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INFLUENCE OF FLUORIDE AND STRESS ON THE MECHANICAL PROPERTIES OF NICKEL-TITANIUM COILS

by

Jennifer Roloff, DDS

A Thesis submitted to the Faculty of the Graduate School, Marquette University, in Partial Fulfillment of the Requirements for the Degree of Master of Science

Milwaukee, Wisconsin

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ABSTRACT INFLUENCE OF FLUORIDE AND STRESS ON THE MECHANICAL PROPERTIES OF NICKEL-TITANIUM COILS

Jennifer Roloff, DDS

Marquette University, 2015

Objective:

The objective of this study was to determine the influence of fluoride and stress on the mechanical properties of nickel-titanium open coil springs.

Materials and Methods:

Sentalloy nickel-titanium open coil springs (15 mm length) were divided into five groups. A control group was tested mechanically in as-received condition. The other four experimental groups were exposed to artificial saliva (Fusayama-Meyer solution) at 37° C for 12 weeks in a 2x2 factorial design with fluoride exposure and stress (compression) as the two factors. Those springs which were under compression were held at 5 mm in length for the entirety of the testing period. Fluoride exposure was two minutes per day in a 1500 ppm NaF solution. In each group, n=10. The experimental groups were then tested mechanically via compression on a universal testing machine following the 12-week exposure period to measure force characteristics.

Results:

No significant differences in activation force were seen between the coil groups, whereas significant (p<0.05) differences were observed in deactivation forces between the control and compressed/fluoride group and also between the control and the non-compressed/non-fluoride group. Ranges of mean deactivation force values between all groups were less than only 6 and 14 grams of force at 4 mm and 8 mm respectively.

Conclusions:

Clinically relevant daily fluoride exposure (1500 ppm) and continuous compression for twelve weeks did not result in clinically significant changes in force values of the nickel-titanium open coil springs, indicating they are likely to deliver consistent forces during the course of treatment.

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Jennifer Roloff, DDS

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CHAPTER 1 INTRODUCTION

Coil springs are a commonly utilized auxiliary in orthodontics. They are used in conjunction with archwires and brackets to redistribute space within the dental arch. While coil springs have traditionally been made of stainless steel, the 1980s saw the introduction of coil springs fabricated from the novel nickel-titanium alloy (Miura et al., 1988). The stainless steel coil springs had been successful in opening and closing space, though testing determined that the recommendations for the amount of activation in these springs lead to a much greater force than that which would be needed or desired for orthodontic tooth movement (Chaconas et al., 1984). Force levels generated by activating the stainless steel coil springs by one third of their original length as directed lead to forces ranging from 270-540 grams which was a force within the orthopedic range, and not one which was desirable for orthodontic tooth movement (Ryan, 1995). As such, the development of a new material with the potential to produce the light and continuous forces desired in orthodontics was considered to be a great advance.

Originally developed for use in the United States Space Program, the nickeltitanium alloy soon found a greater use in the specialty of orthodontics (Proffit, 2007). Stainless steel coil springs traditionally showed a linear load-deflection relationship, whereas the springs fabricated from nickel-titanium demonstrated a constant, light and continuous unloading force (Ryan, 1995). This desirable unloading force characteristic stems from the two unique properties of nickel-titanium: superelasticity and shape memory. These characteristics are directly related to the two unique phase structures of nickel-titanium, austenite (high temperature and low stress) and martensite (low temperature and high stress), and the relatively low transition temperature between the two (Proffit, 2007). Nickel-titanium also possess the unique ability to avoid plastic deformation unless undergoing heat treatment. As such, it has the ability to produce the required forces over a very wide range of activations, making it an extremely versatile material (Proffit, 2007).

These unique properties made the nickel-titanium alloy a logical choice for use in the fabrication of auxiliaries such as coil springs. Both open and closed coil springs are currently fabricated, being used to open and close space within the dental arches, respectively. To date, in vitro studies have shown springs of both types to reliably produce the light and continuous forces that would be expected (Ryan, 1995). These springs are commercially available in various lengths, with multiple options in wire diameter, pitch of the coils and lumen size, all of which have been shown to have an effect on the activation forces required and the tooth movement forces which are generated (Bourke et al., 2010).

While in vitro studies have shown promising results depicting the capabilities of nickel-titanium coil springs, it is important to remember that the oral environment is a dynamic one, with numerous variables constantly being introduced (saliva, various chemicals and compounds from the patient's diet and hygiene practices along with fluctuating temperatures). Han et al. (1993) tested the force characteristics of nickel-titanium coil springs which had been held in artificial saliva at body temperature for 6 weeks, and found that the simulated oral environment did not affect the forces generated.

