EFFECTS OF TOPICAL FLUORIDE PROPHYLACTIC AGENTS
ON THE MECHANICAL PROPERTIES OF ORTHODONTIC
NICKEL-TITANIUM CLOSED COIL SPRINGS AND
STAINLESS STEEL CLOSED COIL SPRINGS

A THESIS IN
Oral and Craniofacial Sciences

Presented to the Faculty of the University
of Missouri-Kansas City in partial fulfillment of
the requirements for the degree
MASTER OF SCIENCE

by
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Kansas City, Missouri
2014
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ABSTRACT

The purpose of this study was to investigate the effects of topical fluoride prophylactic agents on the mechanical unloading properties of nickel-titanium (NiTi) and stainless steel (SS) closed coil springs. Spring were stored at 37°C under static load in phosphate buffered saline (PBS) and treated with either neutral sodium fluoride (NaF) or acidulated phosphate fluoride (APF) five days per week for two minutes. Mechanical testing was done in a dH₂O bath at 37°C at 0-, 1-, 4-, 8-, and 12 weeks. Unloading forces for NiTi and SS springs were measured at 9-, 6-, and 3 mm and 2-, 1.5-, and 1 mm, respectively. Scanning electron microscopy was used to evaluate surface topography of selected springs after 12 weeks.

Based on a 1-Factor ANOVA and Dunnett’s post hoc, 3M NiTi springs showed a significant decrease (p <0.01) in the unloading force at each extension following exposure to both fluoride treatments, but only after 12 weeks. The AO NiTi springs showed a significant
decrease in unloading force at each extension after 12 weeks following exposure to NaF. However, with SS springs, there was no significant effect of either fluoride treatment on the SS springs at any extension or time point. SS also springs showed no significant surface topography changes, irrespective of storage conditions, which correlates with the lack of fluoride effects on SS mechanical property effects. In contrast, while there were NiTi surface topography changes (pitting and mottling) following PBS+APF exposure, those changes could not be directly linked to the observed changes in mechanical properties.

Results suggest topical fluoride used with NiTi springs could potentially lead to prolonged treatment time due to decreased unloading properties. However, topical fluoride used with SS springs should not affect treatment duration.
The faculty listed below, appointed by the Dean of the School of Dentistry, have examined a thesis titled “Effects of Topical Fluoride Prophylactic Agents on the Mechanical Properties of Orthodontic Nickel-Titanium Closed Coil Springs and Stainless Steel Closed Coil Springs,” presented by Brittany Carpenter, candidate for the Master of Science degree, and certify that in their opinion it is worthy of acceptance.

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ACKNOWLEDGEMENT

I would like to thank and express my deep gratitude to:

Dr. Mary Walker, for her endless patience, knowledge, commitment and support throughout this process.

Dr. Jeff Nickel, for his encouragement, support and expertise.

Dr. Ying Liu, for her generosity, statistical knowledge and input.

Dr. Yasuyoshi Ueki, for his feedback, generosity and time.

John Fife, for his organization and help to make this process run smoothly.

Rachel Reed, for her help preparing specimens and collecting data.

Corey Hastings, for his help collecting data.

My husband and parents, for their unconditional love and support throughout these many years of school. I could not have reached this point without them.
CHAPTER 1

INTRODUCTION

Alloys in Orthodontics

Stainless steel, nickel-titanium, and beta-titanium alloys have a variety of applications in orthodontics from brackets to archwires to springs, their main use being for archwires (Kapila and Sachdeva 1989; Kusy 1997). In terms of archwire applications, various amounts of stiffness, springback, formability, biocompatibility and surface friction are what make these alloys desirable (Kapila and Sachdeva 1989). These archwires should provide a light, continuous force to move teeth. These light forces help to decrease patient discomfort, tissue hyalinization, and undermining resorption. Ideally, the various archwires will exhibit an elastic behavior over a long period of time (Kusy 1997). During the initial stage of treatment, nickel-titanium or multi-stranded stainless steel archwires work best due to their flexibility, low load deflection rate, and good springback (Sandhu et al. 2012). Beta-titanium and stainless steel archwires are useful during the later stages of treatment. These alloys provide more applied force and can incorporate bends and loops when necessary in treatment (Sandhu et al. 2012).

Besides archwires, alloys are also used to fabricate orthodontic coil springs. Whether closed coil springs or open coil springs, springs are often used in the mechanics of orthodontics. Currently, the alloys used for springs include nickel-titanium and stainless steel. These two alloys are used due to their mechanical properties and added versatility in orthodontic treatment. With the versatility these various alloys bring, the orthodontist can choose the appropriate alloy and achieve predictable treatment results.
**Stainless Steel Alloy**

Stainless steel (SS) is now available in over 120 different compositions for use in a variety of applications. This alloy is iron-based and contains varying amounts of chromium and nickel (Izquierdo et al. 2010). The addition of chromium increases the corrosion resistance and the addition of nickel helps to stabilize the austenitic phase which increases the toughness. This alloy, when containing 10 to 13% chromium, forms an oxide layer that passivates the surface and makes it “stainless”. When at least 8% of the composition is nickel, the austenite phase is stabilized and corrosion resistance is increased (Kusy 1997). Austenitic stainless steels have come to be a mainstay in orthodontic brackets, wires, and springs. Environmental stability, stiffness, resilience, and formability are some of the desired properties of this alloy that make it appropriate for orthodontic wires and springs. The high yield strength and modulus of elasticity of stainless steel are derived from the carbon interstitial hardening and cold working of the alloy. Any remaining stresses in a SS used for wires or springs can negatively affect the elastic properties; therefore, heat treatment is used in stress-relieving after forming the alloy into an arch, loops, or coils. This heat treatment helps to increase the elastic properties of the alloy (Kapila and Sachdeva 1989).

**Nickel-Titanium Alloys**

In the early 1960’s, an investigation into a nonmagnetic, salt resisting, waterproof alloy led to the development of nickel-titanium alloy (Thompson 2000). This equiatomic alloy, 50% titanium and 50% nickel, was introduced by the Naval Ordnance Laboratory (Kusy 1997). The thermodynamic properties of this intermetallic alloy allow it to exhibit a shape memory effect when specific, controlled heat treatment is applied (Thompson 2000).
Nickel-titanium, like stainless steel, also forms an oxide layer that passivates the surface and lends to its biocompatibility. However, instead of chromium, it is the titanium component that reacts with oxygen to form a protective titanium oxide layer (Lee et al. 2010). Nickel-titanium (NiTi) alloy was introduced into orthodontics in 1971 and brought favorable properties such as biocompatibility, ductility, corrosion resistance, low elastic modulus, superelasticity, and shape memory effect (Andreasen and Hilleman 1971). NiTi alloys can be made into a specific shape and when deformed into a different shape, it can be subsequently heated and return to its original shape. This unique shape-memory property is a result of a transformation of NiTi from its austenitic phase to its martensitic phase when it undergoes temperature change (Kusy 1997; Santoro et al. 2001). It shifts from the austenitic body-centered cubic lattice structure to an intermediate rhomboidal phase. This intermediate structure then shifts to the martensitic hexagonal close-packed lattice structure. When the arch wire is heated, the reverse occurs and there is a shift from martensitic phase to austenitic phase (Fernandes et al. 2011). Applied stress can also induce a transformation from the austenitic to martensitic phase. This phase change results in an increase in strain with little change in stress, which is referred to as superelasticity (Fernandes et al. 2011). As the alloy returns to its original shape, load is applied to the teeth which results in tooth movement related to archwire or spring applications (Pun and Berzins 2008).

