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Surface corrosion and fracture resistance of two nickel–titanium-based archwires induced by fluoride, pH, and thermocycling. An *in vitro* comparative study

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SUMMARY The present comparative study aimed to evaluate the surface corrosion and fracture resistance of two commercially available nickel–titanium (NiTi)-based archwires, as induced by a combination of fluoride, pH, and thermocycling.

One hundred and ten rectangular section NiTi-based archwires were used, 55 of each of the following: thermally activated Thermaloy® and super-elastic NeoSentalloy® 100 g. Each of these was divided into five equal subgroups. One of these five subgroups did not undergo any treatment and served as the control, while the other four were subjected to 30 days of incubation at 37°C under fluoridated artificial saliva (FS) at 1500 ppm fluoride treatment alone (two subgroups) or combined with a session of thermocycling (FS + Th) treatment at the end of incubation (two subgroups). Within each of the Thermaloy® and NeoSentalloy® groups, the FS and FS + Th treatments were performed under two different pH conditions: 5.5 and 3.5 (each with one subgroup per treatment). Analysis of the surface topography and tensile properties by means of scanning electron microscopy (a single sample per subgroup), atomic force microscopy, and a universal testing machine for ultimate tensile strength were carried out once in each of the control subgroups or immediately after the treatments in the other subgroups for 10 of the archwires. Non-parametric tests were used in the data analysis.

Significant effects in terms of surface corrosion, but not fracture resistance, were seen mainly for the Thermaloy® group at the lowest pH, with no effects of Th irrespective of the group or pH condition.

Different NiTi-based archwires can have different corrosion resistance, even though the effects of surface corrosion and fracture resistance appear not to be significant in clinical situations, especially considering that thermocycling had no effect on these parameters.

Introduction

To obtain optimal orthodontic tooth movement, fine control of the forces exerted by orthodontic appliances is required (Krishnan and Davidovitch, 2006). In particular, a continuous and constant force produces more efficient orthodontic tooth movement compared with a discontinuous force (van Leeuwen *et al.*, 1999). With the aim of obtaining such constant forces, the use of nickel–titanium (NiTi) alloys was introduced in orthodontics (Andreasen and Hilleman, 1971). Over the last decades, the use of NiTi-based orthodontic devices, primarily as archwires, has seen a further increase, and they are likely to become even more important in the search for the most efficient orthodontic tooth movement.

However, a side-effect of NiTi-based alloys is surface corrosion in the oral cavity, which is of importance because of the potential toxic effects of Ni (Gursoy *et al.*, 2007; Kao *et al.*, 2007). The corrosion resistance of these alloys is primarily based on their surface titanium oxide (TiO_2) layer, the formation of which is referred to as passivation (Eliades and Bourauel, 2005; Clarke *et al.*, 2006). However, this oxide layer can be destroyed by fluoride ions (Huang, 2007) and this aspect renders dental prophylaxis procedures based on fluoridated agents as possible causes of toxic ion release from NiTi-based orthodontic devices into the oral cavity.

The surface roughness of commercial NiTi-based archwires with similar surface chemical structures has been reported not to correspond with differences in surface corrosion resistance (Huang, 2005). In combination with the variability in surface topography that has also been reported among NiTi-based archwires from different manufacturers (Schiff *et al.*, 2002; Fischer-Brandies *et al.*, 2003; Huang, 2005), this concept makes investigations into corrosion resistance and other mechanical features of such archwires of importance. Indeed, the dissimilarity in corrosion resistance of such archwires has only been partially investigated, especially when considering the wide variability among the study designs used to resemble the *in vivo* situation, in terms of pH conditions and fluoride concentrations. As a consequence, previous *in vitro* and *in vivo* studies have reported contrasting results in terms of corrosion of NiTi-based archwires following fluoride treatment, with significant (Schiff *et al.*, 2006; Ramalingam *et al.*, 2008), non-significant (Petoumeno *et al.*, 2008), and variable (Ahn *et al.*, 2006; Huang, 2007) effects seen.

A further issue is the effect of temperature on NiTi-based archwires, particularly as these alloys have various temperature-dependent phases. Previous studies have reported that the corrosion rate of various NiTi-based archwires increases with temperature (Pun and Berzins, 2008), and intraoral temperature variations have been implicated as the cause of mechanical deterioration of NiTi-based archwires (Eliades and Bourauel, 2005). However, the effects of cycles of different temperatures on surface corrosion and fracture resistance of NiTi-based archwires, as opposed to a constant temperature, have not been determined. This particularly applies to consideration of the critical role the mechanical properties of these archwires have in clinical practice.

