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**The Effects of Autoclave Sterilization on the  
Cyclic Fatigue Resistance of Various Heat-  
Treated Nickel-Titanium Instruments Under  
Varied Kinematic Conditions**

Sultan Saleh Al Saif

The Graduate School  
Yonsei University  
Department of Dental Science

**The Effects of Autoclave Sterilization on the  
Cyclic Fatigue Resistance of Various Heat-  
Treated Nickel-Titanium Instruments by  
Under Varied Kinematic Conditions**

A Master's Thesis

Submitted to the Department of Dentistry

And the Graduate School of Yonsei University

In partial fulfillment of the

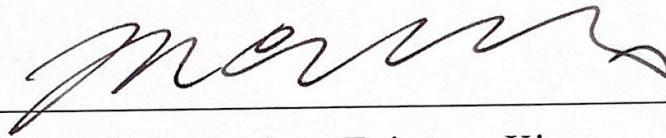
Requirements for the degree of

Master of Dental Science

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December 2022

This certifies that the Master's Thesis  
of Sultan Al Saif is approved



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Yonsei University

December 2022

## Dedication

To my beloved parents, who overwhelmed me with love and the sacrifices and who have provided me with confidence and support throughout of my life.

To my beloved wife and my adorable sons, "fruit of my heart and hope of future (Asser & Obay)", whose love gave to me the strength to achieve my goal.

To my dearest brothers and sisters, who encouraged me until the completion of my scientific trip.

To my dearest professors, friends and colleagues.

## Acknowledgments

My utmost gratitude goes first to Allah the most merciful for his help and guidance all through my life.

I would also like to express my deepest appreciation and thanks to my mother, my late father, may Allah have mercy on him and all my family members, especially, my wife, brothers, sisters and my little sons. And special thanks to my country's government, all my professors, friends and colleagues.

Sincerely grateful to my supervisor professor Euseong Kim for his continuous support and valuable recommendations since the beginning of my residency training program and master's degree on conservative dentistry specialty and I express many thanks to all my professors in Conservative Dentistry Department at Yonsei University for their supervision and continuous support. And I would like to thank all my colleagues and friends during my Specialty Residency Program and Master's degree.

December 2022

Sultan Saleh Al Saif

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## Abstract

# **The Effects of Autoclave Sterilization on the Cyclic Fatigue Resistance of Various Heat-Treated Nickel-Titanium Instruments Under Varied Kinematic Conditions**

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This study aimed to evaluate and compare the effects of autoclave sterilization on static and dynamic cyclic fatigue tests of various heat-treated Ni-Ti instruments in single artificial stainless-steel canals. Three brands of heat-treated Ni-Ti rotary instruments were used for this study: TruNatomy (TRN; prime, 26/.04 taper), WaveOne Gold (WOG; primary, 25/.07 taper), and ProTaper Gold (PTG; F2, 25/.08 taper). All files used in this study were 25 mm long. A total of 144 instruments for each system were divided into two groups (n = 72 each)

according to the cyclic fatigue testing modes (static mode and dynamic mode). Each group was subdivided into three subgroups ( $n = 24$  each) according to sterilization condition (no-sterilization [NSC], five-cycle-sterilization [5SC], and ten-cycle-sterilization [10SC] subgroups). All the static and dynamic cyclic fatigue resistance tests were performed in two customized devices (AendoS; DMJ System, Busan, Korea). In static mode, there were no statistically significant differences in the mean numbers of cycles to fracture (NCFs) between the three subgroups with either WOG or TRN. However, with PTG, there were significant differences between the mean NCFs of the sterilization-condition subgroups. In dynamic mode with WOG, there were no statistically significant differences between the mean NCFs of the three subgroups. However, there was a significant difference between the mean NCFs of the NSC and 10SC subgroups with TRN and PTG. Between TRN, WOG, and PTG, there were no significant differences in mean file fragment length, regardless of sterilization condition, in both static and dynamic modes. The study findings revealed that, compared with static mode, cyclic fatigue resistance was significantly higher in dynamic mode for all investigated Ni-Ti instrument systems. Within the limitations of this study, it can be concluded PTG autoclaved ten times had higher cyclic fatigue resistance than unsterilized instruments in tests conducted in both static and dynamic modes. However, TRN autoclaved ten times had lower cyclic fatigue resistance than unsterilized instruments. With WOG, there were no significant differences between any of the sterilization-condition subgroups in either static or dynamic mode.

# **The Effects of Autoclave Sterilization on The Cyclic Fatigue Resistance of Various Heat-Treated Nickel-Titanium Instruments Under Varied Kinematic Conditions**

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The Graduate School, Yonsei University

(Directed by Professor Euseong Kim, D.D.S., M.S.D, Ph.D.)

## **I. Introduction**

Nickel-titanium (Ni-Ti) alloy, also known as nitinol, was initially used in endodontics 30 years ago when Walia et al. used Ni-Ti archwire to make a root canal file in 1988 (Walia et al. 1988). The Ni-Ti alloy used in endodontic instruments has an atomic ratio of almost one to one (equiatomic) and contains approximately 56% Ni and 44% Ti (Thompson 2000).

There are two types of crystalline structures that change depending on temperature. The phases of change are termed austenite and martensite phases. In the austenitic phase, the Ni-Ti alloy is inflexible and hard, and it has remarkable super plasticity . On the other hand, in the martensitic phase, the Ni-Ti alloy is soft, easily deformable , flexible and has shape memory. Ni-Ti endodontic files have become popular due to their superior elasticity, cyclic fatigue resistance, and torsional resistance compared with conventional stainless-steel files (Shen et al. 2013).