**Applications of Orthodontic Alloys for Springs**

As already mentioned, besides archwire applications, stainless steel and nickel-titanium are also used as springs in orthodontics. Before the discovery of NiTi for use in orthodontics, SS was the main alloy used in appliances. Since the introduction of NiTi with
its superior springback and superelastic properties, it has now become the alloy of choice for coil springs (Angolkar et al. 1992). However, both NiTi and SS springs are still used in practice. They are utilized as both open-coil and closed-coil springs. These springs are used to close space, open space, protract teeth, distalize molars, and to provide traction for impacted teeth. When evaluating force generated during loading and unloading by coil springs of either nickel titanium or stainless steel, the important aspect lies in the force upon unloading. This is the clinically relevant phase because this is the time when tooth movement is generated. Many variables can affect this force including type of alloy, wire size, lumen size, and pitch (Angolkar et al. 1992).

Before specific studies on the mechanical properties of coil springs were investigated, mechanical properties of arch wires were applied to springs. A previous study compared SS and NiTi archwires and reported that in tension, SS had the least maximum springback compared to NiTi (Drake et al. 1982). This is important because higher values of springback allow for increased range of activation clinically. When comparing these two alloys in bending and torsion, SS had the highest springback values (Drake et al. 1982). Miura et al. found that when a NiTi archwire was subjected to load and a deflection of the wire was created, the load stayed constant due to the alloy’s superelasticity. Even when a large amount of deflection was created, a permanent deformation did not occur because the archwire had high springback properties. Also, with use of controlled heat treatment, it was possible to bend the wire and change the load of superelastic activity to a preferred level without changing the mechanical properties (Miura et al. 1988a). Since desirable properties are found in NiTi and SS archwires, it is reasonable to believe that these properties might also be found in coil springs of the same alloy.
Mechanical Properties of SS and NiTi Springs

Coil springs differ from archwires in that springs go through an additional procedure, called winding, during the manufacturing process, which could affect their mechanical properties. Moreover, torsional and tensional forces are applied to springs as well as bending that also could affect mechanical properties. However, load-deformation properties of archwires and springs appear to be similar. Deformation of archwires is usually exhibited as angular bending or linear deflection and deformation of springs is usually exhibited as elongation or compression (Han and Quick 1993). As previously stated, tooth movement by means of light, continuous forces is the preferred modality for treatment in orthodontics. Tooth movement of 0.5 mm/wk for a short time with light continuous forces of 75 – 100g has been the ideal suggestion (Storey 1973). Therefore, it is important to look at the unloading mechanical properties of coil springs for achieving this ideal type of movement. Various factors affecting the force associated with springs have been researched. As wire size and pitch increase, the load-deflection rate has been found to increase. Conversely, as lumen size and length increase, load-deflection rate decreases (Miura et al. 1988b; Boshart et al. 1990). A larger lumen size, longer wire length, smaller wire size, and shorter pitch, therefore, produce more favorable lighter forces (von Fraunhofer et al. 1993; Bourke et al. 2010).

Just as with archwires, when evaluating force of coil springs on a load-deflection curve, the unloading curve is of most importance for orthodontists. This is the part of the curve produced as the spring returns to its resting length. The load-deflection curve of available SS coil springs shows a linear relationship; whereas, NiTi coil springs show a non-linear load-deflection behavior (Miura et al. 1988b; Wichelhaus et al. 2010). It is thought that a favorable load-deflection diagram would have a horizontal unloading curve that
produces a more uniform, continuous force over several millimeters of spring deflection (von Fraunhofer et al. 1993; Ren et al. 2004). Stainless steel springs have been shown to produce high forces that decay rapidly over a short range of tooth movement. SS closed coil springs were shown in a study by Angolkar et al. (1992) to lose 17.3% of their initial force in the first 24 hours after activation. After initial loss, minimal force decay occurred, 20-21%, by the end of 28 days (Angolkar et al. 1992). Nickel titanium, however, has been shown to produce light, continuous forces over a long range of activation (von Fraunhofer et al. 1993). NiTi closed coil springs were shown to have 3.3% force decay after 4 hours and 8.6% after 7 days. Thereafter, the force remained relatively constant (Angolkar et al. 1992).

Although manufacturers report force characteristics of springs, there have been several investigations indicating that the force reports are unreliable. For example, Manhartsberger and Seidenbusch (1996) evaluated NiTi coil springs and found that the forces produced were actually greater than the forces reported by the manufacturer. The coil springs should have produced 150 g of unloading force when compressed up to 80% of their length, but instead were found to produce 300 g of force (Manhartsberger and Seidenbusch 1996). Bourke et al. (2010) and Maganzini et al. (2010) found similar unreliable reported force characteristics. Bourke et al. (2010) tested NiTi open coil springs from three different manufacturers. The properties measured in this study were maximum force, average force during deactivation, load-deflection ratio, and force degradation at 0 hours, 24 hours, 4 weeks, 8 weeks, and 12 weeks. Results showed that the springs had significantly lower average force and higher maximum force than the reported values from the manufacturers. The springs also exhibited a high load-deflection ratio, which is similar to the non-superelastic characteristic of SS springs (Bourke et al. 2010). Maganzini et al. (2010)
compared NiTi closed coil springs from five different manufacturers. They found that the majority of the springs exhibited inconsistent forces during the unloading phase; therefore, failing to produce expected peak load forces and constant unloading forces (Maganzini et al. 2010). In agreement with these studies, Brauchli et al. (2011) found only 4 out of 23 open coil NiTi springs exhibited superelastic behavior. The remaining springs showed a linear force-deflection diagram similar to SS springs (Brauchli et al. 2011). No such studies involving unreliable manufacturer force values for SS springs have been reported. NiTi coil springs have been favored due to their unique properties and constant unloading forces, but with studies showing these springs to produce characteristics similar to SS springs perhaps one spring should not be favored over the other. Along with inconsistent force of as-received springs, other factors will affect spring force such as the oral environment and various types of corrosion.