Therefore, the present comparative study aimed to evaluate the surface corrosion and fracture resistance of two commercially available NiTi-based archwires, as induced by a combination of fluoride, pH, and thermocycling.

Materials and methods

Samples and study design

One hundred and ten NiTi-based rectangular section archwires were used, 55 of each of the following: thermally activated Thermaloy® (code AO7304; Rocky Mountain Orthodontics, Denver, Colorado, USA) and super-elastic NeoSentalloy® 100 g (code 02-522-052; GAC International, Central Islip, New York, USA). According to the manufacturers' indications, the wire dimensions are 0.016 × 0.022 inch and 0.018 × 0.025 inch, respectively. Although not reported for the Thermaloy® archwires, the chemical composition of the NeoSentalloy® archwires is given as 52.9/47.1 (Ni/Ti).

The study design is illustrated in Figure 1. Each of the Thermaloy® and NeoSentalloy® archwires, from different batches, were randomly assigned to five equal subgroups of 11 archwires. Each wire was then cut at one of the terminal portions into a 20 mm length. One of these five subgroups was left untreated and served as the control (as received), while the other four subgroups were subjected to 30 days of incubation under fluoridated artificial saliva (FS) treatment alone (two subgroups) or in combination with a session of thermocycling (FS + Th) at the end of incubation (two subgroups). Within each of the Thermaloy® and NeoSentalloy® groups, the FS and FS + Th treatments were performed under two different pH conditions: 5.5 and 3.5 [each as one subgroup per treatment (i.e. FS and FS + Th)]. For 10 of the archwire samples within each subgroup, analyses of the surface topography and tensile properties were carried out, once as the samples were received and once after treatment by means of atomic force microscopy (AFM) and a universal testing machine for ultimate tensile strength (UTS), respectively. Moreover, the remaining single archwire sample per subgroup was used for scanning electron microscopy (SEM) analysis of the surfaces, again as received and after each treatment.

Incubation and thermocycling treatments

Single samples of the archwires were placed into polypropylene vessels containing 60 ml of FS for both the FS incubation and Th, where performed. The artificial saliva contained: $CaCl_2$ (0.078 g/l), $MgCl_2$ (0.041 g/l), KH_2PO_4 (0.544 g/l), NaN_3 (0.020 g/l), 4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid (4.766 g/l), and KCl (2.236 g/l; Pashley *et al.*, 2004), with an incubation temperature of 37°C throughout the 30 day period. The fluoride concentration used was 1500 ppm and was obtained by adding sodium fluoride (NaF) to the artificial saliva. The different pHs of 5.5 and 3.5 were obtained by adding sodium

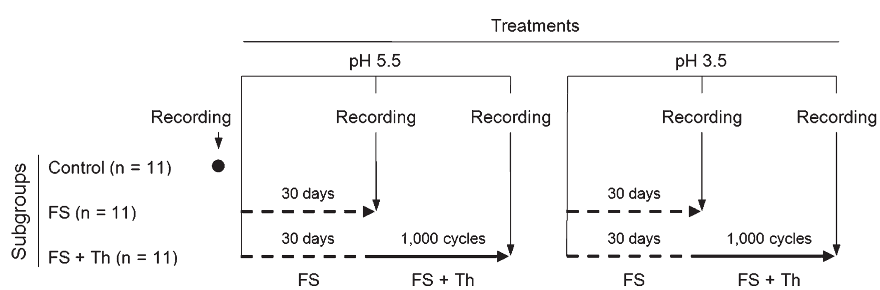

Figure 1 The study design. FS, incubation in fluoridated artificial saliva; FS + Th, incubation in fluoridated artificial saliva followed by thermocycling. The same study design included five subgroups within each of the Thermaloy® and NeoSentalloy® archwire groups: control, FS (pH 5.5), FS + Th (pH 5.5), FS (pH 3.5), FS + Th (pH 3.5). Recording was performed once in each subgroup and consisted of atomic force microscopy and ultimate tensile strength for 10 archwire samples per subgroup and scanning electron microscopy for the remaining archwire sample per subgroup.

hydroxide or lactic acid and measured using a pH metre (MM-41; Crison Instruments SA, Alella, Spain).

The thermocycling treatment included a single session performed after incubations under FS and included 1000 temperature cycles from 5°C to 55°C (Thermo Haake Willytech; SD Mechatronik Feldkirchen-Westerham, Germany), with the archwire samples cyclically immersed in each of the thermal baths for 30 seconds, with 2 seconds at air temperature between immersions (Vidoni *et al.*, 2010). This thermocycling was designed to reproduce the temperature variations that can occur in the mouth when consuming cold and hot food and beverages.

