Thermal Properties of Copper Nickel-Titanium Orthodontic Archwires

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THERMAL PROPERTIES OF COPPER NICKEL-TITANIUM ORTHODONTIC ARCHWIRES

by

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ABSTRACT
THERMAL PROPERTIES OF COPPER NICKEL-TITANIUM ORTHODONITC ARCHWIRES

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Introduction: Copper Nickel-Titanium (CuNiTi) is a relatively new composition of Nickel-Titanium (NiTi) archwires that was originally patented in 1991 as part of the Ormco line of wires. The patent has now recently expired and many other orthodontic companies are making their own CuNiTi wires. Advertisement claims have focused on the laboratory benefits of adding Copper (Cu) to the NiTi, however few independent laboratory testings have been conducted on these new wires to verify claims. The purpose of this study was to conduct thermal analysis of CuNiTi for all currently available wires in two Austenite Finish (Af) variants and two commonly used archwire dimensions.

Materials and Methods: Ten as-received wires of 27°C and 35°C CuNiTi were tested in 0.018” and 0.016” x 0.022” archwire dimensions. The wires examined were Ormco Copper Nickel Titanium (Ormco, Orange, CA, USA), FLI Copper Nickel Titanium (Rocky Mountain Orthodontics, Denver, CO, USA), Copperloy Nickel Titanium (GAC, York, PA, USA), Copper Nitanium (Henry Schein/Ortho Organizers, Carlsbad, CA, USA), Truflex Copper Nickel Titanium (Ortho Technology, Tampa, FL, USA), and Tanzo Copper Nickel Titanium (American Orthodontics, Sheboygan, WI, USA). Segments of archwire were investigated by differential scanning calorimetry over the temperature range from -100°C to 100°C at 10°C per minute.

Results: There were significant differences for all values when comparing across different brands in regards to Heating endset, onset, and enthalpy as well as cooling endset, onset, and enthalpy. Some brands were very close to advertised values, however others were as far away as 4°C from advertised. In addition the difference between higher and lower Af values were as close as 1.5°C for certain brands when expecting 8°C.

Conclusions: One cannot expect to have CuNiTi wires perform similarly across different brands even when they are of the same Af and archwire dimension. For certain brands there may be very little difference between higher and lower Af variants.

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I would also like to thank Dr. Meggie Rosen for doing her thesis in combination with mine by testing the mechanical properties of these same CuNiTi wires.

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CHAPTER 1
INTRODUCTION

Wires have been used in orthodontics as the principal means of applying force to teeth. The ideal force to move teeth has been shown to be light and continuous (Proffit et al., 2013). Over the years biomaterials have improved in orthodontics to allow practitioners to get closer to the ideal of light continuous forces to achieve tooth movement. Orthodontic treatment can be broken into phases with the first phase being to level and align the teeth. This involves resolving rotations and vertical discrepancies. An ideal initial archwire would have low stiffness to deliver light forces upon activation, good range, be able to exert force over long periods, resist permanent deformation, easily engage misaligned brackets, and be affordable (Proffit et al., 2013). Once initial alignment has been achieved heavier archwires can be used to accomplish larger and more difficult movements. Currently the most popular wire for the first phase of treatment has been to use a Nickel-Titanium (NiTi) alloy (Jian et al., 2013).

Nickel-Titanium was introduced to orthodontics in the 1970s by Andreasen and since then it has been the most popular wire for initial leveling and alignment (Andreasen & Hilleman, 1971). This original near equiatomic alloy of Nickel and Titanium was shown to have a lower modulus of elasticity and greater springback compared to stainless steel (Burstone et al., 1985). These properties are possible because of the unique phase transformations demonstrated by the alloy. The two phases are martensite, stable at low temperature and high stress, and austenite, stable at high temperature and low stress. The reversible change between these two phase states allow NiTi alloys to exhibit properties of superelasticity and shape memory. The original alloy by Andreasen named *Nitinol* did indeed have a lower modulus of elasticity and was not as stiff compared to Stainless Steel but it did not exhibit either of the desirable properties of superelasticity or shape memory. Burstone (1985) and Miura (1986) came out with new nickel-titanium alloys, Chinese-NiTi and Sentalloy, which exhibited true superelasticity.

The most recent improvement to the NiTi alloy has been to add Copper (Cu) to the nickel-titanium alloy. Copper nickel-titanium (CuNiTi) was introduced by the Ormco Company (Glendora, Calif) to orthodontics in 1991 and a patent on the alloy was filed at that time. In the initial patent filed by Sachdeva in 1991, the CuNiTi formulation was formally introduced. In the patent they outlined that adding Cu to the standard binary alloy improved desired physical and mechanical characteristics. There were both mechanical and thermal properties that were theoretically improved. The mechanical properties involved reducing the stress hysteresis, having a predetermined maximum loading and minimum unloading forced, as well as reduced fatigue effects upon cyclic loading. The favorable thermal properties mainly included being able to more accurately control the Austenite Finish (Af) temperature so the wire can exhibit true shape memory. The patent has recently expired and now there is an increase in competition amongst companies to produce CuNiTi orthodontic archwires. The main reason other companies have looked to produce CuNiTi is because they have favorable laboratory characteristics that could theoretically translate to more efficient orthodontic tooth movement, and thus be advertised to do so.

One of the most common mechanical topics discussed when advertising CuNiTi is the reduced stress hysteresis. Stress hysteresis when measuring mechanically, rather than thermally, is the difference in loading and unloading stress. A synonym often used for loading is activation force and unloading is deactivation force. In the original patent, they point out that the unloading stress can be increased by increasing the amount of copper in the alloy. The thermal advantages of the Cu addition were to control Af temperature more precisely to allow the alloy to exhibit Shape Memory. Shape memory is exhibited in CuNiTi and other martensitic active alloys because of temperature regulated phase transformations. The wire is formed at a temperature well above the Af and as a result when the wire is at room temperature the grain structure imparted upon manufacturing is “remembered”. In clinical situations, when the wire is placed, the wire is often below Af and mostly martensite so the wire can be more easily engaged into misaligned brackets, but as the oral temperature approaches the Af the wire will become stiffer and revert back to its original archform.

Figure 1 shows a thermogram that is produced when CuNiTi archwire is analyzed by Differential Scanning Calorimetry (DSC). The purpose of scanning a wire by DSC is to verify the manufacturer claims of mainly the Austenite Finish (Af) temperature but other values can be deduced as well. The 35^{o}C CuNiTi wires are predominately martensitic at room temperature (21^{o}C) and go to austenite when warmed in the oral environment (37^{o}C). The heating onset represents when the wire is completely martensite and starts the transition to austenite. During this phase transformation the energy needed for phase transformation, or enthalpy, can be measured. Occasionally during the thermal energy peak there is a second ‘peak’ on the graph which is thought to represent the presence of an R-phase during the phase transformation. From the thermogram the heating endset can be seen, which represents the completion of the phase transformation to austenite. In the cooling aspect of the thermogram the phase transformation from austenite back to martensite can be seen. Again there is a similar spike in thermal energy that represents the energy needed for the phase transformation from Austenite to Martensite. Mf represents when the wire has fully transformed back to martensite.

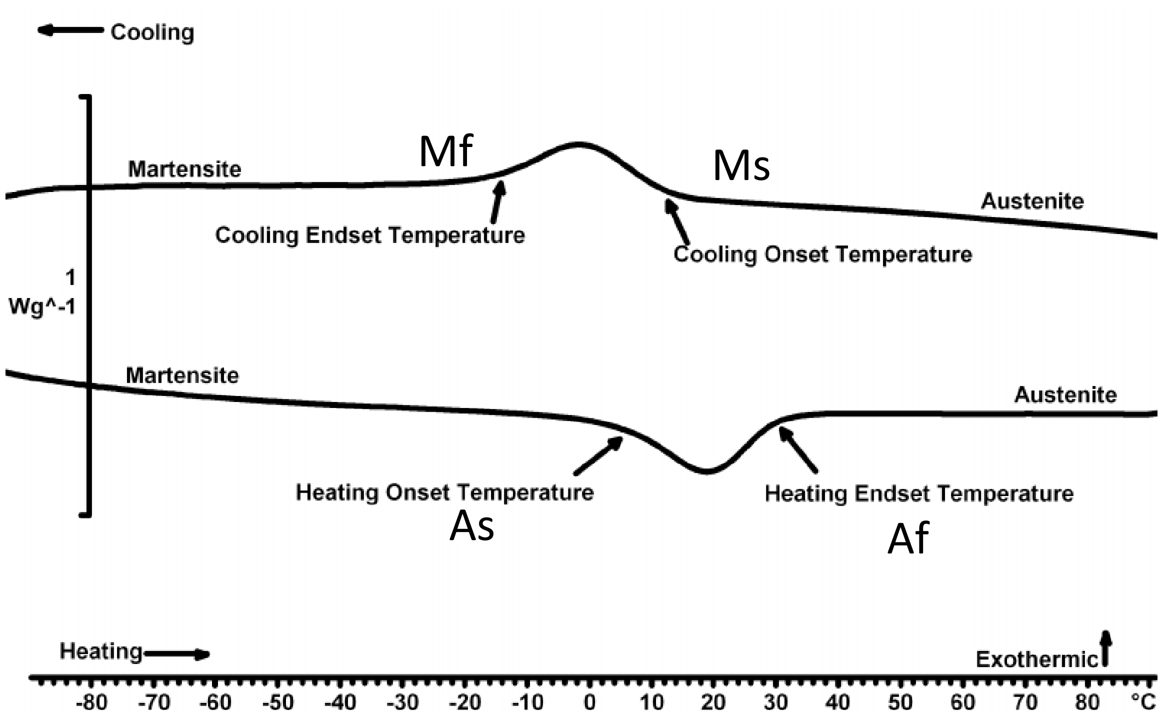

Figure 1: Example of DSC Thermogram

This new formulation of the NiTi alloy is very desirable and as a result many companies are now making CuNiTi since Ormco's patent has expired. Currently there are six companies that offer CuNiTi orthodontic archwires, all of which also offer Af variants which indirectly control force delivery. The wires that were examined are Ormco's original Copper Nickel Titanium (Ormco, Orange, CA, USA), FLI Copper Nickel Titanium (Rocky Mountain Orthodontics, Denver, CO, USA), Copperloy Nickel

Titanium (GAC, York, PA, USA), Copper Nitanium (Henry Schein/Ortho Organizers, Carlsbad, CA, USA), Truflex Copper Nickel Titanium (Ortho Technology, Tampa, FL, USA), and Tanzo Copper Nickel Titanium (American Orthodontics, Sheboygan, WI, USA). No studies in the literature have looked at the temperature transition ranges and validated the companies' claims for all the new archwires on the market. Some have been done with small sample sizes (Pompei-Reynolds & Kanavakis, 2014) and others with only the Ormco product (McCoy, 1996; Biermann et al., 2007, Kusy & Whitley, 2007), however this study tested 10 wires per variant per size.

