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**INVESTIGATION OF STATIC VERSUS DYNAMIC
CYCLIC FATIGUE RESISTANCE IN NITI
ENDODONTIC INSTRUMENTS WITH DIFFERENT
ALLOY TREATMENTS**

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ABSTRACT

Investigation of static versus dynamic cyclic fatigue resistance in NiTi endodontic instruments with different alloy treatments

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Background: Endodontic rotary instruments can be used in a variety of canal conditions; fatigue fracture is a risk. This study looked at the cyclic fatigue resistance of two batches of nickel–titanium (NiTi) dental rotary files with same geometry and different heat treatments operating in clinically simulated root canal.

Aim: To compare the deference in the cyclic fatigue resistance between heat treated and non-heat treated NiTi rotary instruments in static and dynamic modes.

Materials and Methods: Cyclic fatigue tests were performed on PTU and PTG instruments with a curvature of 35° and a radius of 6 mm in static and dynamic mode at body temperature (EndoC: DMJ mechanism, Busan, Korea). The number of cycles to fracture (NCF) was rerecorded. A scanning electron microscope was used to examine the fracture surfaces of all fragments.

Results: Depending on the motions performed, the cyclic fatigue resistance of the PTU and PTG varies (static or dynamic). When PTU and PTG were compared, they showed statistically significant differences in cycle fatigue resistance. PTG had higher cyclic fatigue fracture resistance on both modes tasted, but dynamic mode of testing of testing was associated with a high NCF in both files tested.

The transverse and longitudinal sections of SEM micrograph of the specimens were examined after cyclic fatigue testing for PTG files and PTU files. The micro crack initiation regions, overload quick fracture areas, and microcrack areas in both were identified. PTG was discovered to have a lower concentration of microcracks.

DEDICATION

This dissertation is dedicated to my beloved parents (Ahmad and Badria) for their endless love, sacrifices, prayers, support, and advice. You are my strength. Thank you for always believing in me. Without you, I would not be the person I am today.

My brothers (Aziz, Talal, Mohamed and Bader) and sister (Afarah) for their continued encouragement, love, and support. They have never left my side and are very special to me.

To my best friend (Dr. Omar Nawaf), who shared his words of advice and encouragement with me, without you, it would have been very difficult to overcome all the challenges.

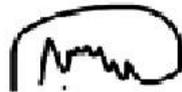
Finally, I would like to thank Allah Almighty for everything in my life; without his guidance and protection, I would not be able to accomplish anything in my whole life.

DECLARATION

I declare that all the contents of this thesis are my work. There are no conflicts of interest with any other entity or organization.

Name: Tareq Abdulkareem

Signature

A handwritten signature in black ink, appearing to be 'Tareq Abdulkareem', enclosed within a hand-drawn oval shape.

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1.REVIEW OF THE LITERATURE

1.1 Objectives of cleaning and shaping root canal system

Cleaning and shaping the root canal system is considered one of the most critical steps in root canal treatment and is essential for an effective treatment. It involves enlarging and shaping the intricate endodontic space, as well as disinfecting it¹. According to Kakehashi (1965) and Siqueira (2001), endodontic failure is thought to be caused by a persistent microbial infection within the root canal system. As a result, extensive chemo-mechanical preparation is required for root canal treatment to be effective^{2,3}.

Schilder's root canal cleaning and shaping concept aimed to create a three-dimensional continuous funnel tapering in multiple planes with sufficient apical enlargement while maintaining foramen position and size⁴. These objectives were classified as mechanical and biological. The mechanical objectives were to create a continuous tapering funnel from the access cavity to the apical foramen, to maintain the initial course of the main canal during root canal preparation, to maintain the original position of the apical foramen, and to keep the apical opening as small as possible. The biological goals were to limit instrumentation to the root canals only, to ensure that no necrotic or instrumentation debris was pushed beyond the apical foramina, to achieve optimal root canal debridement, and, eventually, to create a sufficient space for intracanal medications⁴.

In 2007, Young et al. investigated the concept of chemo-mechanical planning. Biological goals include removing microorganisms from the root canal system, removing pulp tissue that may promote microbial growth, and preventing debris from passing through the apical foramen, which may cause inflammation⁵.

