Differential Scanning Calorimetric (DSC) Study of New and Sterilized Nickel-Titanium Rotary Endodontic Instruments

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DIFFERENTIAL SCANNING CALORIMETRIC (DSC) STUDY OF NEW AND STERILIZED NICKEL-TITANIUM ROTARY ENDODONTIC INSTRUMENTS

by

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Marquette University,
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ABSTRACT

DIFFERENTIAL SCANNING CALORIMETRIC (DSC) STUDY OF NEW AND STERILIZED NICKEL-TITANIUM ROTARY ENDODONTIC INSTRUMENTS

Michael Dyriw, D.D.S.

Marquette University, 2015

Introduction: Nickel-Titanium (NiTi) files have become popular in endodontic procedures compared to traditional stainless steel hand files owing to their increased flexibility which is especially advantageous in curved canals. Due to the associated costs, endodontic instruments are frequently re-used, which requires sterilization between uses. With the application of temperature NiTi has the ability to undergo phase transformations, altering the properties of the instruments. The purpose of this study was to investigate phase transformations of four brands of NiTi rotary endodontic instruments in the as-received condition and after multiple sterilization cycles under steam sterilization, using differential scanning calorimetry.

Materials and Methods: Four brands of nickel-titanium endodontic rotary files size 35, .04 taper were evaluated in this study: ProFile (Dentsply Tulsa Dental Specialties, Tulsa, OK), ProFile Vortex (Dentsply), Vortex Blue (Dentsply), and HyFlex CM (Coltène/Whaledent, Cuyahoga Falls, OH). The transformation temperatures and phase transformations of these files were determined in the as-received condition and after 1, 3, and 5 cycles of steam sterilization by differential scanning calorimetry. Measurements were analyzed using a two-way ANOVA.

Results: There were significant differences between file brands in austenite finish temperature ($p < 0.05$) but the only statistical significant differences in thermal properties when comparing files before and after 1, 3, and 5 cycles of steam sterilization were found with onset heating ($p < 0.05$).

Conclusion: Repeated cycles of steam sterilization do not appear to influence the phase transformations of NiTi endodontic instruments.

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Michael Dyriw, D.D.S.

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INTRODUCTION

Nickel-Titanium (NiTi) files have become popular in endodontic procedures compared to traditional stainless steel hand files, for instrumentation of root canals, due to their increased flexibility and resistance to fatigue, which is especially advantageous in curved canals (1, 2). Nitinol, designed by the Navy for use in the space program (3), was further suggested by Walia et al. in 1988 as an alternative to stainless steel files as it had two to three times more elastic flexibility in bending and torsion, as well as superior resistance to torsional fracture when compared with size #15 stainless steel files (2).

Manufacturers of these NiTi files are continually trying to improve the design of files as well as the process in which the files are produced such as electropolishing, heat treatment, and other modifications to the inherent metallic and surface properties in order to enhance performance, durability and safety. Instruments with greater flexibility may avoid undesirable outcomes in maintaining the shape of curved canals (4). For clinicians, knowledge of how these properties affect use allows the user to choose the appropriate file system as cases dictate.

Due to the associated costs, endodontic instruments are frequently re-used, which requires sterilization between uses. As well, some NiTi instruments arrive non-sterilized from the manufacturer, requiring an initial sterilization. Little research has evaluated what the effects of sterilization are on the mechanical properties of these NiTi instruments. At this point, there is a mix of publications with regards to the effects of heat sterilization with some authors stating an increase in the number of rotations-to-breakage of NiTi instruments (5) while others suggest no effects in the properties of the instruments (6, 7). Some authors have stated that heat sterilization leads to a decrease in the cutting efficiency of NiTi files (8, 9).

NiTi instruments have the ability to undergo a phase transformation, from austenite to martensite, with application of temperature or stress, giving rise to properties such as shape memory and superelasticity. A temperature-induced martensitic transformation is created when changes in the crystal structure occur when the alloy is cooled through a critical transformation temperature range (TTR). The shape memory effect is created when this deformation is reversed to its austenitic phase by heating the alloy above the TTR. Stress-induced martensitic transformation takes place as austenite transforms to martensitic NiTi with the application of stress, creating a superelastic NiTi. NiTi reverts back to austenite when the stress is relieved.

Phase transformations are associated with changes in mechanical properties of the alloy. Differential Scanning Calorimetry (DSC) is a thermoanalytical technique that has been used to evaluate phase transformations of popular NiTi endodontic instruments (10, 11). This is accomplished by analyzing the amount of heat required to increase the temperature of a file sample versus a blank reference measured as a function of temperature. Transformations are discovered as endothermic peaks on the heating DSC curves and as exothermic peaks on the cooling DSC curves. This allows us to gain knowledge of which phase of the NiTi alloy exists at a given temperature.

Onset, end-set, and peak temperatures, along with change in enthalpy for each file sample are calculated for both heating and cooling by analyzing the thermogram (Figure 1). The first peak (H1) on the heating DSC curve represents the transformation from martensite to the intermediate R-phase, while the second peak (H2) represents the transformation from R-phase to austenite. Upon cooling, the single peak (C1) represents the transformation from austenite to martensite. The intermediate R-phase may or may not appear on heating and cooling curves. The enthalpy of the transformation can be calculated by integrating the area under the peaks. The start temperature on heating (R_s) represents the temperature at which martensite starts to transform to the R-phase, while the finish temperature on heating (A_f) is the temperature at which austenite has completely formed. Upon cooling, the start temperature (M_s) represents the initial transformation from austenite to martensite, and the finish temperature (M_f) designates the completion to martensite.

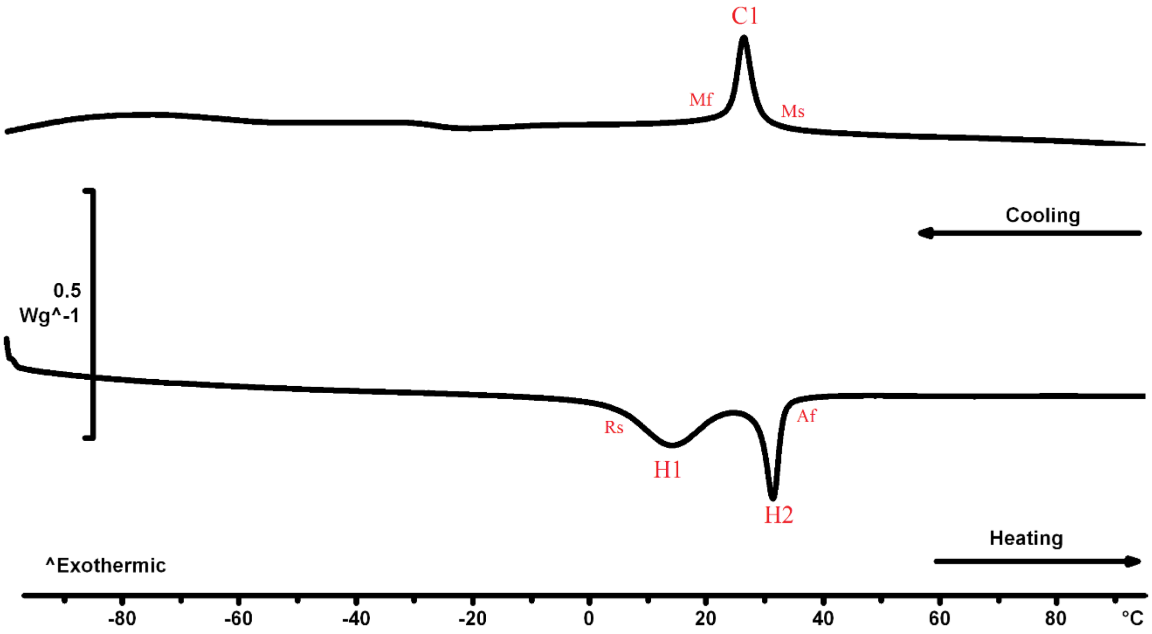

Figure 1: DSC thermogram for heating and cooling of file samples.

