The Effect of Strain on Transformation Temperature Range of Orthodontic Nickel-Titanium Archwires

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THESIS

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This thesis is dedicated to my parents, Tony and Sandy Liu. They have been my steady support system through every chapter of my life and have taught me that with hard work and perseverance, I can accomplish my dreams. They are my heroes and have paved the way for my success. I would also like to dedicate this thesis to my sister, Jasmine, who has become one of my best friends and confidants.

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LIST OF ABBREVIATIONS

ADA	American Dental Association
ANSI	American National Standards Institute
A_s	Austenite start temperature
A_f	Austenite finish temperature
BFR	Bend and Free Recovery
CuNiTi	Copper Nickel-Titanium
DSC	Differential Scanning Calorimetry
FDA	Food and Drug Administration
ISO	International Organization for Standardization
LVDT	Linear variable differential transducer
M_s	Martensite start temperature
M_f	Martensite finish temperature
Ni-Ti	Nickel-Titanium
RTTA	Recovery Temperature Testing Apparatus
R_s	Rhombohedral start temperature
RMO	Rocky Mountain Orthodontics
SIM	Stress-induced martensite
TTR	Transformation temperature range

SUMMARY

Since its introduction to the orthodontic field in the 1970s, Nickel-Titanium (Ni-Ti) has become the predominant material for the initial leveling and aligning phase of orthodontic treatment. The alloy's ability to reversibly transform between two lattice structures determines the material's properties, and thus, its clinical practicality. This transformation occurs as a result of a change in ambient temperature or in the area of a locally applied stress. The range of temperatures over which Ni-Ti transforms from one lattice structure to the other is called its transformation temperature range (TTR). Consequently, TTR is largely responsible for the clinical application of Ni-Ti.

The aim of this study was to examine how varying levels of strain affected the transformation temperature of two brands of Ni-Ti archwires using the bend and free recovery (BFR) method. Strain was altered in this study by two different techniques. The first technique was by varying the diameter of the mandrel over which the Ni-Ti wire was bent, where a smaller diameter mandrel results in a higher strain placed on the archwire. The second technique of varying wire strain was by bending two different dimensions of wires over the same size mandrel, where a larger wire results in a higher strain placed on the archwire. Additionally, the manufacturer-reported transformation temperatures of the wires were compared to the transformation temperatures obtained in this study.

The results demonstrated that TTR values are affected by strain. Increased strain resulted in increased transformation temperature values and decreased range. While this was the trend for increased strain whether by decreasing mandrel diameter or increasing wire size, one company did not have similar results when strain was increased by increasing wire dimension. This may have been a result of only testing two production lots. Additionally, there was a statistically significant difference between the obtained values in this study and those reported by the manufacturers.

I. INTRODUCTION

A. Background

Nickel-Titanium (Ni-Ti) archwires have evolved significantly since the material's introduction into the orthodontic field in the 1970s. The development of these wires has also altered their properties to produce archwires that have a larger range and deliver lighter forces on the dentition (Burstone and Choy 2015). These wires take advantage of the ability Ni-Ti has of changing between two lattice structures, martensite and austenite, in response to the ambient temperature. The period over which the wire is transforming, in which both lattice structures exist in equilibrium, is termed transformation temperature range (TTR).

Other than temperature, stress can also affect the conversion between the austenite and martensite structures. An austenitic wire that is deflected under the application of stress generates a stress-induced martensitic transformation in the area of the deflection. In this phenomenon, the martensite crystalline structure is localized to the stressed area while the rest of the wire remains in the austenitic phase. Some literature demonstrates that the formation of stress-induced martensite (SIM) results in increased transformation temperatures (Santoro, Nicolay, and Cangialosi 2001). Applying a stress to the wire keeps the deflected portion in the martensite phase, and thus, more energy (i.e. heat) is then required for the phase transformation. As Santoro et al. states, "This means that if the values of the TTR provided by the manufacturers are not calculated under proper conditions of deflection, those values might be underestimated and could fail to correspond to the actual TTR values existing in orthodontic applications" (Santoro, Nicolay, and Cangialosi 2001)

In orthodontic literature of TTR, differential scanning calorimetry (DSC) is used most commonly as the method of testing TTR. However, the DSC method does not have the ability to change the amount of strain that is placed on the wire. Orthodontic studies that test the effect of strain on transformation temperature have needed to fabricate loading devices for each testing specimen out of an electronically insulated material, such as plexiglass. Then the change in electrical resistance is measured to determine transformation between martensite and austenite. On the other hand, the bend and free recovery (BFR) test method can subject archwires to varying prescribed strains and can be performed on as-received archwires, without any modification. Additionally, BFR is commonly used to test transformation temperatures of devices in the medical field (Drexel, Proft, and Russell 2009), such as Ni-Ti stents.

B. <u>Statement of the Problem</u>

TTR is a critical determinant of the properties that a given Ni-Ti archwire will exhibit. Despite the importance of TTR, some manufacturers do not provide accurate information when it comes to TTR (Santoro, Nicolay, and Cangialosi 2001); furthermore, some do not even report this information (Spini et al. 2014). The American National Standards Institute (ANSI) approves American Dental

Association (ADA) standards and is also U.S. member to the International Organization for Standardization (ISO). These standards, however, are not enforced by the U.S. Food and Drug Administration (FDA) (Obaisi 2013).

Moreover, clinically, when an orthodontic archwire is ligated to brackets, different amounts of crowding from patient to patient will result in various amounts of strain placed on the archwire. If strain has an effect on the transformation temperatures, then the properties of the archwires could vary from patient to patient as well. Thus, the clinician would need to consider how the crowding would affect the wire properties when selecting an archwire that would best suit the patient's needs.

C. Purpose of the Study

The purpose of this study was to examine how varying levels of strain affected the transformation temperature of Nickel-Titanium orthodontic archwires from two different manufacturers using the bend and free recovery (BFR) test. Strain was altered by varying the diameter of the wire and by varying the diameter of the mandrel of the BFR machine's mandrel. Additionally, the transformation temperatures were compared to the temperatures reported by the manufacturer. Clinically, this information can help orthodontists to determine which Ni-Ti archwire would best suit the patient if there is more crowding, and as a result, more strain on the archwire.

D. Hypotheses

1. There is no mean difference in A_s and A_f with varying strain levels and wire sizes.
2. There is no mean difference in A_s and A_f between tested values and the manufacturer-listed values.

II. CONCEPTUAL FRAMEWORK AND RELATED LITERATURE

A. History of Nickel-Titanium

Ni-Ti was first developed by W.F. Buehler in the early 1960s. It was developed at the Naval Ordinance Laboratory in Silver Springs, Maryland. It was thus named Nitinol, for its chemical components and location of development (Thompson 2000). The alloy's orthodontic application then came about in the early 1970s by Andreasen and his colleagues (Santoro, Nicolay, and Cangialosi 2001). After extensive research published primarily in material science and orthodontic literature, Ni-Ti has found a place in multiple areas of dentistry, including endodontic files, prosthodontic castings for crowns and denture construction, and surgical bone plates (Thompson 2000).

The original Nitinol does not exhibit phase transformation due to the work-hardening in the manufacturing process. Despite not having this thermoelastic effect, the original alloy was still useful in cases with considerable deflections due to its low modulus of elasticity and high working range. In the 1980s, a new form of Ni-Ti was introduced that was able to demonstrate phase transformation. The TTR of these wires was very low. Thus, during clinical application, these austenitic wires were subject to stress-induced phase transformation, but not temperature-induced phase transformation. With further technological advances, the TTR of Ni-Ti wires can now be set at specific temperatures, allowing for both temperature- and stress-induced phase transformations during clinical use of the wires (Santoro, Nicolay, and Cangialosi 2001).

B. <u>Properties of Nickel-Titanium</u>

Ni-Ti exists in two lattice phases, the body-centered martensite phase and the face-centered austenite phase, which allow for the unique ability to return to its original shape before deformation. Later, an intermediate phase with a simple hexagonal lattice was discovered and referred to as the rhombohedral or R phase (Santoro, Nicolay, and Cangialosi 2001; Bradley, Brantley, and Culbertson 1996). In the martensite phase, the wire has high formability and is easily deformed. It can hold a bend in the wire until it undergoes a phase change into the austenite form. In comparison, a wire in the austenite phase has a higher stiffness and is able to resume a preformed shape, such as a designated archform (Santoro, Nicolay, and Cangialosi 2001). The ability of Ni-Ti to reversibly transform between these two phases is dependent on temperature and/or stress.

