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**METALLURGIC COMPOSITION, PHASE TRANSFORMATION, AND  
MECHANICAL PROPERTY ANALYSIS OF HEAT-TREATED VERSUS  
CONVENTIONAL NICKEL-TITANIUM ENDODONTIC ROTARY FILES**

by

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## Metallurgic Composition, Phase Transformation, and Mechanical Property Analysis of Heat-treated Versus Conventional Nickel-Titanium Endodontic Rotary Files

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### Abstract

**Gray, TL, Lien, W, de Guzman, J.** Metallurgic Composition, Phase Transformation, and Mechanical Property Analysis of Heat-treated Versus Conventional Nickel-Titanium Endodontic Rotary Files. *International Endodontic Journal* x, xx-xx, 2021

**Aim:** To analyze the metallurgic composition, phase transformation and mechanical properties of three heat-treated nickel-titanium (NiTi) endodontic rotary files: EdgeEndo X7 (X7), EdgeSequel™ Sapphire (ESS), and Vortex Blue (VB) compared to a conventional NiTi rotary file: Profile (P). The null hypothesis is there is no difference in metallurgic composition, phase transformation and mechanical properties of heat-treated and conventional NiTi files. **Methodology:** File size #35 was selected. Metallurgic composition was analyzed using EDS (n=3). Phase transformation was analyzed using DSC (n=1). Elastic modulus (EM) and hardness (H) were obtained using nanoindentation (n=3). Torsion testing was completed by fixating the handle and 7 mm of the file tip into separate ends of an electromechanical torsion instrument (n=10) and subjecting samples to continuous torque at a rate of 5 degrees of rotation per second until fracture. Statistical analysis was performed by one-way ANOVA test, with Tukey-HSD post hoc comparison,  $\alpha = 0.05$  and unequal variance t-test. A p-value  $< 0.0001$  was considered significant. **Results:** X7, ESS, and VB had significantly higher Ni/Ti mass ratio than P. X7, ESS, and VB displayed an R-phase; P did not. X7 and ESS had significantly higher EM than VB and P. X7, ESS, and P had significantly higher H than VB. X7 exhibited the largest maximum degree of rotation before fracture and P had the smallest. ESS and P had significantly higher maximum torque applied prior to fracture compared to X7 and VB. All file systems had significantly different degrees of rotation at the maximum torque applied before fracture; X7 was the largest, and P was the smallest. High Ni/Ti mass ratio, presence of an R-phase, and a high EM resulted in larger maximum degree of rotation and higher degree of rotation at the maximum torque applied before fracture. Low Ni/Ti mass ratio, absence of R-phase, and a high H resulted in higher maximum torque applied before fracture. **Conclusions:** The null hypothesis was rejected. Heat-treated NiTi endodontic files exhibited different metallurgic compositions, phase transformations, and mechanical properties than conventional NiTi files.

**Key words:** Heat treatment, metallurgic composition, nanoindentation, nickel-titanium endodontic instruments, phase transformation temperatures, torsional fatigue resistance

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## Introduction

Endodontic files were primarily fabricated from stainless steel up until the late 1980s when Walia and colleagues first used orthodontic nickel-titanium (NiTi) wire to fabricate endodontic files (Walia *et al.* 1988). This novel NiTi endodontic file demonstrates superior elastic flexibility in bending and torsion fatigue resistance compared to stainless steel (Walia *et al.* 1988). The increased flexibility of NiTi is attributed to the alloy's lower elastic modulus (Thompson 2000). This flexibility allows the files to navigate root canal morphology with respect to the natural anatomy and curvatures and decrease clinical errors commonly seen with the use of stiffer stainless steel files. It is imperative for clinicians to use instruments with superior mechanical properties to maintain the root canal anatomy and avoid grossly enlarging the canal space due to preparation errors. The superior flexibility of rotary NiTi instruments over stainless steel instruments decreases the likeliness of zipping, ledging, perforation, or transportation of the canal which could potentially reduce the long-term prognosis of the tooth (Zupanc *et al.* 2018). The clinical performance and mechanical properties of NiTi endodontic rotary instruments are heavily impacted by the file's cross-section, flute design, raw material, and different manufacturing processes (Shen *et al.* 2013).

Nickel-titanium was developed in the 1960s by W.F. Buehler who was working for at the Naval Ordnance Laboratory in the space program (Thompson 2000). The composition of the NiTi endodontic files consists of approximately 56% (weight) nickel and 44% (weight) titanium, resulting in a 1:1 atomic ratio, near equiatomic, and is generically coined 55-Nitinol (Thompson 2000). Energy dispersive x-ray spectroscopy (EDS) is an analytical technique used for elemental analysis of a sample and provides a quantitative elemental composition profile of a NiTi rotary file. Literature contains conflicting information pertaining to the elemental percentage composition of NiTi alloys and its impact on the alloy's properties and behaviors. Aminsobhani *et al.* reported that a nickel-rich NiTi alloy would be influenced by secondary phases found in the alloy that would have an impact on phase transformation temperatures, mechanical properties, and shape memory behaviors of the instrument (Aminsobhani *et al.* 2016). It has been shown that a one percent change in nickel content causes a 100°C change to the martensitic starting temperature or the austenite finishing temperature (Yahata *et al.* 2009). Contrary to these findings, Zinelis *et al.* reported that deviations from the standard 1:1 atomic ratio NiTi alloy were most likely caused by raw material variations during manufacturing the alloy and do not affect the mechanical properties of the instrument (Zinelis *et al.* 2010). It was found that the proprietary thermomechanical processing by the manufacturers of NiTi rotary files contributes more to the mechanical properties and transformation temperatures of the alloy (Zinelis *et al.* 2010). Therefore, NiTi alloy should not be described by its elemental composition, i.e. 55-Nitinol, but should be classified based on the transformation temperature instead (Zinelis *et al.* 2010).