The purpose of this study was to investigate phase transformations of four brands of NiTi rotary endodontic instruments in the as-received condition and after multiple sterilization cycles under steam sterilization, using differential scanning calorimetry.

LITERATURE REVIEW

In order to prevent or cure apical periodontitis, conventional endodontic treatment involves complete cleaning, disinfection, and shaping of the root canal (12). Although success is based on many factors, canal preparation is one of the most important steps in root canal treatment as it determines the effectiveness of the subsequent procedures (13). Instrumentation includes removing infected tissue, debris, and dentin, creating a space for irrigation, and obturation (12, 13).

A factor that inhibits proper cleaning and shaping is the complex anatomy present in the root canal system first described by Hess in 1921 (14) and still evaluated by many others (15, 16). In addition to the complexity of the anatomy, Kakehashi et al.’s study on the germ-free and conventional laboratory rats has shown us that it is in fact the presence of bacteria that leads to the infection of the root canal system (17).

Considering that instrumentation is so important, these instruments have continually progressed from the very first made of annealed piano wire by Fouchard in 1746 (18), to the watch springs used in 1838 by Maynard (19). This progression continued, and in 1875, the first commercially available instruments were manufactured for cleaning and shaping (19). At this time in history, instruments were only designed to remove debris in order to allow for intracanal medicament placement, and so failures were rather high (12).

A standardization of instruments began in 1958 with Ingle due to the inconsistent mechanical preparation, and was eventually accepted by the American Association of

Endodontists in 1962 (20). Stainless steel hand files, offered in a 0.02 taper, have an inability to maintain the shape and natural curves of a canal system, especially in large tip sizes, due to their stiffness (21, 22). Literature has shown that in order for irrigation to reach the apical third of the canal, the canal must be enlarged sufficiently to sizes of at least a #35 or #40 file (23-28). Within the apical region lies most of the canal curvatures and an inability to maintain the original canal anatomy leads to complications such as canal transportation, zipping, ledging, and instrument separation (21). Lim and Weber further determined that these apical transportations and undesirable outcomes could be avoided with the use of more flexible files (29).

Nickel-titanium (NiTi) was originally developed in the 1960's by William J. Buehler for use in the space program and was called Nitinol (*Ni*ckel-*Ti*tanium-*N*aval *O*rdinance *L*aboratory) (3). Its main use in dentistry came in 1971 when it was specifically used as an orthodontic archwire (30). It was not until 1988 when Walia et al. suggested using NiTi orthodontic archwires to create endodontic files (2). Due to the lower elastic modulus and wide elastic working range compared with stainless steel, Walia et al. proposed that these new metallurgic properties could achieve better outcomes with less procedural errors when dealing with canal curvatures (2).

Increased flexibility of NiTi compared to stainless steel files is one of the most important characteristics for rotary instruments to negotiate curved root canals (31). Despite the NiTi alloy being quite flexible when compared with stainless steel in terms of elastic modulus, flexibility is also inversely related to the cross-sectional dimensions. This suggests that as size and taper increase, rigidity also increases. Although manufacturers have taken this into consideration and made changes to instrument design, canal transportation does still occur when the elastic limit of the NiTi instrument is exceeded in very curved canals causing the instrument to straighten (32).

NiTi rotary instruments are not impervious to fracture or separation which can be problematic during clinical use (33). When compared to stainless steel, NiTi instruments have a lower yield strength and ultimate tensile strength causing them to fracture at lower loads than for stainless steel instruments (34). Furthermore, it has been reported that these NiTi instruments can fracture unexpectedly without visible changes such as unwinding (35). Examination of clinically used NiTi instruments demonstrated fracture frequency to be anywhere from 5% as shown by Parashos et al. in 2004 (36) up to as much as 21% in another study (37). In an attempt to remove a fractured instrument from a canal, excessive dentin might be removed as well as the possibility of ledging, perforating or even extruding the instrument out the apex, most likely requiring surgery (38, 39). If left behind, there is a potential of not removing necrotic tissue if it occurred during the initial stage of instrumentation. This all leads to a reduced prognosis for success when separation occurs (40).

Much research was conducted in the early 1990’s in order to investigate NiTi instruments and reasons for their failures. It was concluded that instruments used in a rotary motion fracture in two distinct ways: torsional fracture and cyclic flexural fatigue (41). Torsional fracture refers to how much a file can rotate before the plastic limit is reached and the instrument fractures (41). Cyclic fatigue occurs when a metal is subjected to repeated cycles of tension and compression that causes its structure to break down leading to fracture (32, 37, 41).

NiTi files, in addition to instrument separation, have other shortcomings as well. NiTi instruments have been shown to wear faster and have decreased cutting efficiency when compared to stainless steel hand files (1, 31). In comparing NiTi and stainless steel K-files in step-back preparations of curved canals in resin blocks, Coleman and Svec showed that while NiTi instruments transported less, NiTi instruments took significantly longer to prepare the blocks than stainless steel hand files (42).

The alloy composition used for producing NiTi instruments is about 55% nickel and 45% titanium (wt. %) (43). There are three major phases in the NiTi alloys. These phases are what give NiTi its unique properties. The austenite phase, known as the parent phase, has a body-centered cubic structure and exists at higher temperatures and lower stresses (44). At lower temperatures and higher stresses, it exists in a monoclinic crystal structure known as the martensite phase (daughter phase) (44). Transforming between the two phases is reversible and is known as twinning (43). The R-phase forms during the transformation between austenite and martensite and is known as an intermediate phase (44). When the alloy is austenite and begins to cool, it is known as the martensite start temperature (M_s) and when transformation is complete, it is called the martensite finish temperature (M_f). Conversely, when the alloy is fully martensite and is subject to heating, austenite begins to form at the austenite start temperature (A_s) and when completely formed, it is known as the austenite finish temperature (A_f) (44).

The designs of files are continually changing as manufacturers strive to produce instruments that will work more efficiently and safely. Instruments with greater flexibility may avoid undesirable outcomes in maintaining the shape of curved canals (4). Having a knowledge base of the properties of files marketed allows the user to choose the appropriate file system in certain cases. Endodontic instrument variations have an effect on the properties such as cutting efficiency, torsional strength, and flexibility (45).

The ProFile (Dentsply Tulsa Dental Specialities, Tulsa, OK) series of instruments are manufactured from the most commercially pure form of Nitinol (Nitinol SE508) by Nitinol Devices and Components Inc (Fremont, CA) for instruments produced in the United States (46). ProFile Vortex NiTi files (Dentsply) represent the next generation of ProFile instruments. ProFile Vortex Rotary Files feature M-Wire NiTi which is claimed by the manufacturer to result in optimum performance in terms of efficiency, flexibility, and resistance to cyclic fatigue (47). The M-Wire alloy is composed of 508 nitinol that has undergone a proprietary method of treatment consisting of drawing the raw wire under specific tension and heat treatments at various temperatures, resulting in a material that includes some portion in both the martensitic and the premartensitic R phase while maintaining a pseudoelastic state (46, 48). Johnson et al. compared conventional ProFile NiTi instruments with new M-wire NiTi and found them to be nearly 400% more resistant to cyclic fatigue than traditional ProFile (46). Furthermore, other studies have shown M-Wire to be superior in cyclic fatigue resistance in comparison with those made of conventional superelastic NiTi alloys (48-51).