Further studies examined the effects of various food substances and temperature extremes, all finding that the nickel-titanium coil springs maintained their desirable

properties regardless of environment in which they were placed (Nattrass et al., 1998 and Espinar et al., 2013). However, very few studies aimed to directly examine the effect of fluoride on the force characteristics of coil springs. Based on the most recent Public Health Recommendation from the Centers for Disease Control, the recommended concentration of fluoride in public water sources is 0.7 ppm, down from the previously recommended 0.7-1.2 ppm (U.S. Department of Health and Human Services Federal Panel on Community Water Fluoridation, 2015). Higher concentrations of fluoride are present in dentifrices, most of which disclose a concentration of 1000-1500 ppm, with higher concentrations only being available by prescription (Rozier et al., 2010). As such, it is difficult to avoid some contact with fluoride on a daily basis in most cases.

In 2013, Walker et al. examined the effect of stress and fluoride prophylactic agents on the mechanical properties of nickel-titanium closed coil springs. The fluoride agents studies were high concentration, in-office prophylactic agents, and it was found that the unloading forces were negatively affected, potentially leading to greater treatment time. The authors postulated that the adverse effects seen in the mechanical properties of the coils stemmed from an increase in corrosion of the nickel-titanium alloy in the presence of fluoride.

In response to these findings, this study aims to determine the effect of stress and non-prescription strength fluoride exposure on nickel-titanium open coil springs, with the hypothesis that this lower fluoride concentration, meant to mimic a typically daily exposure, will not negatively affect the forces generated.

CHAPTER 2 LITERATURE REVIEW

Incidence of Malocclusion and Dental Crowding

The prevalence of malocclusion in today's society is several times greater than it was only a few hundred years ago. Evolutionary trends, as discovered through fossil records, have shown a decrease in tooth size and number, as well as a decrease in overall jaw size during the last 100,000 years (Proffit, 2007). Should this tooth and jaw size reduction be unmatched, then dental crowding will result. Historically, such dental crowding was rare, likely due to the high demands on the oral apparatus in regards to mastication of primitive foods – teeth were simply of greater importance in a primitive diet (Proffit, 2007). It has been postulated that malocclusion has become more prevalent due to the introduction of our modern way of life, including much softer foods, though direct correlations are hard to pinpoint.

Based on the data from the NHANES III study, only 35% of adults have wellaligned mandibular incisors, while 15% of adults have such severe incisor irregularity that it is socially and functionally detrimental (Proffit, 1998). In such instances of severe dental crowding, part of the orthodontic treatment plan often calls for the extraction of permanent teeth to create room for the alignment of those remaining. Historically, extraction rates for the treatment of crowding and malocclusion have ranged from 30% in 1953, to 76% in 1986, to 28% in 1993 (Proffit, 1994). These extraction rates, however, vary greatly among individual practitioners, with estimates ranging from 5% to 87% of total patients within a given practice (Weintraub et al., 1989). If extractions are pursued, the clinician is then left with the task of closing the extraction space. If extractions are foregone in a case with any sort of crowding present, the clinician must then make space within the arches to align the teeth, often by distalizing specific teeth within the dentition. While there are many auxiliaries which can be used to close or create space, there are few that can be applied in both situations. Nickel-titanium coil springs, however, are one such auxiliary that can be used with duality, depending on the type of coil used (i.e. open or closed).

Nickel-Titanium Alloy

The nickel-titanium (NiTi) alloy was initially developed for use within the United States Space Program, though its properties were soon found to be very beneficial within the field of orthodontics (Proffit, 2007). Most uniquely, NiTi possesses shape memory and superelastic properties which are not found in any other dental material to date (Proffit, 2007). Such properties make the alloy ideal for use in archwires during the initial leveling and aligning phase, as the superelastic nature of the material allows for constant, light forces which are most desirable in orthodontics.

These desirable properties of nickel-titanium stem from its two crystallographic structures, martensite (low temperature or high stress phase) and austenite (high temperature or low stress phase), and the fluctuations between them (Schneevoigt et al., 1999). The shift from one phase to the other can be brought about by temperature or stress, and for the case of a stress-induced change can be visualized as the points at which the stress/strain force diagram plateaus. Additionally, the nickel-titanium alloy demonstrates a low Young's Modulus of elasticity, a yield strength of 8% and a typical hysteresis loop – all desirable properties in the fabrication of an ideal orthodontic material (Bourauel et al., 1997).

The steady force plateau during the unloading phase is what has made nickel titanium such a desirable material for orthodontic purposes. To date, the vast majority of NiTi alloys are adjusted in their composition to accommodate for the average temperature of the mouth $(37^{\circ}C)$ – the phase transformation temperatures are strategically set so as to make optimum use of the superelastic properties of the alloy (Schneevoigt et al., 1999). The exertion of a nearly constant force over a wide range of deflection and activation has made the nickel titanium alloy extremely valuable in the field of orthodontics.

Nickel-Titanium Coil Springs

It was not until 1988 that Miura et al. conducted a study in which their Japanese NiTi wires were coiled into springs, and a new use for the alloy emerged. Both open and closed coils were fabricated, and it was found that both types demonstrated the maintenance of the desirable properties of the NiTi alloy when placed under compression and tension respectively (Miura et al., 1988). Variable force levels and ranges of superelastic activity were achievable based on the lumen size, wire diameter, austenite/martensite transformation temperatures and pitch of the coils (Miura et al., 1988).