Ni-Ti endodontic files provide several benefits over conventional stainless-steel files for use in endodontic clinical procedures, such as reduced preparation time, improved cutting efficiency, and better canal centering (Es-Souni et al. 2001). Despite these benefits, Ni-Ti instruments are prone to fracture or deformation, which may negatively impact treatment outcomes (Peters et al. 2001). Fracturing of rotary Ni-Ti instruments happens because of torsional or cyclic fatigue. Torsional fractures occur when the tip or another component of the instrument becomes entangled in a canal while the shank continues to spin. A cyclic fatigue fracture takes place when the file does not bind but instead freely spins in a curve. This causes tension and compression cycles at the point of extreme flexure, which eventually leads to fracturing (Sattapan et al. 2000).

Endodontic instruments made of Ni-Ti alloys can be classified as either having a martensitic phase (Blue and Gold heat-treated Ni-Ti, CM-Wire) and those that primarily contain the austenitic phase (conventional Ni-Ti, M-Wire, R Phase) (Shen et al. 2013). Dentsply Tulsa Dental Specialist introduced the M-Wire file in 2007. During the

production procedure, it goes through a series of heating and cooling cycles, and this cycling mechanism helps to keep nitinol's crystalline structure in its more martensitic state at body temperature. M-Wire contains the austenite, martensite, and R phases. M-Wire has an austenite finish temperature of 45 to 50 °C at room temperature; the temperature range for the phase transition indicates that M-Wire instruments are martensitic (Christia et al. 2017). In 2008, SybronEndo created the first fluted Ni-Ti file using a plastic deformation process similar to that used in the production of most stainless-steel K-files and reamers. This new manufacturing process aimed to convert raw Ni-Ti wire from the austenitic phase to the R phase and stabilize it at higher temperatures. The R phase has a lower shear modulus than martensite and austenite, and it is entirely austenite at body temperature (Haapasalo & Shen 2013, Christia et al. 2017). CM-Wire (controlled-memory Ni-Ti wire) was first used in 2010. The thermomechanical manufacturing method aimed to improve stability, raise transformation temperatures to around 50 °C, reduce shape memory, and achieve stable martensite at body temperature (Plotino et al. 2011). Tulsa Denta introduced the first blue-colored endodontic instrument, Vortex Blue, in 2011. There are two gold-heated Ni-Ti systems (ProTaper Gold [PTG] and WaveOne Gold [WOG]) and two blue-heated Ni-Ti systems (ProTaper Blue and WaveOne Blue) (Vortex Blue and Reciproc Blue). These instruments are deformable and have a controlled memory effect. The primary distinction between CM-Wire and heat-treated gold and blue instruments is that these files are ground before being heat-treated following machining. Gold and blue heat-treated files have improved flexibility and fatigue resistance relative to standard Ni-Ti and M-Wire

instruments, which could be attributed to their martensitic state (Blum et al. 1999, Pereira et al. 2012, Zupanc et al. 2018). MaxWire (martensite-austenite-electropolish-flex), a new thermomechanically treated Ni-Ti alloy, was recently introduced by FKG Dentaire. It has shape memory and superelasticity in clinical use. The XP-endo Shaper and XP-endo Finisher exemplify these properties. At room temperature, these instruments are straight, but when subjected to intracanal temperature, they curve due to a phase change to an austenitic state (Zupanc et al. 2018).

The mechanical characteristics of Ni-Ti instruments are heavily impacted by phase transition behavior, which is easily changed by parameter variations, such as in terms of heat treatment, chemical composition, and manufacturing procedure. One of the most essential techniques for altering the transition temperatures of Ni-Ti alloys and influencing the fatigue resistance of Ni-Ti endodontic files is heat treatment (thermal processing) (Yahata et al. 2014). Previous studies have found an inverse association between the amount of metal in the file cross section and cyclic fatigue, and it has been proposed that thermal and surface treatments of the Ni-Ti alloy during manufacturing enhance cycle fatigue resistance. The instrument's flexibility may be enhanced by phase changes associated with heat treatment, which can improve the metallurgical characteristics of Ni-Ti files by enhancing cyclic fatigue resistance (Yahata et al. 2009, Zinelis et al. 2010, Plotino et al. 2010, Capar et al. 2017). De Vasconcelos et al. (2016) discovered that increasing the martensitic content of a Ni-Ti alloy made an instrument more flexible and resistant to fatigue. This led to an improvement in the crystal structure arrangement of the

alloy as well as a change in the relative proportion of phases that were present in the alloy (De Vasconcelos et al. 2016). Electropolishing is an electrochemical procedure that removes surface imperfections and microcracks that occur during the grinding process. This improves the instrument's fracture resistance and corrosion resistance (Mohammadi et al. 2014).

Different methods can be used to evaluate the cyclic fatigue resistance of Ni-Ti files. A cyclic fatigue test can be performed to assess Ni-Ti file fracture resistance by using either a static or dynamic mode, but typically a static mode is used under well-controlled experimental conditions. The compression and tensile loads are concentrated in a specific location of the instrument in static modes. In dynamic modes, however, these pressures are dispersed over a larger area along the instrument shaft (Larsen et al. 2009, Al Shwaimi 2018, Tanomaru-Filho et al. 2018, El Feky et al. 2019). Li et al. (2002) investigated the static and dynamic cyclic fatigue resistance of ProFile (size 25/.04 taper) instruments with 1- to 3-mm vertical amplitudes. They conclude that the time of cycle to fracture in dynamic mode was approximately 20% to 40% longer than in static mode (Li et al. 2002). Many studies have confirmed this finding of a long time of cycle to fracture in dynamic mode relative to static mode (Rodrigues et al. 2011, Lopes et al. 2013). During cyclic fatigue resistance testing in dynamic mode, the phase transformation of Ni-Ti alloys spreads along the instrument and prevents microcrack formation at a specific place of the instrument, resulting in a longer lifespan of Ni-Ti instruments (Zupanc et al. 2018). Dynamic movement extended the lifespan of endodontic rotary instruments. In the static mode, the

alternating tensile and compressive stresses are directed at the same zone of the endodontic instrument without any axial movement. The stresses are cumulative and generate microstructural variations in the metallic type of the alloys used in the file. (Li et al. 2002 , Ray et al. 2007, and Hulsmann et al. 2019)