CHAPTER 2
LITERATURE REVIEW

History of Nickel-Titanium

In the 1970s, a new orthodontics alloy of Nickel and Titanium was introduced by Andreasen and colleagues (Andreasen & Hilleman 1971). Andreasen was the first to recognize the orthodontic applications of the alloy that was originally developed by WF Buehler for the space program at the Naval Ordnance Lab (Buehler et al., 1963). This alloy received its commercial name *Nitinol* to represent its origins (Ni, Nickel; Ti, titanium; NOL, Naval Ordnance Lab). Dr. Andreasen was ahead of his time when noting that nickel-titanium archwires were quite different from stainless steel archwires in that they require less archwire changes, less chair time, may reduce treatment time through more efficient leveling and rotation control, and reduce patient discomfort (Andreasen & Morrow, 1978). This first generation nickel-titanium alloy was near-equiatomic nickel and titanium and was commercially available through the Unitek Corporation (Monrovia, CA). Early nickel-titanium wires were marketed as having shape memory however the true shape memory effect was in fact suppressed by cold working during manufacturing (Kusy, 1997). Cold working caused *Nitinol* to become passive in the martensitic stabilized structure and lose the ability for shape memory. In spite of the wire not having 'true shape memory' these wires were lauded for their low modulus of elasticity and extremely wide working range.

Improvements NiTi, and was marketed by the Ormco Company (Glendora, Calif). Burstone (1985) saw the potential for NiTi and noted that the low-load deflection rate, high springback, and relative constancy of force delivery during deactivation offer a highly useful future in orthodontic treatment. About one year later Miura (1986) introduced Japanese NiTi that was marketed by GAC (York, PA) under the name Sentalloy. The superelastic wires were reported to be austenitic active and underwent a reversible stress-induced transformation to martensite during activation and returned to austenite over a constant deactivation force. This differed from the non-superelastic wires, such as nitinol, which had stable work-hardened martensitic structures. The two 'superelastic' alloys produced on *Nitinol* were first introduced by Burstone (1985) in the form of Chinese NiTi, or characteristic stress-strain curves that had not been seen in orthodontics to that point (Kusy, 2002).

Physical Properties of Nickel Titanium

What makes nickel-titanium deliver light continuous forces is its ability to readily and reversibly change between crystalline or lattice structures. Martensite has a distorted monoclinic, triclinic, or hexagonal structure, and is more stable at low temperatures and higher stress. Austenite has an ordered bcc structure that is more stable at higher temperatures and low stress (Brantley & Eliades, 2001). The different crystalline structures of the single alloy allow transition to occur as a result of either stress or a change in temperature (Santoro, 2001). The temperature at which the alloy converts from one phase to another is known as the Temperature Transition Temperature (TTR). The range starts with an Austenite Start (As) temperature, which is the temperature in which martensite starts converting to austenite, and ends with Austenite Finish (Af) temperature, which is the temperature at which the alloy is all austenite. The mechanical analogue is called stress induced martensitic transformation (SIM). Alternatively, a NiTi alloy can be manufactured in a stable form so there are no phase transformations occurring. The transition between the two phases allow nickel-titanium archwires to exhibit two different properties termed Shape Memory and Superelasticity.

Superelasticty is the initial reason nickel-titanium became so popular in initial leveling and alignment of arches. The wire will exert the same force upon deactivation regardless of how far the initial deflection is. Superelastic wires are austenitic alloys that undergo a transition to martensite in a response to stress and during deactivation revert back to austenite. Superelasticity refers to the non-linear stress strain curve of nickel-titanium archwires that show low deactivation forces (Proffit et al., 2013).

Shape Memory materials “remember” their original shape after deformation. Shape memory occurs because the wire is originally formed well above the Af temperature and when it is cooled below the Temperature Transition Temperature (TTR) it can be plastically deformed but the original shape is restored when the wire is heated back to the austenitic crystal form. By controlling the elements of the alloy the Af temperature can be below the oral environments’ 37^{o}C. Nickel-Titanium is unique because phase transformation occurs at exceptionally low temperatures. Shape memory is a thermal reaction while superelasticity is a mechanical reaction but the two are inherently linked because of the fully reversible phase transformations between martensite and austenite. Additionally, an intermediate R-phase was identified. The R-phase has a rhombohedral crystal structure and may form between the reversible transformations of martensite to austenite (Leu et al., 1990).

Copper Nickel-Titanium

With time people experimented and altered the ratios in the nickel-titanium alloy. Myazaki (1989) reported on the Martensite Start (Ms) temperature and found it was constant when copper was substituted for Ni while it decreased with increasing substitution of Cu for Ti. Certain alloys raise Ms such as Au, Pd, and Zr while others lower the Ms such as Fe, Al, Co, V, Mn, and Cr. Other advantages of Cu addition are its ability to vary the stress-hysteresis and stabilize the superelasticity characteristics against cyclic deformation. In the early 1990s Ormco introduced Copper Nickel-Titanium (CuNiTi) archwires with several claims and different temperature variants according to their Af temperatures. A patent was issued for this new alloy in 1991 and as a result Ormco was the only company producing CuNiTi until recently (Sachdeva 1990).

CuNiTi was available at different temperature variants of 27^{o}C, 35^{o}C, and 40^{o}C, corresponding to the austenite-finish temperatures for the completion of the martensite to austenite transformation. In theory the variants will affect the amount of austenite active in the alloy when the wire is in clinical situations. For example one would expect the 27^{o}C to be almost entirely austenite NiTi in the oral environment while the 40^{o}C variant would be more martensitic in the oral environment of 37^{o}C. The amount of austenite also will affect the forces levels so one can expect the 40^{o}C variant to exert less force clinically than a 27^{o}C variant. The three copper NiTi variants have very similar compositions of approximately 44% nickel, 51% titanium, slightly less than 5% copper, and 0.2-0.3 % chromium (Brantley & Eliades, 2001). The transition temperature is mostly affected by the addition of the chromium atoms to the alloy or manufacturing variables (Kusy, 1997). Other factors involved in the temperature transition range also include the amount of cold working and work hardening as the wire is manufactured (Brantley & Eliades, 2001).

Gil & Planell (1999) reported the effect of Cu addition on the superelastic and shape memory aspect of CuNiTi as it applies to orthodontics. In general they determined that the addition of copper was effective in narrowing the stress hysteresis and in stabilizing the superelasticity characteristic against cyclic deformation. As previously discussed stress hysteresis is the difference between the critical stresses; stress for inducing martensitic transformation due to loading and the reverse transformation upon unloading. This stress hysteresis is much narrower for CuNiTi alloys (~70 MPa) than for the binary alloy (~150 MPa). What a narrower stress hysteresis means clinically is that the force applied to the teeth upon deactivation for a given design and wire cross-section will be higher for a Cu variant than other NiTi alloys. Cu addition also produced greater stability of the transformation temperature and the stability Cu imparted has the potential for more consistent manufacturing that is less sensitive to exact proportions in the alloy.

Recently Pompei-Reynolds & Kanavakis (2014) investigated to see if there are similar wire properties amongst different manufacturers and within the same manufacturers' different 'lots'. Statistically significant interlot variations in austenite finish were found in 27^{o}C and 35^{o}C wire categories, and in austenite start for the 35^{o}C wire category. In addition, significant variations in force delivery were found between the 2 manufacturers for the 0.016 inch 27^{o}C, 0.016 inch 35^{o}C, and 0.016 x 0.022 inch 35^{o}C wires. This recent study brings to light the difficulty in manufacturing and that the clinician should be aware that CuNiTi wires may not always deliver the expected forces as claimed by the manufacturer. To test the manufacturer claims a thermal test called differential scanning calorimetry (DSC) may be conducted.

Differential Scanning Calorimetry

DSC is part of a general class of thermal analysis methods that include thermomechanical analysis (dilatometry), thermogravimetric analysis, and differential thermal analysis (Brantley & Eliades, 2001). According to the International Standard Organization, an accepted method for thermal analysis to determine the TTR of alloys is to utilize DSC. Thermal energy is applied to specimens at specific intervals and the resulting thermal power differences are related to the changes in the specific heat of the material under study. DSC can study the variations in these phases with temperature and determine the enthalpy changes associated with the phase transformation (Brantley et al., 2003). Leu et al. (1990) first utilized DSC to analyze the austenitic-martensitic transformations of superelastic NiTi wires. Transformation temperatures were determined for early superelastic nickel-titanium and an intermediate rhomboidal phase or R-phase was discovered as the wire transformed from martensite to austenite. Another method similar to DSC in that it can determine austenite/martensite phases is X-ray diffraction (XRD). The advantage of DSC compared to XRD is that DSC measures the bulk material while XRD measures the top 50 um of the specimen (Thayer et al., 1995).

Bradley et al. in 1996 used DSC to look at as-received NiTi archwires (superelastic, nonsuperelastic, and shape-memory) to determine TTR for the austenitic, martensitic, and rhombohedral structure phases. They found that superelastic NiTi alloys (Nitinol SE and NiTi) undergo austenitic transformations involving R structure which begin below 0^{o}C. NiTi (Ormco/Sybron, Glenora, Calif) is almost entirely austenite and Nitinol SE (Unitek/3m, Morovia, Calif) is a mixture of austenite and R structure in the oral environment. Nonsuperelastic alloy Nitinol is mainly martensite at both room temp and oral environment. The shape memory alloys (NeoSentalloy and Titanal LT) showed that they were almost entirely austenite in the oral environment. The results of their DSC investigation were in good agreement with the manufacturers claims however a criticism of the study can be they only used ‘as-received’ archwires and these may not correlate with *in vivo* conditions.

This subject was investigated by Biermann et al. in 2007 and it was determined that no large differences in thermal activity was present between as-received and clinically retrieved wires tested by DSC. The only difference was with 27^{o}C retrieved wires that had a significant reduction in heating enthalpy associated with the martensite to austenite transition. Valeri (2013) also used DSC to compare as-received NiTi wires with those that were clinically used. A total of 61 patients were recruited for the study and they were randomly allocated to receive one of the four types (n=15) of NiTi archwires. After 4-12 weeks in a clinical setting they were compared to control ‘as-received’ wires. There were no statistically significant differences in thermal properties when comparing archwires before and after clinical use. Berzins and Roberts (2010) performed an *in vitro* test using thermocycling and found that there were some differences in wire properties after thermocycling. Fluctuations in oral temperatures from hot or cold beverages could possibly affect mechanical properties, but evidence is still lacking at this point.

