could be an important factor in the breakdown of the alloy protective oxide layers, leading to potential hydrogen absorption and associated mechanical property changes. Accordingly, fluoride ion concentration would appear to be related to the beta-Ti and SS wire mechanical property degradation associated with the neutral fluoride agent in this study.

Except for the current study and one previous NiTi investigation[28] also reporting a decrease in unloading properties following fluoride exposure, other previous fluoride/wire mechanical property investigations[14–17] have not examined loading and the more clinically relevant unloading properties. Instead, previous investigations have examined properties such as tensile strength at fracture. However, just as the decrease in wire tensile strength has been linked to hydrogen absorption following fluoride treatment,[14–17] trapped interstitial hydrogen might be associated with the decrease in the unloading mechanical properties of both beta-Ti and SS wire after exposure to APF and NaF topical fluoride agents. Why only the unloading properties would be affected might be related to absorbed hydrogen molecules not causing any evident effect within the lattice until the wire has been loaded beyond the elastic range, when lattice dislocations and slip occur. Although this has not been studied in orthodontic wire, it has been reported that absorbed hydrogen can be trapped within the SS lattice vacancies, potentially affecting both plastic flow and recovery.[32] A similar situation might occur with hydrogen absorbed into the beta-Ti lattice structure. This explanation could help account for why only changes in unloading mechanical properties would be detected following fluoride exposure and potential associated hydrogen absorption. However, in order to confirm that phenomenon, future studies using hydrogen thermal analysis and x-ray diffractometry would be necessary.

As with any in vitro investigation, the protocol cannot exactly simulate clinical conditions. The 1.5-hour fluoride exposure in this study attempted to simulate 3 months' accumulation of 1-minute daily topical fluoride applications. In the clinical application, fluoride exposure would be repeated, shorter exposures with dilution of the fluoride with saliva. Therefore, a future study could address the effect of cumulative, shorter treatments and perhaps use the fluoride agents mixed in some measured ratio with an artificial saliva agent.

Even though the reported decrease in beta-Ti and SS unloading mechanical properties may not seem large enough to be clinically significant, in spite of the limitations of replicating in vivo fluoride exposure, actual in vivo wire fluoride exposure could potentially be greater than that of the 3-month simulation. For instance, wires may remain in the oral cavity for 6 months or more while exposed to topical fluoride, fluoridated water and toothpaste, and fluoride-releasing bracket-bonding materials.[38] Despite dilution with saliva, exposure time per 1-minute topical fluoride treatment may also be greater, because instructions indicate to avoid rinsing for at least 30 minutes after the application, in addition to recommending that the product be used before bedtime. This could be important because it has been previously reported that there is a linear increase in hydrogen absorption and potential alloy mechanical property degradation with increased fluoride exposure time.[17]

CONCLUSIONS

- Both beta-Ti and SS orthodontic wires exhibited qualitative surface topography changes following exposure to neutral or acidulated phosphate fluoride agents.
- After exposure to either neutral or acidulated prophylactic fluoride gels, beta-Ti and SS wire showed a statistically significant decrease in unloading mechanical properties. Because unloading forces produce tooth movement, this decrease may be clinically relevant.
- Using topical fluorides with SS or beta titanium orthodontic wire might be a factor in prolonged orthodontic treatment time.

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