1.2 Development of endodontic strategies

Regardless of the technique used to clean and shape the teeth, the use of conventional steel instruments results in a variety of iatrogenic root injuries. Several preparation errors are included in this list, including elbow, zip, ledge, and perforation⁶. Numerous techniques for canal preparation have been developed for this purpose. The step-back technique begins by preparing the root canal's apical area with smaller files, followed by coronal flaring with larger files to allow for obturation⁷. Crown-down techniques include widening canal orifices with Gates-Glidden drills and then removing organic canal material from the canal orifice to the apical part with manual files⁷. When compared to the step-back method, the crown-down method produces less apically extruded debris⁸. The balanced force technique enables the preparation of larger curved canals using modified stainless-steel files. NiTi rotary instruments have virtually eliminated the risk of iatrogenic instrumentation that is frequently associated with endodontic steel instruments⁸. In 2001, Peters et al. investigated micro-computed tomography (CT) scans before and after mechanical instrumentation. They discovered that regardless of instrumentation technique, 35% or more of root canal surfaces remained uninstrumented⁹. Tan and Messer reported in 2002 that rotary NiTi instrumentation produced a significantly cleaner canal in the apical 3 mm to a larger file size than hand instrumentation¹⁰. In a 2011 in vitro study, Paqué and Peter used CT to compare the efficacy of ProTaper files and stainless-steel hand files in the distal oval canals of lower molar teeth. Regardless of the files or methods used, they indicated that unprepared areas in the sample ranged from 59.6 to 79.9 percent, with no statistically significant difference between the groups. Additionally, the increased root canal volume was consistent across all groups ($P=0.001$)¹¹. Hilal et al. (2015) used computed tomography (CT) to assess the shaping abilities of the ProTaper system and nickel-titanium hand files in young permanent teeth. He discovered that unprepared canal surface areas ranged from 40.6 to 46.2 percent of total canal

surface area¹². Schäfer and Zapke (2018), on the other hand, found an un-instrumented region with residual debris in all canals regardless of preparation technique in a curved root canal of extracted human teeth prepared with either rotary NiTi or stainless-steel hand files¹³.

1.3 Endodontic instruments

1.3.1 Stainless steel instrument

Until 1960, root canal instruments were made of carbon steel. Alloys of stainless steel are now widely used¹⁴. Traditionally, canal shaping has been accomplished manually with ISO-standard 0.02 tapered stainless steel tools¹⁴. Initial canal negotiation, defining an endodontic glide path, radiographically or with the aid of electronic apex locators determining working length, and checking patency are all still performed with stainless-steel manual files¹⁵. These instruments are limited in versatility, particularly in larger sizes, which frequently results in procedural errors and a lower success rate for endodontic treatment¹⁶. In 1991, Briseno and Sonnabend reported that no hand stainless-steel instrument produced ideal results, despite the fact that the nine instruments compared produced clinically appropriate canal shapes¹⁷.

1.3.2 Nickel-titanium alloy (NiTi)

W. F. Buehler worked at the Naval Ordnance Laboratory in Silver Springs, Maryland, USA, in the early 1960s, manufacturing nickel-titanium alloys for space programs¹⁸. Andreasen and Hilleman pioneered the use of NiTi wires in orthodontics, observing differences in the physical properties of Nitinol and stainless steel orthodontic wires that allowed for the use of lighter forces¹⁹. Endodontic files were developed by Walia, Brantley, and Gerstein using nitinol orthodontic wires. In comparison to comparable stainless-steel files, these files had two to three times the elastic strength in bending and torsion, as well as a higher resistance to torsional fracturing²⁰.

1.3.3 Basic metallurgy of nickel-titanium (NiTi)

The nickel-titanium alloy used in root canal therapy contains approximately 56% nickel and 44% titanium. As with other metallic systems, the resulting mixture has an atomic ratio of one to one (equiatomic), and the alloy exists in a variety of crystallographic forms¹⁸. These alloys are frequently referred to as 55-Nitinol. It exists in two crystal structures that are temperature dependent. Nitinol is an austenitic material at elevated temperatures. Nitinol has a martensitic crystalline structure at low temperatures. These two distinct characteristics are the result of austenite being transformed into martensite in the NiTi alloy. They are referred to as super-elasticity and shape memory. These are brought about by changes in temperature and stress^{18,21}. Due to NiTi's super elasticity, deformations of up to 8% strain can be recovered. Stainless steel, on the other hand, can withstand a maximum pressure of less than 1% before permanent deformation occurs.