**Corrosion and the Effects on SS and NiTi Alloy Properties**

Corrosion resistance in an orthodontic alloy becomes extremely important with regards to biocompatibility. Environmental stability allows the alloy to demonstrate desired mechanical properties which, in turn, results in a predictable behavior (Kapila and Sachdeva 1989). In both stainless steel and nickel titanium, the formation of a surface passivation layer leads to the resistance of corrosion. In nickel titanium, a passive film of titanium oxide forms when exposed to air, while for stainless steel, a protective layer of chromium oxide forms. These protective layers prevent the diffusion of oxygen resulting in resistance to corrosion (Huang 2003; Watanabe and Watanabe 2003). When various environmental factors disrupt this passivation layer, the alloy becomes susceptible to corrosion.
Orthodontic alloys are susceptible to many different types of corrosion. The most common type of corrosion among all alloy applications is uniform attack. When interacting with the environment, hydroxides or organometallic compounds form and can dissolve large amounts of the alloy (Eliades and Athanasiou 2002). However, with brackets and archwires, pitting corrosion is more commonly seen. The pit is defined as a pore that has a depth equal to its width. These pores have been found in NiTi and SS wires before use in the oral environment and could serve as areas more susceptible to corrosion. Pitting corrosion has also been seen in NiTi archwires placed in a 1% saline solution (Eliades and Athanasiou 2002). Crevice corrosion can arise when nonmetallic products such as an elastomeric chain are placed on metal and a difference of metal ion or oxygen concentration occurs between the crevice and surrounding area. Crevices can reach up to 2-5 mm in some instances (Eliades and Athanasiou 2002). Stainless steels are subject to intergranular corrosion that affects the microstructure. This type of corrosion affects the solubility of chromium carbide which precipitates at the interface of the grains. Fretting corrosion refers to a process that occurs at the contact area of materials under load. The mechanism deals with the cold welding at the interfaces under pressure and results in the rupture of contact points (Eliades and Athanasiou 2002). An example of this lies within a bracket’s slot-archwire interface.

Orthodontic alloys are subjected to a myriad of intraoral environmental factors that can result in corrosion especially considering that orthodontic appliances are routinely left in the intraoral environment for long periods of time. As a result of corrosion, both NiTi and SS alloys have been shown to release nickel, iron, and chromium ions (Milheiro et al. 2012). Nickel has been reported as one of the most common causes of contact dermatitis and its incidence can be as high as 20-30% (Blanco-Dalmau et al. 1984; Dunlap et al. 1989).
However, another important consideration is the effect of corrosion on orthodontic alloy surface integrity and mechanical properties and the potential impact on tooth movement. Numerous studies evaluated different environmental conditions such as pH, temperature, and stress, and their effect on the corrosion of orthodontic alloys. In a study by Kao and Huang (2010), corrosive breakdown of NiTi and SS archwires were reported in three different artificial saliva solutions. One of the solutions contained sodium fluoride at a pH of 4 while the other two solutions did not contain fluoride and were at pH 4 and a pH 6. The NiTi and SS archwires showed corrosion in the artificial saliva solution with pH 4. They also reported that NaF corrosion potential was lower than in either of the other solutions. SEM results showed defects and pitting corrosion in all three media for NiTi and SS archwires. Therefore, decreasing the pH led to an increase in corrosion potential (Kao and Huang 2010). Oshida et al. (1992) found similar results. They tested corrosion rate of NiTi archwires in 0.9% NaCl solution at varying pHs of 3, 7, and 11. As pH decreased, the corrosion rate of the archwires increased (Oshida et al. 1992).

It has also been reported that corrosion rate in NiTi and SS wires increases with temperature. Pakshir et al. (2011) investigated corrosion behavior in NiTi and SS archwires in Ringer’s solution at different temperatures ranging from 15 to 55°C. In both NiTi and SS archwires, as temperature increased the corrosion rate increased. Results also showed that NiTi had greater corrosion resistance compared to SS (Pakshir et al. 2011). Similar findings have been reported for NiTi archwires that were tested in 0.9% NaCl solution at temperatures of 3, 37, and 60°C. An increased corrosion rate was found when temperature was increased (Oshida et al. 1992). Surface topography of NiTi wires were examined by Ahn et al. (2006). NiTi archwires were subjected to fluoride solutions with temperatures of 5, 37, 60°C. They
found the archwires to be severely corroded when subjected to the solution with a concentration of pH 3.5 at 37°C and 60°C. However, negligible corrosion was found, regardless of pH, when the temperature of the solution was 5°C (Ahn et al. 2006).

Finally, another important factor is the application of load and the potential effect on corrosion, as well as corrosive effects on mechanical properties. Corrosive mediums like saliva, with varying pHs and temperatures can increase the reduction in fatigue resistance. As fatigue resistance decreases, SS and NiTi archwire become more susceptible to fracture (Eliades and Athanasiou 2002). Stress applied to NiTi wires in an artificial saliva solution at pH 2 and pH 5.3 cracked the protective oxide layer. This allowed the exposed metal to interact with the surrounding environment and undergo corrosion (Liu et al. 2007). These appliances, such as coil springs, become subject to corrosion fatigue after repeated cyclic stressing (Eliades and Athanasiou 2002).

Hydrogen absorption has been reported to degrade mechanical properties and increase fracture susceptibility. It can affect most metals, especially titanium and steel, causing a limited capability for plastic deformation and a loss in strength (Rogers 1968). Hydrogen ions can easily be absorbed in the bioenvironment due to presence of these ions in saliva and galvanic currents. Hydrogen ions are associated with the repassivation of alloys, therefore, fretting corrosion may also be a contributor (Yokoyama et al. 2001). Once corrosion and degradation of the passive layer has occurred, the alloy will absorb hydrogen readily (Hirth 1980). Hydrogen embrittlement often represented as a decrease in strain, tensile strength, and fracture (Yokoyama et al. 2004). When NiTi absorbs hydrogen, the stress of the martensitic transformation increases. It has been suggested that as critical stress increases, so does the orthodontic force. As hydrogen absorption increases, ductility of NiTi alloy
decreases. Additionally, as strain restriction exceeds the fracture strain the wire becomes brittle and will often lead to fracture (Yokoyama et al. 2003). SS alloys also show propensity for hydrogen absorption and subsequent embrittlement leading to stress corrosion cracking. Although no studies have been done on orthodontic wires, absorbed hydrogen has been shown to become trapped in SS lattice vacancies and may have an effect on plastic flow and recovery and ultimately the mechanical properties (Hirth 1980).