NiTi exists in three different phases based on the crystalline structure. The crystal arrangement of the alloy is temperature dependent. At high temperatures, the alloy is in an austenite (or parent) phase and has a cubic B2 crystal structure (Zupanc *et al.* 2018). At low temperatures, the alloy is in a martensite (or daughter) phase and has a monoclinic

B19' crystal structure (Zupanc *et al.* 2018). Martensitic transformation occurs when the alloy transforms from austenite to martensite phases. This transformation is stress or temperature induced and occasionally results in an intermediate R-phase (rhombohedrally distorted phase) prior to transitioning to martensite (Zupanc *et al.* 2018). The martensitic transformation gives the NiTi alloy two physical properties: superelasticity and shape memory (Zupanc *et al.* 2018). A NiTi alloy file that is primarily in the austenite phase will be stiff, hard, and present with superelasticity and has the tendency to spring-back to its original form after deformation (Zupanc *et al.* 2018). Superelasticity allows the NiTi instrument to deform up to 8% strain and be capable of full recovery (Thompson 2000). Those that are primarily in the martensitic phase will be soft, ductile, easily deform, and have shape memory characteristics when heated (Zupanc *et al.* 2018).

When a martensitic NiTi alloy is heated, it begins to change its crystal structure into an austenitic phase. The temperature at which this phase transformation starts is the austenite transformation start temperature ( $A_s$ ). The temperature at which the alloy has completely converted to austenite is the austenite transformation finish temperature ( $A_f$ ). When an austenite phase alloy is cooled, it will transform into a martensitic phased alloy. The temperature at which this phase transformation begins is the martensitic start temperature ( $M_s$ ) and the temperature at which phase transformation is complete is the martensitic finish temperature ( $M_f$ ). These transformation temperatures critically influence the mechanical properties and behaviors of the NiTi files. The temperatures can be altered by a change in alloy composition, impurities within the alloy, and heat treatments during the manufacturing process (Shen *et al.* 2013). Differential scanning calorimetry (DSC) analysis can be utilized on endodontic NiTi rotary files to determine the phase transformation temperatures. This valuable data allows a clinician to know what phase the file is in during use and what physical and mechanical properties the file may exhibit during endodontic treatment.

Some specific mechanical properties of interest for NiTi endodontic files include elastic modulus (EM) and hardness (H). EM is the stiffness of a material and H is the resistance of a material to plastically deform, typically measured by indentation (Anusavice *et al.* 2013). It is also important to consider the ultimate strength of the file, which is the stress applied to a material to the point of fracture. The EM is constant to the material; the thickness of the material may affect the stiffness of that material, but the EM will remain the same (Anusavice *et al.* 2013). There are several benchtop tests used in literature to measure the H of a material. Some examples include Brinell, Rockwell, Vickers, and Knoop hardness tests. Each test employs a different shaped tip for indenting the sample. Oliver and Pharr introduced a new method for determining EM and H of a material from indentation load-displacement data (Oliver & Pharr 1992, Oliver & Pharr 2003). A Berkovich indenter tip, a three-sided pyramid, is used for indentation with an area-to-depth function the same as the Vickers hardness test. The studies reported that using this method yielded an experimental EM within 4% of the values reported in literature for the same material. It was later shown that the Oliver-Pharr method can be applicable to shape memory alloy that exist in different phases such as nickel-titanium (Kan *et al.* 2013). This is important to know since NiTi exists in different phases and stress, i.e. indentation, can cause a phase transformation of the material.

During cleaning and shaping of a root canal system, endodontic files have the potential to separate within the canal. Rotary NiTi files undergo two types of stresses that lead to separation, torsional stress and cyclic fatigue. Torsional stress is applied to the rotary file when part of the file binds in the canal, typically the tip of the file, while the file continues to rotate. Cyclic fatigue leads to separation of files in canals with curvatures. While rotating in the curvature, the file alternates between tension-compression cycles until separation occurs (Jamleh *et al.* 2016). Torsional stress can be visually observed prior to file separation when examining the file after use when the metal is undergoing plastic deformation. On the contrary, cyclic fatigue separation can occur without any signs of deformation of the metal (Arias *et al.* 2019).

It is important to understand the metallurgic composition, NiTi phase transformation, and mechanical properties of the instruments chosen and the impact these things can have on a clinical level. Because NiTi rotary endodontic files can exist in different phases and exhibit different mechanical properties, the clinician needs to research the instruments of choice in order to make a sound decision based on evidence and not just based on manufacturer's claims of superiority. Mechanical and fatigue properties are related to the metallurgy of the alloy as well as the phase the file exists in during clinical use which can be manipulated through heat treatment. Understanding the properties of the alloy of the instruments will give the clinician some insight to the performance capabilities and limitations of that instrument and chose the best file system for a given clinical situation.