Scanning electron microscopy

The archwires for SEM analysis were briefly cleaned in an ultrasonic bath with 96 per cent ethanol and then dried and mounted on aluminium stubs. After gold coating (Emitech K 550; Emitech Ltd, Ashford, Kent, UK), they were analysed using a Leo 435 VP SEM (Leo Electron Microscopy Ltd, Cambridge, UK).

AFM and data processing

A commercial AFM (Perception; Assing, Rome, Italy) was used to perform surface analysis of the archwire samples. The scanning areas used in the recordings were 20 × 20 m (300 points × 300 points resolution) to a total theoretical area (i.e. in the absence of roughness) of 400 m^2. The specimen was fixed to a piezo scanner with three translatory degrees of freedom. A tapping silicon cantilever with a nominal tip radius of less than 10 nm was used. Subsequently, Gwyddion software, version 2.15 (http://gwyddion.net/) was used in the AFM data post-processing and analysis. In particular, the raw data were subjected to the following procedures before analysis: (1) levelling, using the 'facet level' function; (2) background subtraction, using the 'polynomial background' function set as a second-order curve; and (3) filtering, using the 'Gaussian' smoothing filter set at 2 pixels. The three-dimensional (3D) reconstructions were thus rendered and the corresponding data extrapolated. Data analysis included the following surface parameters: maximum height of the profile (Ry), average of the roughness profile (Ra), skewness of the roughness profile (Rsk; positive when the surface was mainly a plateau; and peaks and negative when it was mainly a plateau and valleys), and area of surface development (Sa; Bourauel *et al.*, 1998; Huang, 2007).

Tensile testing

A Galdabini SUN500 universal testing machine [Galdabini SpA, Cardano al Campo (VA), Italy] was used to measure UTS, expressed in Newton, produced by the archwire samples according to the distances that the crossheads travelled (strength rates) and the loads generated (tensile forces), which were automatically recorded and plotted as XY scatter plots. After each archwire sample had been mounted on the crossheads of the universal testing machine using a pair of hooks, they were extended at a rate of 1 mm/minute. The different archwire sections were also taken into account by normalizing the UTS to the corresponding area of the archwire sections (Newtons per square millimetre).

Data analysis

The Statistical Package for Social Sciences release 13.0 (SPSS Inc., Chicago, Illinois, USA) was used for data analysis. The archwire sample represented the statistical unit. After testing the normality of the data with the Shapiro–Wilk test and Q–Q normality plots and the equality of variance among the datasets using a Levene test, non-parametric methods were chosen for data treatment. Nevertheless, the mean and standard deviations are reported for descriptive purposes, with the exception of the Rsk, which is reported as the median and 25th and 75th percentiles.

A Kruskal–Wallis test was used to assess the significance of the differences for every surface and tensile parameter among the treatments (including the control subgroup), within each of the pH conditions. When significant interactions were seen, a Bonferroni-corrected Mann–Whitney *U*-test was used for pairwise comparisons between the treatments (including the control subgroup) within each of the Thermaloy® and NeoSentalloy® groups and pH conditions. The same Mann–Whitney *U*-test was used to assess the significance of the differences in every parameter between all of the corresponding Thermaloy® and NeoSentalloy® subgroups. Moreover, to further analyse the changes in the surface parameters, normalized changes seen under FS and FS + Th treatments were calculated. In particular, the corresponding mean values of the control datasets were used to normalize every value of the datasets under the pH conditions and treatments. Therefore, the significance of the differences under these normalized changes between the two treatments within each of the Thermaloy® and NeoSentalloy® groups and pH conditions, and between the groups within each of the treatment and pH conditions, was also assessed using the Mann–Whitney *U*-test. A P value of less than 0.05 was considered statistically significant.

Results

Representative stereo micrographs for all the subgroups are shown in Figure 2. Under the initial conditions (control archwires, as received), characteristic surface textures were evident for each of the two types of archwires, with a greater surface roughness apparent for the NeoSentalloy® archwire as compared with the Thermaloy® archwire.