The temperature-dependent reversible transformation is referred to as thermoelasticity or shape memory effect (Thompson 2000; Santoro, Nicolay, and Cangialosi 2001; Burstone and Choy 2015). Ni-Ti transforms from the martensite phase to the austenite phase as its ambient temperature is raised. The crystalline structure of a wire that is completely in the martensite phase will start to transform into the austenite phase at the austenite start temperature (A_s). As the temperature continues to rise, the wire will have completely transformed into the austenite phase once the temperature reaches austenite finish (A_f). Since this transformation is reversible, a wire can transform from the austenite phase to the martensite phase as temperature is cooled. A wire that is completely in the austenite phase will start to transform into the martensite phase at a temperature, martensite start (M_s). It will be completely transformed into the martensite phase when the temperature reaches martensite finish (M_f). Between start and finish temperatures both crystalline structures exist, which is referred to as the transformation temperature range (TTR).

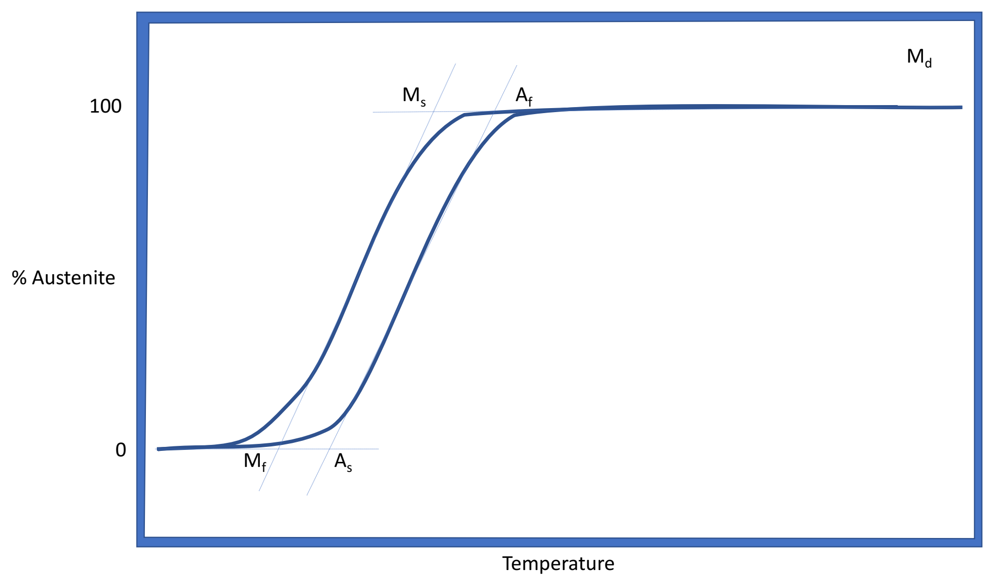

Figure 1. Graphical representation of TTR.

Stress also contributes to the transformation from an austenitic to a martensitic crystalline structure, termed pseudoelasticity, or superelasticity. An austenitic wire that is deflected under the application of stress generates a stress-induced martensite (SIM) in the area of the deflection. In this phenomenon, the martensite crystalline structure is localized to the stressed area while the rest of the wire remains in the austenitic phase. SIM is unstable and even if the temperature remains the same, the SIM will undergo reverse transformation back into the austenitic structure once the stress is removed (Santoro, Nicolay, and Cangialosi 2001; Burstone and Choy 2015).

C. <u>Nickel-Titanium Orthodontic Archwires</u>

Ni-Ti orthodontic archwires have gained popularity since they entered the field, and are now commonly seen as a standard material for the initial stage of fixed orthodontic treatment (Yoneyama and Miyazaki 2008). The initial stage of leveling and aligning in orthodontics typically involves unraveling crowded teeth Thus, it requires a wire that has a low modulus of elasticity in order to engage crowded teeth without permanent deformation as well as adequate force to move teeth, making Ni-Ti alloys the most appropriate choice.

As previously discussed, the original Nitinol did not exhibit phase transformation. It had a very high TTR, remaining martensitic at all times. Compared to the newer forms of Ni-Ti, this wire had a higher stiffness and lower resilience. However, compared to the stainless steel and beta titanium wires that were available at the time, these characteristics were improved for the leveling and aligning stage (Santoro, Nicolay, and Cangialosi 2001; Yoneyama and Miyazaki 2008; Burstone and Choy 2015).

The TTRs of the next generation of Ni-Ti archwires are near or below room temperature (Santoro, Nicolay, and Cangialosi 2001; Burstone and Choy 2015). Consequently, these wires are called superelastic archwires due to their superelastic properties. These wires have TTR values near or below room temperature, making them austenitic at room temperature as well as body temperature and allows for the SIM properties when the wire is deflected. However, these higher stiffness and higher force austenite wires do not utilize thermoelastic properties to make ligation easier. Today, these are the wires that are most commonly used by clinicians for leveling and aligning.

The most recent forms of Ni-Ti allow for the TTR to be set above room temperature. One group of these shape memory, or thermoelastic, wires have the TTR set above room temperature but below body temperature. These wires are martensitic at room temperature, making ligation of the archwire easier with the lower force martensite form. Once engaged in the brackets and the intraoral temperature heats the wire, the wire becomes austenitic. This activation increases the force and stiffness of the wire to result in tooth movement. In areas of severely misaligned teeth, SIM is formed, producing an area of lower force. Once the tooth begins to move, reducing the stress, that area of the wire becomes austenitic again.

Another group of these thermoelastic wires have the TTR set above both room temperature and body temperature. These wires remain in the low-force martensite form as it is ligated as well as during treatment but only have increased forces when the patient consumes hot foods and beverages. These wires are intended to provide more gentle activation for patients with periodontal concerns or those with higher sensitivity to pain (Yoneyama and Miyazaki 2008).

D. <u>Effect of Strain and TTR</u>

Orthodontic literature has demonstrated that deflection of an austenitic wire, which creates an area of SIM transformation, results in increased transformation temperatures (Santoro, Nicolay, and Cangialosi 2001). This finding supports mathematical calculations, since “According to the Clausius-Clapeyron equation, the reverse martensitic transformation finishing temperature (A_f) should be elevated gradually to higher temperatures with an increasing recovery stress” (Zheng et al. 2001). Coluzzi et al. placed two different NiTi alloys under varying bending strains ranging from 0% to 11% (Coluzzi et al. 1996). The authors altered the strain by placing wires into the grooves of circular plexiglass plates of varying radii and measured wire transformation by the change in electrical resistance. They noted that the total change in electrical resistance was higher for the undeformed wire and decreased as strain increased. Additionally, their results indicated that the rhombohedral start temperature (R_s) increased as the loading increased for the two tested wire types. Santoro and Beshers tested five types of orthodontic wires in 1 mm and 6 mm step rectangular plexiglass platforms that represented one displaced anterior mandibular tooth. The platforms were built based on the average of 15 minimal crowding cases and 15 severe crowding cases, respectively (Santoro and Beshers 2000). The authors measured wire transformation by the change in electrical resistance and confirmed the trend of higher A_f temperatures with increased stress.

Outside of orthodontics, Cui et al. studied Ni-Ti embedded in an aluminum matrix in order to create a 4% prestrain in the Ni-Ti. They used DSC to measure the transformation temperatures and found that there was a 15 K increase in A_s in the 4% prestrained Ni-Ti compared to the unstrained specimen (Cui et al. 2000). Also outside of orthodontic literature, Drexel et al. used the amount that the wire is displaced in the bend and free recovery technique to measure phase transformation (Drexel, Proft, and Russell 2009). The authors tested Ni-Ti wires at two different strain levels, at 2.4% and 5.8%. They also found that increasing the outer fiber strain from 2.4% to 5.8% resulted in a shift in transformation temperature by about 1°C.