EndoEdge recently released a series of NiTi rotary files made from their proprietary heat-treated FireWire. The company claims that these files are compatible with other brands of files but are cheaper and have increased cyclic fatigue resistance. According to EdgeEndo's website, the EdgeFile X7 (X7) file is marketed to be comparable to any conventional NiTi rotary file available on the market such as Profile ISO (P) (<https://edgeendo.com/edgefile/>). The author of one study made assumptions that the X7 file exhibited a martensite/austenite composition resulting in the X7 having greater fracture resistance than other tested file systems (Intzes *et al.* 2019). EdgeEndo also claims that their EdgeSequel™ Sapphire (ESS) file is comparable to other marketed "blue" files such as Vortex Blue (VB) but with superior cyclic fatigue results (<https://edgeendo.com/edgesequel/>).

The aim of this study is to analyze the metallurgic composition, phase transformation temperatures and mechanical properties of three heat-treated nickel-titanium (NiTi) endodontic rotary files (X7, ESS, and VB) compared to conventional NiTi rotary files (P). The null hypothesis is that there is no difference in the metallurgic composition, phase profiles and mechanical properties of heat-treated and conventional NiTi endodontic files.

## Materials and methods

### File systems

The conventional NiTi rotary file tested was Profile ISO (P) (Dentsply Sirona, Tulsa, OK, USA). Heat-treated NiTi rotary file systems tested were EdgeFile X7 (X7) (EdgeEndo, Albuquerque, NM, USA), EdgeSequel™ Sapphire (ESS) (EdgeEndo, Albuquerque, NM, USA), and Vortex Blue (VB) (Dentsply Sirona, Tulsa, OK, USA).

### Metallurgic composition

Five files from each system were randomly selected for EDS analysis. Each file was embedded into epoxy resin (EpoxiCure 2, Buehler, Lake Bluff, IL, USA), wet ground with 180-1200 grit size silicon carbide paper (CarbiMet SiC Abrasive Paper, Buehler, Lake Bluff, IL, USA), and polished to 0.05 micron alumina paste (MicroPolish Alumina, Buehler, Lake Bluff, IL, USA) on a polishing machine (Ecomet 6, Buehler, Lake Bluff, IL, USA). Three files from each file set were randomly selected. Each of these three samples were analyzed at the top, middle, and tip of the file's cutting surface to obtain the Ni/Ti mass ratio using the JEOL JSM-IT500 InTouchScope SEM with Live Analysis EDS (JEOL Ltd, Tokyo, Japan) shown in Figure 1.

Figure 1



### Phase transformation temperatures

One file from each system was randomly selected for DSC analysis. Each file was cut into three segments using an orthodontic wire cutter. The segments were weighed and placed into an aluminum crucible. The crucibles were individually placed into the measuring chamber of a DSC823° instrument (Mettler Toledo, Columbus, OH, USA). The samples were cooled to -10°C, heated to 80°C, and cooled to -10°C at a rate of 5°C/min using liquid nitrogen as a cooling gas. Data was analyzed using the STARE software (Mettler Toledo, Columbus, OH, USA).

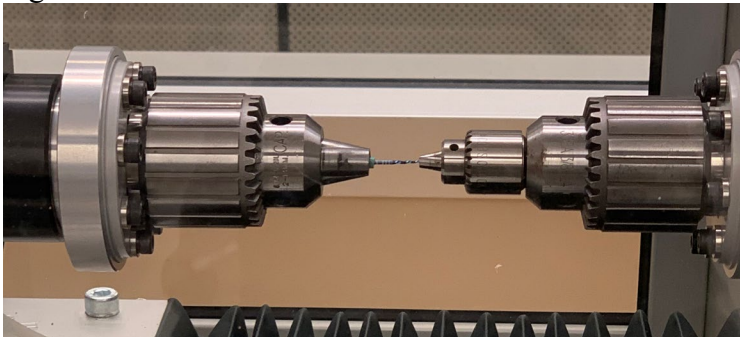
## Nanoindentation

Three files from each system were randomly selected for nanoindentation analysis. Each file was embedded into epoxy resin (EpoxiCure 2, Buehler, Lake Bluff, IL, USA), wet ground with 180-1200 grit size silicon carbide paper (CarbiMet SiC Abrasive Paper, Buehler, Lake Bluff, IL, USA), and polished to 0.05 micron alumina paste (MicroPolish Alumina, Buehler, Lake Bluff, IL, USA) on a polishing machine (Ecomet 6, Buehler, Lake Bluff, IL, USA). Three files from each file set were randomly selected. Each of these three samples were analyzed using nanoindentation (iNano, Nanomechanics, Oak Ridge, TN, USA). A Berkovich tip (#TB26961, Micro Star Technologies, Huntsville, TX, USA) with a 20 nm radius and 65.3° nominal angle was used. The iNano was on a TMC Vibration Control Gimbal Piston table (AMETEK Ultra Precision Technologies, Berwyn, PA, USA). The target load was 10 mN and Poisson's ratio for all specimen was assumed to be 0.33. Data was analyzed with the iNano nanoindent software (iNano, Nanomechanics, Oak Ridge, TN, USA).