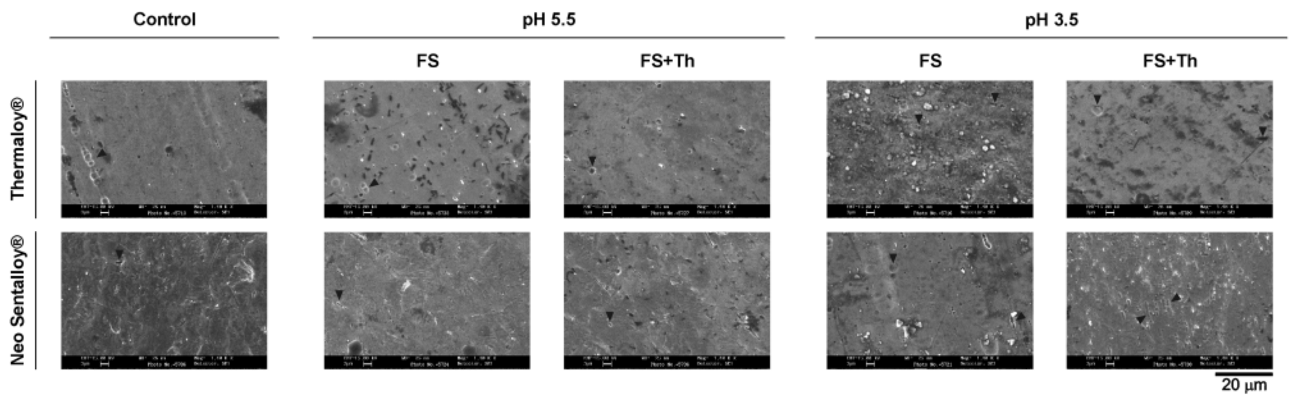

Figure 2 Representative scanning electron microscopy of the experimental archwire groups in relation to pH conditions and treatments. FS, incubation in fluoridated artificial saliva; FS + Th, incubation in fluoridated artificial saliva followed by thermocycling. Each experimental group is represented by data from a single sample. Round or oval pitting (arrowheads) is visible in all the samples, although it is more evident for the archwires treated in FS at pH 3.5, irrespective of Th. Note the changes in surface texture of the Thermaloy® archwire.

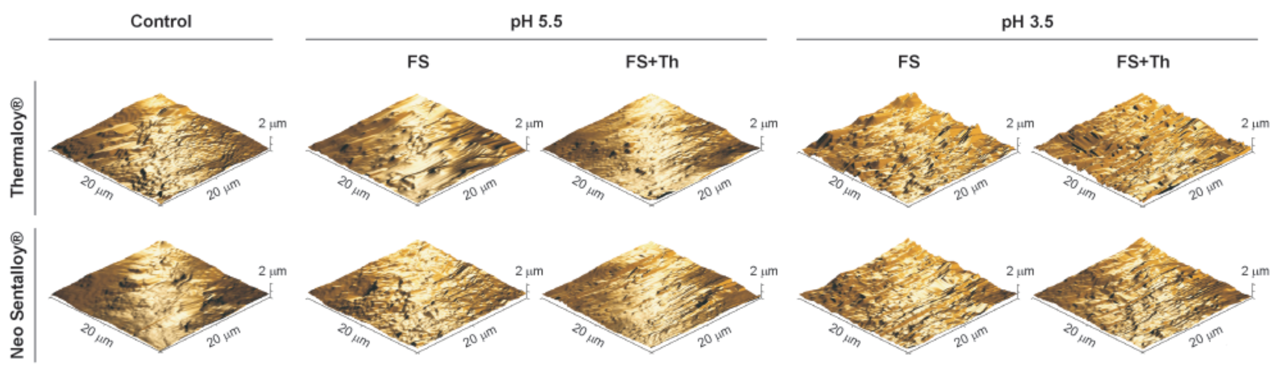

Figure 3 Representative atomic force microscopy three-dimensional reconstructions of the surface recordings of the experimental groups in relation to pH conditions and treatments. FS, incubation in fluoridated artificial saliva; FS + Th, incubation in fluoridated artificial saliva followed by thermocycling.

Round or oval pitting seen on the surface appeared to be deeper in the former. The archwire samples treated by FS and FS + Th at pH 5.5 did not show any notable differences as compared with the corresponding control archwires. In contrast, more irregular surfaces were seen for both archwire types when treated by FS and FS + Th at pH 3.5, where the numbers, dimensions, and deepness of the pitting were greater when compared with those from the corresponding control archwires. Moreover, a significant change in surface texture was evident for the Thermaloy® archwire. No specific differences were seen in any samples treated by FS or FS + Th.

Representative AFM 3D reconstructions for all the subgroups are shown in Figure 3. Less rough surfaces were visible for the control archwires of both the experimental groups. Similar levels of roughness were also seen at pH 5.5, irrespective of the experimental group or treatment. In contrast, an increase in roughness was apparent at pH 3.5 (for both treatments), with more pronounced surface irregularities seen for the Thermaloy® group as compared with the NeoSentalloy® group.

The results of AFM surface and tensile analyses are shown in Table 1. All surface parameters were similar between the groups under the initial conditions, with no significant differences, although the Ry, Ra, and Sa were all greater in the NeoSentalloy® group. The Rsk was nearly zero in both groups, although positive for the Thermaloy® and negative for the NeoSentalloy® archwires.

