COATED AND UNCOATED NICKEL-TITANIUM ARCH WIRES: MECHANICAL PROPERTY AND SURFACE TOPOGRAPHY RESPONSE TO A SIMULATED ORAL ENVIRONMENT WITH AND WITHOUT BRACKET-RELATED LOAD-DEFLECTION INDUCED STRESS

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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2017
ABSTRACT

This study examined the effect of exposure to bracket-related load deflection on the mechanical properties and surface characteristics of esthetic coated and uncoated nickel-titanium archwires. Two types of coated archwires were tested: one with a rhodium ion coating and another with a polymer coating. Corresponding uncoated wires of the same size were also tested. These four different wires were divided into treatment groups based on exposure to bracket-related load deflection and storage in PBS at 37±1°C. A three-point bend test in DI water at 37±1°C was performed on specimens at three time points: 4 weeks, 8 weeks, and 12 weeks. Unloading forces at 1, 2, and 3 mm of deflection were recorded. After 12 weeks exposure, three representative specimens from each treatment group along with untested wires were viewed using scanning electron microscopy (SEM) to provide a qualitative surface topography analysis.

Results of the present study showed that no significant differences (p > 0.05) in unloading force were present in wires exposed to bracket-related load deflection. However, significant differences were observed between coated and uncoated wire types (p < 0.05). Rhodium-coated wires exhibited significantly increased unloading forces compared to their
corresponding uncoated wires. Conversely, polymer-coated wires exhibited significantly decreased unloading forces compared to their corresponding uncoated wires. This may be due to the process of manufacturing the coated archwires or due to differences in the underlying wire itself. SEM analysis revealed that for the rhodium-coated wires, rhodium precipitated out after 12 weeks exposure to PBS at 37±1°C. Furthermore, the degree of this precipitation appeared to be more severe in wires exposed to bracket-related load deflection. Polymer-coated wires showed a complete loss of coating in areas where the archwire contacted orthodontic brackets. This did not occur in wires not exposed to bracket-related load deflection.

The change in mechanical properties, specifically unloading forces, of coated archwires compared to uncoated archwires of the same size could be clinically significant in either increasing or decreasing rate of tooth movement. In addition, coating instability could cause increased friction in the archwire-bracket interface, which could also decrease efficiency of orthodontic tooth movement.
The faculty listed below, appointed by the Dean of the School of Dentistry, have examined a thesis titled, “Coated and Uncoated Nickel-Titanium Archwires: Mechanical Property and Surface Topography Response to a Simulated Oral Environment with and without Bracket-Related Load Deflection Induced Stress,” presented by Kelcey Loveland, candidate for the Master of Science degree, and certify that in their opinion it is worthy of acceptance.

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ACKNOWLEDGMENTS

I would like to express my sincere appreciation and give thanks to:

Dr. Mary Walker, for her encouragement, guidance, and expertise throughout my education at the UMKC School of Dentistry.

Dr. Jeffrey Nickel, for his guidance, expertise, feedback, and time.

Dr. Jeffrey Gorski, for his guidance, expertise, feedback, and time.

Dr. Donggao Zhao, for his expertise and time.

Dr. Joanna Scott, for her expertise and time.

Rachel Reed, for her time and assistance in the lab.

Erin Liljequist, for her support and administrative assistance.

Karina Kearney, for her support and administrative assistance.

Ormco, for their generous donation of brackets.

American Orthodontics, for their generous donation of orthodontic wires.

Dentsply GAC International, for their generous donation of orthodontic wires.

My family, for their love, support, and encouragement throughout my education.
Orthodontic Tooth Movement

Orthodontic tooth movement through alveolar bone occurs by applying forces to teeth. These forces are applied to teeth by the use of brackets and archwires. Brackets are first bonded to teeth in a certain position and in a certain orientation (Jian et al. 2013). A wire is then deflected with a certain amount of loading force into the slot of the brackets and ligated. The arch wire then applies forces as it returns to its original arch form and the ligated brackets take the teeth with them. These forces that are responsible for the movement of teeth are known as unloading forces (Nucera et al. 2014). Therefore, it is the unloading forces applied by archwires that must be understood to evaluate their ability to move teeth. General agreement exists about the nature of these forces, specifically, that light, continuous forces are optimal to accomplish the goals of moving teeth predictably and causing the least amount of harm to those teeth and their supporting tissues (Jian et al. 2013). In order for archwires to function in such a manner, they need to possess certain physical properties including: 1. Low stiffness, 2. High range of activation, 3. High strength, and 4. High formability (Kapila and Sachdeva 1989; Proffit et al. 2013 314-318). Over the past several decades, nickel-titanium archwires are more commonly used because they demonstrate these desirable properties.

Nickel-Titanium Archwires

The development of nickel-titanium (NiTi) archwires changed clinicians’ approach to treating orthodontic patients especially in the initial alignment phase of treatment. In fact, stainless steel archwires are now used by a smaller proportion of orthodontists than ever before (Jian et al. 2013). Andreasen and Hilleman were the first who explored the
incorporation of this alloy into the treatment of patients in 1971 (1971). An important finding made by the pair was that the brackets on malposed teeth could be at least a third farther apart and still be engaged by a NiTi archwire while avoiding plastic deformation compared to stainless steel archwires.

The desirable properties of NiTi alloys exist due to their crystalline composition with two fully-reversible phases. Speaking generically, at high temperatures and low stresses the wire is in the austenite phase, while low temperatures and high stresses favor the martensite phase (Proffit et al. 2013 314-318). Each phase has its own inherent properties. Martensite is not as stiff and is softer compared to the austenite form (Segal et al. 2009). The martensite and austenite phases interchange between each other through an intermediate rhomboidal phase (R-phase) (Ren et al. 2008). This transformation between phases is the underlying mechanism for NiTi archwires' unique shape memory and superelastic characteristics (Proffit et al. 2013 314-318). Shape memory is exhibited as the alloy undergoes a phase change due to alterations in temperature while superelasticity is observed as phase changes occur by applying and releasing stress (Laino et al. 2012). Clinically, it has been very difficult to take advantage of the shape memory properties of the alloy due to the required temperature changes that are not present in the oral environment (Proffit et al. 2013 314-318). On the other hand, clinicians have been able to exploit the superelastic effect of NiTi wires to improve the treatment provided to their patients.