Endodontic instruments must be cleansed and sterilized before use since direct contact with blood, saliva, and necrotic pulp tissues can cause cross-infection between patients. Sterilization refers to the elimination of all bacteria and spores, and autoclave sterilization is widely considered the most effective form of sterilization (Huang et al. 2011). Given that thermal treatment has a major impact on the mechanical properties of Ni-Ti instruments, the additional heat that files are subjected to during autoclave sterilization procedures may affect the mechanical characteristics of these instruments. Yared et al. (2002) found that rotary files can be used safely in up to ten curved canals. According to a recent systematic review, the cyclic fatigue resistance of heat-treated Ni-Ti instruments seems to be influenced by autoclave sterilization procedures (Emmanuel et al. 2020). Zhao et al. (2016) found that although thermally treated HyFlex CM, Twisted File, and K3XF instruments were shown to be more resistant to cyclic fatigue fracture than Race and K3 instruments, autoclaved HyFlex CM and K3XF instruments were found to be more resistant than Twisted File.

Gold heat-treated instruments include PTG (Dentsply Sirona, Ballaigues, Switzerland) and WOG (Dentsply Sirona). PTG files are progressively tapered sequential rotary systems with a convex triangular cross section. According to the manufacturer, PTG instruments

are identical to ProTaper Universal in geometry but have undergone an unique thermal treatment to increase their flexibility and resistance to cycle fatigue(Hieawy et al. 2015, Gagliardi et al. 2015). WOG files are modified WaveOne single-file reciprocating systems. A novel parallelogram cross-sectional design with two cutting edges and gold heat treatment enhance WOG systems (Özyürek et al. 2016, Capar et al. 2017).

TruNatomy (TRN) instruments (Dentsply Sirona) are a new type of heat-treated Ni-Ti instrument with a distinctive design. The slim Ni-Ti wire design is 0.8 mm instead of up to 1.2 mm, which is characteristic of most other variable tapered instruments. The cross-sectional design of the TRN instruments is an off-centered parallelogram, and they are made using a specific Ni-Ti heat-treated wire that, according to the manufacturer, increases the flexibility of the instrument. Because of the instrument geometry, regressive tapers, slim design, and heat treatment of the Ni-Ti alloy, it has been claimed that TRN instruments protect the structural integrity of the tooth and dentinen(van der Vyver et al. 2019, Dentsply Sirona TruNatomy Brochure 2019).

Few published studies have evaluated the cyclic fatigue resistance of heat-treated Ni-Ti instruments after repeated cycles of autoclave sterilization in both static and dynamic modes. Therefore, this study aimed to evaluate and compare the effects of autoclave sterilization on static and dynamic cyclic fatigue tests of various heat-treated Ni-Ti instruments in single artificial stainless-steel canals and to measure broken file fragment length and determine the location of fracture point either below , above or at curvature area.

## II. Material & Methods

### 1. Study design

Three brands of heat-treated Ni-Ti rotary instruments were selected for this study: TRN (prime, 26/.04 taper), WOG (primary, 25/.07 taper), and PTG (F2, 25/.08 taper). All files used in this study were 25 mm long (Figure 1).

A total of 144 instruments for each system were divided into two groups ( $n = 72$  per group) according to the cyclic fatigue testing modes (static mode and dynamic mode). Then, each group was subdivided into three subgroups ( $n = 24$  per subgroup) characterized by their sterilization conditions: the no-sterilization (NSC), five-cycle-sterilization (5SC), and ten-cycle-sterilization (10SC) subgroups. The sterilized instruments were subjected to autoclave sterilization (five and ten cycles) at a temperature of 134 °C for 30 minutes for each cycle (15 minutes for sterilization and 15 minutes for drying) (HS-4085, Hanshin Medical Co., Ltd.).

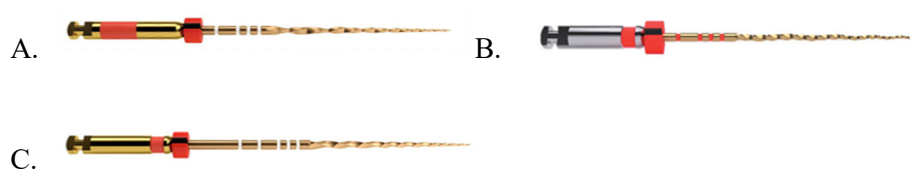


Figure 1. Ni-Ti rotary instruments used in this study

(A) WaveOne Gold (primary), (B) TruNatomy (prime), (C) ProTaper Gold (F2)

## 2.Cyclic Fatigue Resistance

Two customized devices (AendoS; DMJ System, Busan, Korea) were used to conduct all of the static and dynamic cycle fatigue resistance testing (Figure 2). All instruments were subjected to cyclic fatigue testing using artificial custom-made stainless-steel canal blocks, each with a single curvature of 60°, inner diameter of 1.5 mm, and tip diameters with 1-mm and 5-mm radii (Figure 3). An artificial canal block was prepared before each test to reduce friction between the files and the canal walls of the metal block by applying synthetic oil (WD-40 Company, San Diego, CA, USA). There were three types of Ni-Ti file systems evaluated according to the manufacturer's instructions. TRN and PTG filing were conducted using continuous rotation (500 and 300 rpm, respectively). WOG filing was conducted using reciprocation rotation (350 rpm, 150° counterclockwise, 30° clockwise). The dynamic cyclic fatigue resistance testing was conducted with 4-mm up-and-down oscillation motion, 8 mm/s speed, and 50 ms of dwell time. The time to fracture was measured visually and audibly by video recording. All tests were conducted at room temperature. The following formula was used to determine the number of cycles to fracture (NCF):

$$\text{NCF} = \text{revolutions per minute (rpm)} \times \text{time to fracture (s)} / 60$$

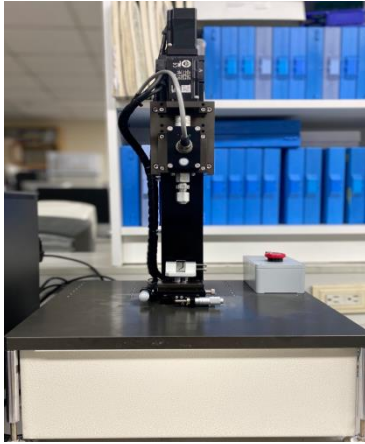


Figure 2. Customized device (AEndoS; DMJ System, Busan, Republic of Korea)



Figure 3. Artificial custom-made, stainless-steel canal with single curvature

### 3. Scanning Electron Microscope Examination

Scanning electron microscopy was used to examine and identify the topographic characteristics of the file fracture surfaces during the cyclic fatigue resistance tests for the three heat-treated Ni-Ti instruments after ten cycles of sterilization in both static and dynamic modes (S-3000N; Hitachi High Technologies, Tokyo, Japan).