Since their introduction, NiTi coil springs have proved very beneficial as a means of intra-oral force delivery for the creation or elimination of space. Open coil springs have spacing between each coil, and are utilized to open space when compressed between two teeth; closed coils have no spacing between each coil and are thus activated in tension with the goal of closing space (Ryan, 1995). Both types of NiTi springs demonstrate a light, constant and continuous unloading force (Miura et al., 1988), while traditional coils fabricated from stainless steel do not. During unloading, NiTi coils have a plateau area, indicating light and continuous forces present over an extended period of time, whereas stainless steel coils show a very high initial force with rapid decline (Ryan, 1995). The light, continuous forces allow for more efficient and gentle tooth movement, greatly reduced risk of periodontal trauma and root resorption, increased patient comfort with low compliance required, and reduced chair time due to long lasting application (Ryan, 1995). With such ideal properties and force characteristics, NiTi coil springs have come of use in numerous facets within orthodontics.

Applications: Nickel-Titanium Open Coil Springs

Perhaps the most common use of NiTi open coil springs is to open up space for a tooth within an arch so as to bring it into alignment. Utilizing these springs in this manner generates lower, more physiologically compatible forces than in the use of stainless steel coil springs (Steinbach et al., 2006). The reactivation intervals can also be extended when NiTi springs are used as they maintain their activity over greater periods of time. Such a finding demonstrates how an auxiliary spring fabricated from NiTi, rather than stainless steel could allow for less chair time due to less of a need for return visits for reactivation.

Additionally, NiTi open coil springs can be of use in extraction cases to assist in distalizing the canine prior to en masse retraction of the four-tooth anterior segment. This technique is of particular benefit when there is also a blocked out lateral, which allows the coil spring to serve as a means of redistributing the space (Kumar et al., 2009).

During canine retraction, bodily distal movement is most ideal, however utilizing an open coil spring too effectively "push" the canine to the distal has the potential to lead to a greater tipping movement, especially in an adult population (Kumar et al., 2009). Careful monitoring of the level of activation allows for greater control of this undesirable tipping, and a greater overall translatory movement.

In cases of Class II malocclusion, molar distalization is one way of achieving a Class I molar relationship. The utilization of extra-oral forces (i.e. headgear) have been shown to be effective in distalizing molars over the years, but are heavily reliant on patient compliance (Erverdi et al., 1997). In an effort to minimize the compliance required and to maximize results, open coil springs can be inserted anterior to the first molar brackets/bands and be activated to gain the desired distalization. Utilizing a compressed NiTi open coil spring, maxillary molars can be distalized 1-2mm per month and moved into an ideal Class I relationship, though a portion of the movement is credited to distal tipping of the molar and anchorage loss in the premolars (Ozturk et al., 2005). This tipping can then be rectified with uprighting bends during the final, complete phase of orthodontic treatment to maximize the stability of the result.

Applications: Nickel-Titanium Closed Coil Springs

The most common use of a NiTi closed coil spring is in the closing of extraction spaces. This auxiliary is ideal for this type of retraction due to its nearly constant unloading force due to the superelastic properties of the material (Ryan, 1995). According to the guidelines set forth in the Sentalloy Coil Spring Information Booklet (GAC), these coils can be activated from 3mm to 15mm with no deformation or change in forces produced, making it ideal for a situation where there will be longer periods of time between patient visits. In the very first studies on canine retraction with NiTi coil springs, it was found that complete retraction could be achieved in five months with only a single activation of the spring at the time of placement (Miura et al., 1988).

Additionally, in some studies NiTi closed coil springs have been found to produce faster tooth retraction than elastics or elastic retraction modules (i.e. powerchains). In comparing NiTi closed coil springs with powerchains, Samuels et al. found the springs closed spaces at a rate of 1.1 mm/4 weeks, while the powerchains closed spaces at a rate of 0.7 mm/4 weeks. When comparing traditional intra- or interarch elastics with NiTi closed coil springs, the retraction rate was found to be 2 mm/4 weeks for the spring and 1.1 mm/4 weeks for the elastics (Ryan, 1995). While this does indeed demonstrate a clinical significance in favor of the coil springs, it must also be remembered that elastics rely greatly on patient compliance, which is a large factor contributing to this difference.

More current research which has been done on the same topic demonstrates less of a significant difference between rates of space closure. Bokas and Woods (2006) did find that NiTi springs had a mean rate of space closure greater than that of elastomeric chains (1.85 mm/month versus 1.68 mm/month), however it was not of statistical or clinical significance. The mean rates of anchorage loss were also measured for both auxiliaries and were not found to be statistically significant. Thus it was stated that, with regular reactivation of the elastomeric chains, they can produce clinically similar results in similar amounts of time.