Super-elastic alloys include copper-zinc alloys, copper-aluminum alloys, gold-cadmium alloys, and nickel-niobium alloys. None of these, however, have the extent of strain or heat recovery, general corrosion resistance, human tissue compatibility, or fluid body compatibility that nitinol has^{16,18,22}.

1.3.4 Benefits of nickel-titanium instruments in endodontics

Nickel-titanium rotary files have become a mainstay of clinical endodontics because they can shape root canals more quickly and with fewer procedural complications⁵. Numerous experiments have demonstrated that when rotary NiTi instruments are used on extracted human teeth, they retain the original canal curvature better than stainless steel hand instruments²³. Short and Gluskin et al. found that rotary NiTi instruments, particularly in the apical region of the root canal, maintain the original canal curvature better than stainless-steel hand instruments in terms of shaping ability^{24,25}. Esposito and Cunningham discovered that NiTi files were significantly more effective at preserving the initial canal course than

stainless steel hand files when the apical preparation was extended beyond ISO size 30²⁵. NiTi instruments, according to in vitro research, produce significantly less straightening and more oriented practices than stainless steel hand files, lowering the risk of iatrogenic errors. Petiette et al. instrumented 40 teeth with NiTi hand files or stainless-steel K-files and discovered that NiTi instrumentation better preserved the initial canal shape. When the two groups were compared one year after endodontic surgery, the investigators discovered that teeth prepared with NiTi files had a slightly higher healing rate (as assessed by changes in the densitometric ratio)²⁶. Tan and Messer discovered that using rotary NiTi instruments to instrument larger file sizes produced significantly cleaner canals in the apical 3 mm compared to hand instrumentation¹⁰. Using NiTi rotary instruments was found to minimize the occurrence of procedural errors such as insufficient working volume, instrument divergence, canal transportation, zip or elbow shape, strip perforation, and unnecessary root weakening are minimized^{27,28}. Furthermore, when rotary nickel-titanium endodontic instruments are used, the success rate is higher than when only stainless-steel hand instruments are used²⁹. Two recent studies at the University of Jordan found that using NiTi rotary files improved the overall technical performance of posterior molar root canal fillings. Their initial experience proved to be more reliable and effective than performing it manually³⁰. Furthermore, as a result of the new technique's widespread acceptance, undergraduate students demonstrated superior knowledge and satisfaction, highlighting the importance of systematic integration of rotary NiTi instruments and methods into undergraduate teaching and future clinical practice³¹.

1.3.5 Different generations of NiTi alloy.

In the 1990s, the first commercially available nickel-titanium rotary files appeared on the market. Each file system generation's mechanical classification was addressed³².

1.3.5.1 First-generation files

In 1992, Dr. John McSpadden introduced the first rotary NiTi instrument with a 0.02 taper to the market³³. By 1994, he had created the ProFile Line, which featured two distinct tapers, 0.04 and 0.06. They have fixed tapers and passively cut radial lands that allow the file to remain balanced in canal curvatures. Following that, LightSpeed rotary files were created³³. The primary limitation of this generation of NiTi rotary instruments is that achieving these goals and complexity requires multiple files³⁴.

1.3.5.2 Second-generation files

In 2001, the generation of NiTi rotary files hit the market³⁵. These instruments differed from the previous generation in that they had active cutting edges without radial lands, resulting in greater cutting efficiency and the use of fewer instruments to prepare a canal³⁵. They form super-elastic wire blocks with a solid martensite phase under clinical conditions. In alloy manufacturing, thermal processing alters the crystal structure arrangement and the phase composition of the alloy. Heat treatment typically results in finely distributed NiTi particles in the matrix and an increase in the alloy's Af (austenite finish), resulting in a different martensite and/or austenite crystallographic percentage near body temperature³³. The ProTaper rotary files are part of the latest generation of NiTi files and feature a redesigned guiding tip as well as multiple levels of rising and decreasing taper³⁵.