#### **4. Statistical Analysis**

The numbers of cycles to fracture (NCFs) and file fragment lengths (FLs) associated with the three Ni-Ti instruments were compared using one-way ANOVA including all subgroups. Post hoc Tukey test analysis was performed for subgroup comparisons of both NCF and FL data. Statistical significance was established at a confidence level of 95%. All of the statistical analyses were performed using GraphPad Prism, version 8.0.2 for Mac (GraphPad Software, San Diego, CA, USA). In all experiments, a P value of less than 0.05 was considered statistically significant.

### III. Results

#### 1. Effects of Autoclave Sterilization on the Cyclic Fatigue Resistance of Three Heat-Treated Ni-Ti Instruments in Static Mode

The NCF means and standard deviations associated with the WOG, PTG, and TRN instruments in all three subgroups (according to sterilization condition) in static mode are shown in Table 1.

With WOG and TRN, there were no statistically significant differences between the mean NCFs of the three sterilization-condition subgroups. However, with PTG, the subgroups varied significantly in terms of their NCF values. The NCF means and standard deviations with PTG were as follows: NSC subgroup,  $522 \pm 18.09$ ; 5SC subgroup,  $599.5 \pm 64.54$ ; and 10SC subgroup,  $630.5 \pm 46.41$  ( $P = 0.0035$ ). The bar chart in Figure 4 shows the mean NCFs associated with the three heat-treated Ni-Ti instruments (categorized in sterilization-condition subgroups) in static mode.

The FL means and standard deviations associated with the WOG, PTG, and TRN instruments in all sterilization-condition subgroups in static mode are shown in Table 2.

There was no statistically significant difference in mean FLs between the three sterilization-condition subgroups in association with all three Ni-Ti heat-treated instruments. The bar chart in Figure 5 shows the mean FLs associated with the three heat-treated Ni-Ti instruments (categorized by sterilization-condition subgroups) in static mode.

Table 1. The means and standard deviations of NCF for three heat-treated Ni-Ti instruments in static mode.

Static Mode	Sterilization Condition			P Value
	No Sterilization	Five Cycles of Sterilization	Ten Cycles of Sterilization	
	NCF	NCF	NCF	
	MEAN $\pm$ SD	MEAN $\pm$ SD	MEAN $\pm$ SD	
WaveOne Gold	2733.2 $\pm$ 425.2	2749.25 $\pm$ 270.94	2997.75 $\pm$ 336.13	NS 0.3766
ProTaper Gold	522 $\pm$ 18.09	599.5 $\pm$ 64.54	630.5 $\pm$ 46.41	** 0.0035
TruNatomy	888.3 $\pm$ 179.79	876.67 $\pm$ 74.6	730.83 $\pm$ 54.16	NS 0.0683

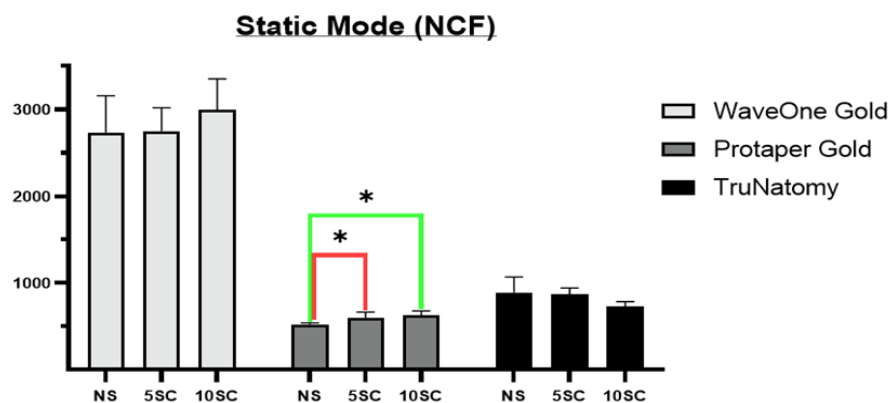


Figure 4. The mean numbers of cycles to fracture (NCFs) for WOG, PTG, and TRN in all sterilization-condition subgroups in static mode.

The horizontal axis (x-axis) represents the types of Ni-Ti heat-treated instruments with sterilization conditions, and the vertical axis (y-axis) represents the number of cycles to fracture.

Table 2. The means and standard deviations of FL for three heat-treated Ni-Ti instruments in static mode.

Static Mode	Sterilization Condition			P Value
	No Sterilization	Five Cycles of Sterilization	Ten Cycles of Sterilization	
	FL	FL	FL	
	MEAN $\pm$ SD	MEAN $\pm$ SD	MEAN $\pm$ SD	
WaveOne	5.43 $\pm$ 0.90	4.43 $\pm$ 0.67	5.06 $\pm$ 0.82	NS
Gold	(=)	(-)	(=)	0.9847
ProTaper	6.62 $\pm$ 0.69	6.92 $\pm$ 0.17	6.78 $\pm$ 0.3	NS
Gold	(+)	(+)	(+)	0.9997
TruNatomy	5 $\pm$ 0.46	5.5 $\pm$ 0.70	5.31 $\pm$ 0.53	NS
	(=)	(=)	(=)	0.9938

Marker (-) meaning the fracture point below curvature area, Marker (=) meaning the fracture point at curvature area and Marker (+) meaning the fracture point above curvature area.