In a clinical situation, with a superelastic NiTi archwire, as the wire is deflected into a bracket slot a shift occurs in the structure from the austenitic phase through the intermediate R-phase to the martensitic phase. The wire then returns through the R-phase to the austenitic phase upon unloading (Ren et al. 2008). Due to the fact that these phases can coexist, superelasticity (also described as plateau behavior) is displayed in the wire (Jian et
Superelastic archwires have changed the way that clinicians have approached treating patients. Proffit proclaimed that superelasticity is an “extremely desirable” characteristic and illuminated the idea that the unloading curve and loading curve differ from one another. Clinically this translates to the wire exerting a different force on the teeth than was applied to the wire to insert it into the bracket slot. Hysteresis accounts for the lost energy (Proffit et al. 2013 314-318). In this manner superelastic wires are able to experience large deflections, for a malposed tooth, with relatively high forces and return to their original shape while delivering low and nearly constant forces to the teeth (Oltjen et al. 1997; Parvizi and Rock 2003). Superelasticity allows the clinician, therefore, to reduce the number of times the arch wire is changed as long as the constant low force level remains above the minimum required force for tooth movement. This is especially helpful in the initial phase of leveling and aligning as one archwire can be used over a wider range of activation (Segner and Ibe 1995; Segal et al. 2009).

**Esthetic Demand in Orthodontics**

Increasingly in today’s society, the demand for esthetic orthodontic appliances has grown. The increased adult patient population is the main reason for this increased demand. Brackets and wires have traditionally been made of stainless steel, cobalt-chromium, or titanium alloy metals (Fallis and Kusy 2000). While the invention of ceramic brackets has satisfactorily addressed this issue both clinically and esthetically, problems still exist for finding an esthetic archwire that possess the desired clinical and esthetic properties. Attempts have been made to manufacture archwires from a composite resin material. However, the archwires have failed to achieve the mechanical properties possessed by the alloy metal group. Kusy, in a past publication, commented on this pursuit of a clinically
acceptable esthetic archwire: “Practitioners assert that esthetics are desirable but that function is paramount” (2002).

**Esthetic Archwires**

As mentioned earlier, the first esthetic wires used had a polymeric base. These wires met the esthetic goals that were set. However, their tendency to be brittle and have poor mechanical properties prevented their wide spread use by clinicians (Iijima et al. 2012). Due to the highly desirable mechanical properties of NiTi archwires, coatings have been developed to improve the esthetics of NiTi wires. Currently, several different types of coated NiTi archwires are available. The materials used to coat the wires include metal alloys, a variety of polymers, and Teflon (Farronato et al. 2012; Iijima et al. 2012). Few studies have evaluated these coated archwires and no studies have reported the effects of clinically applied stress on these coatings and the underlying wire.

There have been several comparative evaluations of different mechanical properties of NiTi archwires with esthetic coatings (Elayyan et al. 2010; Alavi and Hosseini 2012; Iijima et al. 2012; Katic et al. 2014). For example, mechanical properties of epoxy-coated NiTi archwires were investigated in a 2010 study. As-received coated and non-coated wires were subjected to a three-point bend test and were found to produce differing loading and unloading forces. Coated wires were shown to deliver lower forces than their non-coated counterparts. This finding led those who designed the study to conclude that the decreased diameter of the esthetic archwire core was probably to blame. The decreased diameter was necessary to allow space for the outer epoxy coating. Thus, the recommendation to use larger diameter wires than normal when considering coated esthetic archwires came out of the study (Elayyan et al. 2010). In 2012, Alavi and Hosseini found that polymer-coated and non-coated NiTi archwires produced similar forces when subjected to a three-point bend test.
(2012). Epoxy-coated NiTi archwires, however, produced forces that were significantly lower than those produced by the polymer-coated and non-coated NiTi versions. A similar hypothesis about the smaller diameter of the epoxy-coated versions being the culprit was developed. Another 2012 study compared as-received polymer-coated and rhodium-coated NiTi archwires using a three-point bend test at room temperature (Iijima et al. 2012). Rhodium-coated archwires proved to have a lower mean unloading force than those with no coating. On the other hand, the polymer-coated versions showed to possess a higher mean unloading force than those with no coating. Superelastic properties were demonstrated by all of the NiTi archwires used in the study. In another study, rhodium-coated archwires were tested at body temperature – 37° C – most recently in 2014 and found to have increased stiffness and higher loading forces than those with no coating. It was also shown that the rhodium coating did not have any effects on the unloading aspects of the wire (Katic et al. 2014).

Coating Durability and Surface Topography

As already mentioned, numerous studies have evaluated the mechanical properties of coated NiTi wires, other studies have also evaluated the durability of the coating and subsequent surface topography of the coated wire. In a study examining coating durability, four NiTi as-received round wires were examined using atomic force microscopy. Two were coated and two were non-coated. Among these NiTi archwires, it was found that Teflon-coated wires were the smoothest, even smoother than the non-coated varieties, while the rhodium-coated wires were the roughest of the four (D'Anto et al. 2012). This finding of a large amount of surface roughness being found in rhodium-coated archwires was confirmed more recently in a 2014 study (Katic et al. 2014). Field-emission scanning electron microscopy was used in the study to test an as-received rhodium-coated NiTi wire and an
electrochemically tested wire of the same type. A different study looked at epoxy-coated Niti archwires and, using contact stylus profilometry to measure surface roughness and optical and scanning electron microscopy to evaluate surface topography, compared as-received archwires to retrieved archwires. Delamination and discoloration of the coating was seen in this study. In fact, a 25% loss of coating, including ditching and cracking of the epoxy layer, was shown after 4-6 weeks of intraoral exposure. This in vivo study found that these archwires showed much greater surface roughness and that surface morphology deteriorated to a large degree after being retrieved from function in the oral cavity (Elayyan et al. 2008). Alavi and Hosseini’s findings are congruent with these as well (2012). They incubated both polymer and epoxy coated NiTi archwires in Bioxtra artificial saliva and used thermocycling model to simulate the oral environment. A three-bracket bending test machine was then used to apply a load to the wires and then allow the wire to exert its unloading forces over the span of a few minutes. The wires were examined both before and after testing using stereomicroscopy. Both epoxy and polymer coated wires were shown to exhibit poor coating longevity. Both coatings proved to tear and peel away from the underlying wire. Although coating loss was greatest in the polymer varieties, both wires demonstrated sufficient damage as to warrant concern for accumulation of plaque or bracket entrapment which would lead to unsatisfactory tooth movement. If one were to simulate clinically applied stress, similar outcomes to those that evaluated retrieved archwires may be seen.