None of the surface parameters showed any significant changes between the two treatments under the initial conditions and at pH 5.5, although slight increases were generally seen in the treated archwires. However, comparison between the groups with FS treatment showed significantly greater Ry and Ra for the NeoSentalloy® archwires as compared with the Thermaloy® archwires, and significantly greater (positive) Rsk was seen in the Thermaloy® archwires as compared with the NeoSentalloy® archwires (which was negative). A different behaviour was observed when

Table 1 Surface and tensile parameters of the experimental archwire groups in relation to pH conditions and treatments.

Parameter	Group	Control	pH 5.5		Difference	pH 3.5		Difference
			FS	FS + Th		FS	FS + Th	
Ry (nm)	Thermaloy®	833 ± 159	889 ± 134	1092 ± 402	NS	1533 ± 411***	1581 ± 450***	***
	NeoSentalloy®	942 ± 203	1064 ± 138	1401 ± 670	NS	1282 ± 386	1097 ± 236	NS
	Difference	NS	*	NS		NS	**	
Ra (nm)	Thermaloy®	98 ± 12	88 ± 31	94 ± 40	NS	138 ± 38*	129 ± 33*	*
	NeoSentalloy®	110 ± 24	114 ± 18	116 ± 45	NS	123 ± 33	115 ± 23	NS
	Difference	NS	*	NS		NS	NS	
Rsk ($\times 10^3$)	Thermaloy®	0.056 (−0.106 to 0.365)	0.280 (0.031 to 0.515)	0.051 (−0.671 to 0.266)	NS	0.885 (0.456 to 1.255)**	0.295 (0.021 to 0.589)	***
	NeoSentalloy®	−0.083 (−0.191 to 0.273)	−0.054 (−0.210 to 0.042)	0.178 (0.018 to 0.743)	NS	0.269 (0.180 to 0.652)	0.039 (−0.119 to 0.193)****	*
	Difference	NS	*	NS		NS	NS	
Sa (m^2)	Thermaloy®	411 ± 6	410 ± 8	414 ± 8	NS	433 ± 17**	449 ± 26***	***
	NeoSentalloy®	413 ± 11	416 ± 7	423 ± 17	NS	432 ± 21*	418 ± 4	***
	Difference	NS	NS	NS		NS		
UTS (N/mm^2)	Thermaloy®	989.0 ± 11.6	1008.4 ± 43.4	1016.3 ± 9.0***	**	1105.3 ± 10.6***	1104.4 ± 9.0***	***
	NeoSentalloy®	1065.0 ± 5.3	1089.1 ± 23.7*	1098.8 ± 21.2**	**	1125.0 ± 23.7***	1146.3 ± 28.8*	***
	Difference	***	**	***		NS	***	

FS, incubation in fluoridated artificial saliva; FS + Th, incubation in fluoridated artificial saliva followed by thermocycling. Ry, maximum height of the roughness profile; Ra, average of the roughness profile; Rsk, skewness of the roughness profile; Sa, area of the roughness profile; UTS, ultimate tensile strength. The data are presented as means ± SDs (Ry, Ra, Sa, and UTS) or as median (25th and 75th percentile; Rsk; $n = 10$). Difference, significance of the differences among the time points within the groups and pH conditions (columns) or between the groups at baseline (rows). Results of the pairwise comparisons between the controls and the treatments within each of the experimental groups and pH conditions: statistically significantly different from the control (*$P < 0.05$; **$P < 0.01$; ***$P < 0.001$) or FS treatment (****$P < 0.05$) value. NS, difference not statistically significant.

comparing the same parameters between the initial conditions and treatment under pH 3.5: all the surface parameters showed statistically significant differences with treatment (including the control) for the Thermaloy® group, and similarly, the Rsk and Sa of the NeoSentalloy® group showed significant differences. In spite of the statistical significance of the pairwise comparisons, all surface parameters recorded for the FS and FS + Th treatments were greater than the corresponding control scores, in both of the groups. In more detail, all the surface parameters in the Thermaloy® group were significantly greater for both the FS and the FS + Th treatments as compared with the control archwires, with the only exception being for Rsk with FS + Th treatment, which was similar to that seen for the control archwires. In contrast, in the NeoSentalloy® group, only the Sa for FS treatment was significantly greater (positive score) as compared with that of the control archwires (negative score). No significant differences were noted between the FS and FS + Th treatments for any of the surface parameters, with the exception of Rsk in the Neo Sentalloy® group, which was greater with FS treatment. Finally, irrespective of FS or FS + Th treatment, all parameters were greater in the Thermaloy® group as compared with the NeoSentalloy® group, although only the Ry and Sa reached statistical significance.