Alapati et al. suggested that M-Wire contains all 3 crystalline phases, including deformed martensite, R-phase, and austenite (52). Ye and Gao also concluded that M-Wire endodontic instruments are expected to have higher strength and wear resistance than similar instruments made of conventional superelastic NiTi wires because of its unique nano-crystalline martensitic microstructure (53).

An enhancement on the ProFile Vortex file, the Vortex Blue rotary file (Dentsply) uses a unique method of processing of the NiTi wire that results in a distinctive blue color, a result of a visible titanium oxide layer. The processing of Vortex Blue is claimed to reduce its tendency to stay straight allowing them to maintain the shape given to them (47). Gao et al. compared Vortex Blue with M-Wire NiTi, and conventional NiTi with regards to cyclic fatigue, torsional properties, flexibility and Vickers microhardness (54). Vortex Blue showed superior results in flexibility and cyclic fatigue, outperforming both M-Wire and conventional NiTi (54). This study was followed up by Plotino et al. which directly compared cyclic fatigue resistance of ProFile Vortex with Vortex Blue consisting of identical instruments in tip size and taper (15/.04, 20/.06, 25/.04, 25/.06, 30/.06, 35/.06, and 40/.04). They concluded that Vortex Blue showed a significant increase in cyclic fatigue resistance when compared with the same sizes of ProFile Vortex (47).

HyFlex CM (Coltène/Whaledent, Cuyahoga Falls, OH) represents what can be considered the latest generation of NiTi rotary files and has earned the term “Controlled Memory” from its manufacturer. This instrument was developed in order to increase flexibility and efficiency as a result of its superior mechanical properties. Ninan and Berzins compared shape memory files with conventional and M-Wire NiTi and found that shape memory files show greater flexibility (55). Other differences of HyFlex CM files is that they exhibit a lower percent in weight of nickel (52 Ni wt %) compared with other commonly used NiTi instruments (54.5-57 Ni wt %) (56). This change in nickel content results in a softer metal with a decreased hardness (54). The idea behind a softer metal is that it may be less aggressive in cutting dentin but has the ability to stay centered within the canal (56). CM wire is made from NiTi that has been subjected to thermal changes during processing resulting in a more martensitic phase of the metal (57). The martensitic phase is a more flexible form which allows greater flexibility and resistance to cyclic fatigue (58). These attributes may lead to less risk of ledging, transportation or perforation.

When it comes to using rotary NiTi instruments, there are many opinions on how many times they should be used in the clinic. Manufactures of these instruments recommend single use and similar suggestions were proposed by Arens et al. to reduce fracture frequency (59), whereas other studies have suggested that NiTi rotary instruments can be used up to ten times in simulated canals without fracture (60, 61). However, due to the reduction of their cyclic flexural fatigue resistance over prolonged clinical use, this should be used with caution, especially in the presence of curved canals (62-64).

It has been shown that sodium hypochlorite, used for disinfection and lubrication during root canal treatment, is quite corrosive to NiTi alloys (65). This corrosion becomes present in the varying concentrations of sodium hypochlorite from 1.2% to 5.25% and creates corrosion pits which can be detrimental to low-cycle fatigue (66, 67). Busslinger et al. studied the effect of the differing concentrations of sodium hypochlorite after 30 minutes of immersion of a NiTi instrument on elemental leaching and showed a significant increase in titanium release in 5% sodium hypochlorite, compared with 1% sodium hypochlorite (66). Considering that most clinicians use a concentration of 5.25% this might be a concern. Some researchers have suggested that the bulk mechanical properties of NiTi instruments remain unchanged when immersed in sodium hypochlorite, however, this effect is still controversial (68-70).

Heat sterilization of new or used NiTi instruments exposes them to repeated heating and cooling cycles. Several studies have examined the effects of sterilization on NiTi instruments but the topic is somewhat debatable as to the true outcome. Looking at effects of autoclave and dry-heat sterilization on the bending properties of three types of NiTi superelastic wires, Alavi et al. concluded that steam and dry-heat sterilization cause changes in the mechanical properties of superelastic NiTi arch wires (71). Hilt et al. compared stainless-steel and NiTi instruments and sterilized the instruments in increments of 10 cycles, using a full cycle and fast cycle autoclave. The files were tested by twisting each file until fracture. The findings of this study indicate that neither the number of sterilization cycles nor the type of autoclave sterilization used affects the torsional properties, hardness, and microstructure of stainless-steel and nickel-titanium files (6). King et al. concluded in their study that repeat autoclaving of unused GT Series X files (Dentsply) between three and seven times resulted in a significant reduction in torsional strength, while there was no effect observed for Twisted Files (SybronEndo, Orange, CA) (72). Alexandrou et al. noted an increase in surface roughness of instruments that underwent multiple sterilizations. DSC measurements comparing files in the as-received condition and after 11 sterilizations suggested that they were completely austenite in the oral environment temperature. They concluded that they are capable of superelastic behavior in appropriate clinical conditions (73). In a study by Canalda-Sahli et al., it was suggested there is a slight decrease in flexibility of NiTi files after 10 cycles of heat sterilization, however all files satisfied minimum ISO specifications for angular deflection (74). Serene et al. reported that heat sterilization increases the fatigue life of NiTi files through an increase in hardness and torsional resistance (75). This was corroborated by Chaves Craveiro de Melo et al. which demonstrated that the fatigue resistance of ProFile instruments to cyclic rotation in simulated canals as well as microhardness were increased after five cycles of heat sterilization (5).

Differential scanning calorimetry (DSC) is a thermoanalytical technique which measures that amount of heat needed to increases the temperature of a sample and reference at the same rate and is measured as a function of temperature. Transformations are represented as peaks on a DSC thermograph which represent phase transformations. These phase transformations are either endothermic or exothermic, depending on the amount of heat needed to flow through a sample. Computer software accompanying the DSC can be used to analyze the thermographs and determine phase transformation temperature ranges and enthalpy for heating and cooling processes (44).

DSC was first used by Leu et al. to evaluate austenitic-martensitic transformations of superelastic NiTi wires (76). During phase transformation of superelastic nickel-titanium from martensite to austenite, an intermediate rhomboidal or R-phase was discovered. Bradley et al. in 1996 studied superelastic, nonsuperelastic, and shape-memory NiTi wires in as-received condition through DSC to determine the transformation temperatures for the austenitic, martensitic, and R structure phases of each (44). This study concluded that nonsuperelastic wires were almost entirely martensite at room temperature and only contain small amounts of austenite intraorally. As well, superelastic NiTi is almost entirely austenite in the oral cavity but undergo transformation below 0^{0}C to austenite. An intermediate R phase was also evident in the superelastic wires. Lastly, the shape-memory wires were reported to be entirely austenite intraorally (44).

Brantley et al. in 2002 studied ProFile and Lightspeed rotary endodontic instruments in the as-received condition and noted that the instruments would be completely austenitic at room temperature (10). During clinical usage, the instruments undergo superelastic behavior, where stress on the file causes transformation to martensitic phase and back to austenite upon release of such stress unless more than about 10% tensile strain takes place (77). Brantley et al. also found that although differences in the transformation temperatures and enthalpies existed between the two files, both have A_f being near 25°C to permit superelastic behavior during clinical use (10).