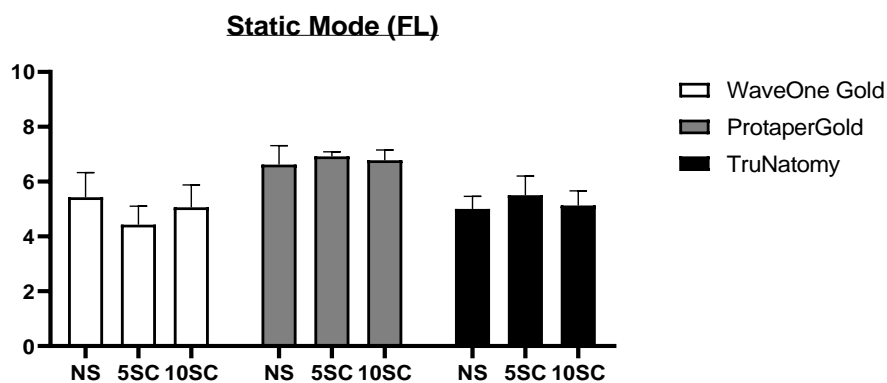


Figure 5. The mean file fragment lengths (FLs) for WOG, PTG, and TRN in all sterilization-condition subgroups in static mode. The horizontal axis (x-axis) represents the types of Ni-Ti heat-treated instruments with sterilization conditions, and the vertical axis (y-axis) represents the file fragment length.

## **2. Effects of Autoclave Sterilization on the Cyclic Fatigue Resistance for Three Heat-Treated Ni-Ti Instruments in Dynamic Mode**

The NCF means and standard deviations for WOG, PTG, and TRN in all three sterilization-condition subgroups in dynamic mode are shown in Table 3. There was no statistically significant difference in mean NCF between the three sterilization-condition subgroups in association with WOG. However, PTG and TRN were significantly different in terms of the mean NCFs of the NSC vs. 10SC subgroups. The NCF means and standard deviations for PTG were as follows: NSC subgroup,  $1367.5 \pm 101.76$ ; and 10SC subgroup,  $1785 \pm 293.50$  ( $P = 0.0302$ ). The NCF means and standard deviations for TRN were as follows: NSC subgroup,  $2076.67 \pm 411.4$ ; and 10SC subgroup,  $1220 \pm 307.06$  ( $P = 0.0031$ ). The bar chart in Figure 6 shows the mean NCFs for the three heat-treated Ni-Ti instruments (categorized in sterilization-condition subgroups) in dynamic mode.

The FL means and standard deviations for WOG, PTG, and TRN in all sterilization-condition subgroups in dynamic mode are shown in Table 4. There were no statistically significant differences in FL between the three sterilization-condition subgroups in association with all three Ni-Ti heat-treated instruments. The bar chart in Figure 7 shows the mean FL findings for the three heat-treated Ni-Ti instruments (categorized in sterilization-condition subgroups) in dynamic mode.

Table 3. The means and standard deviations of NCF for three heat-treated Ni-Ti instruments in dynamic mode

Dynamic Mode	Sterilization Condition			P Value
	No Sterilization	Five Cycles of Sterilization	Ten Cycles of Sterilization	
	NCF	NCF	NCF	
	MEAN $\pm$ SD	MEAN $\pm$ SD	MEAN $\pm$ SD	
WaveOne Gold	2283.17 $\pm$ 275.3	2222.5 $\pm$ 185.6	2325.75 $\pm$ 160.04	NS
ProTaper Gold	1367.5 $\pm$ 101.76	1631.5 $\pm$ 288.74	1785 $\pm$ 293.50	* 0.0302
TruNatomy	2076.67 $\pm$ 411.4	1595.83 $\pm$ 343.4	1220 $\pm$ 307.06	** 0.0031

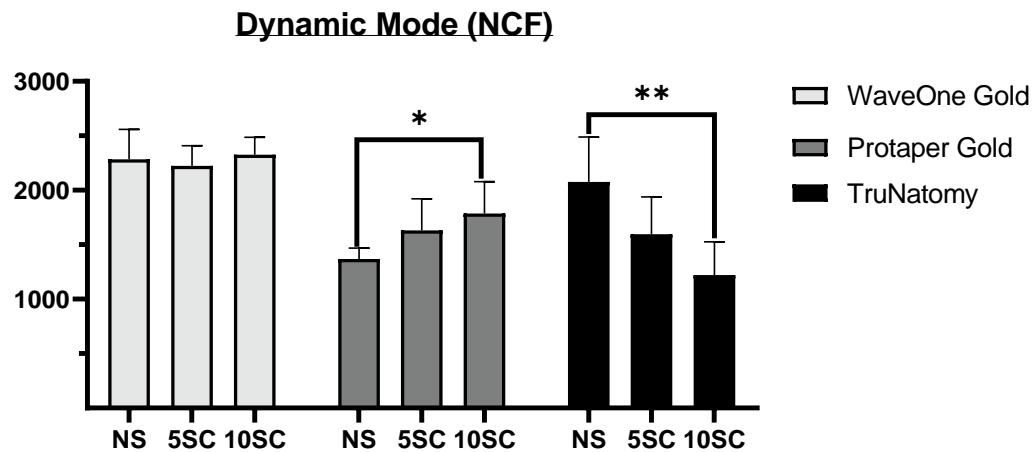


Figure 6. The number of cycles to fracture (NCFs) for WOG, PTG, and TRN in all sterilization-condition subgroups in dynamic mode

The horizontal axis (x-axis) represents the types of Ni-Ti rotary instruments with sterilization conditions, and the vertical axis (y-axis) represents the number of NCFs.