**Effects of Fluoride on NiTi and SS Alloys**

Orthodontic patients with inadequate hygiene resulting in plaque accumulation are seen frequently. Bacterial plaque buildup results in demineralization of enamel leading to white spot lesions. Therefore, topical fluorides are routinely used by orthodontists to prevent decalcification and caries (Kokich 2001; Vo et al. 2010). Fluoride reacts with enamel hydroxyapatite crystals to form fluoroapatite, which is more resistant to acid dissolution. Therefore, the presence of fluoride serves to promote remineralization of the enamel surface and decrease enamel solubility leading to demineralization (Alexander and Ripa 2000). Over-the-counter and prescription prophylactic fluoride products include foams, gels, toothpastes, and mouthwashes. These topical fluoride solutions vary not only in pH but in fluoride ion concentration as well. Acidulated phosphate fluoride (APF) and neutral sodium fluoride (NaF) are two commonly used prophylactic fluoride agents for orthodontic patients. They can be any combination of: fluoride ion concentration (0.0198% - 0.05%) and pH (acidic, 3.5 to neutral, 7) (Kokich 2001). Acidulated fluoride products are sometimes used due to more rapid uptake of fluoride into enamel (Brudevold et al. 1963).
Fluoride inhibits oral bacteria by reacting with the bacteria acid by-product and forming hydrofluoric acid; however, this hydrofluoric acid can ultimately degrade titanium alloys by disruption of their protective passive oxide layer (Nakagawa et al. 1999). As stated previously, once the passive oxide layer is degraded, hydrogen embrittlement can occur.

Acidic pH of different fluoride agents is also important for the breakdown of the oxide layer of titanium-based alloys leading to corrosion (Yokoyama et al. 2003; Schiff et al. 2004). Kao and Huang found that metal SS brackets and NiTi archwires in the presence of acidic conditions, pH 4, and NaF media have shown pitting corrosion (Kao and Huang 2010).

Effects of corrosion on mechanical property degradation have also been reported at a neutral pH and fluoride concentration of 0.5% or greater (Walker et al. 2005). Therefore, fluoride concentration in addition to pH may be of importance in oxide layer breakdown and hydrogen absorption (Walker et al. 2007). As the pH decreases, hydrofluoric acid concentration increased along with hydrogen ion concentration (Watanabe and Watanabe 2003). Lee et al. (2010) found that concentrations of 0.5% and greater of NaF lead to significant effect on corrosion resistance of NiTi archwires leading to damage of passive oxide layer (Lee et al. 2010). Walker et al. (2007) found that after APF and neutral fluoride exposure, SS wires showed a decrease in unloading properties. The same results were found for NiTi wires after exposure to topical fluoride agents (Walker et al. 2005). In both of these archwires, unloading elastic modulus and yield strength were decreased as well corrosive surface topography changes were demonstrated that appeared to be linked to the mechanical property changes (Walker et al. 2005; Walker et al. 2007).

Kaneko et al. (2004) investigated performance degradation of SS and NiTi wires as a result of hydrogen absorption after being immersed in acidic fluoride solution at 37°C for 60
minutes. They found the tensile strength of the NiTi archwire decreased and fractured before yielding. Tensile strength of the SS archwires only decreased slightly (Kaneko et al. 2004). In another study done by Vo et al. (2010), mechanical properties of NiTi archwires were also tested. Wires were exposed to a fluoridated agent and non-fluoridated agent and force degradation at different deflections were measured. The fluoride exposed archwires showed larger force degradation at 3.1 mm and 3.0 mm, but smaller force degradation at 0.5 mm and 1.0 mm deflection. Topical fluoride agents, therefore, resulted in decreased unloading properties at larger deflections. However, with lower deflections, the unloading properties were found to increase (Vo et al. 2010). Previous studies have focused on corrosion and mechanical properties of orthodontic archwires, but similar corrosion related changes could also affect coil spring mechanical properties.

**Problem Statement**

Many studies have focused on effects of simulated oral environments on NiTi and SS archwires. Some have even looked at effects of simulated oral environments on NiTi and SS coil springs (Harris et al. 1988; Han and Quick 1993). To date, no investigations have evaluated the effects of a simulated oral environment with phosphate buffered saline in combination with topical fluoride agents on NiTi and SS coil springs. As importantly, no topical prophylactic fluoride studies have incorporated loading of the springs during experimental exposure to attempt to account for spring functional application. The effects of fluoride on mechanical properties of coil springs are of importance in orthodontics, since tooth movement relies on the unloading properties. If these properties are negatively affected, the rate of tooth movement can be delayed therefore prolonging treatment time.
NiTi and SS springs can be utilized intra-orally for up to six months. During treatment, prophylactic fluoride application can be routinely done and may affect mechanical unloading properties. This study will investigate prophylactic topical fluoride application and static load on mechanical unloading properties of NiTi and SS coil springs. Changes in surface topography will also be evaluated to evaluate surface integrity.

**Hypotheses**

1. There will be a difference in force at varying extensions during unloading of nickel titanium and stainless steel coil springs stored over time in saline, saline in combination with APF gel, or saline in combination with NaF gel with static loading of the spring.

2. There will be a qualitative difference in the surface topography of nickel titanium and stainless steel coil springs as a function of the storage solutions with static loading of the spring.
CHAPTER 2
MATERIALS AND METHODS

Closed Coil Springs

NiTi Closed Coil Springs

The NiTi closed coil springs\(^1\) (Table 1) used in this investigation were approximately 50% nickel and 50% titanium in composition. NiTi springs from two different manufacturers were tested. The NiTi springs were 12 mm in length with an outer diameter of 1.26 mm and an inner diameter of 0.76 mm. The diameter of the wire is 0.25 mm and the radius is 0.63 mm. The springs included eyelets at each end. If springs showed any defects, they were excluded from the investigation.

SS Closed Coil Springs

The SS closed coil springs\(^2\) (Table 1) used in this investigation are derived from a 3’ spool of SS closed coil spring. The springs were measured with a digital caliper and cut to 12 mm in length. Excess coils at each end of the spring were turned up and used as eyelets by which the springs were extended. The SS springs have an outer diameter of 1.26 mm and an inner diameter of 0.76 mm. The diameter of the wire is 0.25 mm and the radius is 0.63 mm. If springs showed any defects, they were excluded from the investigation.