E. <u>Methods for Testing TTR</u>

There are several testing methods that exist for determining TTR, with the most commonly used being differential scanning calorimetry (DSC) and bend and free recovery (BFR). While testing for this important wire property in dental literature has typically utilized differential scanning calorimetry (DSC), BFR is "by far the most simple and often the most useful method to measure A_f" (Pelton, Dicello, and Miyazaki 2000). Pelton et al. compared the two testing methods and described that in the BFR technique, the wire is cooled to a low temperature, bent to a prescribed strain (2-3%), and allowed to return to its original shape as it is heated. Comparatively, DSC measures the amount of heat that is released and absorbed by the wire during the phase transformation since a martensitic transformation is exothermic while an austenitic transformation is endothermic. The DSC method tests wires after they are cut and processed, and does not give the ability to change the amount of strain that is placed on the wire without an external medium, such as an aluminum or cement composite, as seen in studies conducted by Cui et al. (Cui et al. 2000) and Zheng et al. (Zheng et al. 2001). Thus, the test is not representative of the alloy's clinical use. In contrast, BFR is able to measure transformation temperatures after the wire is placed under varying bending strain, which more closely approximates its clinical application. In the previously discussed studies that investigate the effect of strain on transformation temperature, the studies needed to create loading devices for each testing specimen that is made from an electrically insulated material, such as plexiglass. Stated in the ASTM F2004 for DSC/thermal analysis in the "Significance and Use" section, "transformation temperatures derived from differential scanning calorimetry (DSC) may not agree with those obtained by other test methods due to the effects of strain and load on the transformation" (ASTM International 2017). Additionally, both the "Significance and Use" and "Rationale" sections of ASTM F2082 for BFR states that "transformation temperatures measured by this test method will differ from those measured by thermal analysis or other techniques as a result of the effects of strain and load"

(ASTM International 2016). Studies utilizing other methods of testing have indicated that strain increases transformation temperature. Thus, temperatures reported with DSC as the testing method may be significantly underestimating the actual transformation temperatures of the wire during clinical application.

Due to these statements in the standards for TTR testing of Ni-Ti wires, it would be very beneficial to determine how strain and load actually affect the transformation temperature of Ni-Ti. Since the amount of strain placed on the testing wire cannot be altered in the DSC method without placing the wire in an external medium, this study will utilize the BFR technique. Therefore, this study will examine the effects of strain on the transformation temperature ranges of various thermoelastic Ni-Ti orthodontic archwires using BFR testing.

III. MATERIALS AND METHODS

A. Design

This study used commercially available archwires from two manufacturers: 27°C Copper Ni-Ti (CuNiTi) from Ormco (Glendora, California) and Thermaloy Ni-Ti from Rocky Mountain Orthodontics (RMO, Denver, Colorado). Each manufacturer was asked to provide two different sized wires: 0.014 inch (0.3556 mm) and 0.019 inch x 0.025 inch (0.4826 mm x 0.635 mm). Manufacturers were asked to ensure that wires of the same size were all from two different production lots (Ormco 0.019 inch x 0.025 inch lots 071738551 and 091790478; Ormco 0.014 inch lots 061711895 and 071728831; RMO 0.019 inch x 0.025 inch lots F1617891 and F1700432; RMO 0.014 inch lots F1700205 and F1617792). All specimens were stored at room temperature prior to testing. Similar to the study completed by Obaisi et al., "since a closed BFR testing system was not used, [the specimens were] randomized to account for the potential environmental differences within the laboratory at different times…[specimens were numbered by an outside participant and the] numbers were then randomized using the randomization feature in Microsoft® Excel (Redmond, WA, USA) to determine the order of testing" (Obaisi et al. 2016).

Strain is the percentage of physical deformation of a material in response to stress; it is defined as the change in length of the material due to stress divided by the initial length of the material. In terms of the BFR test method, the wire being tested is experiencing compression on the surface in contact with the mandrel of the apparatus and tension on the surface further from the mandrel. As a result, the amount of bending strain is equal to the diameter of the wire divided by the sum of the diameter of the wire and the diameter of the mandrel of the apparatus. Thus, either varying the diameter of the wire or the diameter of the mandrel can alter strain.

In order to alter the strain by the diameter of the mandrel of the testing apparatus, the two wire sizes were bent around mandrels with diameters of 0.625 inch (15.875 mm) and 0.4 inch (10.16 mm). Also, when wires have different dimensions, bending them around the same mandrel produces different strains. Therefore, in order to alter the strain by the diameter of the wire being tested, wires with a dimension of 0.019 inch x 0.025 inch (0.4826 mm x 0.635 mm) were compared to wires with a dimension of 0.014 inch (0.3556 mm). This creates eight total subgroups, as illustrated in Figure 2. Based on previous studies (Obaisi et al. 2016), each subgroup had 12 wires tested from two different production lots.

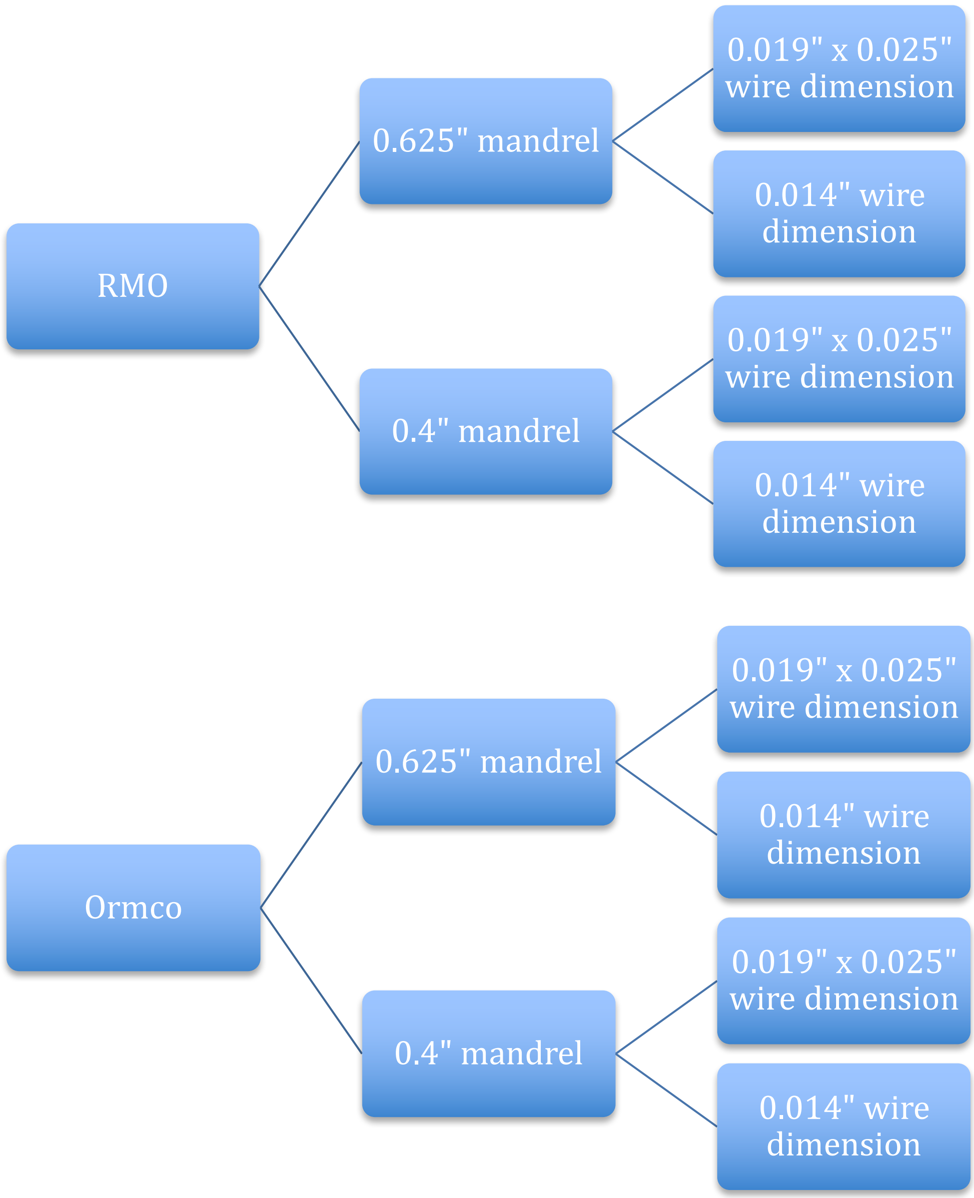

Figure 2. Sample groups for each company. Eight subgroups with 12 wires from two different production lots tested in each subgroup.

Bending strain was then calculated for each subgroup and reported as a percentage using the equation

$$\varepsilon = (d)/(R + d),$$

where d is the diameter of the wire and R is the diameter of the mandrel. The strain calculations for each of the wire-mandrel combinations are listed in Table I.

TABLE I

STRAIN CALCULATIONS

	0.625” mandrel	0.4” mandrel
0.014” wire	ε = (0.014)/(0.625 + 0.014) **ε = 2.19%**	ε = (0.014)/(0.4 + 0.014) **ε = 3.38%**
0.019” x 0.025” wire	ε = (0.025)/(0.625 + 0.025) **ε = 3.85%**	ε = (0.025)/(0.4 + 0.025) **ε = 5.88%**

B. <u>BFR Procedure</u>

The BFR procedure was adapted from the study completed by Obaisi (Obaisi 2013). The wires were tested as they were received from the manufacturer (without being cut) in order to prevent adding additional stresses to the archwire.