**Corrosion of Non-Coated and Coated NiTi Archwires**

One disadvantage of NiTi wires centers around its corrosion susceptibility. Corrosion takes place in all base metals and is high in nickel alloys relative to gold alloys (Blanco-Dalmau et al. 1984). Corrosion occurs when unstable metals release ions into solution until a state of equilibrium is reached (Kim and Johnson 1999). Resistance to corrosion is vital to
the mechanical properties and biocompatibility of NiTi. It has been well documented that an alloy’s biocompatibility is directly related to the passive film on the surface (Yoo et al. 2008; Baker et al. 2009). This film is present due to titanium’s high chemical reactivity and the formation of a thin, stable oxide (TiO2) layer that occurs within 1 ms of exposure to air. With the formation of this layer on the external surface of the wire, known as “passivation”, the diffusion of oxygen is prevented giving the material corrosion resistance (Albrektsson and Jacobsson 1987). Therefore, corrosion cannot take place as long as this oxide film remains intact (Kim and Johnson 1999). It can be understood then that a resilient passive film is advantageous within the oral cavity and its acidic nature (Lee et al. 2010).

Many types of corrosion take place in NiTi alloys in varying environmental conditions. Crevice corrosion has been seen in instances where a nonmetallic appliance, an elastomeric chain for instance, has been in contact with a metal or NiTi appliance. Differences in metal or oxygen concentration of the crevice and its surroundings cause the formation of this type of corrosion. Pitting corrosion can be defined as a pore that has equal depth and width. This type of corrosion has been shown to occur when NiTi archwires were placed in a 1% saline solution and then studied electrochemically. Fretting corrosion occurs within areas of contact of materials under load. Cold welding and ultimately rupture of contact points takes place where the interfaces are under pressure, the bracket slot-archwire interface for example (Eliades and Athanasiou 2002).

Studies have also looked at corrosive properties of coated archwires. In 1999, investigators showed that less corrosion was demonstrated by epoxy-coated NiTi archwires than non-coated wires when both were exposed to a solution of sodium chloride (Kim and Johnson 1999). Another study exposed wires to anodic polarization in a modified saliva to speed up corrosion processes. Ion-implanted and polyethylene coatings showed decreased
corrosion compared to wires with no coating in this study. In the same study, Teflon coated wires were shown to be free of corrosion entirely. However, Teflon coated wires also demonstrated more defects following cyclic mechanical loading suggesting that clinical use may be problematic for coating durability (Neumann et al. 2002).

The aforementioned results of studies indicate that surface coatings when applied to NiTi archwires can degenerate, leaving the underlying metal wire exposed to the environment. The esthetics of the wire would obviously be compromised in this situation. Additionally, as was mentioned previously, increased plaque accumulation and decreased predictability of tooth movement are likely to occur (Elayyan et al. 2008). Most studies concerning coated archwires, including those mentioned, have mainly been designed to assess corrosion due to exposure to some environmental agent. Few studies have addressed corrosion of mechanical properties and surface topography of NiTi archwires due to bracket-related load deflection, and even fewer have evaluated the effects of stress on coated NiTi archwires.

**Influence of Clinically Applied Stress on Superelastic Archwires**

Even though it does not mimic the clinical use of archwires in which the wires are engaged into bracket slots resulting in load-deflection and resultant stress on the wire, most studies on the corrosion of orthodontic archwires have been carried out in the absence of an applied stress. Only a couple of studies have even addressed the concept of stress on corrosion of regular NiTi archwires. Huang looked at whether pitting potential, protection potential, and passive current density of an equiatomic NiTi wire were significantly affected by three different force quantities. The study used cyclic potentiodynamic and potentiostatic tests done in modified Fusuyama artificial saliva at 37°C to simulate oral conditions. Huang concluded that the applied loads of 50, 100, and 150 g had no significant impact on the
aforementioned properties of conventional NiTi archwires (Huang 2003). Another study done by Rondelli and Vincentini, showed that tensile strains of about 4% on superelastic NiTi archwires had no appreciable effect on the wire’s corrosion resistance. Artificial saliva was used as the storage medium for the experiment (Rondelli and Vicentini 2000). Segal et al. further examined the effect of stress and phase transformation on the corrosion of superelastic NiTi archwires. This group constructed an acrylic device designed to simulate a 5-bracket system. At both the second and fourth bracket position the apparatus made it possible to deflect the wire by 0.75, 1.5, or 3.0 mm. The medium chosen to perform these deflections in was phosphate buffered saline to simulate the oral aqueous environment at 37° C. It was found that the corrosion rate of conventional NiTi wires was increased due to the application of stress and that changes in stress/strain that accompanies phase transformation in superelastic NiTi might change the corrosion rate differently compared to wires that do not experience phase transformation (Segal et al. 2009). Studies interested in the effects of corrosion often use phosphate buffered saline to simulate the corrosive effects of the electrolytes in saliva, which is composed of 99% water with several main electrolytes including sodium, potassium, chloride, and phosphate. (Humphrey and Williamson 2001). Dulbecco’s phosphate buffered saline (PBS) is an example of a storage medium commonly used in studies interested in corrosion as it contains potassium chloride, monopotassium phosphate, sodium chloride, and disodium phosphate. Additionally, the experiments were performed at 37° C, the temperature of the oral environment, which is important because corrosion of the NiTi archwires increases as temperature increases (Pun and Berzins 2008). In each of the aforementioned studies, however, the time period for which the archwires experienced the applied stress was not specified.
Stress Applied Over Clinically Relevant Time Periods

While few studies have focused on investigating the effects of stress on NiTi archwires, even less have applied this stress over time periods reflective of clinical use. Only one study, mentioned above, looked at surface characteristics and mechanical properties of retrieved coated NiTi that were used in vivo for a period of four to six weeks (Elayyan et al. 2008). This study failed to investigate a defined stress level as each retrieved archwire was from a different patient with no mention of the degree of malpositioned teeth. Still other studies that have evaluated coated NiTi archwires under a repeatable amount of stress have failed to look at the variable of clinically relevant time periods. Both Rondelli and Vincenti’s and Huang’s studies made no mention of the amount of time that stress was applied to the tested NiTi archwires (2000; 2003). Segal’s study, furthermore, also failed to specify the amount of time that archwires experienced stress in the 5-bracket apparatus prior to testing (2009). Alavi and Hosseini stated that stress was applied to their coated NiTi wires at a rate of 0.5mm/minute and that the wires were deflected 2mm and then unloaded at the same rate (2012). In total, stress was applied for four minutes to a maximum deflection of 2mm and for another four minutes as the wire unloaded back to its original position.