McCoy (1996) was the first to use DSC to investigate CuNiTi wires. The goal was to determine the TTR and also investigate if chemical composition of the wire or manufacturing variables altered the TTR. The temperature variants of 27, 35, and 40^{o}C CuNiTi (Ormco, Sybron, Glendora, Calif) were used in addition to a heat activated shape memory alloy (Neo Sentalloy, GAC) and a cold worked nonsuperelastic alloy (Nitinol, 3M/Unitek). Chemical composition was determined with energy-dispersive spectroscopy (EDS) and it was determined that all CuNiTi variants had essentially identical compositions (44 Ni-51 Ti-5 Cu at%). DSC results indicated that the Af temperatures were within 3^{o}C of what Ormco claimed. From this information it was assumed that the manufacturing differences amongst the variants is what caused the changes in Af. DSC also showed thermal hysteresis for CuNiTi wires was about three times greater than NeoSentalloy.

Kusy (2007) also used DSC to elucidate the TTR of Stainless Steel, TMA, and nickel-titanium archwires. Of the 5 NiTi alloys, 2 were thought to be stabilized martensitic alloys in which processing prevented further transformations, and 3 were thought to be martensitic active CuNiTi alloys. The DSC revealed no transitions in the temperature regime of the oral cavity for the Steel, TMA, and Nitinol Classic (3M Unitek, Monrovia, Calif) as expected, however Orthonol (Rocky Mountain Orthodontics, Denver, Colo) product had a small endothermic (or exothermic) peak on heating (or cooling). After performing Dynamic Mechanical Analysis (DMA) Kusy was able to adduce that Orthonol is about 20% thermoelastic active martensite, with the rest stable passive martensite. The agreement amongst the tests illustrates the sensitivity and reliability of the DSC test. The three CuNiTi wires used were the 27, 35, and 40^{o}C

(SDS/Ormco) Austenite finish variants. As expected, the CuNiTi 27, 35, and 40°C products manifested austenitic finish temperatures of 29.3°C, 31.4°C, and 37.3°C, respectively from DSC and 27.4°C, 35.8°C, and 39.6°C by DMA. For each CuNiTi product, the magnitude of ΔH increased as the transition temperature increased from the 27°C to the 40°C products, independent of heating or cooling. The enthalpy for the 27°C, 35°C, and 40°C variants were 2.47, 2.86, and 3.18 cal/g, respectively. The reported numbers agree with previous reports (McCoy, 1996). In clinical relevance Kusy asks the question if 27 and 35°C variants are clinically necessary when the laboratory values are so similar.

Clinical Implications

Advances in biomaterials are often faster than the scientific community can keep up with. As a result there are manufacturer claims that are unsupported by evidence. These claims often are assumptions based of laboratory findings such as increased tooth movement and less patient discomfort with new ‘space-aged’ wires such as CuNiTi. *In vitro* results need to be verified through properly designed clinical trials taking into account the temperature range of testing, method of ligation, interbracket distance, bracket type and length of wire (Santoro et al., 2001). Many superelastic wires show no superelastic properties *in vivo* because of the exceedingly high force level at the plateau that is not seen in clinical conditions (Schumacher et al., 1992). There are more mechanical and laboratory studies in the literature but the few clinical ones including a Cochrane Review attempt to translate the laboratory testing to clinical conditions.

Dalstra & Melsen in 2004 set out to study if the transition temperatures of CuNiTi archwires affect the amount of tooth movement during alignment. They conducted a split mouth design that was randomly selected from patients being treated at an orthodontic residency program. Fifteen randomly selected patients with similar crowding were picked to have specially manufactured CuNiTi wires with one half being 27^{o}C and the other being 40^{o}C put into the maxillary arch. Tooth movement was larger on the 40^{o}C side; however only in case of the total translation of the premolars was this difference significant. It is interesting that the side experiencing less force had greater tooth movement. This further supports the general consensus that lighter forces are more ideal for tooth movement. However, even though there was a difference it was so small that it is questionable if it is clinically significant. A proposed benefit of thermoactive wires is that the patient can regulate activation and de-activation of the archwire by rinsing with and drinking of warm and cold beverages. The scientific basis for the use of thermo-responsive wires is that bone remodels more effectively when subjected to a dynamic load in comparison to a static one (Lanyon, 1984). The criticism of this study is that the split mouth design assumes proper manufacturing techniques for all wires, which is unlikely considering there is variation amongst one company when trying to produce the same wire of a single variant. Also the split mouth design does have flaws in that one side of the wire can affect the other. An improvement could be an increase in number of patients and using one arch and comparing to another patient.

In 2009 Pandis et al. conducted a double blind randomized control trial to investigate the efficiency of CuNiTi vs NiTi in resolving crowding of the mandibular anterior teeth. There were 2 groups of 30 patients that received either .016 inch CuNiTi

(Ormco, Glendora, Calif) or .016 inch NiTi (Modern Arch, Wyomissing, Pa). There was good blinding in the study (neither patient nor provider was aware) and the study was followed until full alignment of the lower anterior teeth was achieved. The type of wire had no significant effect on crowding alleviation as CuNiTi aligned in 129.4 days while NiTi aligned in 121.4 days. This study was in agreement with Cobb et al. (1988), which showed no difference in alignment with multi-stranded stainless steel, superelastic NiTi, and ion-implanted NiTi archwires.

In 2013 Jian was the lead author of a Cochrane Review looking into initial archwires for tooth alignment during orthodontic treatment with fixed appliances. Nine RCTs were included in the review and they concluded that all trials were at a high risk of bias. Comparisons were made amongst martensitic stabilized NiTi, multistranded stainless steel, superelastic NiTi, and shape memory NiTi (including CuNiTi). They concluded that there is no reliable evidence from clinical trials that any specific initial archwire material is better or worse than another in regard to speed of initial alignment or pain perception. In future research confounding variables, such as bracket type and ligation system, should be better controlled. In addition the RCTs should report both benefits (speed of alignment) with possible harms (pain and root resorption).

CHAPTER 3
MATERIALS AND METHODS

The wires were matched by size and temperature transition temperature to compare different companies' claims. All of the CuNiTi wires currently available on the market were used in the study. The thermal properties of transition temperature and enthalpy were measured using differential scanning calorimetry (DSC). The wires that were examined were Ormco Copper Nickel Titanuim (Ormco, Orange, CA, USA), FLI Copper Nickel Titanium (Rocky Mountain Orthodontics, Denver, CO, USA), Copperloy Nickel Titanium (GAC, York, PA, USA), Copper Nitanium (Henry Schein/Ortho Organizers, Carlsbad, CA, USA), Truflex Copper Nickel Titanium (Ortho Technology, Tampa, FL, USA), and Tanzo Copper Nickel Titanium (American Orthodontics, Sheboygan, WI, USA). The total number of wires for each test was n=10/company/temperature variant/size. The sizes used were 0.018 inches and 0.016 x 0.022 inches. The temperature variants were 27ºC and 35ºC.

Wires submitted for testing were in the 'as-received' state. Specimen selection for DSC analysis consisted of a 5 mm segment from the posterior area of the archform. The terminal 5 mm was removed from the archform and then the next 5 mm was used for testing. This area was chosen because it is straight and will most likely experience fewer stresses during manufacturing. The wires were sectioned with a low-speed water-cooled diamond saw (Figure 2; Isomet, Buehler Ltd, Lake Bluff, IL) with care taken to avoid mechanical stresses and heating that would alter the microstructure of the wire.

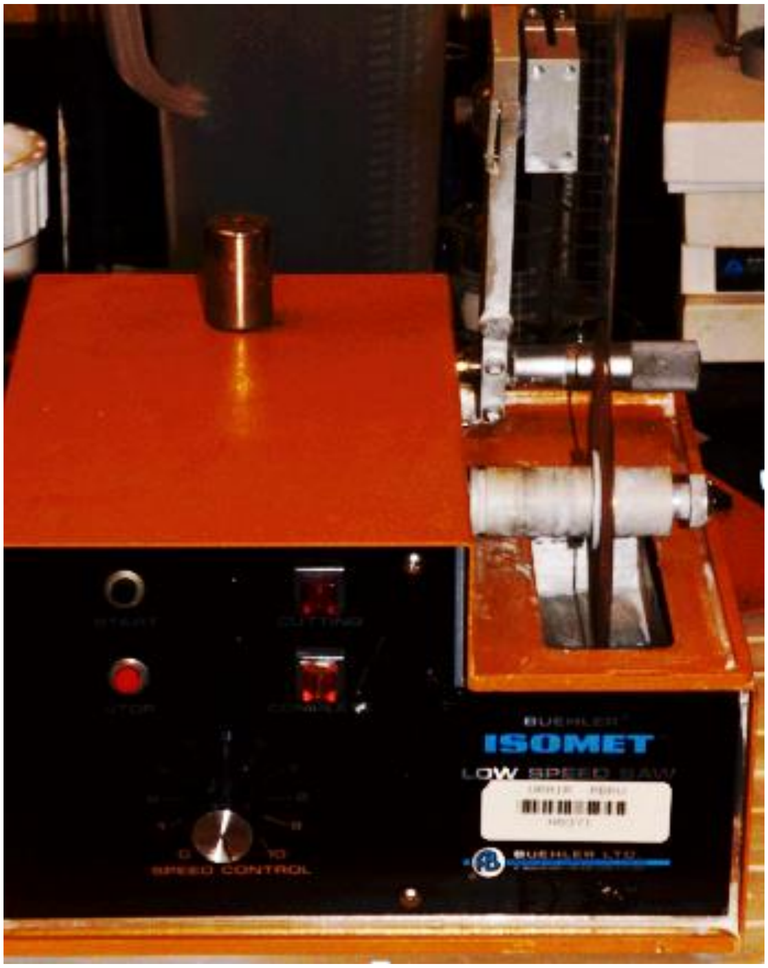

Figure 2: Isomet Diamond Saw used to section wires

The wire segments were weighed (Figure 3) to the nearest 0.01 mg, placed in an aluminum crucible, and sealed.

Figure 3: Scale used to measure wire segments

The crucible was then thermally scanned to obtain DSC measurements (Figure 4; Model 822, Mettler-Toledo Inc, Columbus, Ohio). The temperature of the crucible was scanned from -100^{o}C to 100^{o}C, with liquid nitrogen as a coolant and nitrogen gas for purging, at 10^{o}C per minute for the heating curve and then cooled at the same rate from 100^{o}C to -100^{o}C for the cooling curve.

Figure 4: Mettler Model 822 used to conduct DSC analysis with Liquid Nitrogen as cooling agent

An empty crucible was used as a reference while obtaining DSC data. The DSC manufacturer's software was used to qualitatively and quantitatively analyze the DSC plots. Enthalpy, or ΔH, along with onset and endset temperatures for the various phase transformations were calculated for all the wires. An example of the qualitative analysis is in Figure 5.