Barlow et al. (2008) conducted a systematic review on sliding mechanics and the auxiliaries used to close extraction spaces. Of the modes of space closure which were

examined, elastomeric chains and NiTi closed coil springs were found to be the most efficient, and closed spaces at similar rates. Less efficient means of space closure included active tie backs and interarch elastics. The greatest advantage of NiTi closed coil springs was found to be their long lasting activity, requiring no reactivation during space closure if initially activated to the appropriate length.

Force Characteristics: Nickel Titanium Open Coil Springs

The forces exerted by a given NiTi open coil spring are not merely dependent on the amount of pressure which the clinician places the coil under. Other variables, such as the lumen size, wire diameter and pitch of the coils also affect the forces generated. The greater the diameter of the wire, the greater the force produced. If the wire size is kept constant and the lumen size increased, the force will decrease. The larger the pitch of the wire within the spring, the greater the force which can be generated at a given activation (Chaconas et al., 1984).

While these characteristics of the spring itself do have a significant effect on the forces produced, perhaps the most important component of these springs is the nickeltitanium alloy from which they are made. Traditionally, superelastic NiTi springs would be expected to show a force plateau at the point where its molecular structure shifts from austenite to martensite during activation, and vice versa in deactivation (Brauchli et al., 2011). It is the energy from this transformation which is released during deactivation in a continuous manner, forming the plateau seen in the force diagram. In in vitro testing of numerous brands of NiTi open coils, Brauchli et al. (2011) discovered that most of the springs were showing a linear force deflection when compressed to 25% of the original coil length. In compressing the wire further to 50% of its initial length, the force plateau was seen, indicating that NiTi open coils will likely only demonstrate their desirable superelastic properties when activated at or beyond 50% of their original length.

An additional advantage of utilizing a NiTi open coil spring to create space is that it has been shown to require fewer reactivations to gain the desired space opening. In an in vitro study conducted by von Fraunhofer et al. (1993), it was found that with a single initial activation, a NiTi open coil spring has the ability to generate 7 mm of tooth movement. This minimal need for reactivation is desirable in that it saves chair time in an orthodontic office.

Force Characteristics: Nickel-Titanium Closed Coil Springs

In an extraction case, a force of 150-160 grams of force has been recommended for initial canine retraction into the premolar space (Anglokar et al., 1992). In initial in vitro testing, Miura et al. (1988) recommended extending the NiTi closed coil springs one half to one third of the original length to generate 300 grams of force. This increase in initial force generated, as compared to the force which is generally accepted as necessary, was in compensation for the anticipated amount of force decay within the coil over time.

Further studies regarding amount of activation required for canine retraction found that extension of 5-6mm is adequate to generate 150-160 grams of force (Anglokar et al., 1992). No greater force was required for retraction as the coils demonstrated superelasticity, maintaining their force levels over a long range, even as the space continuously closed. While it is possible to activate the coils beyond the 50% maximum recommendation, it does not provide any long term benefit. In a study comparing light versus heavy forces of NiTi closed coil springs to retract canines, Yee et al. (2009) found that greater activation and greater forces did lead to an increased rate of canine retraction initially, but also lead to greater loss of anchorage. Thus, heavier forces applied to the springs lacked clinical benefit; light constant forces exerted by the closed coil springs were of greatest benefit long term.

Commercially available NiTi closed coil springs are currently available in different lengths and with different force generation capabilities. It has been found, however, that there is no real advantage in using a 200 gram spring over a 150 gram spring as long as activation is done appropriately for the given situation (Barlow and Kula, 2008). Regardless of which length/force NiTi closed coil spring used, they have been shown to be more efficient than interarch elastics or active ligatures in closing extraction spaces. To date, they provide the closest means to the "ideal" light and continuous orthodontic force (Barlow, 2008).

Factors Affecting Desired Forces

While NiTi coils have been shown to maintain their desirable superelastic properties across a wide range of activations, it is important to remember that the actual activations will take place within the dynamic oral environment. Saliva, various chemicals and compounds from the patient's diet and hygiene practices, along with fluctuations in temperatures all contribute to the state of flux within a patient's mouth. Initial in vitro testing to address these variables involved keeping the coils under constant stress at 37°C (body temperature) in an artificial saliva solution for six weeks. After the experimental period, the coils were tested in compression and tension and it was found that no loss of force had occurred when compared to coils tested straight from the packaging (Han and Quick, 1993).

The most critical factor affecting the forces produced by NiTi coils is temperature. In early testing, a pronounced fluctuation in load values of the NiTi coils was found at temperature reversal points, i.e. transitions from heating to cooling and vice versa. In an extension of that study a few years later, the transition temperature ranges of NiTi open and closed coil springs were analyzed to determine when the coils would or would not be within the superelastic range. Springs were found to initially demonstrate superelastic behavior when the temperature increased and the Austenite Transition Start Temperature (As) was reached (9.7-17.1°C). Superelasticity could be lost resulting in a rapid decrease in forced delivery when the coils were cooled to the Martensite Transition Finishing Temperature (Mf) which ranged from 12.7-6.5°C (Barwart et al., 1999). However, as both the Mf and as temperature ranges were below normal room and body temperature, it was concluded that the superelastic property would be maintained within the oral environment during orthodontic treatment.