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1 Nitinol Closed Coil Medium, 3M Unitek, 2724 South Peck Road, Monrovia, CA, 91016
2 NiTi Closed Coil Adjustable Force, American Orthodontics, 1714 Cambridge Avenue, Sheboygan, WI 53081
3 Nubryte SS Closed Coil Spring, Dentsply GAC, 355 Knickerbocker Avenue, Bohemia, NY 11716
4 SS Close Wound Coil Spring, American Orthodontics, 1714 Cambridge Avenue, Sheboygan, WI 53081
TABLE 1

COIL SPRINGS USED IN STUDY

<table>
<thead>
<tr>
<th>Type</th>
<th>Manufacturer</th>
<th>Length</th>
<th>Lot Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiTi</td>
<td>American Orthodontics</td>
<td>12 mm</td>
<td>93546</td>
</tr>
<tr>
<td>NiTi</td>
<td>3M Unitek</td>
<td>12 mm</td>
<td>289243</td>
</tr>
<tr>
<td>SS</td>
<td>American Orthodontics</td>
<td>12 mm</td>
<td>A88271</td>
</tr>
<tr>
<td>SS</td>
<td>Dentsply GAC</td>
<td>12 mm</td>
<td>098945</td>
</tr>
</tbody>
</table>

**Storage Conditions**

A simulated oral environment was achieved by storing the springs in a 0.9% phosphate buffered saline (PBS) solution at 37±1°C. To prevent any microbial growth, 0.002% sodium azide was added to the PBS storage solution. Acidulated fluoride gel (APF) and neutral fluoride gel (NaF), two commonly used prophylactic topical fluoride agents (Table 2), were used in this investigation. These fluoride gels have the same method of application, but different fluoride ion concentrations and different pH values.

TABLE 2

FLUORIDE AGENTS USED

<table>
<thead>
<tr>
<th>Fluoride Agent</th>
<th>Active Agent</th>
<th>[F⁻] (%w/v)</th>
<th>pH</th>
<th>Lot number</th>
</tr>
</thead>
<tbody>
<tr>
<td>PreviDent Gel</td>
<td>1.1% NaF (NaF)</td>
<td>0.5</td>
<td>7</td>
<td>3022USC11A</td>
</tr>
<tr>
<td>Acclean Gel</td>
<td>2.7% NaF in APF</td>
<td>1.23</td>
<td>3.5</td>
<td>41198</td>
</tr>
</tbody>
</table>

---

5 Dulbecco Phosphate Buffered Saline, Sigma-Aldrich™, Inc, 3050 Spruce St., St. Louis, MO, 63103
6 Acclean 60 Second Fluoride Treatment, Henry Schein, 135 Duryea Road, Melville, NY 11747
7 Prevident Gel, Colgate Oral Pharmaceuticals, 300 Park Avenue, NY 10022
**Specimen Preparation**

All NiTi and SS closed coil springs were incubated in PBS at 37±1°C to simulate the aqueous oral cavity. A custom-made rack of 316 stainless steel and polyoxymethylene⁸ (Fig. 1) was used to extend the springs. The custom rack with extended springs was stored and submerged in PBS in a 32-ounce enclosed plastic container⁹. NiTi springs were extended 9 mm during storage, while SS springs were extended 2 mm. The PBS solution was replaced once a week throughout the 12-week time period.

Each rack of springs assigned to an experimental fluoride treatment group was removed from the PBS storage solution in the plastic container and treated with the correct fluoride solution. To simulate 2-min daily prophylactic topical fluoride treatment, the appropriate fluoride gel was brushed onto each individual spring in that treatment group for 2 min. After the springs were treated with the fluoride gel, they were rinsed with PBS and the racks were placed back into the plastic containers filled with PBS solution. Each fluoride treatment occurred once a day for five days per week and was performed at 37±1°C.

---

⁸ Delrin® acetal resin, DuPont™, 1007 Market Street, Wilmington, DE 19898
⁹ GladWare® Containers, Glad®, 1221 Broadway Street, Oakland, CA 94612
Instrumentation and Measurement

A universal testing machine\textsuperscript{10} mechanically tested the springs prior to storage to establish a baseline. The springs were then tested in the same manner following storage in PBS or PBS in combination with APF or NaF after a time period of 1, 4, 8, and 12 weeks. Initial baseline testing was done for both NiTi and SS springs. Any springs with obvious defects or fractures were discarded. A custom made fixture (Fig. 2) consisting of 0.052 mm stainless steel loops was used to suspend the springs between a 50 N load cell and the fixture base. The fixture base was contained in a water bath of dH2O at $37\pm1^\circ$C allowing the springs to be submerged during testing. A preload of 0.1 N was applied to each spring before testing. NiTi springs were extended 9.5 mm for a total length of 21.5 mm at a speed of 15

\textsuperscript{10} Model 5967R4163, Instron Industrial Products, 825 University Ave, Norwood, MA 02062
mm/minute. They were immediately compressed back to their resting length of 12 mm. The SS springs were extended 2.5 mm for a total length of 14.5 mm at a speed of 15 mm/minute. They were immediately compressed back to the original resting length of 12 mm.

Springs were rinsed with dH2O before placement into a 37±1°C water bath where testing occurred. The loaded springs were left on the extension rack and placed directly into the water bath. Load and extension, in newtons (N) and millimeters (mm), was collected during loading and unloading of the springs. A load-extension diagram was generated from the data for NiTi and SS springs (Fig. 3 and 4). Unloading force of the NiTi springs was measured at 3, 6, and 9 mm extension, while unloading force of the SS springs was measured at 1, 1.5, 2 mm extension.

Figure 2. Testing instrument, NiTi closed coil spring.

11 Bluehill® 2 software program, Instron USA, 825 University Ave., Norwood, MA, 02062
Figure 3. Representative load-extension diagram of NiTi closed coil spring. Load phase (blue), unload phase (red). Force measured at 3, 6, and 9 mm extensions during unloading (black dots).
Figure 4. Representative load-extension diagram of SS closed coil spring. Load phase (blue), unload phase (red). Force measured at 1, 1.5, and 2 mm extensions during unloading (black dots).

**Experimental Design**

The experimental design of this in-vitro laboratory study was a two-factor multivariate, randomized design measuring dependent variables (unloading force at 3, 6, 9 mm extension for NiTi and 1, 1.5, 2 mm extension for SS) for each type of spring, respectively. Experimental solution is the first independent variable with three levels: PBS, PBS + APF, and PBS + NaF. The second independent variable is time with four levels: T1 (1 week), T2 (4 weeks), T3 (8 weeks), and T4 (12 weeks). During the 12-week storage period, all springs were exposed to static load at either 9 mm or 2 mm extension for NiTi and SS springs, respectively. Table 3 shows a representative schematic of the research design.
Sample Size

A convenience sample of thirty NiTi springs and thirty SS springs was used for this study. Ten springs of each alloy were assigned to one of the three experimental groups: PBS, PBS + APF, and PBS + NaF.

Scanning Electron Microscopy

Scanning electron microscopy\textsuperscript{12} (SEM) was used to qualitatively examine surface topography of the NiTi and SS springs after the 12-week testing period. Three representative specimens from each experimental group were selected for SEM analysis. Both the end and the middle of each spring were imaged.