Brantley et al. in 2002 then compared ProFile and Lightspeed NiTi rotary files after one, three, and six periods of simulated clinical use in extracted teeth using DSC analysis (11). The analysis of these files showed that they were always in the superelastic condition. They discovered that there was no evident effect after simulated clinical use for both brands tested which they attributed to insufficient mechanical deformation of the files (11). They did find that there were differences when comparing test segments from different positions along the shafts of the instruments with regards to the enthalpy change associated with the transformation from martensitic NiTi to austenitic NiTi for test segments. It was suggested that these variances are attributed to differences in work hardening along the shaft and variations during the productions of the two batches of each instrument (11).

MATERIALS AND METHODS

A total of 128 new rotary NiTi endodontic instruments, taper 0.04, size 35 and 25-mm long were examined: 32 ProFile (Dentsply Tulsa Dental Specialties, Tulsa, OK), 32 ProFile Vortex (Dentsply), 32 Vortex Blue (Dentsply), and 32 HyFlex CM (Coltène/Whaledent, Cuyahoga Falls, OH).

Each set of files were divided into groups of 8 to represent 4 groups: as-received condition, 1 cycle of sterilization, 3 cycles of sterilization, and 5 cycles of sterilization. The sterilizations were conducted in a Getinge Castle 4233 vacuum steam sterilizer at 270^{o}F for 4 minutes followed by a 20 minute drying period and allowed to cool for 1 hour at room temperature.

Files were sectioned 3 mm from the tip and again 4 mm from the previous section (Figure 2) using a water-cooled diamond saw (Figure 3, Buehler, Lake Bluff, IL). The file segments were weighed via an electronic balance (Figure 4, Mettler-Toledo, Columbus, OH) and were sealed into 40 µl aluminum crucibles.

An empty 40 µl aluminum crucible served as a reference during testing (Figure 5). Both crucibles were heated from -100°C to 100°C and subsequently cooled from 100°C to -100°C in the differential scanning calorimeter (DSC822e, Mettler-Toledo) at a rate of 10°C per minute with liquid nitrogen serving as a coolant (Figure 6).

DSC thermogram (Figure 7) plots were constructed by the manufacturer’s software and were quantitatively and qualitatively analyzed. Start, finish and peak temperatures along with transformation enthalpy for each sample were calculated for both heating and cooling. Curves were integrated with a spline baseline and where the tangents along the shoulders of the starting and ending peaks cross the baseline determine onset and end-set temperatures. Integration of these lines provides enthalpy (J/g).

A two way analysis of variance (ANOVA) was performed on each of the measurements to see if there was a difference between sterilization cycles and file types. When the ANOVA test returned a significant result, Tukey’s HSD test was used to determine which of the variants were significantly different.

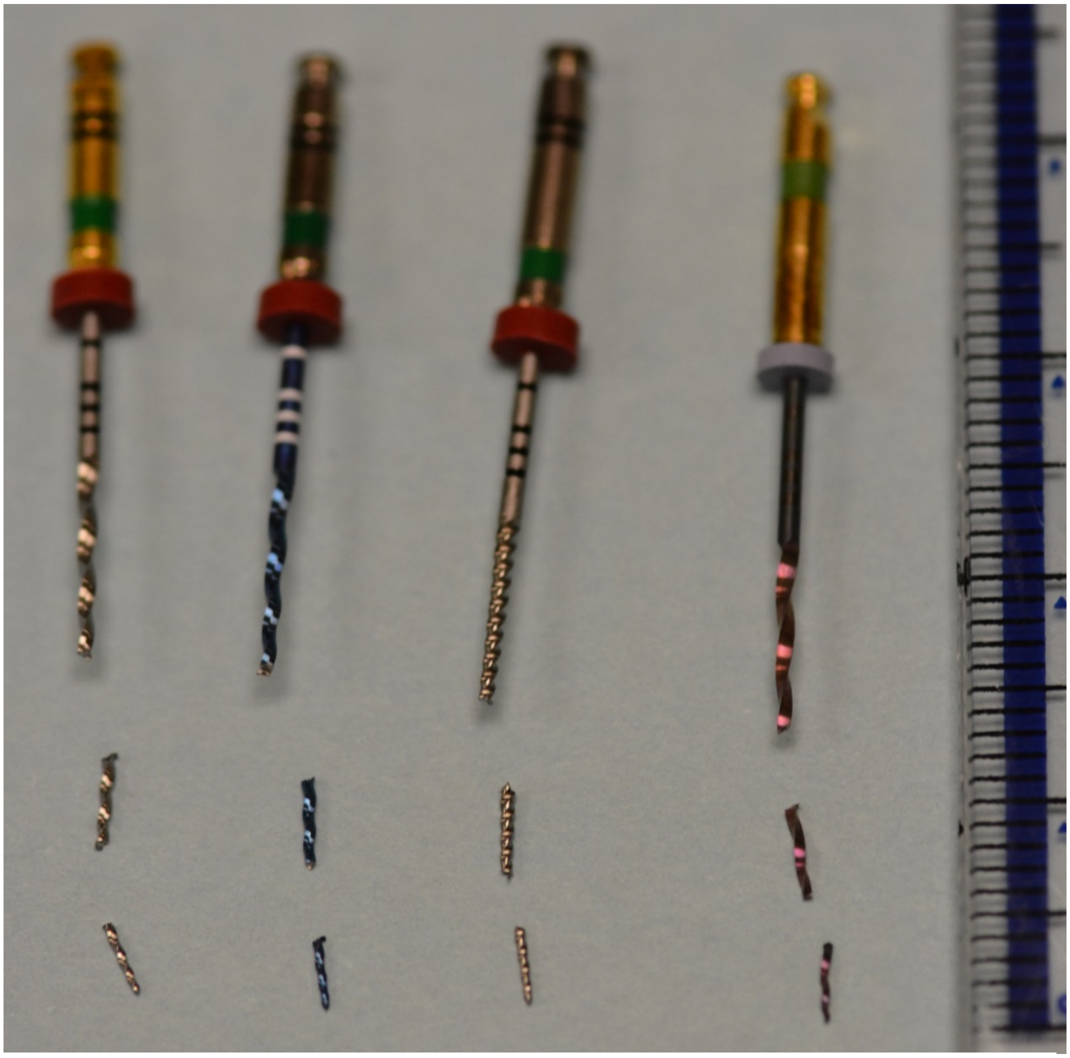

Figure 2: Files sectioned at 3 mm and 7 mm. (Left to right: Vortex, Vortex Blue, ProFile, HyFlex CM)

Figure 3: Water-cooled diamond saw.

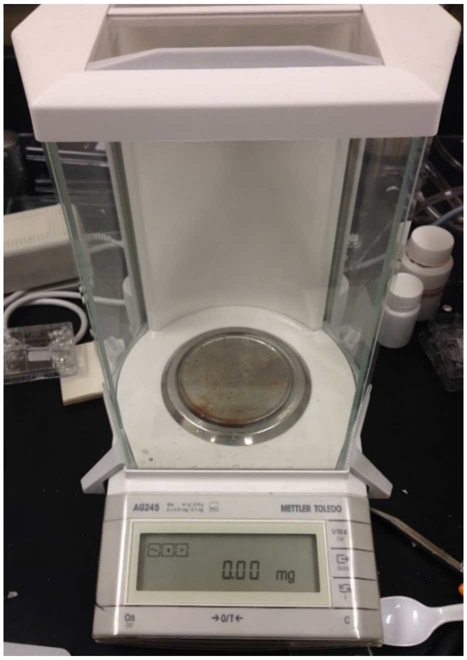

Figure 4: Electronic balance.