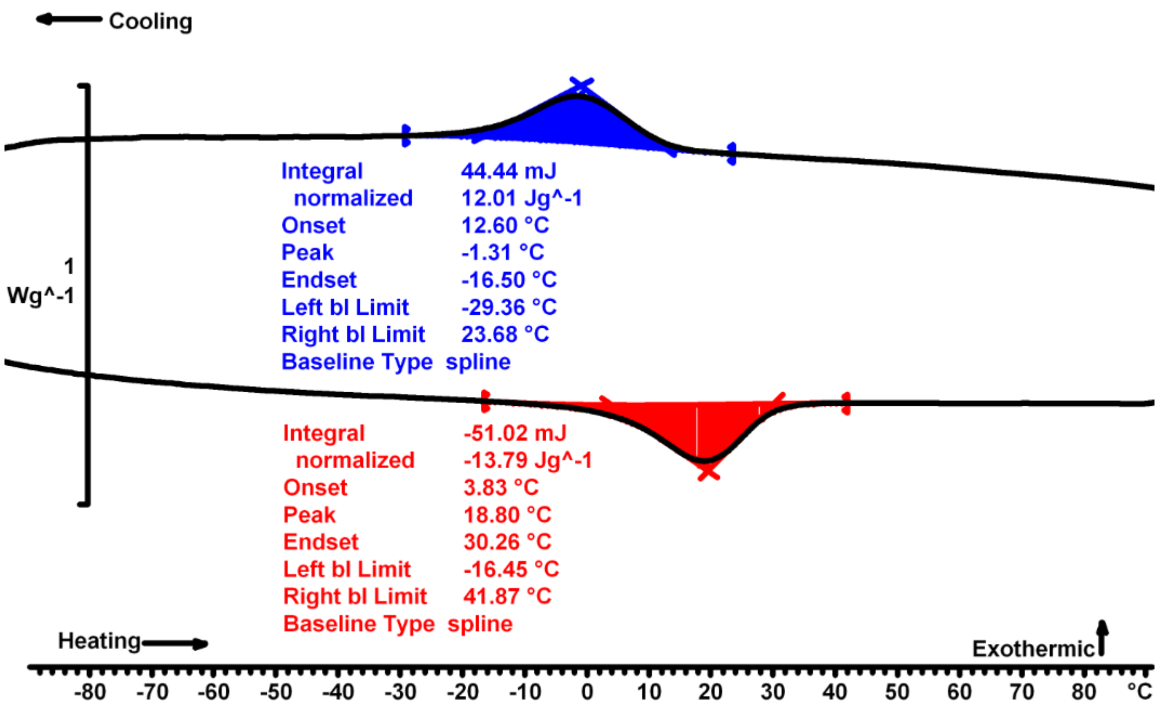

Figure 5: Example of DSC Thermogram Analysis

Some thermograms presented with R-phase in the ‘Heating’ section of the graph. If an R phase was present it was accounted for in the analysis of Heating Endset. An example of an R phase in a thermogram is in Figure 6.

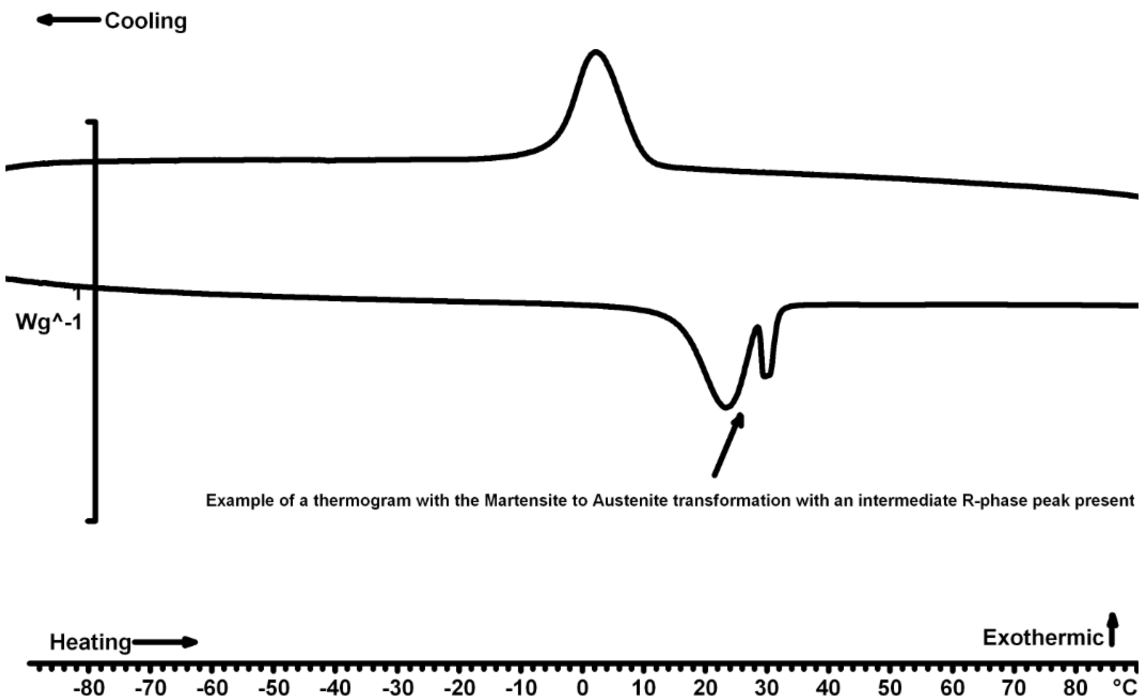

Figure 6: Example of DSC Thermogram with presence of R Phase

Statistical analysis consisted of a three-way ANOVA with brand, temperature variant, and wire size as factors. Due to significant interactions among all factors, a one-way ANOVA was conducted analyzing the different brands within a given temperature variant and size. A post hoc Tukey HSD test, if indicated, was conducted with significance defined as $p < 0.05$.

CHAPTER 4
RESULTS

A three-way ANOVA showed significant differences for temperature, brand, and size, but also significant differences within all interactions. A one-way ANOVA among brands within each temperature variant and wire size combination showed significant differences for all thermal measures. Tables 1-4 present the mean temperature and enthalpy changes for the phase transformations for each group of wires. Results from the Post Hoc Tukey HSD test are noted by different letters and indicate a statistically significant ($p<.05$) difference existed between wires for a given measure.

Table 1. DSC measured temperature and enthalpy changes for phase transformations during heating and cooling of 0.018” 35^{o}C CuNiTi wires

Wire	Heating			Cooling		
	Onset Temperature (^{o}C)	Endset Temperature (^{o}C)	Enthalpy (J/g)	Onset Temperature (^{o}C)	Endset Temperature (^{o}C)	Enthalpy (J/g)
Ormco	-1.0±1.4 C	37.1±2.0 A	-10.3±1.3 B	17.0±0.3 A	-19.3±1.3 D	8.6±0.7 D
American Orthodontics (Low)	11.3±0.8 B	33.1±0.9 B	-15.2±0.5 AB	12.2±0.1 B	-8.8±0.6 C	14.8±0.4 C
GAC (C2)	16.0±0.6 A	32.7±0.8 B	-16.0±1.1 AB	10.6±0.6 ED	-3.5±0.6 B	16.7±0.7 AB
Ortho Organizers	16.1±0.3 A	33.9±1.7 B	-13.8±10.6 AB	11.0±0.2 D	-3.4±0.5 B	16.1±0.8 B
RMO	16.0±0.9 A	34.2±2.0 B	-17.3±0.5 A	11.5±0.3 C	-2.3±0.7 A	17.3±0.4 A
Ortho Technology	15.8±0.5 A	33.2±1.9 B	-15.6±0.5 AB	10.3±0.4 D	-3.7±0.7 B	16.1±0.6 B

Different letters indicate a statistically significant ($p<0.05$) difference exists between wires for a given measure.

For the 0.018” 35°C variants all wires had an Endset Temperature within 2.3°C as advertised. Ormco stood out as significantly different when performing a Post Hoc Tukey HSD analysis and was also the only wire with an Af above the advertised value of 35°C. All other wires were below the expected Af. American Orthodontics and GAC do not advertise a specific Af but instead report wires by expected force values or arbitrary number, respectively. It was hypothesized that both GAC and AO were manufacturing 35°C Af wires and it was supported with the data. Significant differences across other variables can be seen in the data when comparing different companies to each other.

Table 2. DSC measured temperature and enthalpy changes for phase transformations during heating and cooling of 0.018” 27°C CuNiTi wires

Wire	Heating			Cooling		
	Onset Temperature (°C)	Endset Temperature (°C)	Enthalpy (J/g)	Onset Temperature (°C)	Endset Temperature (°C)	Enthalpy (J/g)
Ormco	-0.7±0.7 F	26.9±0.7 C	-8.5±0.6 B	11.9±0.6 A	-20.7±1.3 E	7.4±0.7 D
American Orthodontics (Mid)	4.5±0.3 E	31.7±1.6 A	-12.3±0.3 AB	12.6±0.2 A	-16.0±0.6 D	11.6±0.3 C
GAC (C1)	16.0±0.3 A	31.5±1.1 A	-15.8±0.6 A	9.2±0.2 B	-5.5±0.9 A	15.3±0.6 A
Ortho Organizers	10.0±0.4 D	23.2±0.6 D	-14.7±1.1 AB	6.0±0.3 D	-12.0±0.6 C	13.4±2.6 B
RMO	12.1±2.1 C	28.5±3.4 BC	-13.9±1.6 AB	7.6±1.3 C	-11.4±2.1 C	13.5±2.0 AB
Ortho Technology	14.7±0.2 B	30.5±1.6 AB	-8.9±12.5 B	9.2±0.1 B	-7.4±0.4 B	13.6±0.4 AB

Different letters indicate a statistically significant ($p<0.05$) difference exists between wires for a given measure.

For the 0.018” 27ºC variants all wires were within 4.7ºC of the advertised Heating Endset Temperature. There was more variability amongst the Af in these wires compared to the 35ºC variants, with four different categories produced upon Post Hoc Tukey HSD analysis. The AO and GAC wires, not being advertised at 27ºC, were significantly further away from the hypothesized Af value than all the other wires. A 1.2ºC difference was observed between Heating Endset Temperatures for the C1 and C2 GAC CuNiTi archwires. Significant differences across other variables can be seen in the data when comparing different companies to each other.