1.3.5.3 Third-generation files

NiTi metallurgy has progressed to the third generation. One of the most effective methods for modifying the transition temperatures of NiTi alloys and thus influencing the fatigue tolerance of NiTi endodontic files is heat treatment. The thermodynamic treatment of the wire before or during manufacturing was used to fabricate three different wires³³. They are known as the M-wire, the R-phase, and the CM wire (controlled memory).

1.3.5.4 Fourth-generation files

The reciprocity principle, also known as up-and-down or back-and-forth motion, is a recent advancement in the canal preparation process³⁶. A bidirectional reciprocating file (clockwise and counterclockwise) takes more internal friction to advance and does not cut as well as a rotary file of the same size³⁷. This concept has spawned the fourth generation of canal-shaping instruments. In 2010, a self-adjusting file was introduced. WaveOne and Reciproc were added as single file forming techniques. In both files, the M-wire was used³⁷.

1.3.5.5 Fifth-generation files

The fifth-generation files were designed with a balanced mass or rotational center of mass³⁸. These files with an offset design generate motion waves along the file's active section. The offset design eliminates the taper lock or screwing effect between the file and the dentin, which results in instrument separation³⁴. The asymmetrical cross-section of the Revo-S NiTi rotary mechanism increases the usable volume for upward debris removal³⁵. The asymmetric cross-section of the Revo-S allows for snake-like penetration and root canal shaping that meets biological and ergonomic requirements³⁴.

ProTaper Next (PTN) files have progressive percentage tapers on a single file, as well as M-Wire technology for increased stability and cyclic fatigue resistance. It outperforms ProTaper Universal files in terms of efficiency by using fewer files. It has a variable taper and an off-center rectangular cross section.

1.3.6 Types of heat treatment wires

1.3.6.1 M-Wire

Dentsply Maillefer (Ballaignes, Switzerland) In 2007, introduced the M-Wire file. It undergoes a series of heating and cooling cycles, which helps to maintain Nitinol's crystalline structure in its more martensitic state at body temperature. Austenite, martensite, and R-phase were all components of the M-wire³³. At room temperature, M-Wire has an austenite finish

temperature of 45–50 °C; the temperature range for the phase transition indicates that M-Wire instruments are martensitic. M-Wire instruments include the ProFile GT sequence X, ProFile Vortex, ProTaper Next files (PTN), Proglider, Wave One (WO), and Reciproc³³.

1.3.6.2 R Phase

In 2008, SybronEndo created the first fluted NiTi file, employing a plastic deformation process similar to that used in the production of most stainless steel K-files and reamers³⁴. This new manufacturing process aims to convert raw NiTi wire from the austenitic phase to the R-phase and stabilize it at higher temperatures³³. The R-phase has a lower shear modulus than martensite and austenite, and it is entirely austenite at body temperature³³. R-phase instruments include the TFA, K3XF.

1.3.6.3 CM Wire (Controlled memory NiTi wire)

In 2010, the CM wire was first used. The thermomechanical method aimed to improve stability, increase transformation temperatures to around 50 °C, reduce shape memory, and achieve stable martensite at body temperature³⁹. According to Testarelli et al., CM has less nickel (52 percent Ni wt.) than the more well-known NiTi³⁵. According to a recent study by De Vasconcelos (2016), the austenite finish (Af) temperature of Hyflex CM and Typhoon CM is approximately 47 °C and 55 °C, respectively, leading him to believe that this instrument at body temperature is a mixture of martensitic, R-phase, and austenitic structure. This finding backs up a previous study that found that instruments made of super-elastic NiTi exhibit an austenitic process at room temperature. The MW and CM files, on the other hand, are in the martensite and R-phases, as well as austenite⁴⁰.

1.3.6.4 Gold and Blue heat-treated wire

Dentsply International Inc. is a United States-based dental supply company. Tulsa Dental introduced Vortex Blue, the first blue-colored endodontic instrument, in 2011. There are two NiTi systems with gold heat treatment (ProTaper Gold and WaveOne Gold) and two NiTi systems with blue heat treatment (ProTaper Blue and WaveOne Blue) (Vortex Blue and Reciproc Blue). These instruments are capable of deformation and have a controlled memory effect⁴¹. The primary distinction between CM wire and heat-treated gold and blue instruments is that these files are ground prior to being heat-treated after machining⁴⁰. Both gold and blue heat-treated files demonstrated increased flexibility and fatigue resistance when compared to standard NiTi and M-Wire instruments, which could be attributed to their martensitic state. Only Hyflex EDM files outperformed ProTaper Gold, WaveOne Gold, and Reciproc Blue in terms of cyclic fatigue tolerance¹³.