## Torsional fatigue

Ten unused files from each file system were randomly selected for torque testing. Each file was placed in a torsion instrument (MTS Bionix Electromechanical Torsion Test System, MTS Systems, Eden Prairie, MN, USA) with the handle of the file and 7mm of the tip locked into place as shown in Figure 2. The files were subjected to torque at a rate of 5° of rotation per second until fracture occurred. Results were analyzed using TestWorks software (MTS TestSuite, MTS Systems, Eden Prairie, MN, USA).

Figure 2



## Results

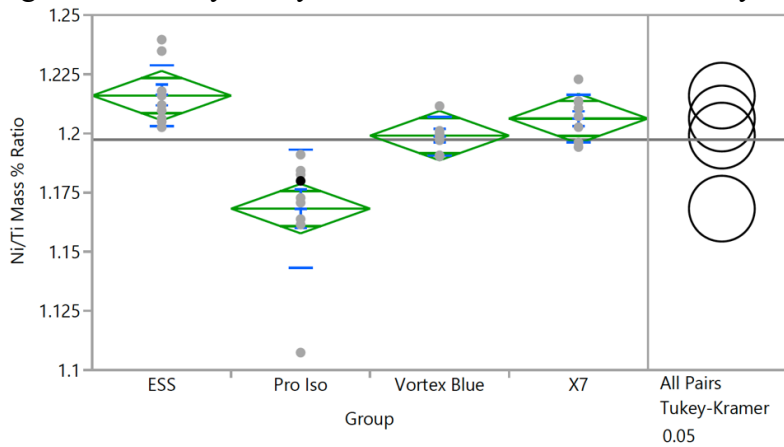
### Metallurgic Composition

The mean Ni/Ti mass ratio of each file system at the top, middle and tip of the cutting portion of the file is shown in Table 1. Figure 3 represents the oneway ANOVA analysis. X7, ESS, and VB had statistically significantly higher Ni/Ti mass ratio than P. The file systems were grouped into two groups according to high (X7, ESS, and VB) or low (P).

Table 1: Ni/Ti Mass Ratio

	Area of File	Ni/Ti Mass Ratio
P	Top	1.179
	Middle	1.181
	Tip	1.144
X7	Top	1.205
	Middle	1.209
	Tip	1.205
VB	Top	1.203
	Middle	1.196
	Tip	1.198
ESS	Top	1.211
	Middle	1.218
	Tip	1.218

Figure 3: Oneway Analysis of Ni/Ti Mass Percent Ratio by Group



### Phase Transformation Temperatures

Figure 4 shows the DSC graphs of each file system. Based on the phase transformation profile of each system, the files were grouped according to the presence (X7, ESS, and VB) or absence (P) of a R-phase. Table 2 shows the phase transformation start and finish temperatures of austenitic, martensitic and R-phases.

Figure 4: DSC Analysis of (a) P (b) X7 (c) VB (d) ESS

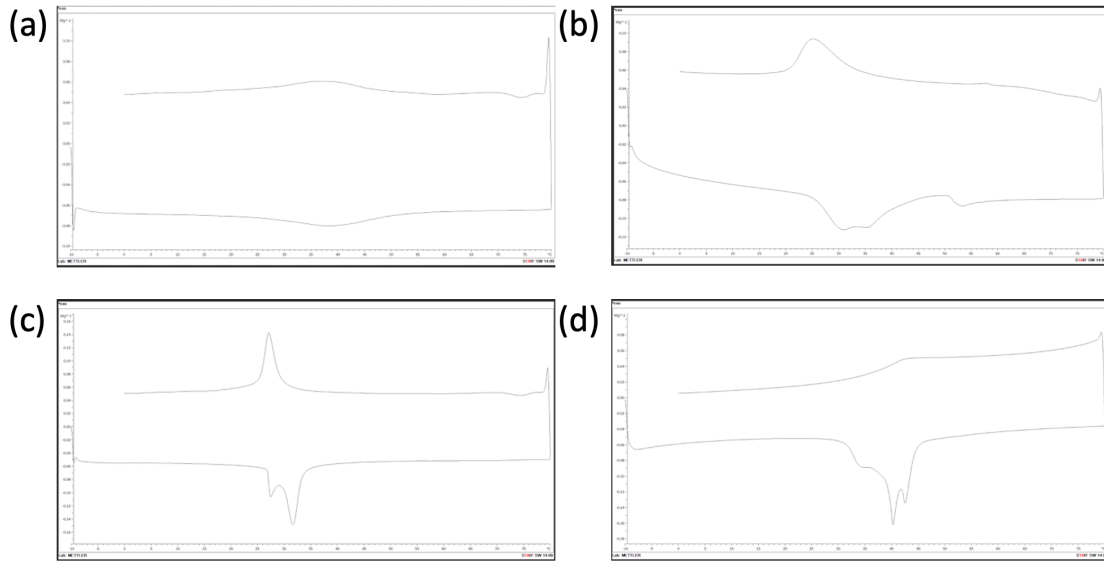


Table 2: Phase Transformation Start and Finish Temperatures

	A <sub>s</sub> (°C)	A <sub>f</sub> (°C)	M <sub>s</sub> (°C)	M <sub>f</sub> (°C)	R <sub>s</sub> (°C)	R <sub>f</sub> (°C)
P	18.18	53.13	52.27	18.79		
X7	50.69	60.83	34.50	20.33	25.09	45.39
VB	29.19	33.98	29.28	25.22	26.41	29.39
ESS	41.77	44.91	56.36	36.09	37.84	41.77