Table 3. DSC measured temperature and enthalpy changes for phase transformations during heating and cooling of .016”x.022” 35ºC CuNiTi wires

Wire	Heating			Cooling		
	Onset Temperature (ºC)	Endset Temperature (ºC)	Enthalpy (J/g)	Onset Temperature (ºC)	Endset Temperature (ºC)	Enthalpy (J/g)
Ormco	11.4±1.8 B	33.8±1.0 A	-16.1±2.3 AB	15.4±0.4 A	-6.9±1.3 DC	14.4±0.7 B
American Orthodontics (Low)	11.1±0.5 B	34.3±2.1 A	-15.0±0.6 B	11.3±0.2 BC	-7.5±0.3 D	16.6±0.7 A
GAC (C2)	15.1±0.5 A	32.0±0.8 BC	-16.6±0.7 A	11.7±0.4 A	-5.0±0.6 A	17.0±1.3 A
Ortho Organizers	14.8±0.4 A	32.8±0.8 AB	-16.7±0.6 A	11.8±0.2 A	-5.2±0.3 A	16.2±0.6 A
RMO	14.0±0.8 A	31.9±1.0 BC	-16.5±0.8 A	11.4±0.6 A	-5.8±0.9 BA	16.2±0.5 A
Ortho Technology	14.8±0.6 A	30.7±1.0 C	-14.9±.6 B	10.8±0.4 C	-6.2±0.7 CB	14.9±.6 B

Different letters indicate a statistically significant ($p<0.05$) difference exists between wires for a given measure.

For the 0.016 x 0.022” 35ºC variants all wires were within 4.3ºC of the advertised Heating Endset Temperature. AO and GAC wires were within 3ºC to the hypothesized 35ºC Af. With the exception of Ortho Technology all wires were within 3.1ºC of the expected Af. In general all the wires were below the expected Af in the 0.016 x 0.022” 35ºC variants. Significant differences across other variables can be seen in the data when comparing different companies to each other.

Table 4. DSC measured temperature and enthalpy changes for phase transformations during heating and cooling of 0.016”x.022” 27ºC CuNiTi wires

Wire	Heating			Cooling		
	Onset Temperature (ºC)	Endset Temperature (ºC)	Enthalpy (J/g)	Onset Temperature (ºC)	Endset Temperature (ºC)	Enthalpy (J/g)
Ormco	7.4±0.1 D	27.1±1.6 C	-13.4±0.2 E	9.6±0.3 A	-16.1±1.0 C	10.8±0.4 E
American Orthodontics (Mid)	8.4±0.3 C	32.8±0.5 A	-14.5±0.5 CD	10.0±0.3 A	-11.6±0.3 B	14.6±0.7 B
GAC (C1)	11.2±0.5 B	25.3±0.6 D	-14.0±0.9 DE	6.8±0.6 D	-10.6±0.7 B	12.4±0.9 D
Ortho Organizers	10.9±1.3 B	25.4±1.1 D	-15.1±0.5 BC	7.4±0.7 C	-11.6±1.8 B	14.8±0.6 AB
RMO	13.7±0.4 A	29.2±1.3 B	-16.0±0.4 B	8.8±0.3 B	-8.2±0.9 A	15.5±0.4 A
Ortho Technology	13.5±0.4 A	29.2±1.6 B	-15.3±0.3 AB	8.6±0.2 B	-8.3±0.4 A	13.5±0.4 C

Different letters indicate a statistically significant ($p<0.05$) difference exists between wires for a given measure.

For the 0.016” x 0.022” 27ºC variants all wires were within 5.8ºC of the advertised Heating Endset Temperature. The biggest outlier in this data set was AO, which deviated from the hypothesized Af by 5.8 degrees. Excluding AO the rest of the wires were within 2.2°C of the advertised Af. AO showed a difference of 1.5°C in the respective heating endsets for the reported Mid and Low Force levels amongst its 0.016” x 0.022” archwires. Ortho Technology, which advertises specific Af values, also had only a 1.5°C difference between its two temperature variants. Significant differences across other variables can be seen in the data when comparing different companies to each other.

In addition to the raw data, thermograms were produced to visually assess any differences amongst the wires and additionally whether R phase presence can be determined. The presented thermograms are represented by a single wire for each temperature/variant that qualitatively represented the group of wires as a whole.

Figure 7 shows the thermograms for 0.018” wires with an Af of 35°C. R phase can be readily appreciated upon heating with all wires except Ormco. Ormco also has less of a pronounced peak when compared to all the other wires on heating and cooling. No R phase was present for cooling with any wire.

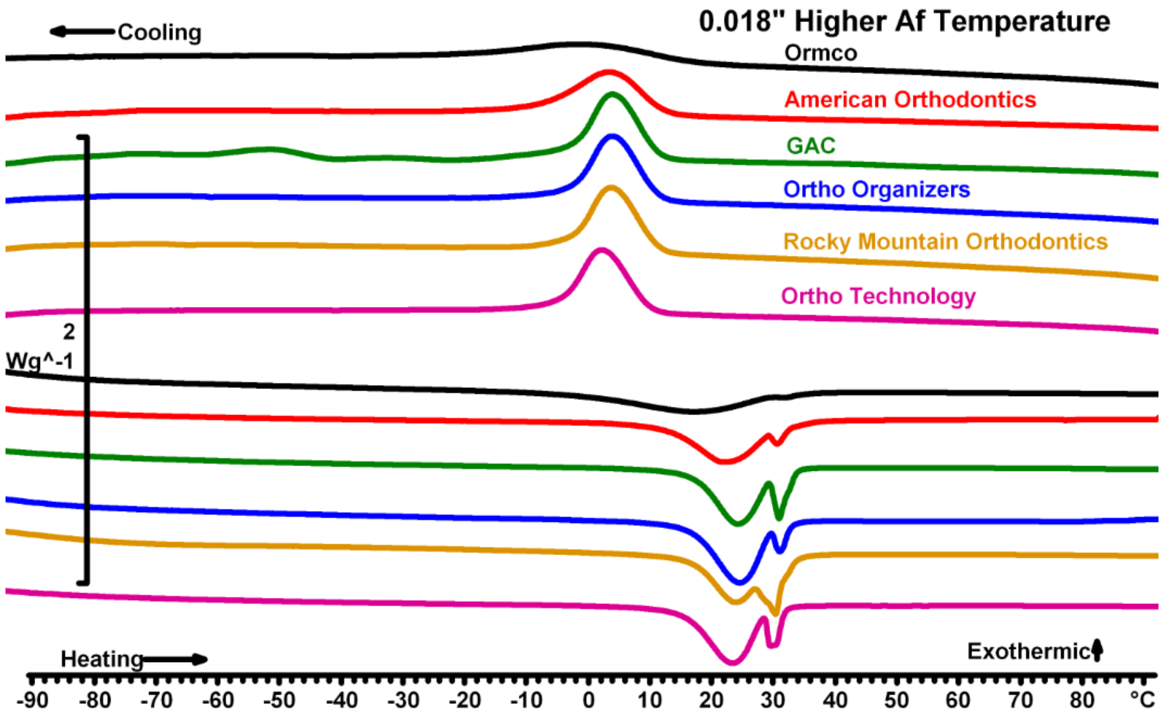

Figure 7: Thermogram for 0.018” wires with Af of 35°C

Figure 8 shows thermograms for all the 0.018” wires with an Af of 27ºC. R phase was present in three of the six wires upon heating. Ormco and American Orthodontics both have less pronounced peaks on heating as compared to the other wires.

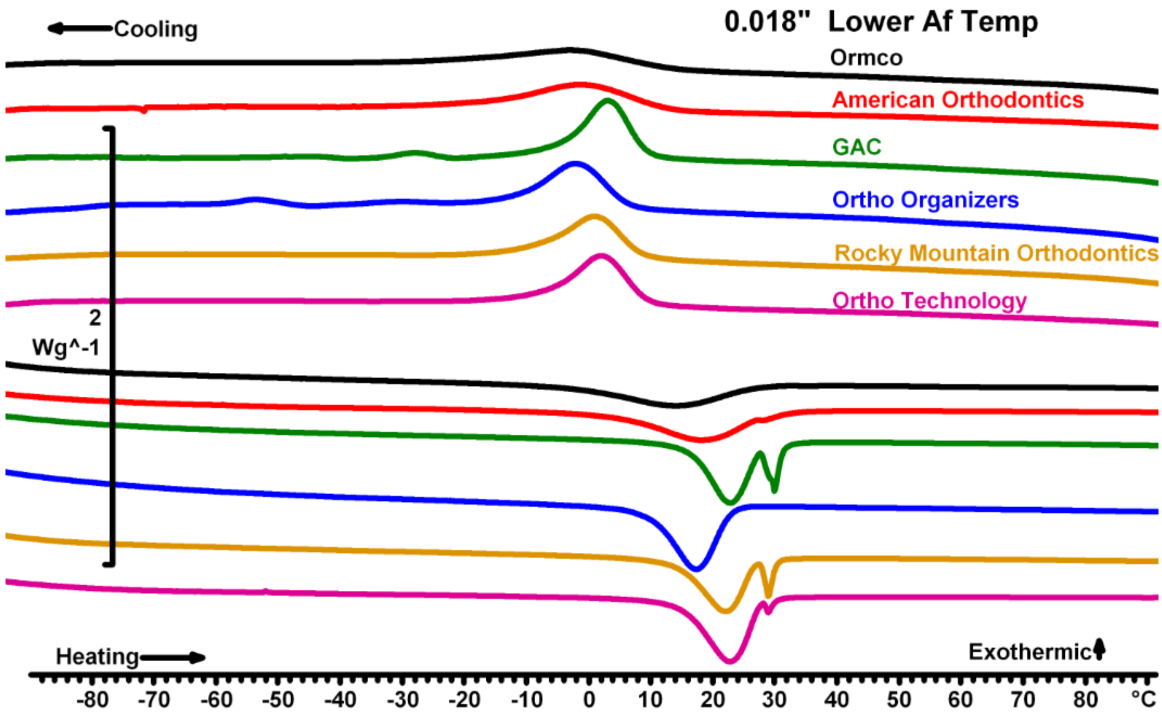

Figure 8: Thermogram for 0.018” wires with Af of 27ºC

Figure 9 shows the thermograms for all the 0.016" x 0.022" wires with an Af of 27ºC. Three of the six wires show an R phase upon heating while there was no R phase upon cooling.