Additional studies delved further into analyzing the effects of temperature changes and found that an 18°C increase in temperature lead to a 30% increase in force generated by the NiTi Coil (Espinar et al., 2013). When the temperature was then decreased back to 37°C, the coil recovered to its initial force level. When the temperature was then decreased further to 15°C, the force levels decreased by 46%. Despite these varying ranges of force levels over the temperature range tested, the springs remained in the superelastic range throughout.

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Nattrass et al. (1998) took another step further and tested the effects of other oral environment factors on NiTi coils. In addition to being held in artificial saliva, the coils were also exposed to water, Coke and a turmeric solution to simulate potential dietary influences. Temperature was also varied and force was measured at 10°C, 22°C and 37°C. Only temperature was shown to have any effect on the force characteristics of the NiTi coils, while all factors detrimentally affected other auxiliaries, i.e. elastomeric chains, rubber bands, etc. (Nattrass et al., 1998).

Another interesting contributor to changes in the oral environment which is continually becoming more prevalent is that of fluoride. Daily fluoride exposure is commonplace, while fluoride prophylactic agents are gaining popularity within dentistry and orthodontics. However, fluoride is known to be a corrosive agent, and specifically has the ability to decrease the corrosion resistance of the otherwise relatively corrosionresistant titanium alloys. Once in the oral environment, the NaF breaks down, forming hydrofluoric acid (HF). It is this HF which leads to the breakdown of the thin oxide layer on the surface of the titanium alloys, leading to an increase in corrosion of the alloy, and a negative effect on its mechanical properties (Fragou and Eliades, 2010).

Numerous types of corrosion have been defined, including uniform corrosion, localized corrosion, galvanic corrosion, flow-induced corrosion, crevice corrosion, pitting corrosion and selective corrosion (Akid, 2004). Uniform corrosion is the most common type seen wherein all areas of the metal corrode at a similar rate. Large concentrations of stress in specific areas of a given alloy prevent this type of corrosion from happening. Localized corrosion, in contrast, only occurs locally, and is the most destructive type of corrosion, damaging the material on both the microscopic and macroscopic levels. The rate of metal loss is unpredictable and dependent on numerous other factors. Galvanic corrosion is seen when there is electrical contact between two dissimilar metals via an aqueous electrolyte. Flow-induced corrosion is seen when a gas or electrolyte transports corrosive agents across the metal surface, pulling solid products away. This could lead to localized corrosion if the flow is uneven, leading to grooves, waves and even holes across the corroded surface. Crevice corrosion is a type of localized corrosion occurring in crevices adjacent to the metal. The rate of this type of corrosion depends on the anatomy of the crevice itself, and is induced by differences in oxygen concentration outside of and within the crevice. Pitting corrosion is very similar to crevice corrosion to occur, there must already be surface damage or heterogeneities on the alloy surface which initiates pit formation. Lastly, selective corrosion is a subtype of corrosion which occurs at selective sites within a metal, due to lack of uniformity in the composition of the metal itself (Akid, 2004).

Regardless of which type of corrosion is to occur, the ultimate result is a loss of the metal, which will have a negative effect on the mechanical properties of the metal in question. While titanium has been known to be relatively corrosion resistant, the strong HF acid which is generated within the oral environment as a result of fluoride intake has the ability to break through the outer protective layer of NiTi alloys and lead to corrosion (Fragou and Eliades, 2010). In an effort to examine the mechanical effects of fluorideinduced corrosion on nickel-titanium, Walker et al. (2013) tested NiTi coils after three months of full-time exposure to artificial saliva at 37°C, with an additional two minutes per day of fluoride exposure. Two fluoride solutions were tested – an acidulated fluoride and a neutral sodium fluoride. It was found that the acidulated fluoride, which would be comparable to an in-office fluoride application, lead to a decreased unloading force, likely due to increased corrosive changes (Walker et al., 2013). As such, it was cautioned that topical fluoride could lead to changes which negatively affected the desirable properties of NiTi coils, though more research is needed in this area.

CHAPTER 3 MATERIALS AND METHODS

For this study, fifty Sentalloy 200 gram nickel-titanium open coil springs (Dentsply GAC, Islandia, NY) were tested. Ten springs were kept in as-received condition as a control group, while the remaining forty springs were divided into four test groups where n=10. The springs in all four test groups were kept in a Fusayama-Meyer artificial saliva solution (pH = 5.8) at 37°C to simulate the oral environment. The saliva solution was made with the following composition: KCl (0.4 g/L), NaCl (0.4 g/L), CaCl₂ (0.6 g/L), NaH₂PO₄ (0.690 g/L), Na₂S•9H₂O (0.005 g/L), and urea (1 g/L). Springs were mounted into their exposure apparatuses according to the 2x2 study design shown in Table 1, with stress and fluoride being the two factors studied.