The wires were tested 15 mm from the end of each archwire in the straight portion of the archwire. Testing was completed in the parallel orientation to the existing arch shape of the wire since the perpendicular orientation introduces additional strain. This then, would mimic the clinical performance of the wire as a tooth moves buccolingually. Thus, strain calculations for the rectangular archwires utilize the width, or larger 0.025 inch dimension, of the wire. The wires were stored at room temperature until the time of testing. The BFR procedure involved the following components:

i. Linear variable differential transducer (LVDT)

ii. Thermocouple and indicator with a resolution of 0.1°C

iii. Automated data acquisition system

iv. Hot plate and stirrer

v. Water bath

vi. Mandrel

vii. Recovery fixture clamp

viii. Wire forming lever

A water-glycerin solution was placed in the water bath to a level that would still completely cover the archwire even after it had recovered to its original shape. The prepared bath had a starting temperature of -20°C or colder. Once the glycerin water bath was prepared, the BFR testing apparatus used in this study, or recovery temperature testing apparatus (RTTA), was calibrated. Using a permanent marker, the wires were marked 20 mm from one end where the wire would be clamped with the recovery fixture clamp, and marked again 15 mm from the same end from where the wire would be measured and where the LVDT core would rest on the wire. The apparatus, including the recovery fixture, mandrel, and wire, were submerged in the water-glycerin bath as seen in Figure 3.

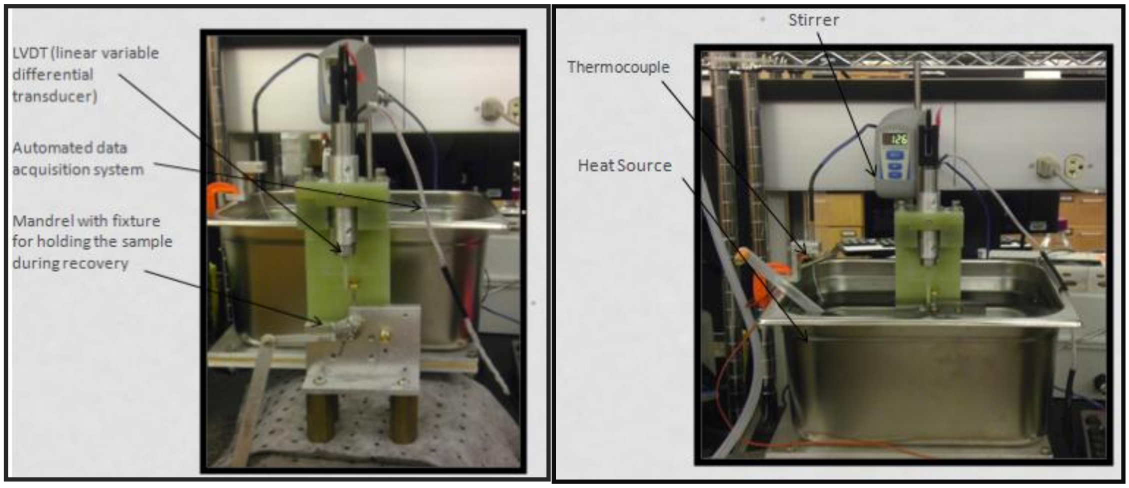

Figure 3. RTTA submerged in water-glycerin bath [Created by Obaisi, Noor Aminah. 2013. "Determination of the Transformation Temperature Ranges of Orthodontic Nickel-Titanium Archwires." Image courtesy of Noor Obaisi.]

The thermocouples were then positioned in the bath as close as possible to the test specimen. In order for the wire and RTTA parts to equilibrate with the water-glycerin bath temperature, the wire and testing apparatus were kept in the water-glycerin solution for a minimum of 3 minutes prior to testing.

After 3 minutes, the wire was bent against the mandrel by moving the wire forming lever over the wire and lowering the LVDT core onto the wire. In order to minimize the effects of any forces applied to the wire as it transitioned from the martensite to the austenite phase, the weight of the LVDT core was counterbalanced to allow no more than 3 grams of force on the wire as seen in Figure 4. The data acquisition system, hot plate, and stirrer were then turned on.

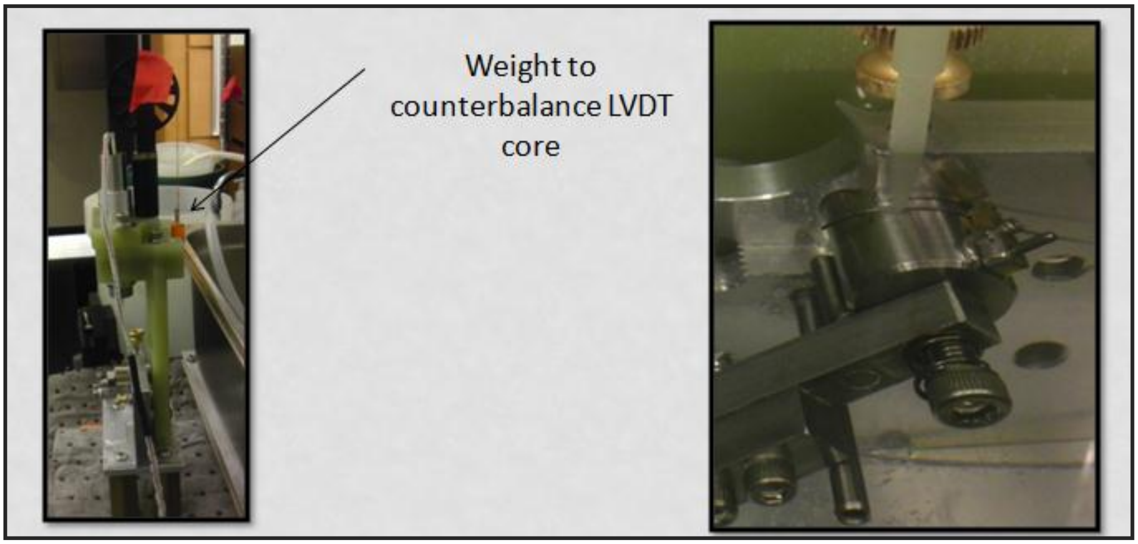

Figure 4. Wire bent around mandrel and counterbalance weight [Created by Obaisi, Noor Aminah. 2013. “Determination of the Transformation Temperature Ranges of Orthodontic Nickel-Titanium Archwires.” Image courtesy of Noor Obaisi.]

The water-glycerin bath was heated to 50°C at a heating rate of 1.40-1.60°C/min. In accordance with the BFR standard, the stopping point for the test should be at 100% recovery, or at least 10°C above the flattened displacement versus temperature graph (ASTM International 2016). Both the hot plate and data acquisition system were stopped at this point in time.

The acquired data were saved as a text file and exported to Microsoft® Excel. A Temperature–Displacement graph was plotted and tangent lines were drawn by the examiner in order to determine A_s and A_f by tangent methodology as seen in Figure 5.

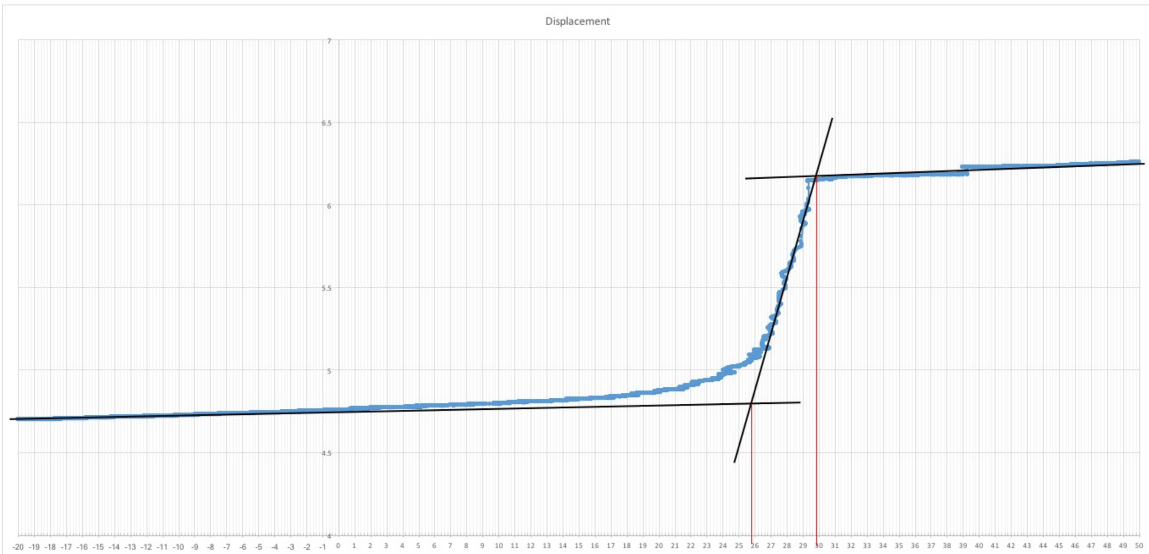

Figure 5. Temperature-Displacement graph. Tangent lines drawn by examiner in order to determine A_s and A_f.