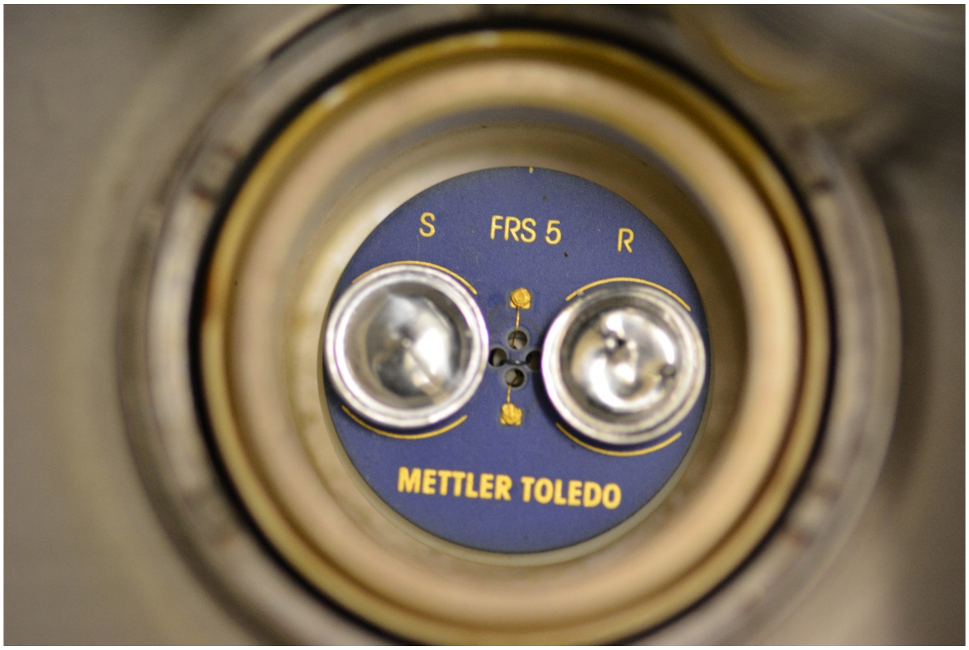

Figure 5: Aluminum crucibles on the sensor of the DSC. The crucible containing the file sample is on the left and the blank reference crucible is on the right.

Figure 6: DSC equipment and liquid nitrogen.

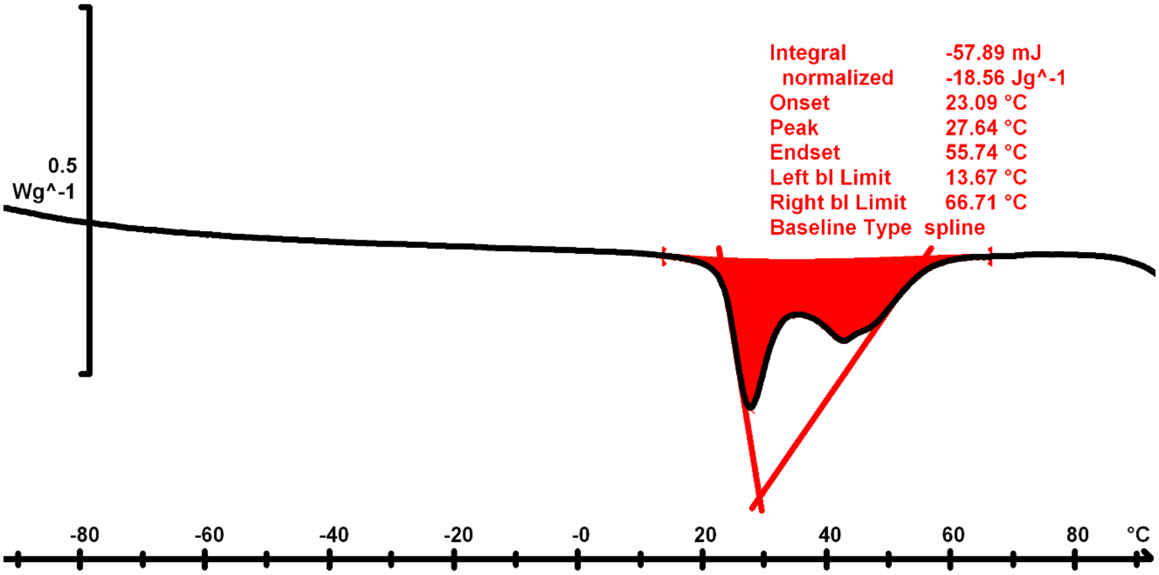

Figure 7: Integration of DSC heating file sample

Results

Figure 8 displays a thermogram comparing an as-received ProFile file with files after 1, 3, and 5 cycles of steam sterilization. Figure 9 displays a thermogram comparing an as-received Vortex file with files after 1, 3, and 5 cycles of steam sterilization. Figure 10 displays a thermogram comparing an as-received Vortex Blue file with files after 1, 3, and 5 cycles of steam sterilization. Figure 11 displays a thermogram comparing an as-received HyFlex CM file with files after with 1, 3, and 5 cycles of steam sterilization. Observation of these thermograms does not show a qualitative difference between them.

Data for mean onset temperatures (°C), end-set temperatures (°C), peak temperatures (°C), and change in enthalpy (J/g) for each file on heating and cooling as determined from the DSC plots are summarized in Table 1 and Table 2. Table 3 summarizes any differences between file types with regards to thermal properties.

Figure 12 displays a thermogram comparing the as-received files of ProFile, Vortex, Vortex Blue, and HyFlex CM. It can be seen that Vortex Blue and HyFlex CM gave more intense peaks. This is consistent with the enthalpy provided in Tables 1 and 2. It can be qualitatively observed from Figure 12 that Vortex and ProFile files exhibited 1 small peak around 10^{o}C and a larger peak around 40^{o}C on heating with just one cooling peak around 40^{o}C. Alternatively, HyFlex CM and Vortex Blue showed 2 connected peaks on heating but Vortex Blue had one peak on cooling whereas HyFlex CM exhibited multiple peaks on cooling.