Problem Statement

To date, a limited number of studies have been done to determine the mechanical and surface characteristics of coated NiTi archwires. For those that have been done, most tested the wires in the as-received condition, without taking into consideration the effects of simulated application conditions that include bracket-related load-deflection and the associated clinically applied stress over a defined time period. No studies have been
undertaken to determine the effects of bracket-related load deflection over relevant time periods on the unloading mechanical properties of coated NiTi archwires. Therefore, the purpose of the present study will be to examine the effects of simulated oral conditions using phosphate buffered saline at 37°C combined with bracket-related load deflection over time periods that are consistent with clinical practice on the mechanical properties and surface characteristics of coated NiTi archwires.

**Hypotheses**

1. There will be a difference in the unloading mechanical properties of non-coated and coated NiTi archwires exposed to simulated oral conditions (phosphate buffered saline at 37°C) with or without bracket-related load deflection over time.

2. There will be a qualitative difference in surface topography of non-coated and coated NiTi archwires exposed to simulated oral conditions with or without bracket-related load deflection over time.
CHAPTER 2

MATERIALS AND METHODS

Archwires

Two types of preformed coated archwires were selected for inclusion in this study: a polymer-coated NiTi wire along with a rhodium ion-implanted NiTi wire\textsuperscript{12}. Corresponding non-coated archwires were also included. Table 1 contains descriptions of the archwires. A polymer-coated NiTi wire was requested from American Orthodontics (AO) with a composition of 55% nickel and 45% titanium coated entirely by Hybrix White \#C57 (Table 2); these wires are referred to as AO Poly. The non-coated version, NiTi Memory Wire, from AO was included and is referenced by AO NC. A rhodium ion-implanted NiTi wire was obtained from Dentsply/GAC International (GAC) with composition of 51% nickel and 49% titanium; these wires are called GAC Rho. These “High-Aesthetic Sentalloy” archwires are coated with 100% rhodium that is applied by ion beam assisted deposition. The non-coated counterpart to the rhodium coated archwire is GAC’s Sentalloy NiTi archwire; these are referred to by GAC NC. All wires were requested in the round variety and in a 0.016 inch diameter for consistency.

\textsuperscript{1} Everwhite Cosmetic NiTi Wire, American Orthodontics, 3524 Washington Avenue, Sheboygan, WI 53081.
\textsuperscript{2} Sentalloy High-Aesthetic Wire, Dentsply/GAC International, One CA Plaza, Suite 100, Islandia, NY 11749.
### TABLE 1

ARCHWIRES USED IN THE STUDY

<table>
<thead>
<tr>
<th>Archwire</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Lot Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Everwhite Cosmetic NiTi Wire</td>
<td>American Orthodontics</td>
<td>55% Nickel, 45% Titanium, Hybrix White #C57 coating</td>
<td>D165</td>
</tr>
<tr>
<td>(AO Poly)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NiTi Memory Wire</td>
<td>American Orthodontics</td>
<td>45-55% Nickel, 45-55% Titanium, 0-0.5% Chromium</td>
<td>E10106</td>
</tr>
<tr>
<td>(AO NC)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sentalloy High Aesthetic Wire</td>
<td>Dentsply/GAC</td>
<td>51% Nickel, 49% Titanium, 100% Rhodium treatment</td>
<td>D80220</td>
</tr>
<tr>
<td>(GAC Rho)</td>
<td>International</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sentalloy NiTi Wire</td>
<td>Dentsply/GAC</td>
<td>51% Nickel, 49% Titanium</td>
<td>H193</td>
</tr>
<tr>
<td>(GAC NC)</td>
<td>International</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### TABLE 2

HYBRIX WHITE #C57 COMPOSITION

<table>
<thead>
<tr>
<th>COMPONENT</th>
<th>PERCENTAGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-methoxypropan-2-ol</td>
<td>20-25%</td>
</tr>
<tr>
<td>n-butyl acetate</td>
<td>23-28%</td>
</tr>
<tr>
<td>Toluene</td>
<td>20-25%</td>
</tr>
<tr>
<td>Aluminum oxide</td>
<td>3-5%</td>
</tr>
<tr>
<td>Silicon dioxide</td>
<td>15-20%</td>
</tr>
<tr>
<td>Methyl methacrylate, n-butyl acrylate, 2-hydroxyethyl methacrylate, dimethylaminoethyl methacrylate copolymer</td>
<td>10-15%</td>
</tr>
</tbody>
</table>
Wire Specimen Preparation

By cutting the two posterior sections away from the four types of as-received archwires, two specimens were procured from one archwire with each specimen measuring 30 mm in length. Each specimen was then randomly assigned to one of two treatment groups: without stress or with stress. The diameter of each specimen was measured with a digital caliper at three points, averaged, and then recorded. This was done before, during, and after treatment. A three-point bend test, according to American Dental Association specification no. 32 protocol (Council on Scientific Affairs 2006), was performed in a deionized water bath (dH₂O) at 37±1°C. The initial data collected prior to treatment was used as a negative control.

In order to simulate the aqueous oral environment, 0.9% phosphate buffered saline (PBS)³ was used as a storage solution. Sodium azide (0.002%) was added to the PBS in order to prevent microbial growth during storage periods. Half of each wire type specimens were stored under no load or function in a vial with PBS. To simulate the load deflection an archwire experiences when ligated into brackets on malaligned teeth, the remaining half of the specimens were loaded and ligated into a custom-made 3-bracket simulation apparatus where the middle tooth was out of alignment so that a deflection in the archwire was necessary (fig. 1). See Appendix A for fabrication protocol for tooth/bracket apparatus. Each of these simulation apparatus specimens were submerged in PBS and stored in a 32 ounce encased plastic container⁴. All specimens, both stressed and unstressed, were incubated at 37°C. At each measurement time point, the PBS solutions were changed for all specimens in order to prevent microbial growth.