Table 4. The means and standard deviations of FL for three heat-treated Ni-Ti instruments in dynamic mode

Dynamic Mode	Sterilization Condition			P Value
	No Sterilization	Five Cycles of Sterilization	Ten Cycles of Sterilization	
	FL	FL	FL	
	MEAN $\pm$ SD	MEAN $\pm$ SD	MEAN $\pm$ SD	
WaveOne Gold	3.68 $\pm$ 1.16	2.75 $\pm$ 1.03	3.5 $\pm$ 1.06	NS
	(-)	(-)	(-)	0.9428
ProTaper Gold	4.5 $\pm$ 1.25	3.18 $\pm$ 0.88	4.75 $\pm$ 1.6	NS
	(-)	(-)	(-)	0.8513
TruNatomy	4 $\pm$ 0.26	3.93 $\pm$ 0.32	4.18 $\pm$ 0.59	NS
	(-)	(-)	(-)	0.9933

Marker (-) meaning the fracture point below curvature area, Marker (=) meaning the fracture point at curvature area and Marker (+) meaning the fracture point above curvature area.

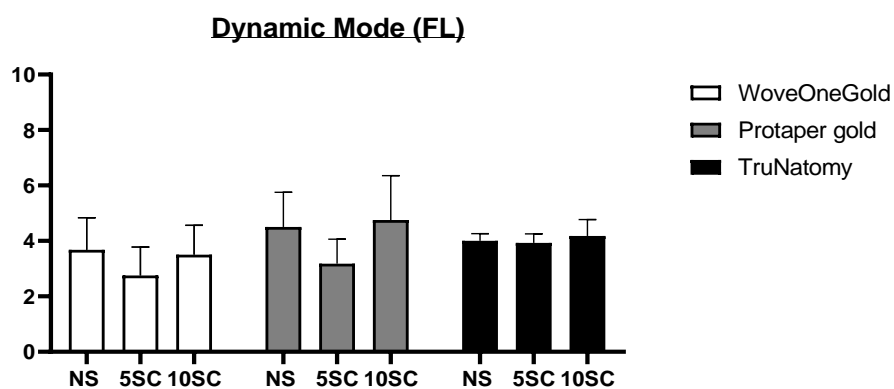
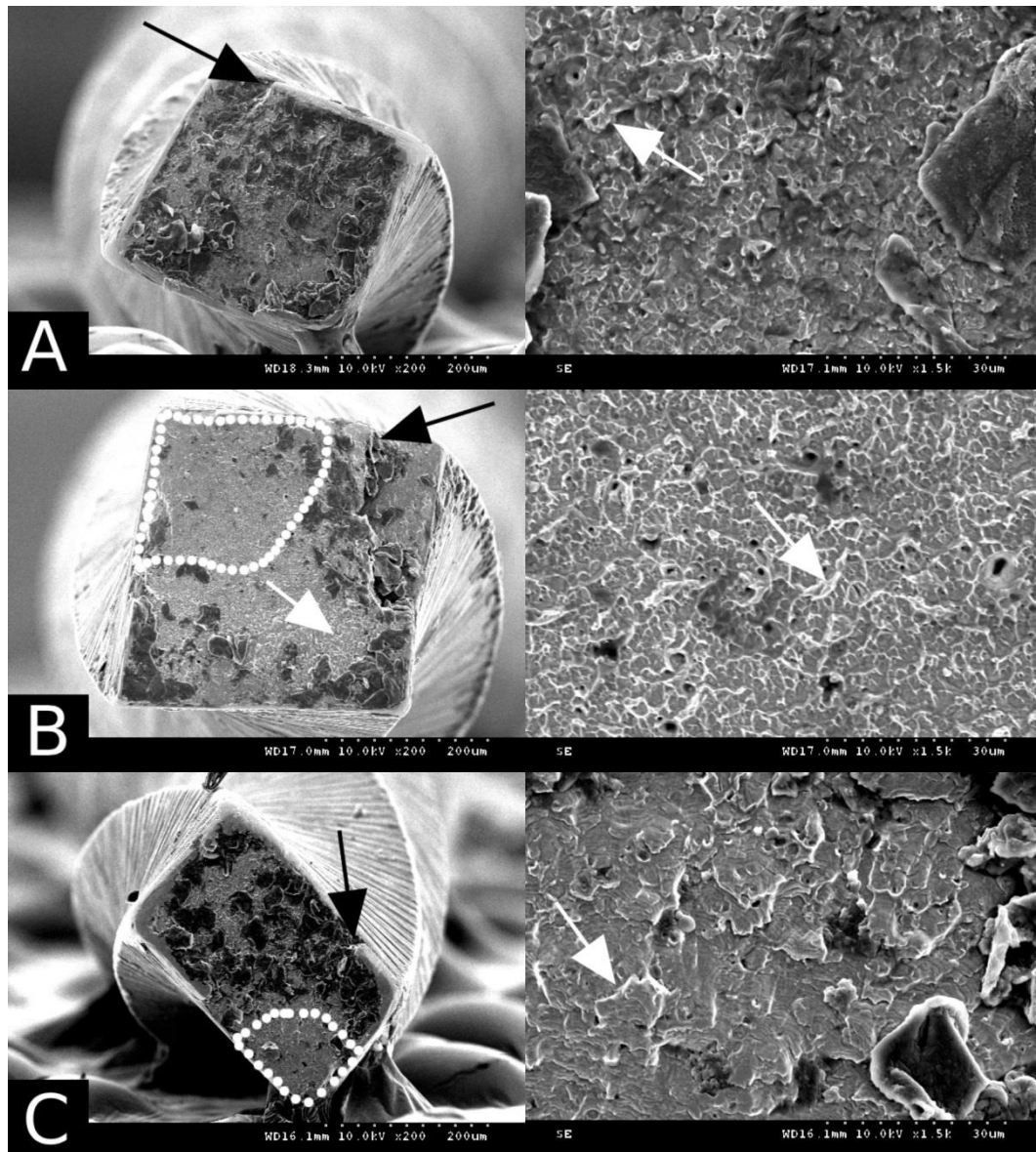
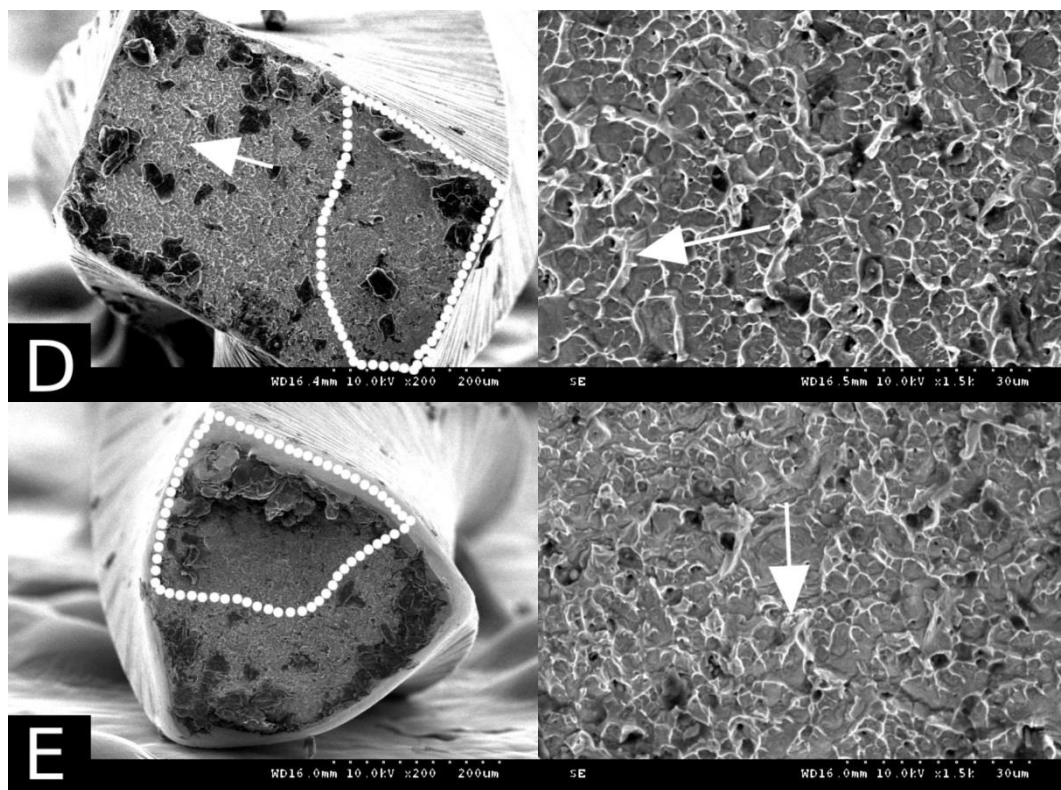