C. <u>Statistical Method</u>

To determine the intra-reliability measurements of the BFR method used in this study, the examiner tested five wires at two separates time points. A_s and A_f temperatures were calculated using the described method. The intra-class correlation was determined for these two variables, and it was determined that the correlation coefficient for the variables was higher than 0.90 (0.997 for A_s and 0.917 for A_f, with a p-value <0.001 and 0.017, respectively), indicating a high degree of reliability for the test-retest measurements.

For the A_s and A_f temperatures obtained from the test specimens, a Shapiro-Wilks test for normality was conducted. The Shapiro-Wilks test found that the transformation temperatures are not normally distributed. Since the data is not normally distributed, nonparametric Mann-Whitney tests for two independent samples were conducted for statistical analysis. Despite using a nonparametric test for statistical analysis, descriptive statistics were run in order to obtain information regarding mean and standard deviation in order to compare the obtained results with other literature and for more clinical application. SPSS (version 22.0, IBM Corp., Armonk, NY) was used for statistical analysis.

IV. RESULTS

A. Comparison Between Mandrel Size

Mann-Whitney tests were conducted in order to determine if a mean difference exists between transformation temperatures (A_s and A_f) of wires that are bent around a mandrel of 0.4 inch or 0.625 inch diameter. Table II summarizes the statistical results:

TABLE II

COMPARISON OF A_S AND A_F BETWEEN MANDRELS

Wire Diameter	Variable by Manufacturer		N	0.4" Diameter Mandrel: Mean Rank	0.625" Diameter Mandrel: Mean Rank	p-value*
0.019" x 0.025"	RMO	A_s	12	18.50	6.50	0.000*
		A_f	12	16.83	6.50	0.002*
	Ormco	A_s	12	16.04	8.96	0.012*
		A_f	12	10.17	14.83	0.114
0.014"	RMO	A_s	12	15.50	9.50	0.039*
		A_f	12	14.33	10.67	0.219
	Ormco	A_s	12	14.08	10.92	0.291
		A_f	12	12.08	12.92	0.799

*p-values statistically significant at $\alpha \leq 0.05$.

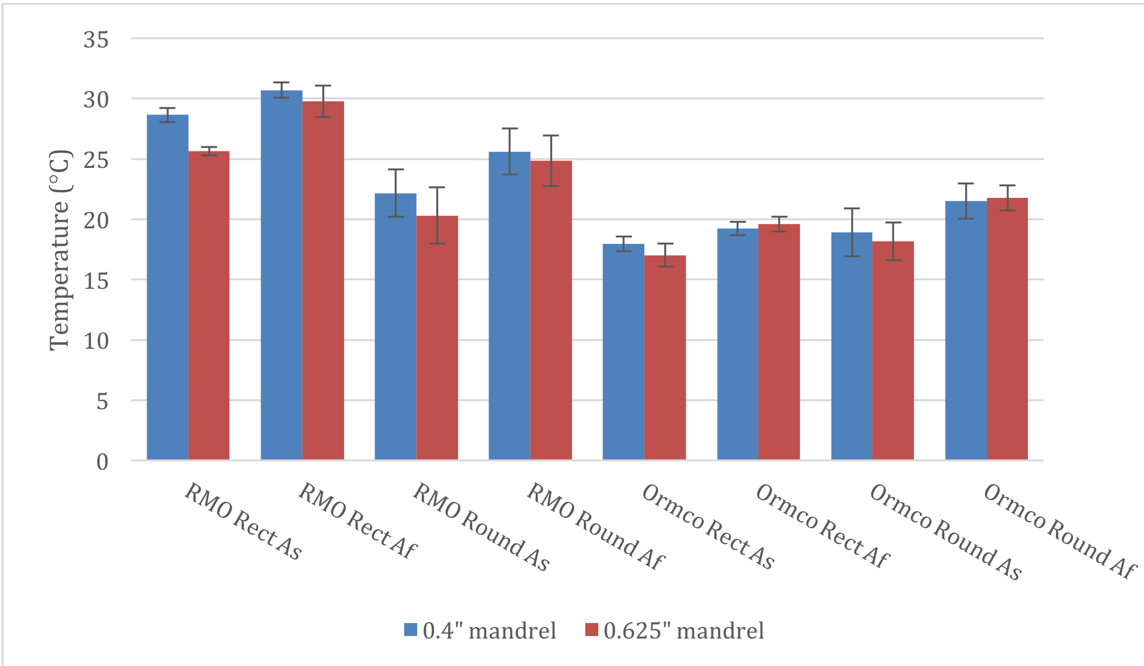

Figure 6. Comparison of A_s and A_f temperatures between mandrels

There was a statistically significant difference for both A_s and A_f of RMO rectangular wires ($p=0.000$ and $p=0.002$, respectively) when testing between mandrel size. For both upper and lower bounds of TTR, the mean rank was comparatively higher for the 0.4 inch mandrel. There was also a statistically significant difference between A_s of RMO round ($p=0.039$) and Ormco rectangular ($p=0.012$) wires when comparing the two mandrel sizes. For austenite start temperatures of both of these wires, the mean rank was comparatively higher for the 0.4 inch mandrel.

B. Comparison Between Wire Diameter

Mann-Whitney tests were conducted in order to determine if a mean difference exists between transformation temperatures (A_s and A_f) when wires of different dimensions were bent around test mandrels of the same diameter. Table III summarizes the statistical results:

TABLE III

COMPARISON OF A_S AND A_F BETWEEN WIRE DIAMETERS

Mandrel Diameter	Variable by Manufacturer		N	0.019" x 0.025" Wire: Mean Rank	0.014" Wire: Mean Rank	p-value*
0.4"	RMO	A_s	12	18.50	6.50	0.000*
		A_f	12	18.50	6.50	0.000*
	Ormco	A_s	12	9.33	15.67	0.028*
		A_f	12	7.54	17.46	0.000*
0.625"	RMO	A_s	12	18.50	6.50	0.000*
		A_f	12	18.50	6.50	0.000*
	Ormco	A_s	12	8.04	16.96	0.001*
		A_f	12	6.96	18.04	0.000*

*p-values statistically significant at $\alpha \leq 0.05$.

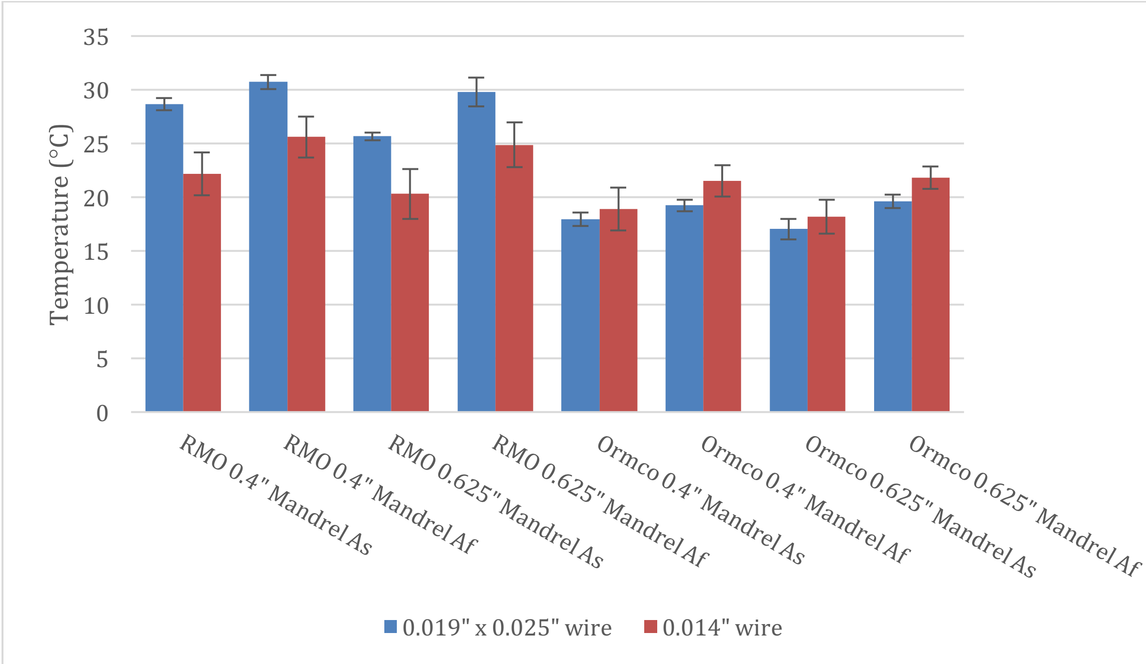

Figure 7. Comparison of A_s and A_f temperatures between wire diameter

There was a statistically significant difference between wire dimensions for both A_s and A_f for both mandrel dimensions and both companies. For all transformation temperatures for RMO wires, the mean rank was comparatively higher for the 0.019 inch x 0.025 inch wires. However, for all transformation temperatures of Ormco wires, the mean rank was comparatively higher for the 0.014 inch wires.