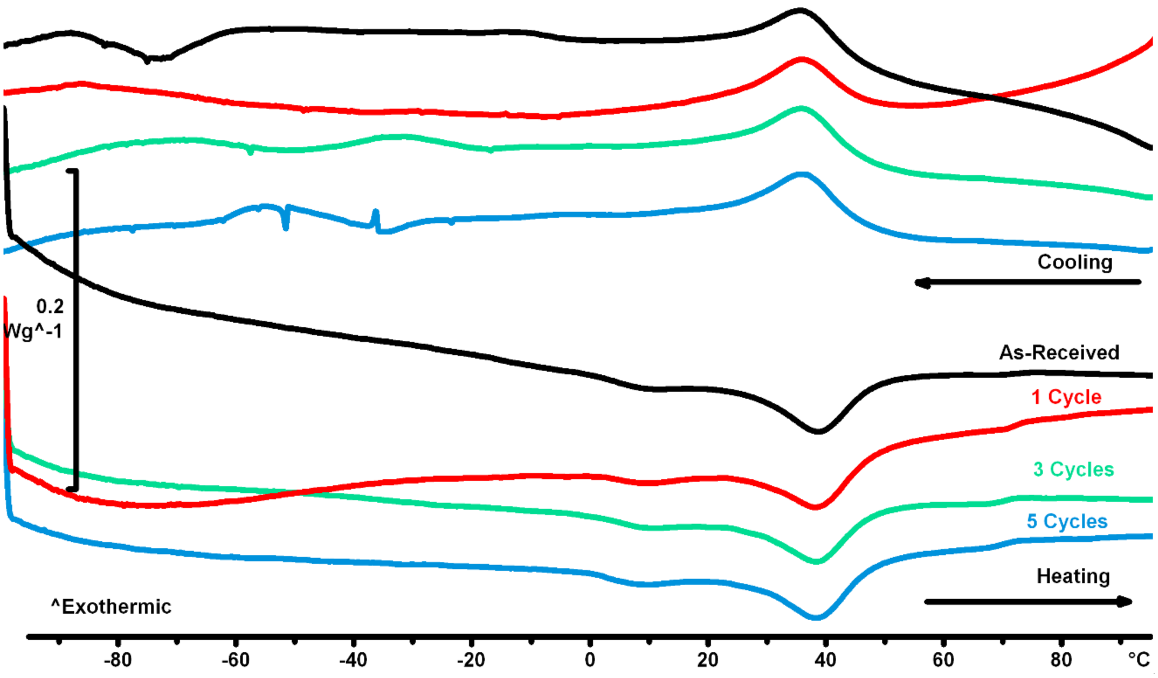

Figure 8: DSC heating and cooling curves for ProFile size 35 with .04 taper.

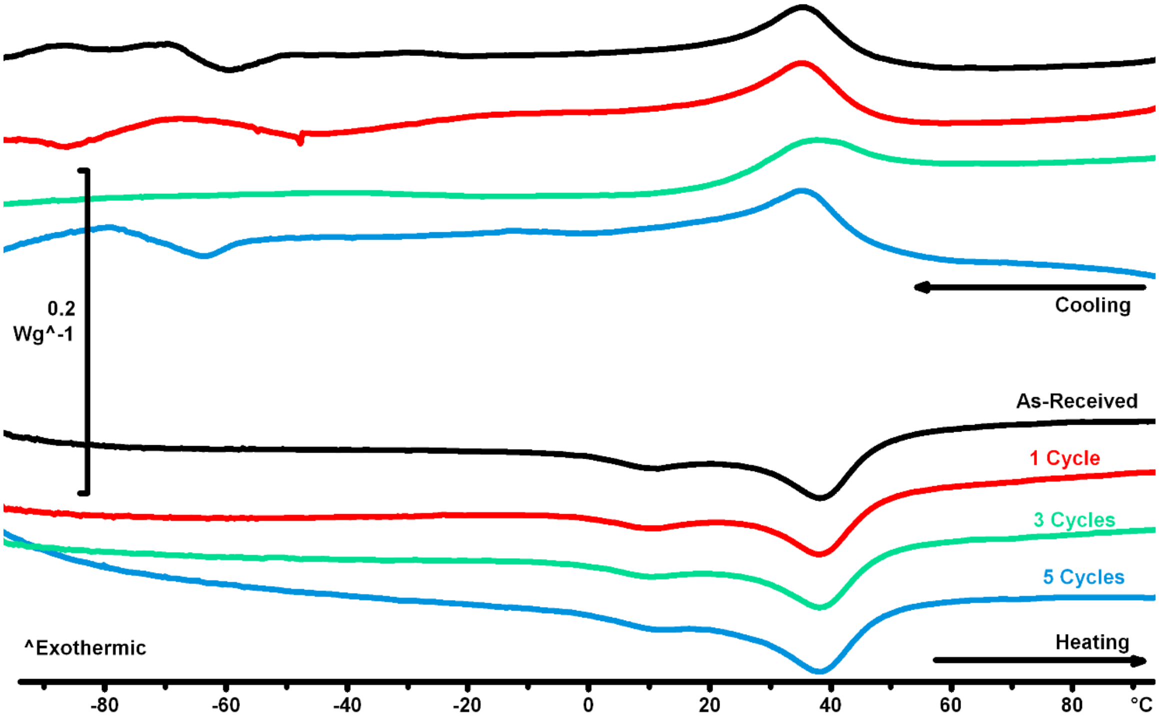

Figure 9: DSC heating and cooling curves for Vortex size 35 with .04 taper.

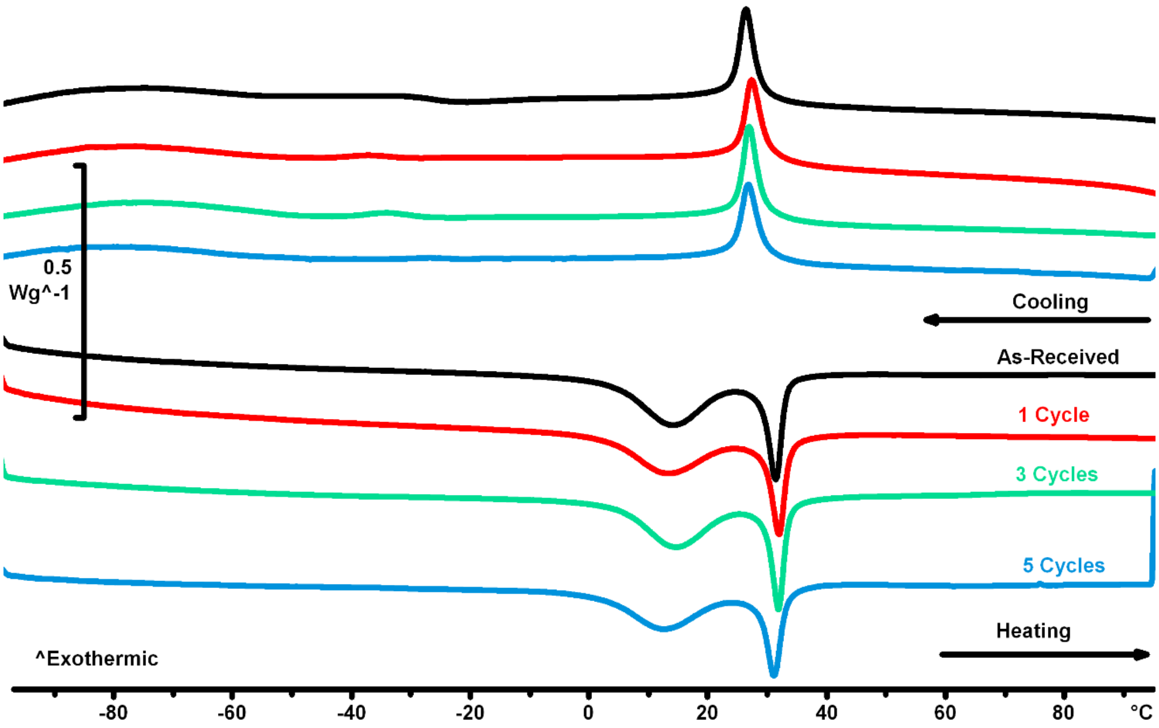

Figure 10: DSC heating and cooling curves for Vortex Blue size 35 with .04 taper.