³ Dulbecco Phosphate Buffered Saline, Sigma-Aldrich™, Inc, 3050 Spruce St., St. Louis, MO, 63103.
⁴ GladWare® Containers, Glad®, 1221 Broadway Street, Oakland, CA 94612.
Figure 1. Three-bracket simulation apparatus.
Mechanical Testing

According to protocol described by ADA specification No. 32 (Council on Scientific Affairs 2006) the three-point bend test, which simulates clinical orthodontic conditions, was performed on all specimens at clinically relevant time points. A universal testing machine\(^5\) was used to perform all tests. Specimens were placed on a fixture with support span measuring 10 mm. The striker and both supports have radii of 0.10±0.05 mm (fig. 2). To continuously simulate the aqueous oral environment, the fixture was placed in a dH\(_2\)O bath where the testing was performed. Specimens were loaded on to the fixture so that arch curvature was concave toward the striker. After applying a 0.1N preload force to maintain consistency, each specimen was bent to a deflection of 3.1 mm with a crosshead speed of 10 mm/min. Unloading force at 1.0, 2.0, and 3.0 mm of deflection were recorded (fig. 3).

\(^5\) Model 5967, Instron Industrial Products, 100 Royal Street, Canton, MA 02021
Figure 2. Three-point bend test setup. A) 0 mm deflection; B) 3.1 mm deflection.
Figure 3. Representative load/deflection curve. Evaluating unloading force at 1, 2, and 3 mm.
Scanning Electron Microscopy

Following mechanical testing at 12 weeks, three representative specimens from each experimental group, coated and non-coated wires with or without bracket-related load-deflection, were selected for imaging using scanning electron microscopy (SEM) at various magnifications. Additionally, SEM images of as-received wires of each wire type were obtained at the same magnifications. A qualitative analysis was used to investigate differences in surface topography and possible related corrosion based on treatment group.

Experimental Design

A two-factor, repeated measures design was utilized in this study. The first independent variable with four levels is wire type. These include GAC Sentalloy® 0.016" Medium Upper High Aesthetic Rhodium Coated NiTi, GAC Sentalloy® 0.016" Medium Upper NiTi (non-coated), American Orthodontics 0.016" EverWhite NiTi, and American Orthodontics 0.016" NiTi Memory Wire (non-coated). The second independent variable is the presence or absence of bracket-related load deflection of the wire. Dependent variables were unloading force at 1, 2, and 3 mm deflection and wire surface topography after 12 weeks. The experimental design is presented in Table 3.

Sample Size

For this study a convenience sample size of 10 wire specimens per treatment group was used. As there were two treatment groups (simulated oral conditions: phosphate buffered saline at 37°C with or without bracket-related load deflection over time) and four different types of archwires, a total of 80 wire specimens were tested.
# TABLE 3

## EXPERIMENTAL DESIGN

<table>
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<tr>
<th>Archwire</th>
<th>Treatment (n=10/group)</th>
<th>Time Point</th>
<th>Unloading force at 1, 2, 3 mm</th>
<th>Surface topography (3 out of 10/group)</th>
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<td>No Load-Deflection</td>
<td>0 (baseline)</td>
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<td>After 12 weeks only</td>
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**Data Analysis**

A two-factor univariate, repeated measures ANOVA was done to evaluate the effect of treatment and wire type on unloading force. The data was analyzed using statistical software\(^6\) with significance set at \(\alpha = 0.05\), while the surface topography analyses were qualitative comparisons of SEM images.

---

\(^6\) SPSS version 21, 233 S. Wacker Dr., Chicago, IL 60606
CHAPTER 3

RESULTS

Mechanical Testing

The mechanical property measurements for each wire type and treatment group can be seen below in Table 4.

<table>
<thead>
<tr>
<th>Wire Type</th>
<th>Treatment Group</th>
<th>Time Point (weeks)</th>
<th>Mean Unloading Force (N) at 1mm</th>
<th>Mean Unloading Force (N) at 2mm</th>
<th>Mean Unloading Force (N) at 3mm</th>
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<tr>
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<td>1.18 (0.12)</td>
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<td>1.08 (0.15)</td>
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<td></td>
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<td>12</td>
<td>1.05 (0.20)</td>
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<td>12</td>
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<td>0.93 (0.15)</td>
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<td>8</td>
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<td>8</td>
<td>0.89 (0.15)</td>
<td>0.94 (0.15)</td>
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<tr>
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<td>1.24 (0.14)</td>
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<td>1.05 (0.07)</td>
<td>1.10 (0.07)</td>
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<td>No Load-Deflection</td>
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<td>0.77 (0.07)</td>
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<tr>
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<td>0.64 (0.25)</td>
<td>0.51 (0.28)</td>
<td>0.56 (0.28)</td>
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</table>
The two-factor ANOVA revealed significant differences (p < 0.05) in unloading forces between non-coated and coated NiTi (rhodium or polymer) archwires exposed to simulated oral conditions with or without bracket-related load deflection over time at each extension. On the other hand, no significant effect (p>0.05) was observed within each wire type as a function of bracket-related load deflection.

Figures 4 and 5 more clearly visualize the unloading force comparisons within each wire type. Figure 4 demonstrates that for all unloading forces measured, rhodium-coated wires showed higher unloading forces than their non-coated counterparts. In fact, the increase in unloading forces was by an average of 42% across all three deflection points and time periods whether the wire was load-deflected or not. Figure 5 demonstrates, conversely, that polymer-coated wires showed lower unloading forces than their non-coated counterparts. The unloading forces for polymer-coated wires decreased by an average of 26% across all three deflection points and time periods for both load-deflected and no load-deflected wires. Both figures also illustrate that no significant differences were observed based on whether wires were subjected to bracket-related load deflection.
Figure 4. Mean unloading forces for rhodium-coated archwires. Measurements were made after 4, 8, and 12 weeks storage in PBS at 37°C combined with or without load deflection of the wires. Each graph shows unloading forces measured at 1, 2, and 3 mm deflection. *Across storage times with or without load deflection, rhodium-coated wires produced significantly (p>0.05) higher unloading forces than uncoated wires at each deflection measurement. However, within each wire type (coated and uncoated), load-deflection was not found to significantly impact unloading forces consistently. To view standard deviations, please see Table 4.
Figure 5. Mean unloading forces for polymer-coated archwires. Measurements made after 4, 8, and 12 weeks, respectively. Each graph shows unloading forces measured at 1, 2, and 3 mm deflection. *Across storage times with or without load defelction, polymer-coated wires produced significantly (p<0.05) lower unloading forces than uncoated wires at each deflection measurement. However, within each wire type (coated and uncoated), load deflection was not found to significantly impact unloading forces consistently. To view standard deviations, please see Table 4.
**Surface Topography Evaluation**