C. **<u>Comparison of TTR</u>**

Using descriptive statistics, the mean of each subgroup was calculated. The TTR was calculated from the difference between values of A_f and A_s. The values are listed in Table IV:

TABLE IV

MEAN AND STANDARD DEVIATION A_S AND A_F WITH TTR

Company	Wire Dimension	Mandrel Diameter	A_f $\bar{X} \pm$ S.D. (°C)	A_s $\bar{X} \pm$ S.D. (°C)	TTR ± S.D. (°C)
RMO	0.014”	0.4”	25.6 ± 1.9	22.2 ± 2.0	3.4 ± 0.9
		0.625”	24.8 ± 2.1	20.3 ± 2.3	4.5 ± 0.9
	0.019” x 0.025”	0.4”	30.7 ± 0.6	28.7 ± 0.6	2.0 ± 0.8
		0.625”	29.8 ± 1.3	25.7 ± 0.4	4.1 ± 1.4
Ormco	0.014”	0.4”	21.5 ± 1.5	18.9 ± 2.0	2.6 ± 0.8
		0.625”	21.8 ± 1.0	18.2 ± 1.6	3.6 ± 1.0
	0.019” x 0.025”	0.4”	19.2 ± 0.5	18.0 ± 0.6	1.3 ± 0.6
		0.625”	19.6 ± 0.6	17.0 ± 1.0	2.6 ± 0.9

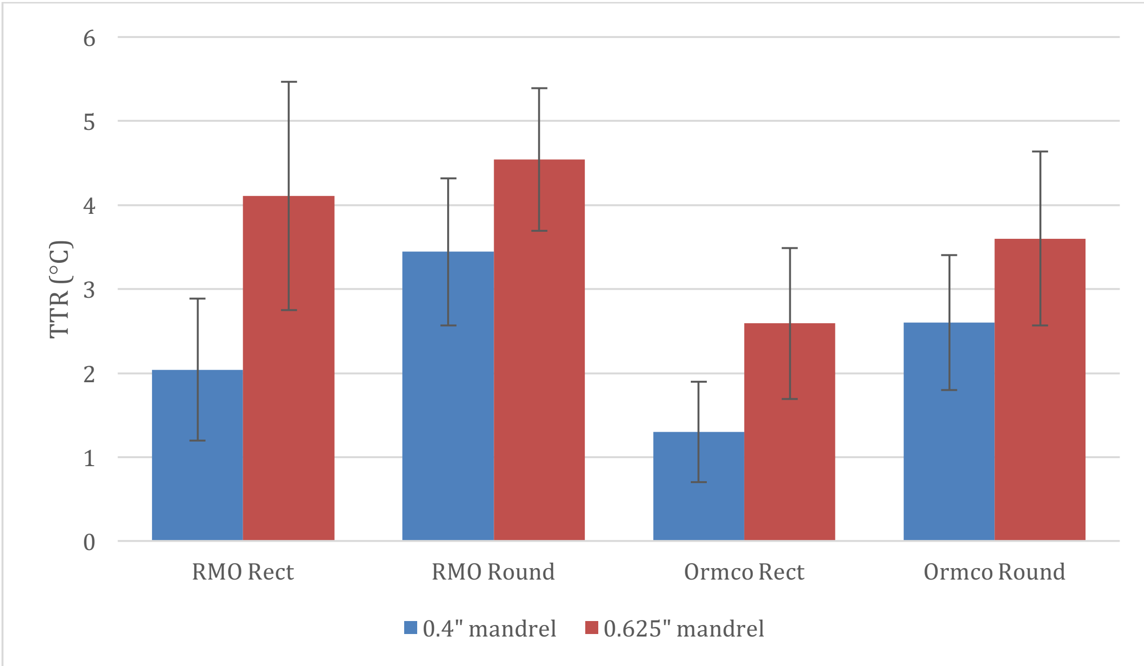

Figure 8. Comparison of TTR between mandrels

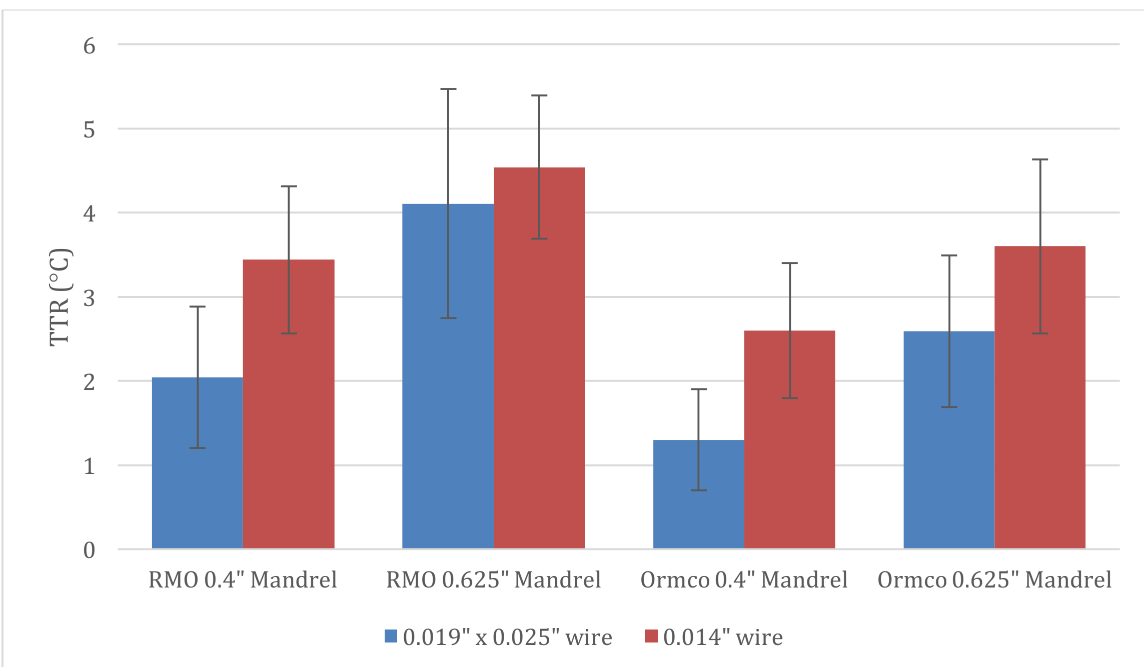

Figure 9. Comparison of TTR between wire diameter

For both companies, regardless of the method of increasing strain, whether via a smaller mandrel diameter or a larger wire dimension, a narrower range for TTR was observed.

D. <u>Comparison Between Tested Value and Manufacturer-Listed Value</u>

The transformation temperatures that were reported by RMO for their Thermaloy Ni-Ti archwires was 13°C ± 5°C for A_s and 23°C ± 5°C for A_f. These temperatures were obtained using the DSC method of testing transformation temperature. RMO also reported an A_f temperature for the Thermaloy wires using a water bath test in which the Ni-Ti wire was strained and the temperature at which the Ni-Ti wire reverted back to its straight length was recorded as A_f. Although this test is similar to the BFR method, this test was completed prior to the ADA/ISO standard being issued. Additionally, no information was given regarding the percentage of strain that was used. Regardless, temperatures from both methods were included for comparison. Ormco reported an A_f of 27°C ± 2°C for their 27°C CuNiTi. Upon further inquiry, information regarding A_s was deemed by Ormco as proprietary information, and thus, not reported. The temperature that was reported was obtained using the DSC method of testing transformation temperature.