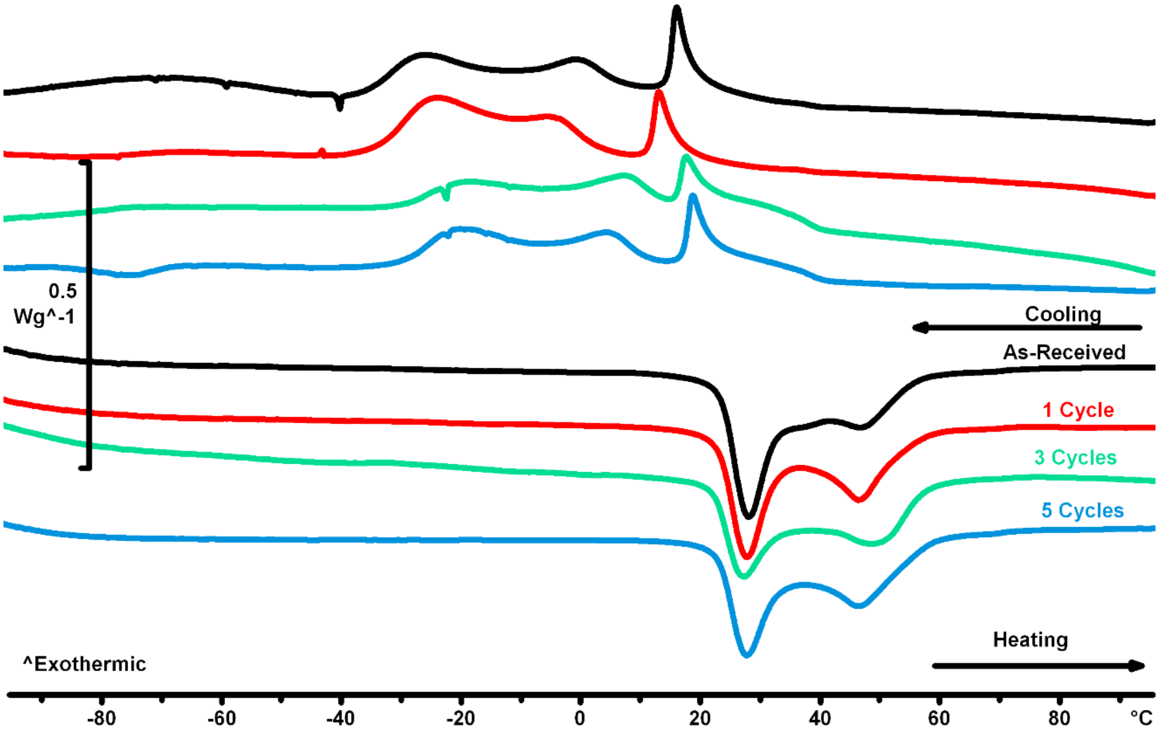

Figure 11: DSC heating and cooling curves for HyFlex CM size 35 with .04 taper.

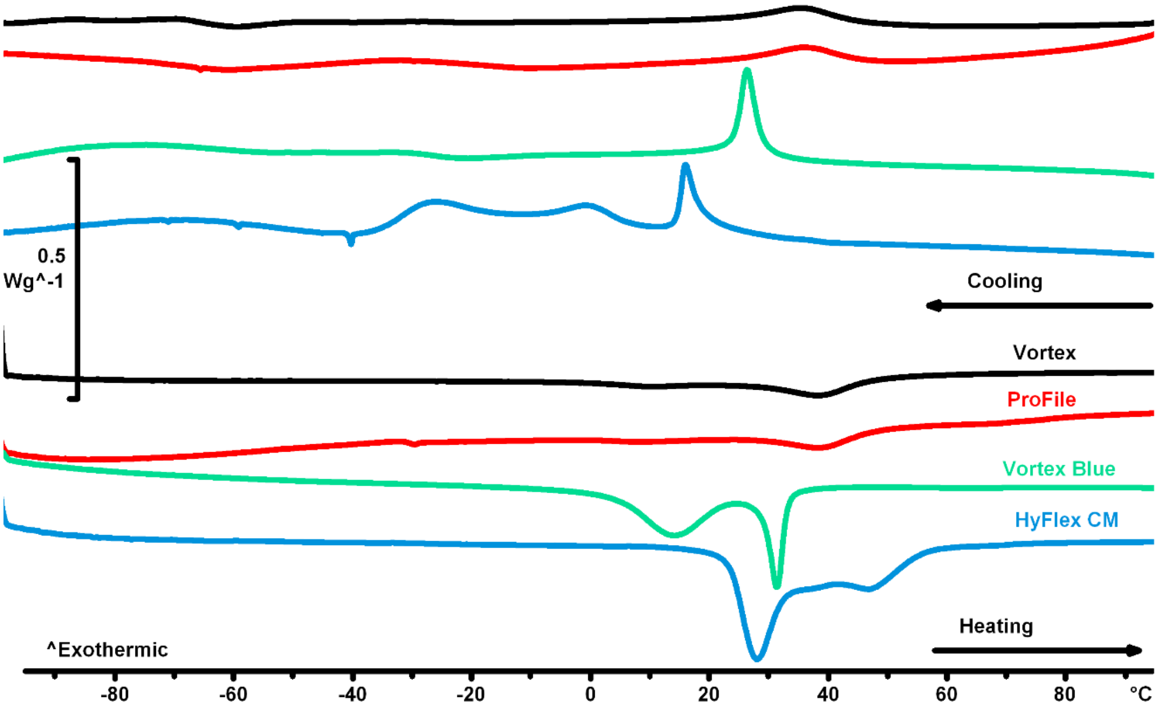

Figure 12: DSC heating and cooling curves for as-received ProFile, Vortex, Vortex Blue, and HyFlex CM size 35 with .04 taper.

File	Cycles	Heating Onset (°C)	Heating Peak (°C)	Heating End-set (°C)	Change in enthalpy (J/g)
ProFile	0	-.4 ± 1.6	38.6 ± 0.2	48.0 ± 0.4	3.11 ± 0.76
	1	2.0 ± 1.1	38.6 ± 0.2	47.6 ± 0.9	2.94 ± 0.45
	3	1.0 ± 1.5	38.6 ± 0.2	47.7 ± 0.4	3.01 ± 0.74
	5	0.3 ± 1.7	38.6 ± 0.2	47.8 ± 0.4	2.96 ± 0.44
Vortex	0	0.2 ± 0.8	38.5 ± 0.3	48.3 ± 1.4	3.10 ± 0.49
	1	0.1 ± 0.4	38.5 ± 0.2	47.7 ± 0.3	3.53 ± 0.86
	3	0.1 ± 0.6	38.4 ± 0.2	47.7 ± 0.2	3.33 ± 0.22
	5	0.3 ± 1.0	38.4 ± 0.2	47.7 ± 0.1	3.33 ± 0.30
Vortex Blue	0	2.5 ± 0.9	31.2 ± 0.4	33.2 ± 0.4	9.80 ± 0.81
	1	3.8 ± 0.7	31.3 ± 0.5	33.3 ± 0.5	10.30 ± 0.51
	3	3.8 ± 0.9	31.4 ± 0.3	33.3 ± 0.4	10.42 ± 0.81
	5	2.5 ± 1.3	31.3 ± 0.4	33.4 ± 0.3	10.29 ± 0.70
HyFlex CM	0	24.2 ± 0.8	37.0 ± 11.9	57.3 ± 4.0	18.01 ± 3.28
	1	24.8 ± 2.0	32.8 ± 9.0	57.8 ± 3.0	17.57 ± 0.82
	3	23.4 ± 1.0	34.3 ± 12.1	58.2 ± 2.1	17.95 ± 1.17
	5	23.6 ± 0.8	34.5 ± 11.9	58.1 ± 2.5	17.97 ± 0.79

Table 1: Differential scanning calorimetry measured temperature and enthalpy changes for phase transformations during heating of files