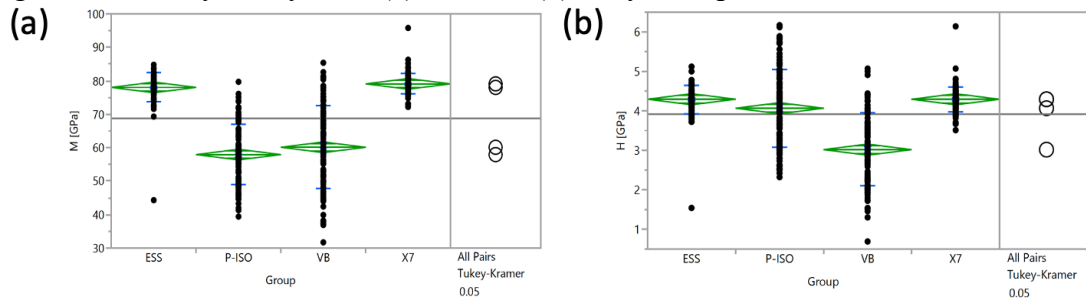
### Nanoindentation

Table 3 shows the mean values and standard deviations of the elastic modulus (EM) and hardness (H) measured in GigaPascals (GPa) from nanoindentation. X7 and ESS had statistically significantly higher EM than VB and P. X7, ESS, and P had significantly higher H than VB. Figure 5 represents the oneway ANOVA of EM and H.

Table 3: Mean Values (std dev) of EM and H from Nanoindentation

	EM (GPa)	H (GPa)
P	57.94 (9.01)	4.07 (0.98)
X7	79.09 (3.07)	4.29 (0.31)
VB	60.12 (12.39)	3.02 (0.92)
ESS	78.03 (4.33)	4.29 (0.37)

Figure 5: Oneway Analysis of (a) EM and (b) H by Group



### Torsional Fatigue

Table 4 shows the mean values and standard deviations of maximum degree of rotation, maximum torque applied, and the degree of rotation at the maximum torque applied before fracture. Figure 6 represents the oneway ANOVA analysis of maximum degree of rotation, maximum torque applied, and the degree of rotation at the maximum torque applied before fracture. X7 exhibited the highest maximum degree of rotation and P had the lowest (Fig 6a). ESS and P had statistically significantly higher maximum torque applied before fracture compared to X7 and VB (Fig 6b). All file systems had statistically significantly different degrees of rotation at the maximum applied torque before fracture; X7 was the highest and P was the lowest (Fig 6c).

Table 4: Mean Values (std dev) of Maximum Degree of Rotation (Max Deg), Maximum Torque (Max T), and Degree of Rotation at Maximum Torque (Deg at Max T) Before Fracture from Torsion Test

	Max Deg (°)	Max T (Nmm)	Deg at Max T(°)
P	238.43 (57.69)	70.87 (6.29)	187.90 (14.07)
X7	819.46 (79.75)	48.72 (6.45)	724.75 (90.89)
VB	461.80 (38.93)	49.72 (12.47)	387.07 (74.04)
ESS	605.01 (79.50)	64.69 (4.60)	533.33 (66.05)

Figure 6: Oneway Analysis of the (a) Maximum Degree of Rotation (b) Maximum Torque Applied and (c) Degree of Rotation at the Maximum Applied Torque Before Fracture by Group