Group 1 (NCNF)	Group 3 (CNF)				
Non-compressed (NC)	Compressed (C)				
No fluoride (NF)	No fluoride (NF)				
n = 10	n = 10				
Group 2 (NCF)	Group 4 (CF)				
Non-compressed (NC)	Compressed (C)				
Daily fluoride exposure (F)	Daily fluoride exposure (F)				
n = 10	n = 10				

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Non-compressed spring set-ups (Fig. 1) held the existing springs at their original 15 mm length, while the compressed springs (Fig. 2) were held at 5 mm in length for the duration of the test period, which totaled 12 weeks. The exposure apparatuses were made with Teflon plates held apart 5 or 15 mm via PVC tubing. The coils were held between the Teflon plates with the guidance of a stainless steel wire painted with nail polish so as to avoid galvanic effects between it and the NiTi coils. The fluoride solution which Groups 2 (NCF) and 4 (CF) were exposed to on a daily basis was a 1500 ppm NaF solution. Daily exposure time totaled two minutes.



Figure 1. Non-compressed (NC) NiTi open coil springs held at 15 mm



Figure 2. Compressed (C) NiTi open coil springs held at 5 mm in length

After 12 weeks of artificial saliva and/or fluoride exposure, all coils were removed from their respective apparatuses and stored separately according to test group. The force characteristics of each coil were then analyzed in compression utilizing a universal testing machine (Instron, Norwood, MA). Each specimen was individually loaded onto the testing apparatus (Fig. 3), which consisted of a stainless steel guiding wire, and a thin aluminum plate with a hole mounted on top of a cylinder. The hole was small enough that it permitted the 0.020" stainless steel guiding wire to pass through, but did not permit the coil spring to pass through. The stainless steel guiding wire was held in place with a Jacobs Chuck. Once in position, each spring was compressed from 15 mm down to 3 mm (Fig. 4) at a rate of 10 mm/minute. The springs were then released at the same rate back to original length so both activation and deactivation forces could be measured.



Figure 3. NiTi open coil on Instron at original 15 mm length



Fig. 4 NiTi open coil on Instron compressed to 3 mm

Utilizing the IBM SPSS Statistics software (Armonk, NY), the raw data was then analyzed using a repeated measures MANOVA with post hoc analysis to compare each test group to the control group using a significance value of p<0.05. Activation and deactivation forces were analyzed separately.

CHAPTER 4 RESULTS

In Table 2 below, the average force values and standard deviation for each coil group across the activation range (1 mm - 12 mm) is shown. Table 3 is a continuation of this, showing the average deactivation forces as the coils were released.

Force (g) 2 3 9 12 Group 1 4 5 6 7 8 10 11 mm Control 108 223 308 338 344 370 397 429 465 502 506 514 ± 6 ± 8 ±9 \pm \pm \pm ± \pm \pm \pm \pm \pm 10 20 27 28 27 25 23 17 30 CF 220 476 510 107 307 341 338 361 399 438 521 539 ± 7 ± 4 ± 7 ± 6 \pm \pm ± ± \pm \pm \pm \pm 21 11 13 16 28 21 24 17 CNF 223 303 343 369 469 497 509 109 336 397 432 518 ± 3 ± 8 ± 6 \pm \pm \pm ± \pm \pm \pm \pm \pm 25 19 15 30 13 18 10 34 13 NCF 110 225 308 346 350 374 407 441 471 505 517 523 ± 3 ± 5 ± 6 \pm \pm \pm \pm \pm \pm \pm \pm \pm 22 25 19 24 17 18 18 33 30 NCNF 114 230 310 341 356 378 408 443 479 519 518 544 ± 4 ± 9 ± 8 ± ± ± ± \pm ± \pm ± ± 22 12 27 29 32 10 20 19 28

Table 2. Activation forces of compressed NiTi open coils

	Force (g)										
Group	11	10	9	8	7	6	5	4	3	2	1
	mm	mm	mm	mm	mm	mm	mm	mm	mm	mm	mm
Control	316	272	235	219	211	207	204	204	212	180	80
	±16	± 16	± 16	± 9	± 6	± 5	± 6	± 6	± 9	± 4	± 7
CF	296	266	228	213	207	204	202	200	209	179	81
	± 18	± 24	± 19	± 19	± 8	± 8	± 8	± 9	± 9	± 6	± 8
CNF	312	269	236	222	211	210	206	205	209	178	81
	± 17	± 16	± 10	± 9	± 11	± 12	± 12	± 13	± 9	± 8	± 9
NCF	307	271	245	227	217	211	206	205	211	179	79
	± 17	± 18	± 13	± 9	± 10	± 9	± 9	± 9	± 9	± 10	± 7
NCNF	307	261	239	223	216	210	208	206	210	184	82
	± 20	± 21	± 17	± 9	± 10	± 11	± 13	± 12	± 11	± 8	± 7

Table 3. Deactivation forces of compressed NiTi open coils

Figures 5-8 graphically demonstrate the forces generated over the activation/deactivation range for each individual test group. In Figure 9, the forces generated by the Control Group (as-received) can be visualized. Finally, Figure 10 compiles all five graphs for comparison. Qualitatively, very little difference is observed in the force vs compression plots between the five groups of coils.