Figure 7. The file fragment length (FL) for WOG, PTG, and TRN in all sterilization-condition subgroups in dynamic mode

The horizontal axis (x-axis) represents the type of Ni-Ti rotary instruments with sterilization conditions and the vertical axis (y-axis) represents the file fragment length (FL).

### 3. Scanning Electron Microscope Examination





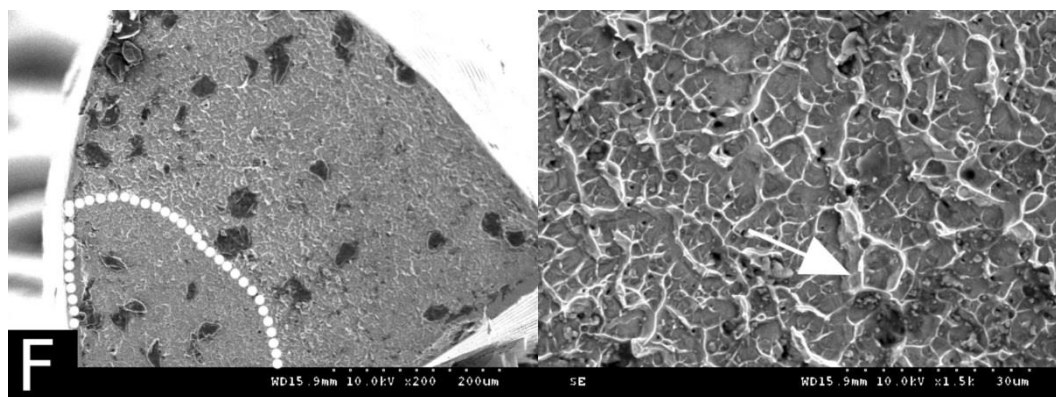


Figure 8. Scanning electron microscope images of file fragments for three-heat-treated Ni-Ti instruments after ten cycles of sterilization in both static and dynamic modes (A) TruNatomy under dynamic testing. (B) TruNatomy under static testing. (C) WaveOne Gold under dynamic testing. (D) WaveOne Gold under static testing. (E) ProTaper Gold under dynamic testing. (F) ProTaper Gold under static testing. The regions of crack initiation are indicated by black arrows. Fatigue zones are indicated by white dotted lines. Overload fast fracture zones and fatigue striation (fibrous dimples) are indicated white arrows.

## IV. Discussion

The cyclic fatigue resistance of Ni-Ti rotary instruments may be affected by a wide variety of factors, such as alloy composition, manufacturing procedures, cross-sectional geometry, and flute design (Kim TO et al. 2009, Kim HC et al. 2010). Modifying the metallurgical structures, heat treatments applied to the files, designs, and kinematic properties of Ni-Ti files can improve their cyclic fatigue resistance. (Shen et al. 2013, Ferreira et al. 2017). Owing to practical and economic factors, most dental practitioners use Ni-Ti endodontic files several times in association with many cycles of autoclave sterilization. Therefore, this study aimed to evaluate and compare the effects of autoclave sterilization on the static

and dynamic cyclic fatigue tests of various heat-treated Ni-Ti instruments in single artificial stainless-steel canals.