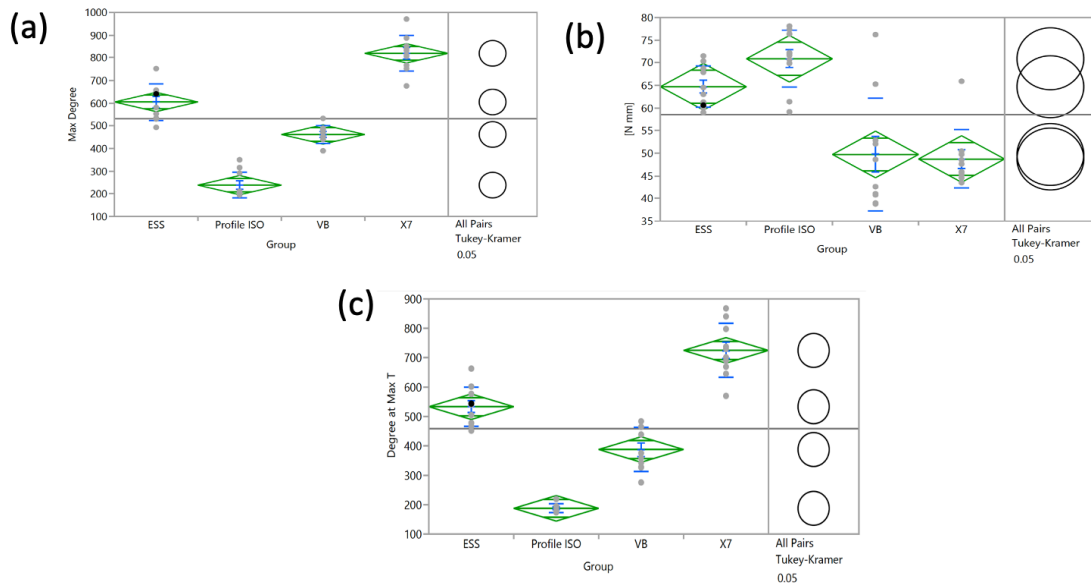


Figure 7 shows the unequal variance t-test comparisons for the maximum degree of rotation before fracture. High Ni/Ti mass ratio, the presence of a R-phase, and a high EM resulted in statistically significantly higher maximum degree of rotation before fracture (Fig 7a,b,c). H had no effect on maximum degree of rotation before fracture (Fig 7d).

Figure 7: Maximum Degree of Rotation by (a) Ni/Ti Mass Ratio (b) Presence of R-Phase (c) EM and (d) H

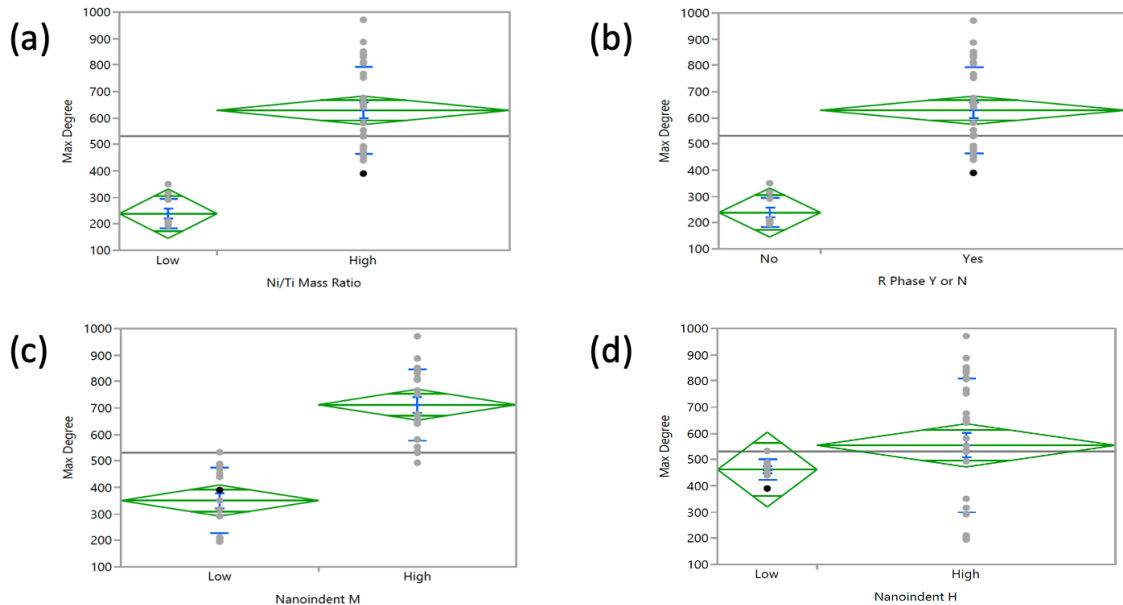


Figure 8 shows the unequal variance t-test comparisons for the maximum torque applied before fracture. Low Ni/Ti mass ratio, the absence of a R-phase, and a high H resulted in statistically significantly higher maximum torque applied before fracture (Fig 8a,b,d). EM had no effect on maximum torque applied before fracture (Fig 8c)

Figure 8: Maximum Torque Applied by (a) Ni/Ti Mass Ratio (b) Presence of R-Phase (c) EM and (d) H