Figure 5. Graph of forces generated by Group 1 (NCNF) coils in activation/deactivation



Figure 6. Graph of forces generated by Group 2 (NCF) coils in activation/deactivation





Figure 8. Graph of forces generated by Group 4 (CF) coils in activation/deactivation







Figure 10. Graph comparing forces generated by all testing groups in activation/deactivation

A repeated measures MANOVA with a post-hoc analysis to compare the force of the four groups against the control were performed. The activation and deactivation analyses are presented separately below.

For the activation (i.e. compression), the result of the within-subject MANOVA is presented in Table 4. The Greenhouse-Geisser statistic for the interaction effect between all five groups (including the control) and the spring compression (1 mm to 12 mm) produced an F-value of 0.896 with a p-value of 0.656. This means that there is no difference between the five groups. The plot of marginal means are given in Figure 11. Since there is no difference between the groups (including control), no post hoc analysis results are presented.



Figure 11. Graphic plot of marginal means during activation across all test groups

		Type III Sum				
Source		of Squares	df	Mean Square	F	Sig.
compress	Sphericity Assumed	8671207.783	11	788291.617	2918.075	.000
	Greenhouse-Geisser	8671207.783	5.233	1656893.301	2918.075	.000
	Huynh-Feldt	8671207.783	6.530	1327929.439	2918.075	.000
	Lower-bound	8671207.783	1.000	8671207.783	2918.075	.000
compress *	Sphericity Assumed	10095 353	44	229 440	849	743
Group		10000.000		220.440	.045	.740
	Greenhouse-	10095 353	20 934	482 255	849	656
	Geisser	10000.000	20.001	102.200	.010	.000
	Huynh-Feldt	10095.353	26.119	386.507	.849	.681
	Lower-bound	10095.353	4.000	2523.838	.849	.502
Error(compress)	Sphericity Assumed	133719.783	495	270.141		
	Greenhouse-	100710 700	005 504	507.004		
	Geisser	133719.783	235.504	567.804		
	Huynh-Feldt	133719.783	293.844	455.070		
	Lower-bound	133719.783	45.000	2971.551		

Table 4. Activation results of within-subject MANOVA

For the deactivation (decompression), the repeated measures MANOVA is presented in Table 5. The Greenhouse-Geisser statistic for the interaction effect yielded an F-value of 1.869 with a significant p-value of 0.027 (p<0.05). Thus, there is a significant difference between the groups. The post hoc analyses yielded a significant difference between the control and the CF group, and between the control and the NCNF group. The plots of marginal means are presented in Figure 12. Although rather slight, greater ranges of mean deactivation forces were observed at greater compression levels. For instance, ranges of mean deactivation force values between all groups were less than 6 and 14 grams of force at 4 mm and 8 mm, respectively. Those force values are less than the standard deviations within the groups at those compression amounts.

		Type III Sum		Mean		
Source		of Squares	df	Square	F	Sig.
Deactivate	Sphericity Assumed	6132828.171	11	557529.834	3646.029	.000
	Greenhouse- Geisser	6132828.171	3.910	1568366.090	3646.029	.000
	Huynh-Feldt	6132828.171	4.710	1302193.467	3646.029	.000
	Lower-bound	6132828.171	1.000	6132828.171	3646.029	.000
Deactivate *	Sphericity Assumed	12572.932	44	285.748	1.869	.001
Group	Greenhouse- Geisser	12572.932	15.641	803.828	1.869	.027
	Huynh-Feldt	12572.932	18.838	667.408	1.869	.018
	Lower-bound	12572.932	4.000	3143.233	1.869	.132
Error(Deactivate)	Sphericity Assumed	75692.555	495	152.914		
	Greenhouse- Geisser	75692.555	175.965	430.157		
	Huynh-Feldt	75692.555	211.933	357.154		
	Lower-bound	75692.555	45.000	1682.057		

Table 5. Deactivation results of within-subject MANOVA



Figure 12. Graphic plot of marginal means during deactivation of all test groups

CHAPTER 5 DISCUSSION

Nickel-titanium open coil springs have been established as an optimal auxiliary for use in the specialty of orthodontics. While the traditional methods of creating space remain effective, studies have shown that nickel-titanium open coil springs possess the ability to produce the light and continuous forces which are most desirable in orthodontics. In this study, the mechanical properties of nickel-titanium open coil springs were analyzed after they were kept in artificial saliva with or without constant compression and with or without exposure to two minutes of daily fluoride.

As visualized from the graphs in Figures 5-10, all coil groups demonstrated the expected superelastic force plateau during deactivation between approximately 10 and 2 mm. This plateau is unique to materials fabricated from nickel-titanium and graphically represents the range over which they will exert a nearly continuous force. Based on the results of this in vitro study, the force plateau occurred over roughly an 8 mm range of deactivation and generated an average force of around 200 grams. This 200 grams of force comes close to the recommended 150–160 grams of force for translating teeth within the arch (Anglokar et al., 1992), and is a much more physiologically acceptable force level than the 270–540 grams of force typically produced by a stainless steel open coil spring (Ryan, 1995). The presence of the superelastic plateau implies that the coils are "austenitic active" and rely on the stress-induced phase change between austenite and martensite. Consequently, they would be expected to have an Austenite Finish Temperature below the testing temperature (37°C) and likely below room temperature as well.