Representative SEM images for rhodium-coated wires and the corresponding uncoated wires may be seen in figures 6 and 7, respectively. For rhodium-coated wires, particulates were observed in greater number on the surface of load-deflected wires (fig. 6C”) compared to the non-deflected wires (fig. B”). However, few to no particulates were observed in the untested (as-received) wires (fig. A”). While particulates were not seen in images of the uncoated wires, pitting corrosion was observed in the load-deflected group of uncoated wires (figs. 7C’-C”). Uncoated wires exposed only to PBS have fewer surface irregularities than the same group of rhodium-coated wires (fig. 6B”, fig. 7B”).

Because particulates were observed in the rhodium-coated load-deflected and non-deflected wires, Energy-dispersive X-ray Spectroscopy (EDS) was used to determine the composition of these particulates. The results may be seen in figure 8. As expected, peaks indicating the presence of nickel and titanium were present in all three wire groups. Load-deflected (fig. 8C) and non-deflected wires (fig. 8B), however, contained a rhodium peak not present in the untested group (fig. 8A). The comparative heights of the rhodium peaks (fig. 8C vs. fig. 8B) along with the comparatively greater number of particulates present in the SEM images, as mentioned above, in the load-deflected wires appears to indicate a much greater amount of rhodium present in the load-deflected wires compared to non-deflected wires. A gold peak was also observed in the spectrum of the load-deflected wires (fig. 8C) and, to a lesser extent, in the non-deflected wires (fig. 8B) but was absent in the as-received rhodium-coated archwires (fig. 8A).

Representative SEM images for polymer wires may be seen in figure 9 juxtaposed with images of its corresponding uncoated wire in figure 10. Surface characteristics for polymer wires appeared to vary depending on exposure to bracket-related load deflection.
Load-deflected polymer wires (fig. 9C) showed complete destruction of the outer polymer coating leaving the underlying wire exposed. Non-deflected wires (fig. 9B') also showed minor interruptions in the integrity of the polymer coating. Untested wires (figs. 9A, 9A', and 9A''), however, showed a completely intact external polymer coating.
Figure 6. Rhodium-coated wire SEM images. Images were made at 50, 1000, and 4000x magnification. A-A". Untested wires; B-B". Non-deflected wires; C-C". Load-deflected wires. Particulates, indicated by the arrows, observed in greater quantity in (C) than in (B) and not present in (A).
Figure 7. GAC uncoated wire SEM images. Images were made at 50, 1000, and 4000x magnification. A-A''. Untested wires; B-B''. Non-deflected wires; C-C''. Load-deflected wires. Pitting corrosion, indicated by the arrows, observed in load-deflected wires.
Figure 8. Energy-dispersive spectroscopy of rhodium-coated wires. A. Untested wires; B. Non-deflected wires; C. Load-deflected wires. Rhodium peak was present in non-deflected (B) and load-deflected wires (C) only (arrows). Height of rhodium peak much greater in C than B. Gold peak (asterisks) follows a similar pattern in the non-deflected and load-deflected wires.
Figure 9. Polymer-coated wire SEM images. Images were made at 50, 1000, and 4000x. A-A". Untested wires; B-B". Non-deflected wires; C-C". Load-deflected wires. Note: While a crack is present in B' and B", complete destruction of the polymer coating can be seen.
Figure 10. AO uncoated wire SEM images. Images were made at 50, 1000, and 4000x. A-A’. Untested wires; B-B’. Non-deflected wires; C-C’. Load-deflected wires.
CHAPTER 4
DISCUSSION

The purpose of this study was to examine the mechanical properties and changes in surface topography of coated and uncoated NiTi archwires when subjected to bracket-related load deflection over clinically relevant time periods. The unloading force of each NiTi archwire was measured using the three point bend test and the surface topography was examined under scanning electron microscopy.

Mechanical Testing

The present study detected significant differences in the unloading forces of the coated wires and their non-coated counterparts. The findings indicate that the rhodium-coated archwires demonstrated increased unloading forces by an average of 42% across the three measured deflection points and time periods across whether or not they were exposed to bracket-related load deflection when compared to their corresponding uncoated archwire of the same diameter. This is in contrast with previous studies that have either noted that rhodium coating had a deleterious effect or no effect on unloading forces. Iijima and colleagues found a significant decrease in unloading forces compared to its uncoated counterpart when they subjected as-received archwires to a 3-point bend test (Iijima et al. 2012). It should be noted that the rhodium-coated wires used were a type that is designed to provide different forces in the anterior region of the archwire compared to the posterior region of the archwire while the uncoated comparison was not. More recently, Katic and colleagues found no significant differences between the unloading forces of rhodium-coated archwires and uncoated archwires of the same diameter (Katic et al. 2014).

While the esthetic coating increased the unloading forces for the rhodium-coated archwires, it had the opposite effect for the polymer-coated archwires. In fact, polymer-
coated archwires showed an average 26% decrease in unloading forces across all deflection points measured and all three time points regardless of exposure to bracket-related load deflection when compared to its uncoated counterpart. This contradicts the findings of Alavi and Hosseini that polymer-coated and non-coated archwires showed no significant differences in unloading forces (Alavi and Hosseini 2012). A separate study by Iijima and colleagues found that polymer-coated archwires demonstrated significantly higher unloading forces than similar non-coated archwires (Iijima et al. 2012). The polymer-coated archwires proved to increase unloading forces in the study by about 11%. The authors state that the application of the thick coating layer on the polymer wire was at fault for these increased unloading forces.