To our knowledge, no published studies have evaluated the cyclic fatigue resistance of WOG, PTG, and TRN after repeated cycles of autoclave sterilization in both static and dynamic modes. In the present study, we evaluated the effects of autoclave sterilization on static and dynamic cyclic fatigue tests on WOG, PTG, and TRN. This study used both static cyclic fatigue and dynamic cyclic fatigue mode to simulate the circumstances of clinical practice and to ensure that the stresses on the file are distributed evenly within the canal.

We found that cyclic fatigue resistance was significantly higher in dynamic mode for all the instrument systems. This result agreed with previous studies (Li et al. 2002, Lopes et al. 2010, Keles et al. 2019). The instrument is subjected to a single location of compression and tensile loads during static tests. On the other hand, in the dynamic tests, these stresses are dispersed over a larger area along the instrument shaft. (Li et al. 2002, Lopes et al. 2010). Notably, A better mimic of the clinical setting is provided by dynamic mode. (Yao et al. 2006).

In the present study, there was a statistically significant difference between the NSC and 10SC subgroups, in association with both TRN and PTG, in both the static and dynamic tests. PTG autoclaved ten times had higher cyclic fatigue resistance than unsterilized PTG. This result was consistent with previous studies (Plotino et al. 2012, Zhao et al. 2016 , Özyürek et al. 2017, and Emmanuel et al. 2020). GT Series, HyFlex CM, PTG, K3XF, and

ProTaper Next have been shown to have relatively high cyclic fatigue resistance after many (ten) sterilization cycles. Several investigator teams have concluded that the mechanical behavior of these instruments cannot be significantly changed by a small number of sterilization cycles. Özyürek et al. (2017) examined the effect of autoclave sterilization on the cyclic fatigue resistance of PTG. They concluded that ProTaper Next and PTG autoclaved ten times had substantially higher cyclic fatigue resistance than new instruments of these types (Özyürek et al. 2017). They found that autoclave sterilization affects cyclic fatigue resistance after ten cycles.

To our knowledge, there have been no published studies examining and comparing the effects of autoclave sterilization cycles on the cyclic fatigue resistance of WOG and TRN. Therefore, the results of this study cannot be directly compared with those of previous studies. For WOG, there was no statistically significant difference between the sterilization-condition subgroups in either static or dynamic cyclic fatigue tests. Moreover, WOG exhibited higher cyclic fatigue resistance than PTG and TRN. This may have resulted from variation in motion patterns between file systems (reciprocation vs. continuous motion) or a differing martensitic composition in WOG (Weyh et al. 2020). Reciprocation motion has been associated with enhanced cyclic fatigue resistance compared with rotation motion (Pedulla et al. 2013, Perez-Higueras et al. 2013). According to Zupanc et al. (2018), instruments with lower taper tend to be more flexible and have higher cycle fatigue resistance. (WOG has a .07 taper, and PTG has a .08 taper). In the present study, the TRN autoclaved ten times had lower cyclic fatigue resistance than unsterilized TRN. Elngly et

al. discovered that in single and double curvature canals, Hyflex and TRN instruments had greater resistance to cycle fatigue than Vortex Blue and Race instruments (Elnaghy et al. 2020). However, Özyürek et al. (2020) found VDW.ROTATE files to have the highest cyclic fatigue resistance; they also found TRN and 2Shape files to have the lowest cyclic fatigue resistance in artificial canals at body temperature.

We also found no published studies examining and comparing the effects of autoclave sterilization cycles on the cyclic fatigue resistance of PTG and TRN. In the present study, PTG autoclaved ten times had higher cyclic fatigue resistance than unsterilized PTG in both static and dynamic modes. However, TRN autoclaved ten times had lower cyclic fatigue resistance than unsterilized TRN. This difference may have resulted from the differing cross-sectional designs and file sizes between PTG and TRN. PTG has a triangular cross-sectional design (Figure 8E and 8F), and TRN has a square cross-sectional design (Figure 8A and 8B). According to Capar et al. (2015), triangular cross-sectional files exhibit higher levels of fatigue resistance than square cross-sectional files (Capar et al. 2015). According to Pedulla et al. (2017), an increase in file size may result in a reduction in cycle fatigue resistance. TRN Prime has a slightly larger diameter (0.26 mm) than PTG F2 (0.25 mm) (Pedulla et al. 2017). Regarding to the mean of fracture file fragment length, There was no significant difference in the mean length of the fracture fragments of files in single curvature. and the most of files fractured at or just below the center of curvature, which is consistent with the previous studies ( Duke F et al. 2015, Elnaghy AM et al. 2018, Topçuoğlu HS et al 2018, Elnaghy AM et al 2020). And most of the tested files fractured

nearly at the starting point of the curved part especially in the apical part of the file. This finding may have resulted from the more stresses around the curved point and the most weakest point of the file in the apical part. In our study, scanning electron microscopy analysis revealed that the fracture surfaces of all evaluated Ni-Ti instruments had similar typical cycle fatigue patterns. Crack initiation sites, crack propagation, fatigue zones, overload fast fracture zones, and fatigue striations were observed in cross sections of cyclic fatigue tests of the 10SC specimens. TRN instruments exhibited more crack initiation and propagation in their cross sections (black arrows, Figure 8A and 8B). Due to cyclic fatigue, fracture surfaces typically have areas of crack initiation and linear striations that are perpendicular to the direction of the tensile stress. (white arrows, Figure 8)

Finally, more research is required to assess the effects of multiple sterilization cycles and uses at body temperature simulations to mimic routine clinical usage. The main limitation of this study was the lack of accuracy in our typical-use simulation.

## **V. Conclusion**

Within the scope of this study, it is possible to conclude that ten cycles of PTG autoclave sterilization was associated with higher cyclic fatigue resistance than that observed with unsterilized instruments in both static and dynamic modes. However, TRN autoclaved ten times had lower cyclic fatigue resistance than unsterilized instruments. Finally, with WOG, there were no statistically significant differences between the sterilization-condition subgroups in either static or dynamic cyclic fatigue tests.

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