The maintenance of the horizontal force plateau during deactivation is most promising for those coils which were exposed to fluoride on a daily basis. Given that all five coil groups, including those which were immersed in fluoride daily, produced a superelastic plateau, fluoride likely does not lead to degradation of the mechanical properties of the nickel-titanium open coil springs. In previous studies, it was found that higher fluoride concentration exposures would lead to decreased corrosion resistance and an increase in the hydrogen absorption of titanium alloys (Fragou and Eliades, 2010). Titanium alloys are known to be relatively corrosion resistant due to the thin, stable oxide layer that they form on their surface, however, fluoride can be a corrosive agent (Walker et al., 2005). When a patient rinses with a fluoride-containing mouth rinse, the bacteria in the mouth is inhibited by the formation of hydrofluoric acid (HF). It is this HF which breaks down the oxide layer on the surface of the titanium alloy, leading to increased corrosion (Fragou and Eliades, 2010) and allowing for the absorption of hydrogen due to the high affinity of the exposed titanium to hydrogen (Yokoyama et al., 2003). The pH of the oral environment and fluoride rinse, the concentration of fluoride and temperature have all been implicated in increasing the amount of HF production, and thereby increasing corrosive changes in titanium alloys (Fragou and Eliades, 2010). Immersion time also plays a role in the degree to which HF causes undesirable changes in titanium alloys – tooth brushing with a fluoridate toothpaste has not been shown to induce corrosive changes (Fragou and Eliades, 2010).

Once the surface oxide layer on the titanium begins to break down, the material is then susceptible to surface and pitting corrosion (Koushik et al., 2011). This corrosion negatively effects the physical properties of the NiTi material, which thereby could negatively affect its clinical effectiveness. More specifically, it is the absorption of hydrogen ions once the protective surface layer has been broken down that leads to an increased susceptibility of nickel-titanium materials to become embrittled, thus becoming less clinically effective (Koushik et al., 2011).

Based on the results of this study, a daily two minute exposure to a 1500 ppm neutral NaF solution (meant to mimic daily exposure outside of a dental office) did not cause negative changes of clinical significance in the mechanical properties of nickeltitanium open coil springs. It has been shown that fluoride exposure through topical, inoffice agents can decrease the unloading mechanical properties of auxiliaries comprised of nickel-titanium (Walker et al., 2005). The goal of this study was to determine if the minor fluoride exposure that a patient encounters on a daily basis through toothpaste, water sources and over the counter mouthrinses would demonstrate the same such effect. As Figure 10 shows, all coils from all five groups demonstrated very similar force diagrams, indicating that a typical daily fluoride exposure does not adversely affect the mechanical unloading properties of nickel-titanium open coil springs. Statistical analysis showed significant differences in deactivation forces between the control group and two other groups (CF and NCNF). However, the actual difference in mean force values at a given distance of compression was less than 7% between the minimum and maximum force values among any groups. Further, these differences were often less than the standard deviations in force values for a given compression. Thus, any significant differences observed in deactivation force between groups is not likely to be clinically significant.

The second aspect analyzed in this study was whether constant compression over a period of 12 weeks would lead to a change in mechanical properties, likely due to force decay over time. To date, most studies concerning force decay have been performed on NiTi closed coil springs in situations of space closure. According to Nightingale and Jones (2003), the nickel-titanium coil springs demonstrated a rapid initial force decay over the first six weeks in the mouth, and then the force amounts leveled out. A follow-up study by Cox et al. (2014) again examined force decay of nickel-titanium coil springs, comparing in vivo and in vitro situations. The same initial force decay was seen over the first 6-8 weeks, after which the forces produced remained stable, leading to the expected rate of space closure of 1 mm per month. There was no significant difference in force loss between in vivo and in vitro situations (Cox et al., 2014). In the current study, the coils compressed for 12 weeks did not differ from the control group in deactivation force in a meaningful amount. However, the complicated oral environment and the clinical loading situation is difficult to replicate in vitro. Further, besides using closed coils, the Cox et al. (2014) study examined the force decay using a different method at the different time points (dynamic mechanical analysis vs a universal testing machine).

CHAPTER 6 CONCLUSION

Exposure to clinically relevant fluoride levels on a daily basis did not show clinically significant effects on the mechanical properties of the nickel-titanium open coil springs. While fluoride is known to be a corrosive agent, it did not detrimentally affect nickel-titanium auxiliaries when supplied in low, daily doses. Continuous compression over 12 weeks similarly did not significantly affect the mechanical properties of the nickel-titanium coils. Based on these results, nickel-titanium open coil springs appear to be mechanically reliable for use as an auxiliary in orthodontic treatment.

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