Data Analysis

Statistical Analysis

A statistical software program\textsuperscript{13} was used to analyze data. One-factor ANOVAs and one-sided Dunnett’s post hoc tests, $\alpha = 0.01$, were also used to identify the effect of storage condition on the decrease in unloading force with PBS in combination with both fluoride treatments as compared to PBS exposure only with each wire.

SEM Qualitative Topographical Analysis

A qualitative analysis of the scanning electron microscopy images of each alloy from each experimental condition were examined to determine if there is a difference in the surface topography of the closed coil springs as a function of the experimental conditions.

\textsuperscript{12} Philips XL 30 ESEM-FEG, FEI Company, 5350 NE Dawson Creek Drive, Hillsboro, OR, 97124

\textsuperscript{13} SPSS Version 18, 223 S. Wacker Dr., Chicago, IL 60606
This analysis investigated a potential corrosion effect as a result of the exposure to the experimental conditions.

### TABLE 3
**RESEARCH DESIGN**

<table>
<thead>
<tr>
<th>Time</th>
<th>Experimental conditions (n = 10 springs/condition)</th>
<th>Unloading Force at 3, 6, 9 mm for NiTi (N)</th>
<th>Unloading Force at 1, 1.5, 2 mm for SS (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 week</td>
<td>PBS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>PBS + APF</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>PBS + NaF</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 weeks</td>
<td>PBS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>PBS + APF</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>PBS + NaF</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 weeks</td>
<td>PBS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>PBS + APF</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>PBS + NaF</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12 weeks</td>
<td>PBS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>PBS + APF</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>PBS + NaF</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
CHAPTER 3

RESULTS

Mechanical Testing

Listed in tables 4 and 5 are the means and standard deviations of unloading force for each experimental condition over time. Figures 5 and 6 represent means at each time point to better visualize trends over time.

Based on the 1-Factor ANOVA and Dunnett’s post hoc, with 3M NiTi springs there was a significant decrease (p <0.01) in the unloading force at each extension following exposure to both fluoride treatments, but only after 12 weeks. Compared to 3M NiTi PBS group at 3 mm extension, the PBS+NaF group had a 33% decrease in unloading force and the PBS+APF group had a 22% decrease. At 6 mm extension, the PBS+NaF group showed a 23% decrease and the PBS+APF group showed a decrease of 14%. At 9 mm extension, the PBS+NaF group had a 16% decrease and the PBS+APF group had an 11% decrease compared to the PBS group. With the AO NiTi springs, there was a significant decrease in unloading force after NaF exposure at 9 mm extension after 12 weeks. In comparison to the PBS group, the AO NiTi NaF group at 9 mm extension had a decrease of 6% after the 12 week time period. At 3 and 6 mm extension the decrease with NaF was also significant only after 12 weeks. There was a 7% decrease at 3 mm and a 4% decrease at 6 mm compared to PBS after 12 weeks.

Another important observation with the 3M NiTi springs was the percentage decrease in unloading force across time irrespective of storage solution. For example with 3M NiTi PBS group at 9 mm extension the unloading force decreased from 1.21 N to 0.64 N. This
represents a 47% decrease in unloading force. In contrast, this decrease in unloading force across time did not occur with the AO NiTi springs. For the AO NiTi PBS group at 9 mm there was only a 1% decrease in unloading force across time.

However, with SS springs, based on the 1-Factor ANOVA and Dunnett’s post hoc (p>0.01), there was no significant effect of either fluoride treatment on the SS springs at any extension or time point. Both SS springs at 2 mm extension demonstrated some decrease in unloading force over time, 7% and 9% respectively for AO and GAC SS springs

**SEM Qualitative Topographical Analysis**

Representative SEM images are shown in figures 7 and 8. From each experimental group, three representative springs were analyzed after the 12 week testing period. The middle section of each spring was imaged and analyzed at 5,000x. Neither 3M nor AO NiTi closed coil springs exposed to PBS+NaF demonstrated any surfaces changes that were different from the respective PBS only groups (fig. 7a, 7c, 7d and 7f). However, both the 3M and AO NiTi springs exposed to PBS+APF showed some surface mottling and pitting (fig. 7b and 7e) that was not evident on the PBS only groups.

All three of the SS experimental groups from each manufacturer showed no significant surface mottling or pitting suggesting that there were no surface changes associated with either fluoride treatment. Machining lines and oils are visible on each of the springs examined with SEM (fig. 8).
Table 4

Mean and SD of Unloading Force for NiTi Springs

<table>
<thead>
<tr>
<th>Brand</th>
<th>Time</th>
<th>Experimental Condition (n = 10 springs/condition)</th>
<th>Unloading Force (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>3 mm</td>
</tr>
<tr>
<td>AO</td>
<td>1 week</td>
<td>PBS</td>
<td>0.87(0.08)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.86(0.03)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.84(0.04)</td>
</tr>
<tr>
<td>AO</td>
<td>4 weeks</td>
<td>PBS</td>
<td>0.89(0.05)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.87(0.03)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.85(0.03)</td>
</tr>
<tr>
<td>AO</td>
<td>8 weeks</td>
<td>PBS</td>
<td>0.87(0.06)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.87(0.03)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.83(0.03)</td>
</tr>
<tr>
<td>AO</td>
<td>12 weeks</td>
<td>PBS</td>
<td>0.87(0.05)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.86(0.03)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.81(0.02)*</td>
</tr>
<tr>
<td>3M</td>
<td>1 week</td>
<td>PBS</td>
<td>0.86(0.07)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.77(0.08)</td>
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<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.81(0.07)</td>
</tr>
<tr>
<td>3M</td>
<td>4 weeks</td>
<td>PBS</td>
<td>0.36(0.14)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.43(0.16)</td>
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<td>PBS + NaF</td>
<td>0.38(0.15)</td>
</tr>
<tr>
<td>3M</td>
<td>8 weeks</td>
<td>PBS</td>
<td>0.29(0.10)</td>
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<td></td>
<td>PBS + APF</td>
<td>0.43(0.08)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.25(0.07)</td>
</tr>
<tr>
<td>3M</td>
<td>12 weeks</td>
<td>PBS</td>
<td>0.27(0.08)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.21(0.04)*</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.18(0.02)*</td>
</tr>
</tbody>
</table>

*There was a significant (p<0.05) decrease in unloading force noted after 12 weeks with AO NiTi springs exposed to PBS + NaF and with 3M NiTi springs exposed to both PBS + APF or PBS + NaF. Significant decreases in unloading force are identified by asterisks.
Figure 5. NiTi springs mean unloading force at each time point and each extension. a. 3M NiTi; Omegas represent significant effects of NaF and APF on unloading force at each extension after 12 weeks, b. AO NiTi; Asterisks represent significant effect of NaF on unloading force at each extension after 12 weeks.
TABLE 5
MEAN AND SD OF UNLOADING FORCE FOR SS SPRINGS