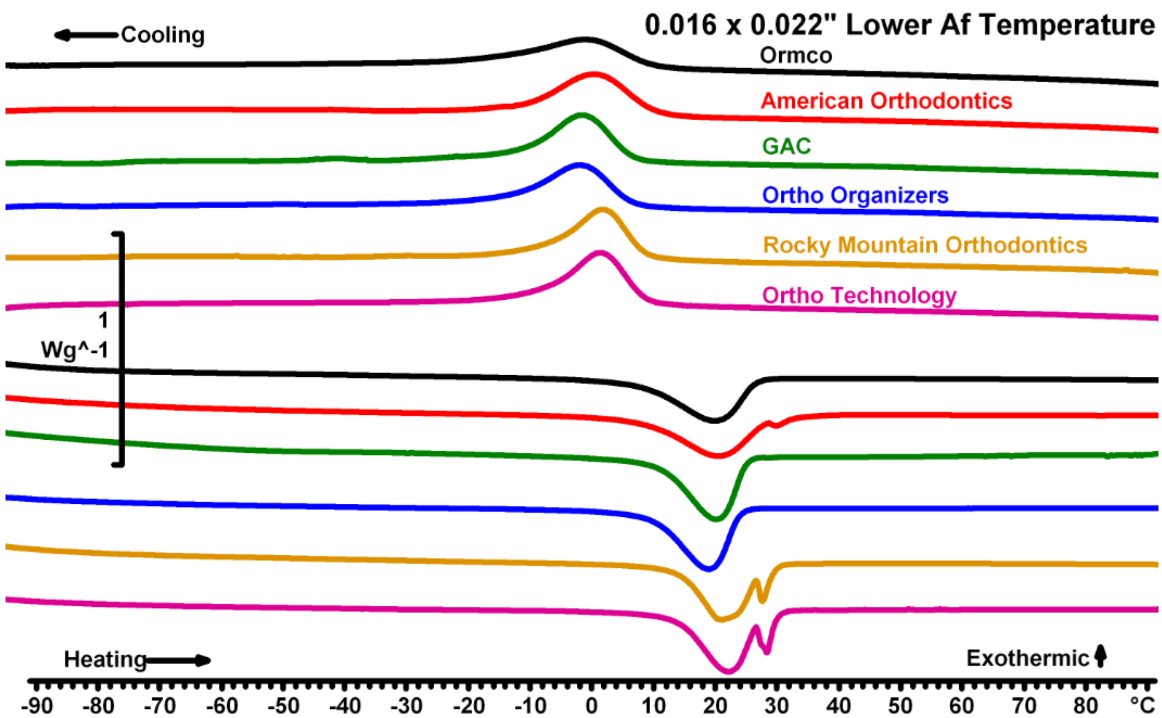

Figure 9: Thermogram for 0.016 x 0.022" wires with Af of 27ºC

Figure 10 shows all the thermograms from 0.016” x 0.022” wires with an Af of 35°C. All wires showed presence of R phase upon heating and there was no R phase present upon cooling.

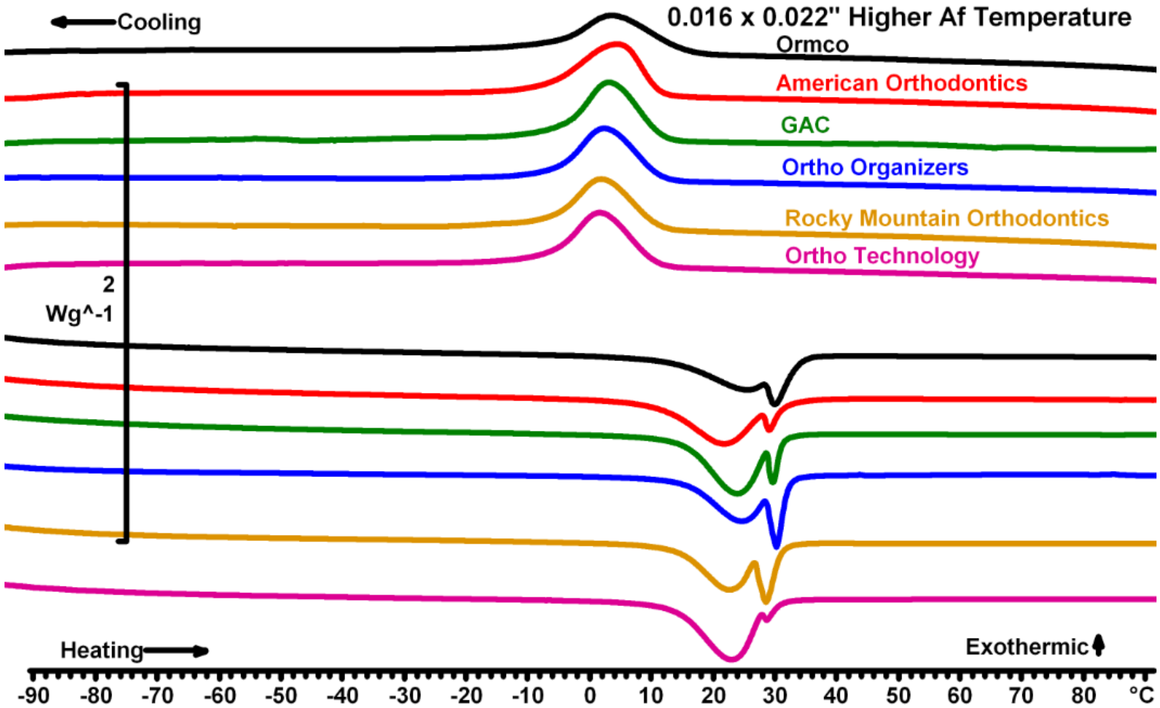

Figure 10: Thermogram for 0.016” x 0.022” wires with Af of 35°C

The next six figures (Figures 11-16) presented are the thermograms for the selected companies with their different sizes and variants.