Since nonparametric tests were used in this study due to the non-normal distribution of the data, median values and Wilcoxon Signed Rank Tests were used to compare study results against the manufacturer reported temperatures.

TABLE V

COMPARISON BETWEEN TESTED AND MANUFACTURER-LISTED VALUES

Company	Testing Method	Variable	Reported Temperature (°C)	Median (°C)	Difference (°C)	p-value*
RMO	DSC	A_s	13	20.55	7.55	0.002*
	DSC	A_f	23	25.4	2.4	0.034*
	Water bath	A_f	32	25.4	-6.6	0.002*
Ormco◊	DSC	A_f	27	22.25	-4.75	0.002*

*p-values statistically significant at $\alpha \leq 0.05$.
◊Ormco deemed A_s values as proprietary information.

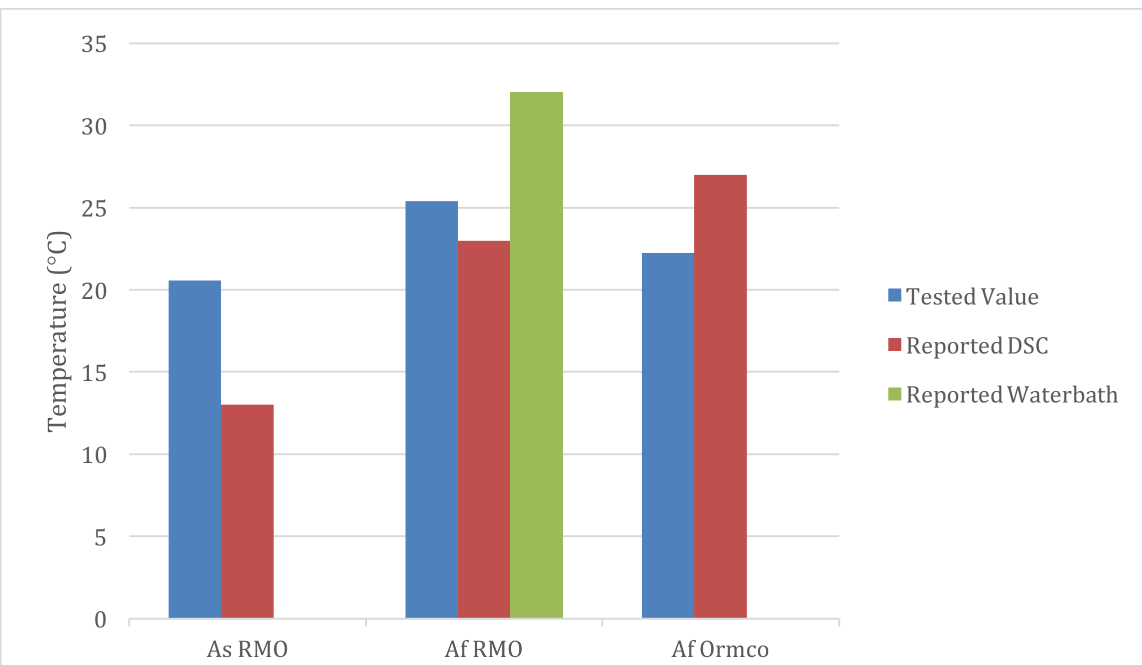

Figure 10. Comparison with manufacturer-listed values

For all transformation temperatures that were reported by both manufacturers, there was a statistically significant difference when compared to the transformation temperatures obtained in this study.

V. DISCUSSION

A. Temperature Variation with Strain

This study varied the strain applied to Ni-Ti wires by two different methods. The first method was bending the wire around mandrels of different diameters, 0.625 inch and 0.4 inch. For a 0.019 inch x 0.025 inch rectangular wire, the smaller diameter mandrel increases the outer fiber strain from 3.85% to 5.58%. Likewise, for a 0.014 inch round wire, the smaller diameter mandrel increases the outer fiber strain from 2.19% to 3.38%. Another method of altering strain is to bend wires with different dimensions around a test mandrel of the same diameter; round wires with 0.014 inch diameter and rectangular wires with 0.019 inch x 0.025 inch dimension were used. For a 0.4 inch diameter mandrel, the larger diameter wire increases the outer fiber strain from 3.38% to 5.58%. Likewise, for a 0.625" diameter mandrel, the larger diameter wire increases the outer fiber strain from 2.19% to 3.85%. See Table I.

When comparing the effect of increasing strain with decreasing the mandrel diameter for the 0.019 inch x 0.025 inch rectangular wire, both companies showed statistically significant difference in temperature. When comparing the effect of increasing strain with decreasing the mandrel diameter for the 0.014 inch round wire, only A_s of the RMO wires showed a statistically significant difference. All statistically significant differences resulted in a higher mean rank for wires bent around the 0.4 inch mandrel compared to the 0.625 inch mandrel. When comparing the effect of increasing strain with increasing the wire dimension, all transformation temperatures for RMO wires showed statistically significant difference in temperature and showed higher mean rank for 0.019 inch x 0.025 inch wires compared to 0.014 inch wires. These trends were expected based on mathematical calculation (Zheng et al. 2001) as well as with other studies that studied TTR with various strains regardless of the method of testing TTR (Coluzzi et al. 1996; Santoro and Beshers 2000; Cui et al. 2000; Drexel, Proft, and Russell 2009). The smaller diameter mandrel and larger dimension wire results in a higher bending strain and recovery stress, and thus, more energy in the form of heat was required for phase transformation.

Coluzzi et al. placed two different NiTi alloys under varying bending strains ranging from 0% to 11% (Coluzzi et al. 1996). The authors altered the strain by placing wires into the grooves of circular plexiglass plates of varying radii and measured wire transformation by the change in electrical resistance. Their results indicated that the rhombohedral start temperature (R_s) increased as the strain increased for both the wire types that they had tested. Their results also indicated that as strain increases, the TTR narrows. Using the descriptive statistics that were obtained, this conclusion agreed with the results of the current study, as seen in Figures 6 and 7.

Santoro and Beshers also measured transformation temperatures with electrical resistance. They constructed plexiglass platforms with 1 mm and 6 mm steps to represent one displaced anterior mandibular tooth (Santoro and Beshers 2000). Despite not having a calculated amount of strain, their plexiglass platforms still found a trend of higher A_f temperatures with increased stress. Additionally, similar to the results in Coluzzi et al. and the current study, Santoro and Beshers also noted a smaller range in TTR with the increase of stress. Cui et al. measured transformation temperatures with DSC, and embedded the Ni-Ti in an aluminum matrix in order to create a 4% strain. They found a 15 K increase in A_s in the strained Ni-Ti compared to the unstrained specimen (Cui et al. 2000). Similar to the current study, Drexel et al., used the bend and free recovery technique to measure transformation temperatures (Drexel, Proft, and Russell 2009). The authors tested Ni-Ti wires at two different strain levels, at 2.4% and 5.8%. They also found that increasing the outer fiber strain from 2.4% to 5.8% resulted in a shift in transformation temperature by about 1°C. Despite all the differences in these studies, these authors all found increases in either upper (A_f) or lower (A_s) bound of TTR as the strain on Ni-Ti was increased.

In comparison, when comparing the effect of increasing strain with increasing the wire dimension, all transformation temperatures for Ormco wires showed statistically significant difference in temperature. However, they showed higher mean rank for 0.014 inch wires compared to 0.019 inch x 0.025 inch wires. This discrepancy may be due to the specific manufacturing lots from which the tested 0.014 inch wires and the tested 0.019 inch x 0.025 inch wires were obtained. The study completed by Obaisi found lot variation between half of the lots that were tested in that study (Obaisi 2013). Additionally, Pompei-Reynolds and Kanavakis studied the consistency of properties of CuNiTi between lots of the same two manufacturers. The authors found “notable differences among production lots of the same wire type from the same manufacturer” and “that certain archwires made by Ormco had greater interlot variabilities in transition temperature and force delivery values” (Pompei-Reynolds and Kanavakis 2014). From the DSC graphs in that study, the authors explain that the more parallel the plots of each wire are, the greater the consistency in transformation temperatures for each wire in every lot. The results indicated that the plots for RMO wires were near parallel and indicated greater consistency, whereas those for Ormco wires were notably less parallel. The authors explained that “wire properties are extremely sensitive to the alloy ratio; small amounts of dissolved interstitial elements act as impurities and disrupt the NiTi crystal matrix and therefore its transformation behavior” (Pompei-Reynolds and Kanavakis 2014). This results in interlot differences even with the same manufacturer since the manufacturing materials and conditions may not be tightly regulated from site to site. Similarly, Fernandez et al. expounds that “a very small excess nickel in structure can reduce TTR and increase the permanent yield strength of the austenite phase by roughly threefold” (Fernandes et al. 2011). Thus, Ni-Ti production is extremely technique sensitive and the variation between production lots even from the same manufacturer could have attributed to the inconsistency of the observed trend for one of the manufacturers in the present study. These observations also draw attention to the need to enforce a standardized testing of TTR for orthodontic wires.