The present study was the first to examine the effects of bracket-related load deflection on the mechanical properties of coated and non-coated NiTi archwires. No wire tested in this study, coated or non-coated, showed any significant difference in unloading forces based on this variable. In 2014 an in vivo study, evaluated unloading forces subsequent to clinical application of archwires in patients (Bradley et al. 2014). There was no way to isolate bracket-related load deflection in the study due to the nature of in vivo studies; however, its presence was an unchangeable fact. In the study, two aesthetic coated NiTi wires and two corresponding uncoated NiTi wires were placed in the mouths of 61 patients for between 4 and 12 weeks. Once retrieved, these wires were subjected to a three-point bend test. The polymer coated wire used in the study showed increased unloading forces following retrieval compared to the as-received samples. This is in contrast to the present study, however, in which polymer coated wires showed no change in unloading forces whether or not they were subjected to bracket-related load-deflection. On the other hand, Bradley and colleagues found that the unloading forces of the uncoated wires showed
no change after retrieval. This finding is consistent with the finding in this study that bracket-related load-deflection had no significant effect on unloading forces.

**Surface Topography and Corrosion Resistance**

In contrast to the mechanical testing, surface topography did vary based on bracket-related load deflection. Scanning electron microscopy (SEM) revealed that the surface topography of rhodium-coated archwires differed depending on exposure to bracket-related load deflection. As mentioned above, particulates were seen in greater number on the rhodium-coated wires exposed to bracket-related load deflection (Figure 6C”) than those only exposed to PBS (Figure 6B”). This suggests that bracket-related load deflection directly correlates to the amount of corrosion that occurred over the 12-week period. Additionally, few to no particulates were seen in as-received rhodium-coated archwires (Figure 6A”) while some particulates were present in archwires exposed only to PBS, suggesting that either exposure to PBS or load deflection associated with the mechanical testing caused rhodium to precipitate out of the surface in the non-deflected group. The amount of particulates and pits on the surface of rhodium-coated wires exposed to PBS suggests disruptions of the external structure. Pitting was also present on the corresponding uncoated archwires that were tested but to a smaller degree. These two findings agree with those of a previous study. Katic and colleagues performed electrochemical impedance spectroscopy testing and found that rhodium-coated wires had more corrosion than their corresponding uncoated wires (Katic et al. 2014). This finding agrees with the finding of the present study that rhodium-coated archwires show a greater propensity to corrode than their uncoated counterpart does.

The particulates discovered on the surface of rhodium-coated archwires was a unique feature to that type of wire. EDS revealed that not only was rhodium present in the
particulates but gold was as well. This discovery manifests that, at least to some degree, the integrity of the rhodium coating could be compromised by the corrosive nature of the oral cavity and to a greater degree by bracket-related load-deflection. Katic and colleagues also concluded that environment had an impact on the surface composition of rhodium-coated archwires (Katic et al. 2014). They observed that surface particulates increased in prominence and number after corrosion testing. Using EDS, they confirmed that the particulates were composed of rhodium and gold, which is in agreement with the findings of the present study and disagrees with Iijima’s study, which found only gold to be present (Iijima et al. 2012). In contrast to the findings of the present student, however, Katic also found that these surface irregularities were present in as-received samples. As-received wires in the current study did not appear to have any particulates present. This could be due to a change in the manufacturing process of the archwires.

Similarly to the rhodium-coated archwires, the surface topography of polymer-coated archwires also varied with or without bracket-related load deflection. As shown in Figures 9B’ and 9B”, minor disruptions of the polymer layer occurred in wires exposed only to PBS and mechanical testing. However, complete loss of the polymer layer occurred in wires exposed to bracket-related load deflection. This suggests that the friction and forces associated with ligation of the wires into the bracket caused the resin to peel off and is consistent with previous studies (Lim et al. 1994; Kusy 2002; Elayyan et al. 2008; Bradley et al. 2014).

**Clinical Implications**

Based on the results of the current study, coated nickel-titanium archwires could have an effect on both the mechanical properties and surface topography of the wires and potentially impact treatment time and the patient experience. Specifically, rhodium coatings
increased unloading forces, while polymer coatings decreased unloading forces of similarly sized archwires.

Increasing the unloading forces of an archwire may or may not be clinically significant. The idea of an optimal force concerning orthodontic tooth movement has evolved over the past 70 years (Ren et al. 2003). The current hypothesis centers on the concept that a force of a certain magnitude and whether that force is continuous or intermittent and constant or declining determines if it is capable of producing a maximum rate of tooth movement without damaging tissues and without causing excessive discomfort. The optimal force may differ from patient to patient and even from tooth to tooth within each patient. Therefore, if the rhodium-coated archwires produce excessive unloading forces, it is possible that bone hyalinization and undermining resorption will begin to take place (Santoro et al. 2001). However, the force levels demonstrated in this study by the rhodium-coated archwires do not appear to be to that extreme.

The decreased unloading forces demonstrated by the polymer-coated archwires may delay tooth movement and increase treatment times. In fact, a previous study found that polymer-coated NiTi archwires that had been retrieved clinically after 4-12 weeks produced lower unloading forces (Bradley et al. 2014). The authors surmised that this is due to either the coating process affecting the mechanical properties or different stock wire being used for each product. Decreased unloading forces have the potential to decrease the rate of tooth movement and thus lead to protracted treatment times.

Sliding mechanics are very important to tooth movement. Several studies have shown that coated NiTi archwires demonstrate higher surface roughness when compared to uncoated NiTi archwires (Iijima et al. 2012). Rhodium-coated NiTi archwires, in a recent study, were implicated as having the highest surface roughness of any other wire included in
the study (Katic et al. 2014). This increased roughness increases friction and hampers the ability of the archwire to slide along the archwire-bracket interface, which effectively limits tooth movement.

The breakdown of coatings and corrosion of coated NiTi archwires, mentioned above, may be sufficient reasons to limit their use clinically. Specifically, in situations where archwire sliding is critical such as correcting rotated teeth or closing a diastema, the use of coated NiTi archwires would lead to treatment delays due to the elevated friction levels and subsequent decrease in rate of tooth movement.

In combination with the resultant effects on tooth movement, it is important to consider other side effects of the corrosion or complete loss of the coating layer intraorally. The amount of coating loss on polymer-coated NiTi wires could increase plaque accumulation on the archwire-bracket-tooth interface that would result in increased decalcification of teeth. Additionally, the large amounts of polymer that peel off have the potential to become lodged in surrounding soft tissues. Finally, at least in some cases, the increased esthetic premise for using coated NiTi archwires may be completely undermined. The deterioration or discoloration of these coatings would negatively impact the esthetics of these archwires when used in a clinical situation. Patients would most likely be dissatisfied within a few days as the appearance of a tooth colored wire gave way to a shiny metal wire.