For UTS, under control conditions, a significantly greater score was observed for the NeoSentalloy® group as compared with the Thermaloy® group. UTS also showed statistically significant differences among the treatments (including the control) for the groups and pH conditions. Pairwise comparisons demonstrated that all UTS scores for both treatments and pH conditions were significantly greater when compared with those of the corresponding control archwires. The only exception was for the Thermaloy® group, in which the UTS for FS treatment at pH 5.5 was similar to that of the control archwires. These UTS scores for the NeoSentalloy® group remained statistically significant as compared with those of the Thermaloy® group, at each comparison within the pH conditions and treatments, with the only exception being for FS treatment at pH 3.5.

The normalized changes of the surface parameters seen with the FS and FS + Th treatments with respect to the corresponding control scores are shown in Figure 4. Generally, no significant changes were seen for either groups or treatments at pH 5.5, with the only exception being for Rsk, which was greater with FS treatment in the Thermaloy® group as compared with the NeoSentalloy® group. A different behaviour was seen between the groups at pH 3.5. In particular, all the normalized changes seen in the Thermaloy® group were greater when compared with those in the NeoSentalloy® group, irrespective of the treatments. Statistically significant differences were seen for the Ry (both treatments), Ra and Sa (FS + Th treatment only). Finally, the only significant difference between the treatments within the same experimental group was seen in the NeoSentalloy® group for the Rsk and Sa, the normalized changes of which were greater with FS treatment as compared with FS + Th treatment.

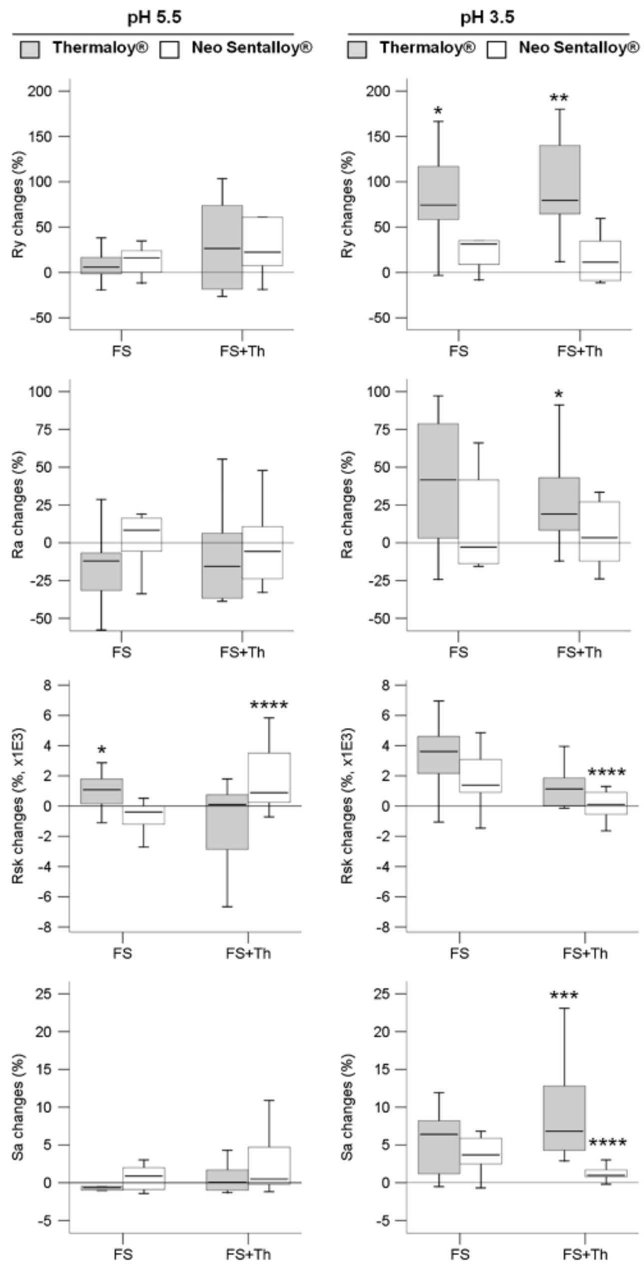

Figure 4 Normalized changes in the surface parameters of the experimental archwire groups in relation to pH conditions and treatments. FS, incubation in fluoridated artificial saliva; FS + Th, incubation in fluoridated artificial saliva followed by thermocycling. Ry, maximum height of the roughness profile; Ra, average of the roughness profile; Rsk, skewness of the roughness profile; Sa, area of the roughness profile; UTS, ultimate tensile strength. The data are presented as box plots (n = 10). Statistically significantly different from the corresponding NeoSentalloy® (*P < 0.05; **P < 0.01; ***P < 0.001) or FS treatment (****P < 0.05) datasets.