File	Cycles	Cooling Onset (°C)	Cooling Peak (°C)	Cooling End-set (°C)	Change in enthalpy (J/g)
ProFile	0	-.4 ± 1.6	38.6 ± 0.2	48.0 ± 0.4	3.11 ± 0.76
	1	2.0 ± 1.1	38.6 ± 0.2	47.6 ± 0.9	2.94 ± 0.45
	3	1.0 ± 1.5	38.6 ± 0.2	47.7 ± 0.4	3.01 ± 0.74
	5	0.3 ± 1.7	38.6 ± 0.2	47.8 ± 0.4	2.96 ± 0.44
Vortex	0	45.7 ± 0.9	35.4 ± 0.4	22.9 ± 2.2	2.28 ± 0.40
	1	46.6 ± 1.7	35.9 ± 0.6	23.9 ± 1.1	2.10 ± 0.50
	3	45.8 ± 1.1	35.6 ± 0.2	24.1 ± 0.6	2.16 ± 0.36
	5	45.5 ± 0.3	35.6 ± 0.3	22.4 ± 2.3	2.40 ± 0.11
Vortex Blue	0	29.4 ± 0.4	26.5 ± 0.4	23.9 ± 0.4	4.02 ± 0.12
	1	29.4 ± 0.5	26.6 ± 0.5	24.3 ± 0.6	4.12 ± 0.11
	3	29.5 ± 0.4	26.7 ± 0.3	24.3 ± 0.4	4.17 ± 0.15
	5	29.6 ± 0.4	26.6 ± 0.4	24.2 ± 0.4	4.08 ± 0.16
HyFlex CM	0	22.2 ± 3.7	18.8 ± 3.2	16.6 ± 4.4	2.25 ± 0.54
	1	18.2 ± 2.9	15.4 ± 6.0	13.0 ± 6.9	2.45 ± 0.54
	3	20.4 ± 2.9	17.1 ± 3.3	14.0 ± 5.9	2.21 ± 0.27
	5	21.2 ± 2.4	17.9 ± 2.6	14.9 ± 5.2	2.55 ± 0.34

Table 2: Differential scanning calorimetry measured temperature and enthalpy changes for phase transformations during cooling of files

Thermal Property	Statistical Significance
Onset Heat	Hyflex CM > Vortex Blue > ProFile = Vortex
Peak Heat	ProFile = Vortex > HyFlex CM = Vortex Blue
End-set Heat	HyFlex CM > Vortex = ProFile > Vortex Blue
Enthalpy Heat	HyFlex CM > Vortex Blue > Vortex = ProFile
Onset Cool	ProFile = Vortex > Vortex Blue > HyFlex CM
Peak Cool	ProFile = Vortex > Vortex Blue > HyFlex CM
End-set Cool	ProFile = Vortex Blue = Vortex > HyFlex CM
Enthalpy Cool	Vortex Blue > HyFlex CM = Profile = Vortex

Table 3: Differences of thermal properties between file types

The 2 factors analyzed in the ANOVA were autoclave cycles and file brand. What the results showed was that there was no difference between cycles except with onset heating ($p < 0.05$). For all thermal parameters, there is a significant difference between brands. However, there is no significant interaction except for onset heating and cooling temperature. The most important factor is that of austenite finish temperature which did not change within file brands. This indicates that each file was similarly not affected by autoclaving.

The post-hoc Tukey's HSD test showed that from as-received to 1 cycle of sterilization and from 1 cycle of sterilization to 5 cycles of sterilization, the onset heating temperature was significantly greater than 5 cycles of sterilization and as-received. It was also seen that 3 cycles of sterilization was equivalent to all other subsets.

DISCUSSION

Nickel-Titanium rotary files have become the gold standard when it comes to cleaning and shaping of the root canal system due to their increased flexibility and resistance to fatigue (1, 2). Manufacturers of NiTi files continually try to improve on the design of files through processes such as electropolishing, heat treatments, and other modifications to produce a file with greater flexibility and torque resistance. This study examined the effect of sterilization on the inherent metallurgical properties of 4 file brands through differential scanning calorimetry in order to gain better insight into their phase transformations.

This study demonstrated that within each file brand, sterilization of 1, 3, and 5 cycles, as compared to the as-received condition creates essentially no difference in the phase transformation behavior. The only difference which was seen after sterilization was that of onset heating. This represents the austenite or R-phase start temperature but is less important than austenite finish temperature which is a critical factor in a files transformation behavior, giving rise to properties such as superelasticity and shape memory (78). This suggests that the structure of the file is unaffected by sterilization. These findings are similar to what Hilt et al. discovered in 2000, when NiTi files were twisted until fracture following increments of 10 cycles of sterilization. They found no change in torsional properties, hardness, and microstructure (6). Alexandrou et al. observed an increase in surface roughness of instruments that underwent multiple sterilizations (73). Any change in a files performance following sterilization is most likely to occur as a result of changes that occur to the outer surface of the file. An increase in surface roughness, debris remaining and surface defects on a files surface can lead to microfractures and an increase in failure rate of the file. This was also demonstrated by Rapisarda et al. in 1999 and Schafer in 2002, both of whom found that repeated sterilizations under autoclave alter the superficial structure of such instruments (8, 9). This likely also explains why King et al. found a significant reduction in torsional strength of GT Series X files upon repeat sterilization (72).

Analyzing between brands, it is evident that there are many differences as can be seen in Table 3. This is understandable as all files are created by different techniques, utilizing different processing. As stated previously, the thermal parameter that is the largest determinant on a file's shape memory and superelasticity properties is A_f. As evidenced in the table, there is a statistical difference between HyFlex CM, Vortex Blue and ProFile/ProFile Vortex. There was no statistical difference between ProFile and ProFile Vortex. This difference in A_f temperatures can most likely be explained by the different manufacturing steps used to produce the files which then results in different proportions of phases and microstructural elements. This is explained by Bradley et al. in which they determined that differences between wires was due to the relative proportions of the metallurgical phases in the microstructures (44). As well, both Vortex Blue and HyFlex CM are heat treated after manufacturing with differing amounts of time and temperature which affect transition temperature. Vortex Blue is produced with a proprietary treatment resulting in a visible titanium oxide layer giving it a distinctive blue color which is claimed to reduce its tendency to remain a straight file allowing them to maintain the shape given to them (47). HyFlex CM on the other hand exhibits a lower weight of nickel (52 Ni % wt) as compared with other commonly used NiTi instruments

(54.5-57 Ni % wt) (56). A lower weight of nickel would also help explain the difference HyFlex CM demonstrated.

Ninan and Berzins similarly found that shape memory files compared with conventional and M-Wire NiTi show greater flexibility (55). This is demonstrated by the fact that it has a more martensitic phase when at room temperature or above. As seen in Table 1, as-received HyFlex CM files have a much higher onset temperature at 24.2 ± 0.8°C and an end-set temperature of 57.3 ± 4.0°C. CM wire is made from NiTi that has been subjected to thermal changes during processing resulting in a more martensitic phase of the metal (57). The martensitic phase is a more flexible form which allows greater flexibility and resistance to cyclic fatigue (58). As-received ProFile, Vortex, and Vortex Blue samples had an onset of -.4 ± 1.6°C, 0.2 ± 0.8°C, and 2.5 ± 0.9°C respectively. This explains why these files are likely stiffer.

SUMMARY AND CONCLUSIONS

This study concluded that autoclaving with steam sterilization up to 5 cycles had no effect on the phase transformations of ProFile, ProFile Vortex, Vortex Blue, or HyFlex CM NiTi files. It is important to note there was no effect on austenite finish temperature.

This study also demonstrated that there are differences in thermal transition temperature between files which would explain why different files behave differently with regards to flexibility and other such properties.

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