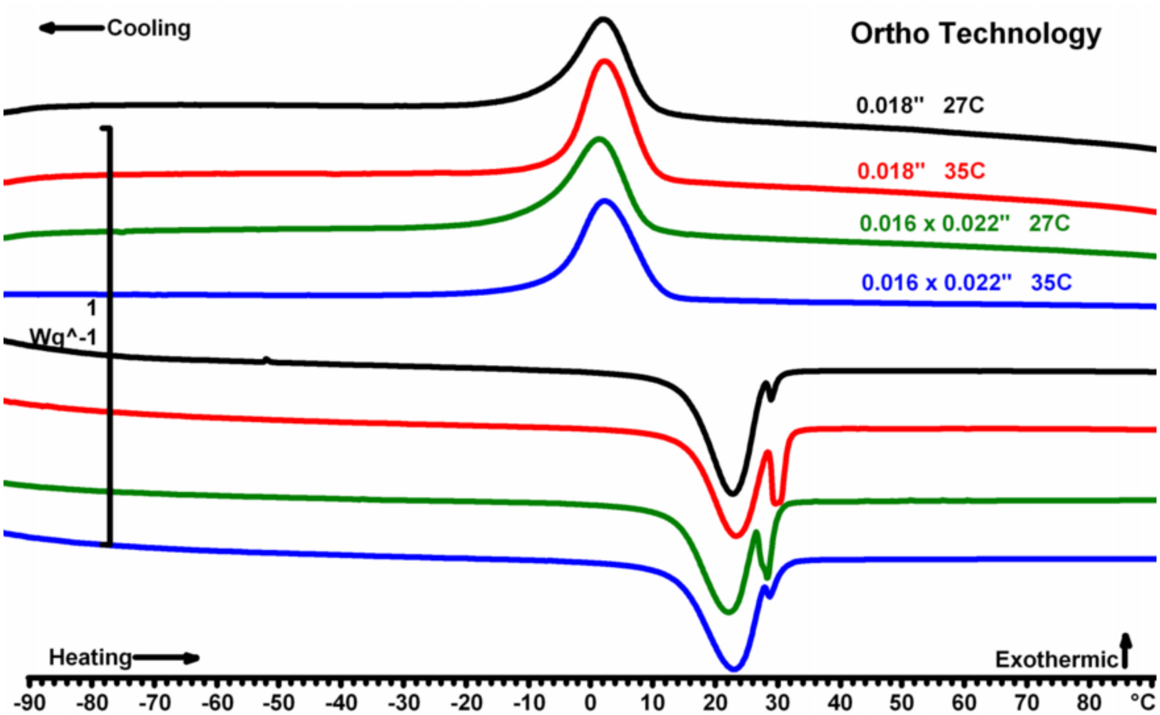

Figure 11: Thermogram for all variants of Ortho Technology CuNiTi wires

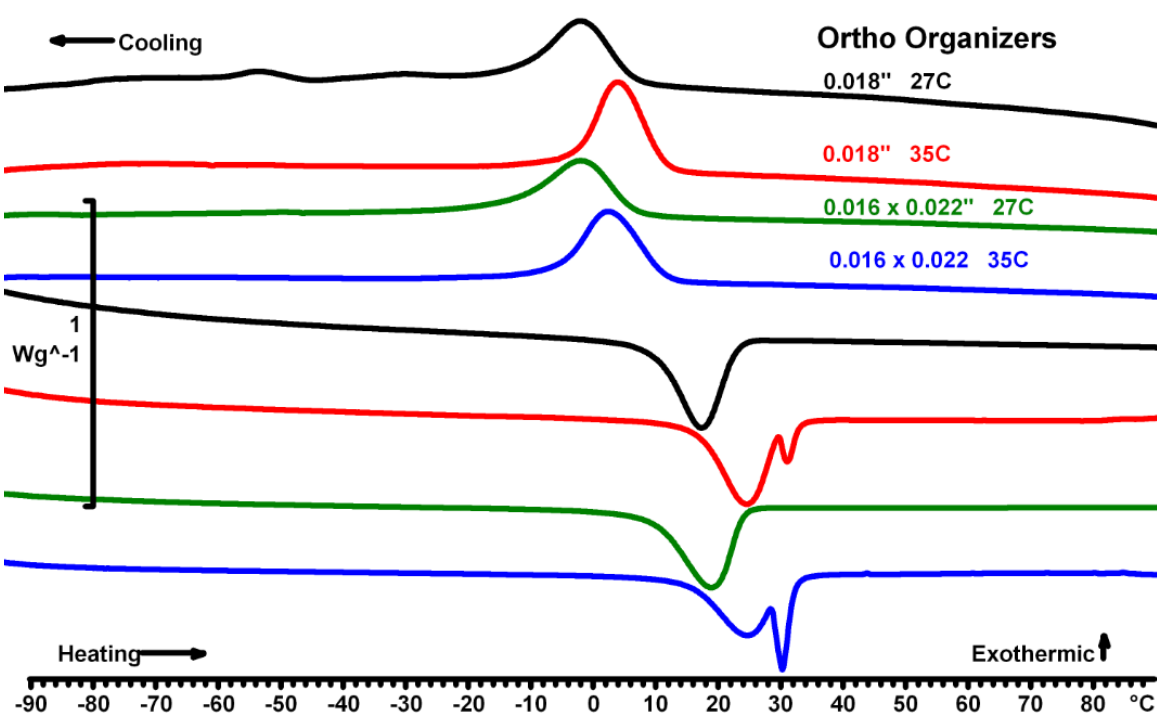

Figure 12: Thermogram for all variants of Ortho Organizers CuNiTi wires

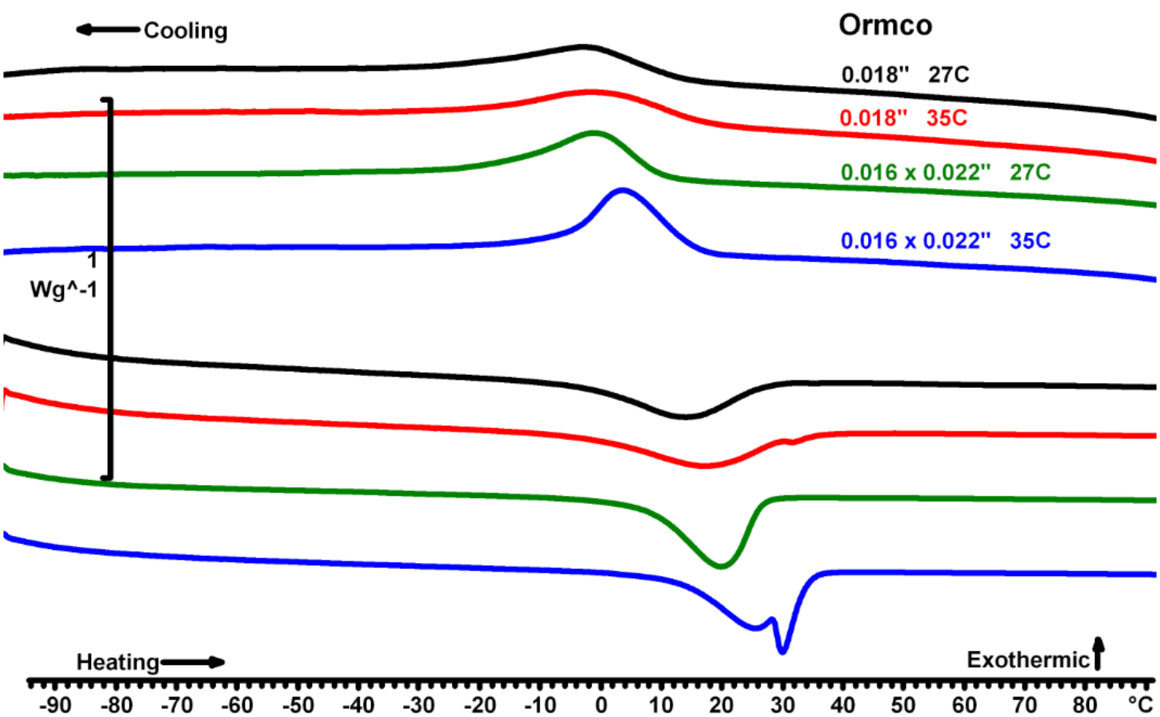

Figure 13: Thermogram for all variants of Ormco CuNiTi wires

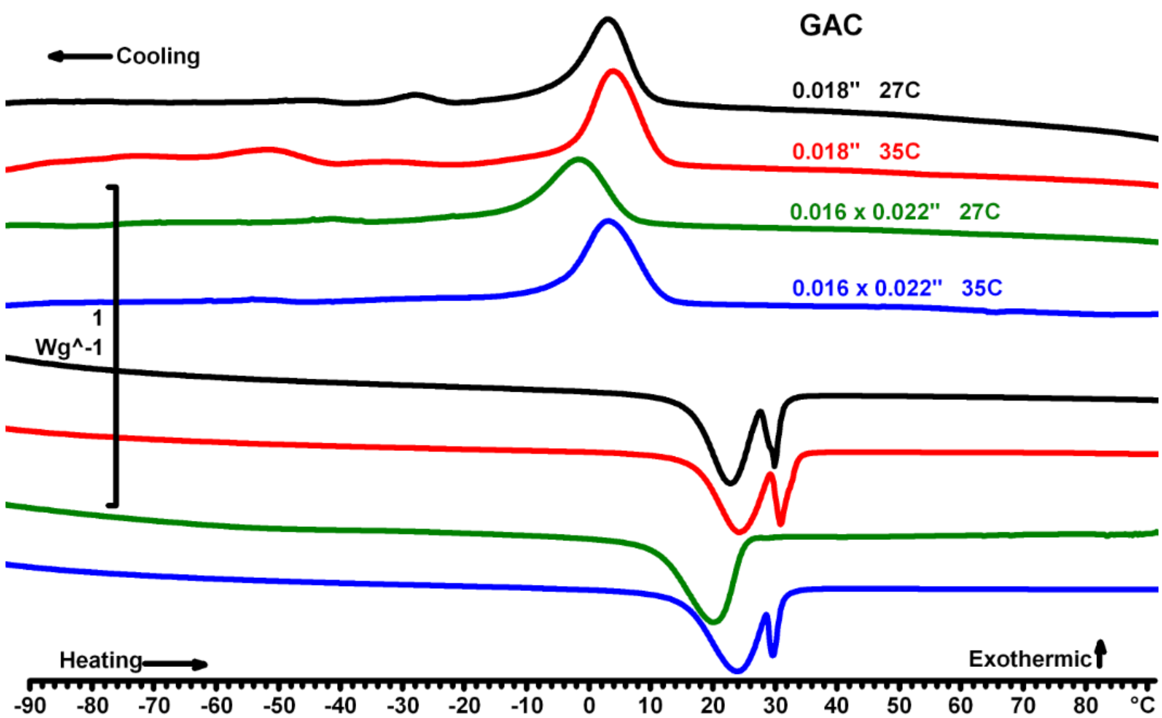

Figure 14: Thermogram for all variants of GAC CuNiTi wires

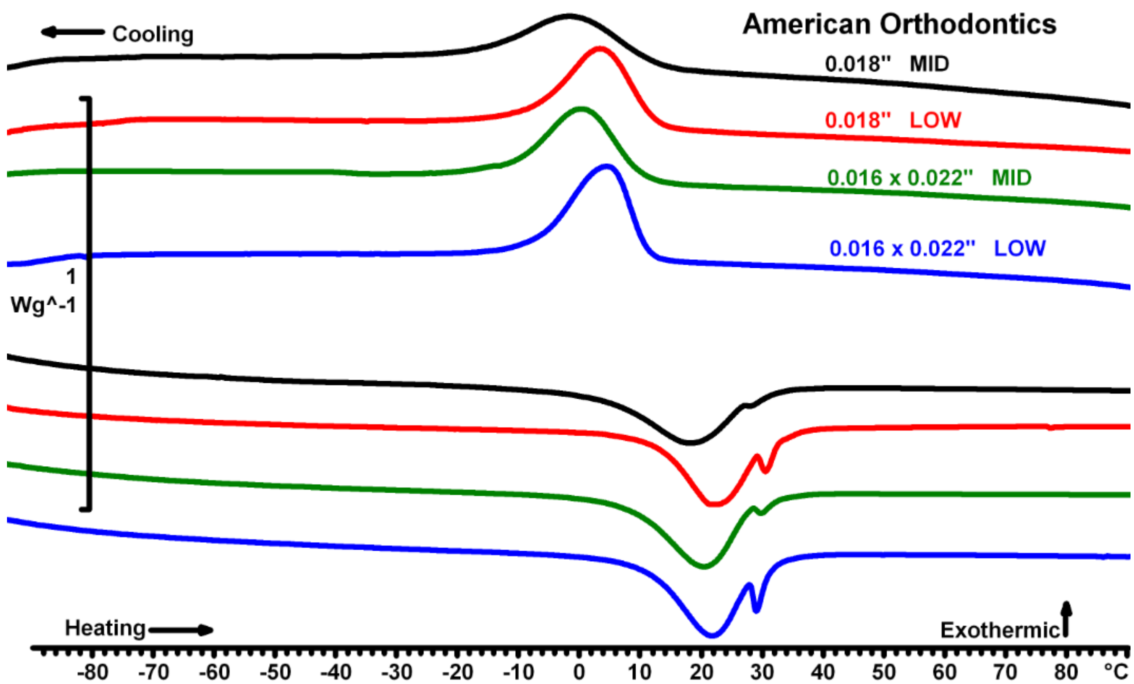

Figure 15: Thermogram for all variants of American Orthodontics CuNiTi wires

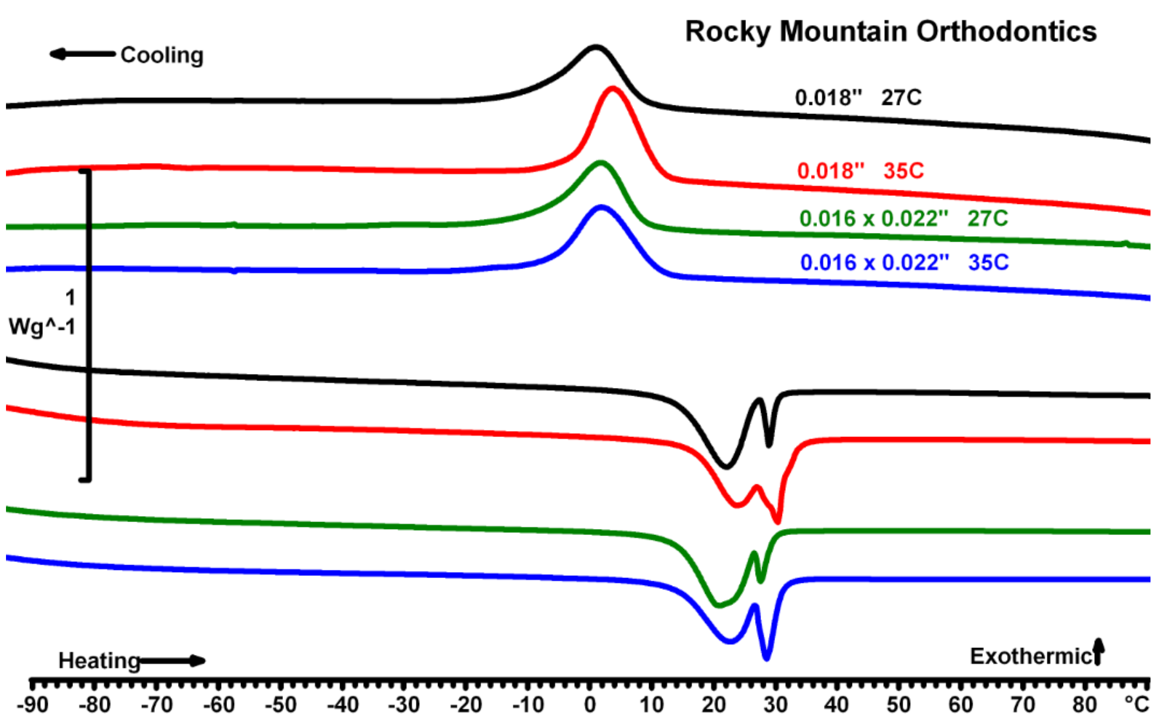

Figure 16: Thermogram for all variants of Rocky Mountain Orthodontics CuNiTi wires

CHAPTER 5
DISCUSSION

This study is one of the first times that more than the Ormco lines of CuNiTi archwires were subjected to independent laboratory testing (Pompei-Reynolds and Kanavakis 2014, McCoy 1996, Biermann et al., 2007, Kusy & Whitley, 2007). From the DSC analysis information on Temperature Transition Range (TTR) could be verified for the six different companies tested using both round and rectangular wire and two temperature variants. One of the reasons that companies produce CuNiTi is that the copper addition to the NiTi alloy allows more accurate control of TTR and as a result shape memory. The more accurate control of Af implies the level of martensite and austenite present in a wire at a given temperature can be controlled. With the Af of 27^{o}C one would expect there to be a mixture of austenite and martensite at room temperature while in the oral environment the wire would be entirely austenite. On the other hand the 35^{o}C variant would be mainly martensite at room temperature with a combination of martensite and austenite in the oral environment depending on external influences such as cold or hot beverage consumption. With this in mind the 27^{o}C variant would be expected to produce more force intraorally than the 35^{o}C variant because the more austenite present would correlate to higher forces exerted by the wire. Ormco originally patented CuNiTi so it was the only company making a CuNiTi archwire for many years until recently when the patent expired. As a result much of the previous literature has only studied Ormco CuNiTi.