Discussion

The present comparative *in vitro* study evaluated the surface corrosion and fracture resistance of Thermaloy® and Neo Sentalloy® NiTi-based archwires after 30 days incubation in FS, at two different pHs of 5.5 and 3.5, followed by thermocycling. In particular, the fluoride concentration used in the present study, at 1500 ppm, was set in the range of, or slightly above, most commercially available toothpastes and mouthwashes to determine the effects of regular fluoride prophylaxis on these NiTi-based archwires. The results showed significant effects in terms of surface corrosion, but not fracture resistance, which were seen mainly for the Thermaloy® group at the lower pH, with no effects of thermocycling irrespective of the group or pH condition.

A previous *in vivo* clinical and histological analysis (Gursoy *et al.*, 2007) demonstrated that low-dose continuous Ni release from orthodontic appliances, as 1.3 g/g of tissue, might be the initiating factor for gingival overgrowth. Moreover, *in vitro* evidence that showed dose-dependent toxic effects of Ni on oral mucosal cells (Gursoy *et al.*, 2007), possibly due to intracellular accumulation of the Ni, should also be taken into account (Ermolli *et al.*, 2001). This is of importance when considering that the average Ni released from a full-mouth fixed appliance can be up to 40 mg/day (Park and Shearer, 1983). Thus, Ni release through surface corrosion should be avoided, or at least minimized, in clinical situations.

The modality of surface corrosion of NiTi-based archwires is mainly characterized by pitting, as seen in the present study for the Thermaloy® group (Figure 2); this is in agreement with previous reports (Eliades and Bourauel, 2005; House *et al.*, 2008). A different scenario was observed for the degree of corrosion. In spite of previous studies that evaluated the degree of surface corrosion of NiTi-based archwires in response to fluoride, pH, and temperature changes, direct comparisons with the present results are difficult due to the differences in the study designs. For instance, some *in vitro* studies used an immersion solution containing very variable fluoride concentrations (from 250 ppm up to approximately 10 000 ppm), which would not always resemble the clinical situation (Nakagawa *et al.*, 1999; Watanabe and Watanabe, 2003; Schiff *et al.*, 2006), while Huang (2007) used a fast electrochemical technique under modified Fusayama artificial saliva with a pH of 6.25. Further studies evaluated the corrosion after 4 weeks of incubation under artificial saliva and/or after sterilization (Mayhew and Kusy, 1988; Thierry *et al.*, 2000; Lee and Chang, 2001). These investigations, therefore, did not always include a low pH incubation, while the sterilization treatment does not resemble the temperature change behaviour in the oral cavity. Only one previous study evaluated corrosion under different pH conditions in combination with variable fluoride concentrations (Huang, 2007). That author reported no significant changes in the surface topography, as recorded using AFM, for four different commercial NiTi-based archwires after 28 days of incubation at pH 3.4 and with fluoride ions of 1483 ppm. The same study reported fluoride concentration as the only parameter that affected the degree of corrosion, while the pH of incubation (from 6.3 to 3.4) was not relevant (Huang, 2007). However, all the fluoride mouthwashes/gels used in that study were diluted up to 10-fold in artificial saliva, making the final fluoride concentration and pH of different solutions of incubation different to those of the original formulations (Huang, 2007). Therefore, these results showing no significant effects in terms of surface corrosion of NiTi-based archwires incubated up to 2500 ppm fluoride have to be considered with caution. A further *in vitro* study also reported differential surface corrosion effects for NiTi-based archwires on immersion in two different commercial mouthwashes that contained only 250 ppm fluoride (Schiff *et al.*, 2006).

In contrast to these earlier results (Huang, 2007), the present study showed that the pH appears to be responsible for increased corrosion in the Thermaloy® group (Table 1 and Figures 2 and 3). These inconsistencies might arise from the different manufacturing processes of the NiTi-based archwires or from the different protocols used. While the present research made use of NaF, Huang (2007) used commercial fluoride-containing mouthwashes/gels in which other molecular contents may well have influenced the corrosion effects produced by the fluoride ions. Interestingly, acidic conditions (in combination with fluoride ions) can accelerate the chemical disruption of the oxide films, which would then reform as the metal surface is again exposed to oxygen (House *et al.*, 2008). It can thus be hypothesized that the synergistic corrosive actions of fluoride and low pH are more important than each of the single, individual treatments. However, in the present and previous studies, the archwires were continuously immersed in the saliva solutions, and not exposed to oxygen from the air during the experiments, conditions that would be more likely to occur *in vivo*. This consideration would support the concept that *in vitro* incubation would be more extreme than the actual clinical situation, at least regarding the repassivation process.