<table>
<thead>
<tr>
<th>Brand</th>
<th>Time</th>
<th>Experimental Condition (n = 10 springs/condition)</th>
<th>Unloading Force (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>1 mm</td>
</tr>
<tr>
<td>AO</td>
<td>1 week</td>
<td>PBS</td>
<td>0.74(0.11)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.71(0.11)</td>
</tr>
<tr>
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<td>PBS + NaF</td>
<td>0.62(0.15)</td>
</tr>
<tr>
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<td>0.75(0.11)</td>
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<tr>
<td></td>
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<td>PBS + APF</td>
<td>0.71(0.09)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.68(0.08)</td>
</tr>
<tr>
<td>AO</td>
<td>8 weeks</td>
<td>PBS</td>
<td>0.68(0.11)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.66(0.09)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.64(0.06)</td>
</tr>
<tr>
<td>AO</td>
<td>12 weeks</td>
<td>PBS</td>
<td>0.64(0.14)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.64(0.07)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.63(0.05)</td>
</tr>
<tr>
<td>GAC</td>
<td>1 week</td>
<td>PBS</td>
<td>0.69(0.04)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.63(0.09)</td>
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<tr>
<td></td>
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<td>PBS + NaF</td>
<td>0.71(0.06)</td>
</tr>
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<td>GAC</td>
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<td>PBS</td>
<td>0.59(0.04)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.55(0.05)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.58(0.02)</td>
</tr>
<tr>
<td>GAC</td>
<td>8 weeks</td>
<td>PBS</td>
<td>0.56(0.04)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.56(0.04)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.57(0.03)</td>
</tr>
<tr>
<td>GAC</td>
<td>12 weeks</td>
<td>PBS</td>
<td>0.55(0.03)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + APF</td>
<td>0.55(0.04)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PBS + NaF</td>
<td>0.57(0.02)</td>
</tr>
</tbody>
</table>

*At all extensions and across time, there was no significant effect of either fluoride treatment on either SS wire.*
Figure 6. SS springs mean unloading force at each time point and each extension.  a. AO SS, b. GAC SS. With either wire, there was no significant effect of either fluoride treatment on unloading force at all extensions across time.
Figure 7. Representative SEM images at 5k magnification of NiTi closed coil springs. Springs were imaged after exposure to PBS only or PBS in combination with APF or NaF after 12 weeks: a. 3M; PBS, b. 3M; PBS+APF, c. 3M; PBS+NaF, d. AO; PBS, e. AO; PBS+APF, f. AO; PBS+NaF.
Figure 8. Representative SEM images at 5k magnification of SS closed coil springs. Imaged after exposure to PBS only or PBS in combination with APF or NaF after 12 weeks: a. GAC; PBS, b. GAC; PBS+APF, c. GAC; PBS+NaF, d. AO; PBS, e. AO; PBS+APF, f. AO; PBS+NaF.
Orthodontists frequently utilize topical fluorides in cases where demineralization and caries are a threat. As previously reported, these fluorides can cause mechanical property changes as well as topographical changes to NiTi and SS alloys (Watanabe and Watanabe 2003; Kaneko et al. 2004; Walker et al. 2005; Walker et al. 2007). Many studies have concentrated on the effects of fluoride on archwire properties (Walker et al. 2005; Walker et al. 2007; Kao and Huang 2010; Lee et al. 2010). However, no studies have evaluated the effects of a simulated oral environment in addition to topical fluoride on coil springs. This investigation not only looked at the effects of a simulated oral environment with exposure to topical fluoride, but did so while the coil springs were under static load to simulate a functional application. After each time period, the NiTi and SS closed coil springs were analyzed. Our study focused on the unloading mechanical properties since this is the force that causes tooth movement.

According to the results of our study, significant effects were seen on the unloading properties of the NiTi closed coil springs from both manufacturers. The 3M NiTi closed coil springs showed a significant decrease in unloading properties at each extension following both fluoride treatments, APF and NaF, after 12 weeks. Similar results were found in a study by Walker et al. (2005) where a significant decrease in unloading properties of NiTi archwires was seen when exposed to NaF and APF solutions (Walker et al. 2005). The decrease in unloading properties of the NiTi coils springs could be the result of hydrogen
embrittlement and fatigue resistance from static load. It has been shown that protective oxide layers crack and degrade as NiTi alloys are exposed to stress and halides such as Cl and F (Nakagawa et al. 1999; Yokoyama et al. 2003; Liu et al. 2007). As the oxide layer is lost, hydrogen can diffuse through the interstitial sites and form hydride phases such as titanium hydride. These body-centered tetragonal structures are thought to be the cause of degradation in mechanical properties (Yokoyama et al. 2001). Due to break down of the oxide layer and the load applied to the alloy, hydrogen absorption not only occurs more readily but is accelerated (Yokoyama et al. 2001). The results of Yokoyama et al. (2004) also imply that hydrogen-related degradation of mechanical properties of NiTi archwires occurs when exposed to neutral and acidulated phosphate fluorides (Yokoyama et al. 2004). This is in contrast to the findings of a recent unpublished study by McGivern (2012) where only a significant decrease was found in the PBS+APF groups. This difference could be due to different manufacturing processes employed by different companies. It has been reported that during manufacturing processes such as electropolishing, NiTi alloys can absorb hydrogen (Kaneko et al. 2004). This could also account for the decrease in unloading force across time regardless of storage solution that was seen in the 3M NiTi coil springs.

However, the AO NiTi springs only showed a significant decrease in unloading properties with the PBS+NaF group after 12 weeks. A significant effect on the AO NiTiAPF group may have been seen if sample size was larger or if the springs were tested again after a longer measurement of time.

Another interesting observation was the percentage decrease in unloading force over time of the 3M NiTi springs irrespective of the storage solution. This could be attributed to
stress relaxation of the springs. As previously discussed, several studies have found that NiTi coil springs exhibit a decrease of force over time. Angolkar et al. found a 14.6% decay of force at the end of 28 days in NiTi closed coil springs. Bourke et al. (Bourke et al. 2010) and Brauchli et al. (Brauchli et al. 2011) also found a decrease in unloading force overtime resulting in a more linear load-deflection curve suggesting behavior similar to SS springs. Since this same decrease was not seen with the AO NiTi springs, it could also be due to a difference in the manufacturing process.