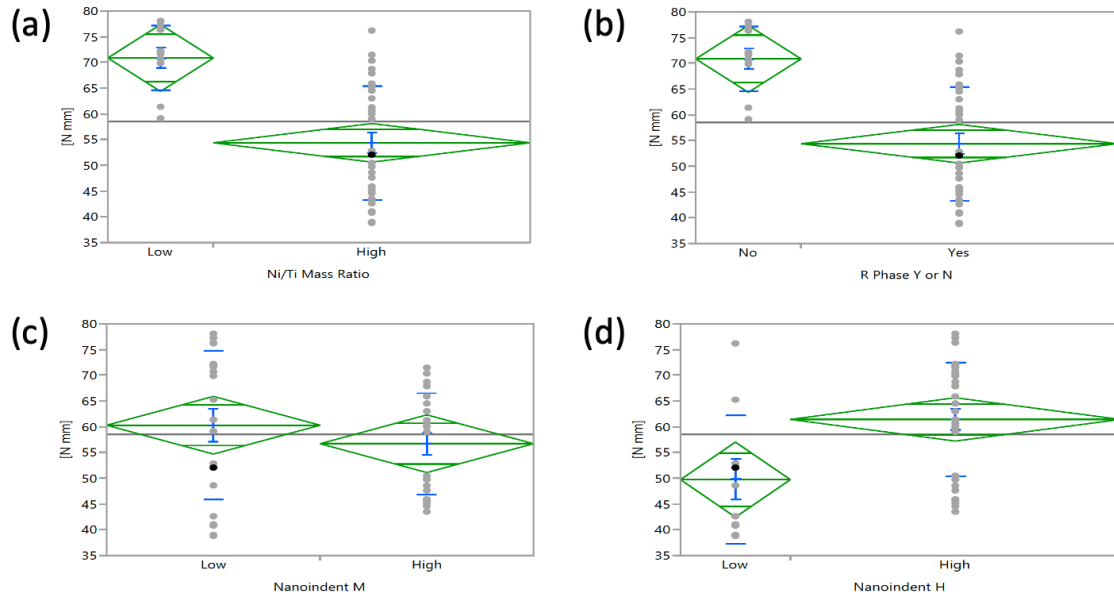
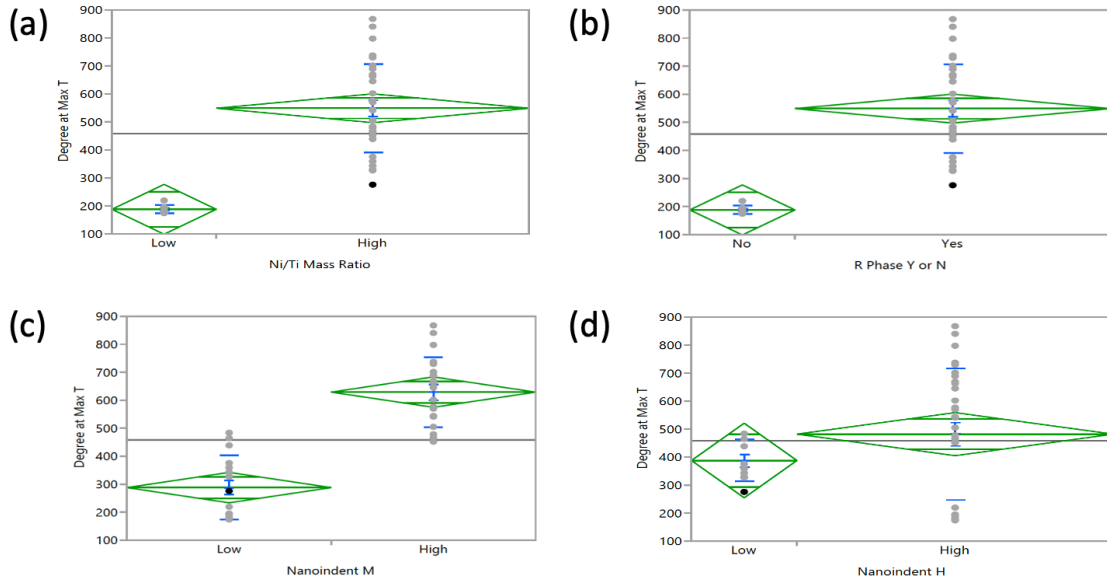


Figure 9 shows the unequal variance t-test comparisons for the degree of rotation at the maximum torque applied before fracture. High Ni/Ti mass ratio, the presence of a R-phase, and a high EM resulted in statistically significantly higher degree of rotation at the maximum torque applied before fracture (Fig 9a,b,c). H had no effect on the degree of rotation at the maximum torque applied before fracture (Fig 9d).

Figure 9: Degree of Rotation at the Maximum Torque Applied by (a) Ni/Ti Mass Ratio (b) Presence of R-Phase (c) EM and (d) H



**Discussion**

Manufacturers are constantly modifying products to have superior properties over competitors. Proprietary heat-treatment of NiTi endodontic rotary files have shown to impact the mechanical properties of these files. This study showed that heat-treated NiTi endodontic files exhibit different metallurgic compositions and phase transformation profiles than conventional NiTi files. Heat-treating NiTi files has a significant impact on some mechanical properties of the files.

This study showed that heat-treated files have a higher Ni/Ti mass ratio than conventional files. The values in Table 1 represent a range of 54-55% weight Ni to 44-45% weight Ti which is in the accepted range found in various other studies (Thompson 2000, Zinelis *et al.* 2010). According to Zinelis *et al.*, higher Ni content correlates to a harder file (Zinelis *et al.* 2010). The results of the present study produced similar findings. X7 and ESS were found to have the highest Ni/Ti mass ratio and the highest H.

An R-phase was present in the heat-treated files in this study. Other studies have shown that VB has an Af temperature of approximately 38°C and Ms temperature of 31°C which is consistent with the results of the present study (Zupanc *et al.* 2018). Another study described the presence of an R-phase resulted in a lower EM and increased flexibility (Shim *et al.* 2017). This is contrary to the present study which resulted in VB having statistically similar EM as P. This previous study also stated that a lower EM was associated with a decrease in maximum torque applied prior to fracture (Shim *et al.* 2017). The present study also contradicted this finding with results of ESS and P being statistically similar with a high maximum torque applied before fracture but statistically different EM. These discrepancies could be due to different testing methods for EM and maximum torque applied before fracture.