The present study also demonstrated a different degree of surface corrosion between the two experimental groups that became more evident when analysing the normalized changes in the surface parameters (Figure 4). Significant differences were seen at pH 3.5, with the Thermaloy® group showing greater corrosion as compared with that of the Neo Sentalloy® group. This different corrosion behaviour between the groups might be due to the various surface treatments since surface oxide thickness has been shown to influence resistance to surface corrosion (Clarke *et al.*, 2006). The present differential surface corrosion seen between the two pH conditions, in particular for the Thermaloy® group, is in agreement with a previous study that followed a different design to evaluate the cytotoxicity of ions released by NiTi-based archwires (Kao *et al.*, 2007).

Increased toxicity was seen on electrochemical corrosive breakdown of the archwires with 0.2 per cent NaF at pH 3.5 but not at pH 6.25 (Kao *et al.*, 2007). Of further note, a recent 12 month *in vivo* study reported saliva with increased Ni ions immediately after orthodontic appliance insertion and for up to 1 month, followed by a stable decay to baseline values (Petoumeno *et al.*, 2008). However, that study did not include any fluoride treatment. Therefore, knowledge of the different resistances to corrosion would be useful in the choice of NiTi-based archwires, at least for those patients requiring heavy fluoride prophylaxis, i.e. those highly susceptible to dental caries, especially if agents with more than 1500 ppm fluoride are to be used.

Another interesting aspect of the NiTi-based corrosion relates to the temperature effects. The corrosion rate of various NiTi-based archwires has been reported to increase with temperature (Lee and Chang, 2001; Pun and Berzins, 2008). However, these studies used incubations at constant temperatures that do not resemble the true oral environment, where the temperature is continuously changing during eating or drinking. For this, thermocycling treatment would better fit the actual clinical situation. The results from the present study, and especially those relating to the Thermaloy® group under both pH conditions, are in agreement with the findings of Ahn *et al.* (2006) in which the samples were immersed in acidic fluoride solutions at pHs of 6.0 and 3.5 and then kept at a constant temperature of 60°C. This previous evidence, along with the present results regarding the minimal effects of thermocycling (Table 1 and Figures 3 and 4) support the concept that pH is more important than temperature in determining the surface corrosion or fracture resistance of these NiTi-based archwires. However, the different temperature treatments, whether variable or constant, have to be taken into account.

While previous evidence showed no significant destruction of the metal components or detrimental effects on the mechanical properties of NiTi-based archwires, in spite of the occurrence of surface corrosion (House *et al.*, 2008), other evidence has clearly shown that with intraoral exposure, these archwires fracture more frequently than expected (Eliades and Bourauel, 2005). The morphometric assessment used in the present study cannot describe in full the corrosion behaviour and mechanical consequences thereof, which has to be taken into account when reappraising the current evidence. These inconsistencies thus require further investigation and reinforce a previous concept according to which the causes of archwire fracture are multifactorial, with corrosion, surface finish, and work hardening during treatment all contributing (House *et al.*, 2008).

Clinical implications

The findings of the present study show that different degrees of corrosion resistance can be seen among different NiTi-based archwires. However, considering that notable surface alterations were observed only at pH 3.5 and at 1500 ppm fluoride after a 30 day incubation (and especially for the Thermaloy® group), with no significant effects due to thermocycling, the problem of surface corrosion is not likely to be clinically significant, as long as the prophylaxis procedures during any orthodontic treatment include mouthwashes or toothpastes with no more than a 1500 ppm fluoride concentration. In contrast, any dental treatment that involves the use of agents with higher fluoride concentrations (e.g. 10 000 ppm) would still have relevant effects in terms of surface corrosion of NiTi-based archwires and must preferably be carried out by the clinician after demounting of the archwire. Of note, the limited surface corrosion observed in the current study would have limited impact on resistance to sliding, even considering the contrasting results (Eliades and Bourauel, 2005). Finally, the fracture resistance of both the archwires included in the present study can be considered of no clinical relevance, although any detrimental mechanical effects that might derive from loading would also need to be taken into account.

Conclusions

The findings of the present study indicate that:

1. Different NiTi-based archwires can show different corrosion resistance during treatment with 1500 ppm fluoride and low pH, with Neo Sentalloy® wires showing no significant alterations.
2. Thermocycling has no effects on the surface corrosion and fracture resistance of NiTi-based archwires.

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