**Limitations of Study**

The present study was the first to test coated NiTi archwires with or without a repeatable amount of bracket-related load deflection. Multiple limitations exist in in vitro studies of this sort. First, being that this was a pilot study, only 10 specimens were included in each group. Another limitation of the study was the inclusion of only two coated NiTi archwires. Clinicians are currently using multiple versions of coated NiTi archwires and in
order to provide more information on the subject, more versions could be tested in future studies. Another limitation of the study involves the testing of these archwires at four different time points and allowing them to return their original shape each time. In a clinical setting, an initial orthodontic archwire would be ligated into malposed teeth and would apply forces to the teeth in a continuous manner for periods of up to twelve weeks. The process of removing the archwires and deflecting them to 3.1 mm and then returning them to the three bracket simulation apparatus every 4 weeks could have contributed to some of the effects seen in this study. Additionally, in a clinical setting with a tooth that is out of alignment, the tooth will begin to align from the day the wire was ligated into the bracket. This would gradually decrease the amount of deflection experienced by the archwire over time. The rigidity of the three bracket simulation apparatus does not translate to the clinical setting. A final limitation of the current study is the qualitative nature of the surface topography analysis. Performing a roughness test would perhaps allow for more direct comparison in a quantitative manner. This would also allow for more comparisons to previous studies that looked at coated and uncoated NiTi archwires.

**Future Studies**

Further investigation into coated NiTi archwires and their potential for corrosion due to bracket-related load deflection is warranted. As mentioned above, instead of testing wires every four weeks, it would be noteworthy to test wire groups once after certain time periods and compare unloading forces as a function of time. This would eliminate the potential confounding variables of removal from the three bracket simulation apparatus, deflection by the universal testing machine, and return to the apparatus. Another development that would allow in vitro orthodontic studies to more closely resemble clinical settings would be the development of a material that would mimic tooth movement within the supporting
periodontium over similar periods of time when exposed to similar forces. The current study used a rigid apparatus that would not allow the wire to return to its shape. If such a material was developed, a different apparatus could be constructed allowing for return of the archwire to its original shape and thus more closely resemble in vivo tooth movement.

Finally, large amounts of polymer coating were lost throughout this study. It has been reported in other studies that as much as 44% of the polymer coating was lost (Bradley et al. 2014). Consequently, future studies should also focus on the potential allergenic or inflammatory side effects of the polymer lodging in intraoral soft tissues.
1. There was a significant difference in unloading mechanical properties of uncoated and coated NiTi archwires exposed to simulated oral conditions (phosphate buffered saline at 37°C) with or without bracket-related load deflection over time. Specifically:
   a. Rhodium-coated archwires showed increased unloading forces compared to their corresponding uncoated archwires
   b. Polymer-coated archwires showed decreased unloading forces compared to their corresponding uncoated archwires. However, bracket-related load deflection had no significant effect on unloading forces.

2. There was a qualitative difference in surface topography of uncoated and coated NiTi archwires exposed to simulated oral conditions with or without bracket-related load deflection over time.
   a. Rhodium particulates were present on rhodium-coated archwires exposed to simulated oral conditions and were more abundant in wires that had been deflected.
   b. Polymer-coated wires showed complete loss of the coating in areas of the archwire-bracket interface.
LITERATURE CITED


Pun DK, Berzins DW. Corrosion behavior of shape memory, superelastic, and nonsuperelastic nickel-titanium-based orthodontic wires at various temperatures. Dent Mater 2008;24:221-7.


APPENDIX

3-BRACKET TOOTH APPARATUS
Twenty sets of three mandibular incisors, namely teeth numbers 23, 24, and 25, were obtained from Kilgore International, Inc. One of each tooth number were placed in wax in alignment vertically, horizontally, and rotationally. Tooth number 24 was then displaced in a lingual manner.

Petroleum jelly was then used to lubricate the internal surface of the blue flask.
Monomer and polymer from Great Lakes Orthodontics was mixed together in a 7.5:15ml ratio.
This mixture was then poured into the blue flask-tooth setup device and allowed to setup.

In order to duplicate this precise tooth setup, Bisico S1 Suhy silicone impression material was used to create a negative of the device.
One of each of the Kilgore tooth numbers mentioned previously were then placed into the impression before being placed onto a blue flask.

The same monomer and polymer were mixed together in the same ratio as above and poured into the impression-blue flask setup repeatedly until twenty devices were fabricated.
After allowing the mixture to set, each device was removed and trimmed.
Ormco Damon brackets were then bonded to each tooth after applying Reliance’s Assure Plus All Surface Bonding Resin to each tooth and 3M Unitek’s Transbond Supreme LV Low Viscosity Light Cure Adhesive to each bracket.
Bisico S1 Suhy silicone impression material was then used to create a negative of the setup in order to precisely duplicate the bracket locations. Brackets were placed in the impression, light cure adhesive was applied to each bracket, and bonding resin was applied to each tooth. Ortholux Luminous Curing Light was used to cure the adhesive.
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EDUCATION:

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<td>2008</td>
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<td>Logan, Utah</td>
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<td>2015</td>
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<td>UNLV School of Dental Medicine</td>
<td>Las Vegas, Nevada</td>
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PROFESSIONAL ORGANIZATIONS:

- 2015-present: American Association of Orthodontists, Member
- 2015-present: American Dental Association, Member
- 2015-present: Omicron Kappa Upsilon National Dental Honor Society, Member
- 2013-2015: SGT. Clint Ferrin Dental Clinic, Clinic Director
- 2012-2015: E Steven Smith Dental Student Honor Society, UNLV, Member
- 2012-2015: American Student Dental Association, Officer

SELECTED HONORS:

- 2015: Innovation and Originality in Dental Research Award
- 2015: Magna Cum Laude Graduate, UNLV School of Dental Medicine
- 2014: UNLV GPSA Competitive Travel Grant
- 2014: Dr. William S. Kramer Award Nominee, Omicron Kappa Upsilon
- 2012: ADA Foundation Scholarship Award
- 2012-2015: Dean’s List