Conventional NiTi alloys are primarily austenite and thermomechanically treated alloys are martensite or R-phase at room temperature (Shim *et al.* 2017). Austenitic alloys possess superelasticity and tend to spring-back to their original shape after deformation. These characteristics result in higher torque values before fracture (Zupanc *et al.* 2018). Table 4 of the current study displays P with the greatest torque to fracture, consistent with literature (Zupanc *et al.* 2018). Martensitic alloys are easily deformed and have shape memory. Files in the martensitic phase tend to have lower torque to fracture which can be seen in the results of this study with X7 and VB (Zupanc *et al.* 2018).

In this study, the maximum degree of rotation is associated with the superelasticity of the file. A high Ni/Ti mass ratio, the presence of an R-phase, and a high EM resulted in high superelasticity, representative of X7, ESS, and VB groups. The strength of the file is directly correlated to the maximum torque applied before fracture. A low Ni/Ti mass ratio, absence of an R-phase and high H resulted in higher file strength, representative of P group. The degree of rotation at maximum torque applied prior to fracture from this study correlates with the strain of the file just prior to fracture. A high Ni/Ti mass ratio, the presence of an R-phase, and a high EM resulted in higher strain prior to fracture, representative of X7, ESS, and VB groups.

One limitation to this study is a small sample size in all testing groups, particularly the DSC analysis due to equipment failure. Torsion fatigue can be tested in several different ways; therefore, future studies could consider alternative torsion testing for comparison. In order to further correlate the results of this study to clinical application, cyclic fatigue testing should be done with these file systems. Those cyclic fatigue results could be compared to the findings of this study in regard to the metallurgic composition, phase transformation profiles and mechanical properties. Statistical analysis of the combined results could credit or discredit EdgeEndo's claim of offering cheaper files with superior performance.

## **Conclusion**

The null hypothesis was rejected in this study. Heat-treated NiTi endodontic files exhibited different metallurgic compositions and phase transformation profiles than conventional NiTi files. Heat-treating these files does have a significant impact on some mechanical properties of the files. The properties exhibited from heat treatment allow for the files to be more flexible which would create a more favorable outcome in endodontic treatment of curved canal systems. Knowledge of the types of NiTi endodontic file systems available could lead to more predictable and successful clinical outcomes.

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## Conflict of Interest

The authors declare that there are no conflicts of interest regarding the publication of this article.

## References

- Aminsobhani M, Khalatbari MS, Meraji N, Ghorbanzadeh A, Sadri E (2016) Evaluation of the fractured surface of five endodontic rotary instruments: a metallurgical study. *Iranian Endodontic Journal* **11**, 286-92.
- Anusavice KJ, Phillips RW, Shen C, Rawls HR (2013) *Phillips' science of dental materials*. 6<sup>th</sup> edn; pp. 48-68. St. Louis, Mo: Elsevier/Saunders.
- Arias A, Hejlawy S, Murphy S, de la Macorra JC, Govindjee S, Peters OA (2019) Variable impact by ambient temperature on fatigue resistance of heat-treated nickel titanium instruments. *Clinical Oral Investigations* **23**, 1101-8.
- Intzes L, Tzima ZD, Gogos C (2019) Comparative evaluation of resistance to cyclic fatigue of three rotary endodontic Ni-Ti instruments. *Balkan Journal of Dental Medicine* **23**, 36-9.
- Jamleh A, Yahata Y, Ebihara A, Atmeh AR, Bakhsh T, Suda H (2016) Performance of NiTi endodontic instrument under different temperatures. *Odontology* **104**, 324-8.
- Kan Q, Yan W, Kang G, Sun Q (2013) Oliver-Pharr indentation method in determining elastic moduli of shape memory alloys – a phase transformable material. *Journal of the Mechanics and Physics of Solids* **61**, 2015-33.
- Oliver WC, Pharr GM (1992) An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *Journal of Materials Research* **7**, 1564-83.
- Oliver WC, Pharr GM (2003) Measurement of hardness and elastic modulus by instrumented indentation: advances in understanding and refinements to methodology. *Journal of Materials Research* **19**, 3-20.
- Shen Y, Zhou H, Zheng Y, Peng B, Haapasalo M (2013) Current challenges and concepts of the thermomechanical treatment of nickel-titanium instruments. *Journal of Endodontics* **39**, 163-72.
- Shim KS, Oh S, Kum KY, Kim YC, Jee KK, Chang SW (2017) Mechanical and metallurgical properties of various nickel-titanium rotary instruments. *BioMed Research International* **2017**, 1-13
- Thompson S (2000) An overview of nickel-titanium alloys used in dentistry. *International Endodontic Journal* **33**, 297-310.
- Walia H, Brantley W, Gerstein H (1988) An initial investigation of the bending and torsional properties of Nitinol root canal files. *Journal of Endodontics* **14**, 346-51.
- Yahata Y, Yoneyama T, Hayashi Y, et al. (2009) Effect of heat treatment on transformation temperatures and bending properties of nickel-titanium endodontic instruments. *International Endodontic Journal* **42**, 621-6.
- Zinelis S, Eliades T, Eliades G (2010) A metallurgical characterization of ten endodontic Ni-Ti instruments: assessing the clinical relevance of shape memory and superelastic properties of Ni-Ti endodontic instruments. *International Endodontic Journal* **43**, 125-34.

Zupanc J, Vahdat-Pajouh N, Shafer E (2018) New thermomechanically treated NiTi alloys – a review *International Endodontic Journal* **51**, 1088-103.