B. Variation Between Tested Value and Manufacturer-Listed Value

According to Pelton et al, with the bend and free recovery method, the Ni-Ti sample should be bent to a prescribed strain of 2-2.5% (Drexel, Proft, and Russell 2009). As result, when comparing the tested transformation temperatures to the manufacturer-listed temperatures, only 0.014 inch wires tested on the 0.625 inch mandrel were used since only this subgroup had a strain between 2-2.5%. There was a statistically significant difference between all tested values and manufacturer-listed TTR values. Only RMO reported an A_s value, which was obtained using the DSC method. The study completed by Obaisi compared DSC and BFR testing methods and found all A_s to be different between the two test methods to a statistically significant degree (Obaisi 2013). Additionally, the study noted that A_s values were consistently lower with the DSC method of testing compared to BFR. This study attributed the discrepancy as a result of interpretation of the DSC graph, "since many [specimens] showed double peaks due to an R-phase…[which makes] determination of A_s more difficult" (Obaisi 2013). Therefore, it may be difficult to distinguish when the rhombohedral phase ends and the austenite phase begins.

The A_f values using the DSC method were reported for both companies but were both statistically significantly different. The difference between the tested value and the manufacturer-reported value for RMO and Ormco was 2.4°C and 4.75°C, respectively. While these values are statistically significant, they may not necessarily be clinically significant since there is such a large range in intraoral temperature (Airoldi et al. 1997; Moore et al. 1999). On the other hand, Ormco markets their wires with three different transformation temperatures: 27°C, 35°C, and 40°C. If 5°C is enough to warrant an additional line of Ni-Ti wires, the argument can be made that a 4.75°C difference is actually clinically relevant.

RMO also reported an A_f value that was obtained using a water bath method. This reported value is similar to the BFR method of testing, which was statistically significantly higher than the tested value in this study. Despite being a more similar method of testing, this value was only included for comparison since the water bath testing was completed prior to BFR standards. Additionally, information regarding the amount of strain that was used in the water bath testing was not obtained, and due to the concluded effects of strain on TTR values, likely was a higher percentage of strain in RMO's water bath testing that may be more comparable to the higher strain groups used in this study.

C. <u>Clinical Significance</u>

The statistically significant differences in temperature indicate that strain does have an effect on the properties of Ni-Ti orthodontic archwires, specifically, transformation temperature and TTR. Clinically, patients present with varying degrees of crowding, which translates into varying levels of strain when the archwire is ligated. This variation in strain, and thus transformation temperature should be taken into consideration when a clinician is selecting an appropriate Ni-Ti archwire with corresponding appropriate transformation temperatures for a patient. Countless factors affect a patient's intraoral temperature, including baseline core body temperature, the temperature of foods and drinks that a patient consumes, mouth-breathing and lip incompetency (Moore et al. 1999). As a result, these factors will also affect wire phase properties. It is not possible for a clinician to control for all of these factors, but the more factors that are taken into account when a clinician selects an appropriate archwire for a patient, the more predictable the treatment outcome will be and the more efficient a clinician will be with the initial leveling and aligning phase of orthodontic treatment. For example, in the study by Moore et al., the authors recorded intraoral temperatures of twenty male subjects over a period of 24 hours (Moore et al. 1999). While archwires with a transformation temperature of 40°C are manufactured, the results of this study indicate that only a few of the subjects even reached an intraoral temperature of 40°C. If these subjects were undergoing orthodontic treatment, the Ni-Ti wires with a transformation temperature of 40°C would not be appropriate, as the wires would be fully active for only a very brief period of time each day. If they had significant crowding, increasing the strain and transformation temperature on the orthodontic archwire, it would be active for an even shorter period of time.

Additionally, the tested TTR values were statistically significantly different compared to the values reported by the manufacturers, which highlights the need for enforcing product standardization and reporting of TTR values. While there are both national and international standards in place, manufacturers are hesitant to divulge this information and deem it as proprietary information and will at best only disclose information when questioned. Having accurate and transparent information regarding the transformation temperature, and thus the wire properties, will provide orthodontists with the necessary information in order to select an appropriate archwire for their patients' needs. Despite the importance of this information and additionally having standards in place, the U.S. Food and Drug Administration (FDA) does not enforce manufacturers to comply with standards or even report the transformation temperatures of wires.

D. Strengths and Limitations to the Current Study

The BFR procedure was adapted from the study completed by Obaisi (Obaisi 2013). As described in that study, the RTTA apparatus was designed for reproducibility and also had the benefit of clinical relevance by testing TTR utilizing the shape memory properties of Ni-Ti. Utilizing the same machine in the study by Obaisi, the entire archwire was tested as-received by the manufacturer. The wire did not need to be manipulated prior to testing in order to avoid introducing additional strain to the wire. Furthermore, one investigator performed all testing in order to limit introduction of any variation in testing technique. An additional strength of the current study was the large sample size of 12 specimens that was tested for each subgroup, which increased the power of the study.

Similar to the study by Obaisi et al., a closed system was not used for testing with the BFR apparatus. Due to the potential variations in the surrounding environment, the tested archwires were randomized by an outside participant to account for possible differences.

One of the main limitations to the current study was the number of production lots that were tested. Since previous studies have illustrated differences between production lots from the same manufacturer (Obaisi 2013; Pompei-Reynolds and Kanavakis 2014), a larger number of production lots would have provided results that are more reflective of the wires made by each manufacturer.

E. Future Research

Further studies can be conducted using the same apparatus with additional production lots, which may have been one of the main limitations to the current study. Additionally, future studies can include additional manufacturers and wire dimensions.

Further testing could also be completed to determine if the intraoral environment has any effect on wire properties by testing wires that have been previously placed in patients. Testing can also be completed to determine the effects of thermocycling on Ni-Ti archwires, such as the study completed by Berzins and Roberts (Berzins and Roberts 2010). The temperature of the oral cavity fluctuates greatly with cold and hot liquid consumption and in general over a 24-hour period, as shown by Airoldi et al. and Moore et al. (Airoldi et al. 1997; Moore et al. 1999). As a result, Ni-Ti archwires undergo multiple phase transformations due to these intraoral temperature changes, which may result in more intermittent forces placed on teeth, as opposed to the light, continuous forces that most clinicians aim to achieve during initial orthodontic treatment.

Additionally, it would be interesting to investigate the force delivery of various wires as they undergo austenitic transformation as a function of temperature and strain. As Yoneyama explains, when an orthodontic archwire approaches A_f, a significant increase in magnitude of force delivery is noted (Yoneyama and Miyazaki 2008). Three-point bend testing is typically used to supplement TTR studies as seen in the study by Pompei-Reynolds and Kanavakis (Pompei-Reynolds and Kanavakis 2014) in order to obtain information regarding force levels. Testing is typically done at a set temperature, 37°C in the Pompei-Reynolds and Kanavakis study, and under a closed chamber. Utilizing the BFR method and three-point bend test, a BFR apparatus modified to measure force delivery may be able to provide further understanding of Ni-Ti properties.

VI. CONCLUSION

1. With exception of A_f values of Ormco archwires, transformation temperature values increased when strain was increased by using a smaller diameter mandrel for the BFR apparatus. The Ormco wires may have had varying results due to manufacturing variations in the two specific production lots that were tested.
2. All tested wires had statistically significant differences when comparing transformation temperature values when strain was increased by using a larger dimension archwire. RMO wires had statistically significant increases in transformation temperature values with increased strain, while Ormco had statistically significant decreases in transformation temperature values with increased strain. Again, the Ormco wires may have had varying results due to the two specific production lots that were tested.
3. An increased strain, regardless from decreased mandrel diameter or increased wire dimension, resulted in a narrower range for TTR for both companies.
4. The difference between tested TTR values and manufacturer-reported TTR values were statistically significantly different for both manufacturers. RMO provided more information regarding TTR values and testing compared to Ormco. While all values were statistically significantly different, they may not all be clinically significantly different.

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APPENDIX

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Regards,
Laura Liu

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