McCoy in 1996 reported Af for Ormco CuNiTi variants first and showed that all three 0.016” x 0.022” variants were within 3^{o}C of manufacturer advertisement. The 27, 35, and 40 variants showed an Af of 29.7ºC, 38.0ºC, and 41.2ºC, respectively. McCoy studied wires in the as-received state only. Biermann et al. in 2007 looked at both as-received and retrieved from clinical use wires for the Ormco line of CuNiTi wires. All wires tested were 0.016” x 0.022” and they tested all three temperature variants, however, only the 27 and 35 variants were tested by DSC after clinical use. The Af for the as-received wires for the 27ºC, 35ºC, and 40ºC were 29.2ºC, 36.0ºC, and 36.3ºC, respectively. After clinical use the 27 and 35ºC variants showed Af values of 29.1ºC and 35.9ºC. All wires showed no significant differences after clinical use except the 27ºC wires exhibited a significant decrease in the heating enthalpy associated with the martensite-to-austenite transition. Pompei-Reynolds & Kanavakis in 2014 also conducted DSC analysis on CuNiTi wires for both Ormco and RMO lines using 0.016” x 0.022” and .016” for all three temperature variants. The primary conclusion of that study was that interlot variation existed from the same company from wire to wire however comparison to average Af can be made. For the 0.016” x 0.022” Ormco wires the 27ºC, 35ºC, and 40ºC had an experimental Af of 25.05ºC, 32.17ºC, and 34.54ºC, respectively. For the 0.016” x 0.022” RMO wires the 27ºC, 35ºC, and 40ºC had an experimental Af of 26.36 ºC, 30.0 ºC, and 33.26 ºC, respectively. All previous studies on DSC for CuNiTi are presented in Table 5.

Study (no. of wire specimens)	Ormco			RMO		
	Experimental Af for 27ºC (ºC)	Experimental Af for 35ºC (ºC)	Experimental Af for 40ºC (ºC)	Experimental Af for 27ºC (ºC)	Experimental Af for 35ºC (ºC)	Experimental Af for 40ºC (ºC)
McCoy 1996 (6)	29.7	38.0	41.2			
Biermann et al. 2007 (6)	29.2	36.0	36.3			
Kusy & Whitley 2007 (3)	29.3	31.4	37.3			
Pompei-Reynolds & Kanavakis 2014 (2-5)	25.1	32.2	34.5	26.36	30.0	33.26
Current (10)	27.1	33.8		29.2	31.9	

Table 5: Presentation of all previous studies on CuNiTi using DSC analysis. Afs presented are for 0.016” x 0.022” CuNiTi archwires

In comparison to the previous studies that tested as-received 0.016” x 0.022” Ormco wires this study showed an Af closer to the 27ºC variant (27.1ºC) than was previously reported by McCoy, Biermann et al., Kusy & Whitley, and Pompei-Reynolds & Kanavakis of 29.7ºC, 29.2ºC, 29.3ºC, and 25.05ºC, respectively. With the 35ºC variant the value in this study for Af was 33.8ºC while McCoy, Biermann et al., Kusy & Whitely, and Pompei-Reynolds & Kanavakis were 38.0ºC, 36.0ºC, 31.4ºC and 32.17ºC, respectively. The differences in these reported values can be explained possibly by improved manufacturing techniques when comparing the 27ºC variants as the Ormco wires tested in this study were only 0.1ºC off from advertised. The 35ºC variant also was only 1.2ºC off and this value was similar to that reported by Biermann et al. however these both were much closer to advertised when comparing to the McCoy and Pompei-Reynolds & Kanavakis studies. Again this can possibly be attributed to improved manufacturing technique as the McCoy study was performed early in the CuNiTi introduction, however, Pompei-Reynolds & Kanavakis was recently published and their reported Af was 32.17ºC. In the Pompei-Reynolds & Kanavakis study they illustrated that there are statistically significant differences amongst wires from the same company in different lots. This manufacturing variability could also account for the seemingly random differences amongst the experimental Af values. In addition to different lots there could be calibration differences in the machines being tested and slight data analysis procedure variation via manufacturer software.

The Afs associated with the wires analyzed in this study are presented in Table 6. In addition to the Af the difference between the two values for a given variant were also calculated.

Brand	.018”			.016”x.022”		
	Lower Af (ºC)	Higher Af (ºC)	Difference (ºC)	Lower Af (ºC)	Higher Af (ºC)	Difference (ºC)
Ormco	26.9 ± 0.7	37.1 ± 0.8	10.2	27.1± 1.6	33. 8± 1.0	6.7
American Orthodontics	31.7 ± 1.6	33.1 ± 0.9	1.4	32.8 ± 0.5	34.3± 2.1	1.5
GAC	31.5 ± 1.1	32.7 ± 0.8	1.2	25.3± 0.6	32.0± 0.8	6.7
Ortho Organizers	23.2 ± 0.6	33.9 ± 1.7	10.7	25.4 ± 1.1	32.8±0.8	7.4
RMO	28.5 ± 3.4	34.2 ± 2.0	5.7	29.2 ± 1.3	31.9±1.0	2.7
Ortho Technology	30.5± 1.6	33.2 ± 1.9	2.7	29.2 ± 1.6	30.7±1.0	1.5

Table 6: Af values for 0.018” and 0.016” x 0.022” wires for both temperature variants as well as the calculated difference between them

Most of the companies, with the exception of American Orthodontics and GAC, advertise a specific Af for a given wire. One purpose of this study was to independently verify the company claims. Table 6 demonstrates the wide range of actual Af values from this study. There is an expected difference of 8ºC between the 27ºC and 35ºC variants however none of the companies for either 0.018” or 0.016” x 0.022” were within 1 degree of the expected 8ºC difference. What is most troubling is that six of the twelve temperature/size combinations were within 3ºC of each other. American Orthodontics in particular showed a 1.4ºC and 1.5ºC difference between their two force levels with their 0.018” and 0.016” x 0.022 wires, respectively. As mentioned previously AO does not claim particular Af values for their wires instead they have different force levels. It was hypothesized that the ‘Low’ force was their 35ºC variant and the ‘Mid’ force was their 27ºC variant. Different force levels could not be assessed in this study however the small difference in the Af for their wires most likely results in very similar force levels experienced clinically. Ortho Organizers and Ormco, based on Af differences, seem to show the most consistent TTR and as a result may have higher quality control standards in manufacturing as compared to the other brands tested.

Another comparison to make from the data is the consistency in TTR for round and rectangular archwires. Would an orthodontist expect the TTR to be more consistent for one archwire size/dimension versus another? From this data it appears to be an inconsistent answer however certain companies, such as GAC, showed larger differences in Af values for their 0.016” x 0.022” archwires compared to the 0.018”. GAC also does not specify their Af for various wires rather they give arbitrary designations such as C1 and C2 with C1 representing a lower Af value. RMO also showed a difference in their round versus rectangular with their round wire being closer to the advertised values and also showing a larger difference between their Af values in the round wire. AO and Ortho Technology showed poor differentiation in Af values amongst their variants for both round and rectangular archwires. The reasons are not clear why there are differences when comparing round to rectangular, but likely different amounts of cold working are needed to form the round compared to the rectangular. Essentially the wires could have slightly different structures before the heating step, so the same protocol may not render the wire to the same exact Af temperature. Less likely but a possibility is inconsistency in the heat treatment step or location of the wires during heat treatment. What this means to the orthodontist is that once again there are variations in reported to actual values for TTR and these differences exist within the same company based on different archwire dimensions.

In addition to Af values other values, such as enthalpy, can be determined by conducting DSC. Enthalpy (J/g) in this situation is essentially the thermal energy required for a phase transformation to occur. In general the enthalpy was similar for each individual wire set in the transition from martensite to austenite upon heating as well as the transition from austenite to martensite upon cooling. Significant differences were seen when comparing different companies' wires within the same archwire dimension and expected Af as well as across different dimensions and Afs. A general trend was seen with their being a higher associated enthalpy with the 0.016" x 0.022" wire compared to the 0.018" wire in both the 27^{o}C and 35^{o}C variants. A higher enthalpy potentially could mean that there is an increase in energy demand for the phase transformation to exist so one could expect that the rectangular wire may not as easily demonstrate phase transformations. The clinical significance of enthalpy affecting usage is not known at this time, however.

By qualitatively looking at the thermograms the presence of an R phase can be observed. When examining all the wires together R-phase was observed in 17 of the 24 wires tested for the heating peak however it was never present in the cooling peak. McCoy observed R-phase to be present in 35°C and 40°C wires but not in 27°C CuNiTi wires and this was consistent with the findings in this study. Of the twelve 35°C wires tested eleven demonstrated R phase while interestingly only six of the twelve 27°C wires showed the presence of R phase. Clinically it is not known if the presence of R phase has any impact, however. These differences may result from different manufacturing techniques as mentioned previously.

When comparing all six companies against each other it was seen that there were significant differences for all interactions of temperature, brand, and size. This demonstrates that a wire from one company will not consistently perform a certain way relative to a wire from another company. This may translate to clinically different force values expressed against the teeth depending on what company, temperature variant, and size the orthodontist is using.

From this study it can be seen that advertising claims of laboratory results often are not completely accurate. It is difficult to determine how these values will impact clinical orthodontics. In a recent Cochrane Review Jian et al. (2013) deduced from the literature that the initial NiTi or stranded stainless steel wire in orthodontic alignment actually has no bearing on treatment. This result is surprising given all the time invested in developing new wires for clinical use. The downfall, however, of review articles is that they are only as strong and current as the studies that they are reviewing. With new clinical trials using these new wires there may be indeed a difference when comparing

CuNiTi to superelastic NiTi or multistranded stainless steel. Also there may be a potential difference when comparing different brands of CuNiTi to others.

CHAPTER 6

CONCLUSION

All brands tested showed significant differences between each other when comparing by size, temperature, and brand. One cannot expect to have CuNiTi wires perform similarly across different brands even when they are of the same Af and archwire dimension. For certain brands there may be very little difference between higher and lower Af variants.

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