In contrast to other studies examining the effects of fluoride on mechanical property degradation, our investigation showed no significant decrease in unloading force for the SS springs from either manufacturer. These results are in contrast to previous studies in which mechanical properties of SS archwires were shown to decrease when exposed to artificial saliva and fluoride agents (Kaneko et al. 2004; Walker et al. 2007). It has been reported that the passive film can be enhanced by the synergistic effects of high nitrogen and molybdenum content (Oh et al. 2004). Therefore, our results could indicate that the composition of the SS springs contains higher amount of nickel and molybdenum resulting in increased corrosion resistance.

Force decay over the 12 week time period for the AO and GAC SS springs was minimal compared to the 3M NiTi springs at only 7% and 9%, respectively. Some stress relaxation and loss of force was not surprising as this has been reported in previous studies. Angolkar found a 20-21% loss of force in SS closed coil springs after 28 days (Angolkar et al. 1992). Hazel et al. (Hazel et al. 1984) also reported stress relaxation in SS archwires at 37°C.
**SEM Topographical Analysis**

In addition to mechanical testing, SEM imaging was done for qualitative evaluation. SS springs from both manufacturers showed clearly visible machining lines and oils on the surface after the 12 week testing period for all storage conditions which correlates with the results of no significant decrease in unloading force with either fluoride treatment.

The 3M and AO NiTi springs exposed to PBS+NaF showed similar surface topography as springs exposed to PBS only, with some machining oils and lines still visible. However, the lack of NiTi surface changes with PBS+NaF exposure does not correlate with the mechanical property results. For example, despite no surface changes, the AO PBS+NaF springs did have a significant decrease in unloading properties after 12 weeks exposure. With the 3M NiTi springs, there was a significant decrease after exposure to both fluoride treatments, but only the PBS+APF springs showed surface mottling and pitting that suggests loss of surface material potentially due to corrosion. Although we did see surface changes with the 3M and AO APF groups, only the 3M NiTi PBS+APF springs showed a significant decrease in unloading properties where the AO PBS+APF springs did not. Therefore, based upon our results, we cannot directly link NiTi surface topography changes and mechanical property degradation. Since changes are seen in surface topography with the PBS+APF groups, this could suggest that the condition may worsen over time. Mechanical results may have been different if the springs were tested again after a longer measurement of time.

**Study Limitations**

Our current in vitro study investigated the effects of topical fluoride solutions on the mechanical properties of NiTi and SS closed coil springs under static load. Although we
tried to replicate the conditions of the oral environment, this still poses limitations as the oral cavity is a dynamic and constantly changing environment. External variables, such as diet and fluctuating pH, could also have a significant effect on mechanical properties. Different compositions of alloys and different manufacturing processes employed by various manufacturers may also play a role in mechanical properties of NiTi and SS springs. As discussed previously, wire size, lumen size and pitch can change also change mechanical properties. In our study we only focused on springs that were one length and size. In addition, the topical fluorides used had different fluoride concentrations and different pH. It could have been beneficial to use fluoride solutions that had equal fluoride concentrations.

**Clinical Significance**

Although our study found some significant decreases in unloading force which can ultimately affect tooth movement and time involved, the results may not be clinically significant. There is such a small difference in force values between the significantly affected groups and the PBS only control groups that it may not affect treatment outcomes. For example, at 9 mm extension after 12 weeks, the AO NiTi PBS+NaF group demonstrated an unloading force of 2.10 N compared to the PBS only group which showed 2.23N unloading force. While this decrease was considered statistically significant, it is unlikely that this difference would significantly slow clinical treatment progress.

Another important consideration is how these springs are marketed by the manufacturers. AO NiTi springs are said to be variable force springs. Therefore, it should not be surprising if the springs do not exhibit a continuous force upon deactivation. However, the 3M NiTi springs are stated to be superelastic by the manufacturer. If these
springs are truly superelastic, then they should have the same force at each extension. In our study and other studies, NiTi springs have exhibited a linear load-deflection diagram suggesting that they behave like SS springs. As importantly, this particular NiTi spring also demonstrated high force decay over time, which again suggests behavior similar to SS springs. In fact, the force decay over time for the 3M NiTi springs was 47% as compared to 7 and 9% force decay for the SS springs. Since greater force decay leads to less efficient tooth movement, this specific NiTi spring would need to be changed more frequently. A clinician, therefore, should not presume that a NiTi spring is superelastic (providing similar force at varying extensions) or providing stable force over time. This is important because there is a large cost difference between the two different alloy springs. The NiTi closed coil springs cost around $10 per spring, whereas, the SS closed coil springs cost around $0.06 per spring. If NiTi and SS springs exhibit similar load-deflection curves and you are not gaining the advantage of superelastic properties that are stable over time, then based on cost alone, it may be more efficient to use SS springs. Our study also found no significant decreases in unloading properties for the SS springs when exposed to either fluoride agent. This could be another reason to consider the use of SS over NiTi springs in clinical practice with patients requiring prescribed fluoride.

**Future Investigations**

Since our study did not see significant decreases in unloading force for any of the SS spring groups or for the AO NiTi PBS+APF group after 12 weeks, it would be interesting to see the mechanical and topographical changes beyond a 12 week time period. Studying these
effects over a longer period of time may give more insight to the potential correlation between surface topography and mechanical properties.

Hydrogen embrittlement has been shown to greatly affect mechanical properties of both NiTi and SS alloys (Rogers 1968; Hirth 1980; Yokoyama et al. 2003). Yokoyama et al. (Yokoyama et al. 2011) reported that addition of small amounts of hydrogen peroxide to acid fluoride solutions inhibits hydrogen embrittlement. It appears that the hydrogen peroxide changes the surface condition of the alloy creating a high oxidation capability and subsequently inhibiting hydrogen absorption. Future studies could apply this theory when testing coil springs treated with fluoride to see if mechanical properties are still affected.

It would also be beneficial to test fluoride treated coil springs that are under a dynamic changing load and not static load. This would more accurately reflect the environment in which the springs are used. Springs are used to move teeth and, therefore, are continuously changing length. A more clinically relevant study could investigate the changes in mechanical properties of springs that are used in vivo. Perhaps, springs that are used to retract canines in a patient who has been prescribed fluoride treatment.
CHAPTER 5
CONCLUSION

1. With the 3M NiTi spring, there was significant a decrease in unloading force at each extension after 12 weeks exposure by both APF and NaF, while the AO NiTi spring demonstrated a decrease at all extensions after 12 weeks but only with NaF. In contrast, the unloading force of both brands of SS springs was not negatively affected by either fluoride agent.

2. Qualitative surface topography changes (pitting and surface material loss) were seen with both brands of NiTi springs after 12 weeks exposure to either NaF or APF. In contrast, there were no surface changes noted with both brands of SS springs after 12 weeks exposure to either fluoride agent.
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