1.3.6.5 MaxWire

MaxWire (Martensite-Austensite-electropolish-flex), a new thermomechanical treated NiTi alloy, was recently introduced by FKG Dentaire. It has shape memory and super-elasticity in clinical use. The XP-endo Shaper and XP-endo Finisher are two of these techniques. At room temperature, these instruments are straight, but when subjected to intracanal temperature, they curve due to a phase change to an austenitic state¹³.

1.3.6.6 Summary of the new thermomechanical treatment for the NiTi alloy

Under clinical conditions, a recent thermomechanical therapy for the NiTi alloy allows for a change in phase structure, resulting in the appearance of the martensite state. The martensitic instruments are lighter, have a higher cyclic fatigue resistance, and have a greater angle of rotation. They do, however, have a lower torque at fracture and a greater angle of rotation than ferrite instruments. These devices are

suitable for canals that are steeply inclined or have a double curvature. They can prebend, which is useful for avoiding ledges¹³.

1.3.7 Fracture of the NiTi instrument

In NiTi rotary files, instrument fracture is a common problem. Stainless steel rotary files, on the other hand, are less versatile and durable than NiTi rotary files.⁹ Separation can occur in the absence of visible evidence of previous irreversible deformation, simply beyond the file's elastic boundary ^{40,42} , reducing the likelihood of achieving the targeted treatment's ultimate goal³. According to a literature review, the average clinical fracture frequency of rotary NiTi instruments is approximately 1% percent, with a range of 0.4–3.7%. The average prevalence of retained endodontic hand instruments (primarily stainless steel files) is approximately 1.6%, ranging from 0.7 % to 7.4 percent ⁴⁰. According to one study⁴³, fractures caused by NiTi rotary instruments were seven times more common than fractures caused by hand instruments during an endodontic residency program. Unfortunately, research on the therapeutic value of preserving fracture files inside treated root canals has produced mixed results. Evidence suggests that in some cases (teeth with necrotic pulp or teeth with periapical lesions), if the instrument fragments, it would be more painful and unfortunate, as healing chances would be reduced⁴⁴. If the endodontic instrument is split in the apical third of the root canal or beyond the root canal curvature, the prognosis of the tooth undergoing root canal treatment is poor. The fragment's removal becomes more difficult. The greater the curvature of the root canal, the more difficult it is to extract the fractured segment^{45,46}.

1.3.8 Factors influencing the mechanism of NiTi instruments

To use NiTi instruments safely in clinics, it is necessary to first understand the basic fracture mechanisms and how they relate to canal anatomy. When used in a rotary motion, they are prone to structural fatigue, which can lead to fracture if the file's resistance exceeds a certain threshold⁴⁷. Sattapan et al. identified two types of fracture in rotary NiTi instruments in 2000: cyclic fatigue (when repeated compressive and tensile stresses act on the outer fibers of a file rotating in a curved canal) and torsional failure (when the tip of the instrument binds). Even so, the file's shank continues to spin. In clinical trials, he discovered that torsional failure (56 percent vs. 44 percent) was more common than cyclic fatigue for a variety of NiTi-based rotary file applications⁴⁸.

1.3.8.1 Torsional stress

Torsional stress fractures occur when the instrument tip or another component becomes lodged in a canal and the instrument's elastic limit is exceeded, resulting in plastic deformation (unwinding, reverse winding) and fracture⁴⁸. It typically occurs when the user applies an excessive amount of apical force to the rotating instrument in the root canal, or when smaller diameter root canal instrumentation produces more torsional tension than larger diameter root canal instrumentation during the cleaning and shaping procedure^{48,49}. Torsional stress fractures can occur when the tip of a spinning instrument enters a narrow root canal. Because of the increased friction, a greater torque is required to rotate the file, putting undue strain on the delicate instrument tip⁴¹. This phenomenon is known as 'taper lock' because it can occur with equally tapered instruments with different tip diameters rather than variably tapered instruments⁵⁰. Torsion stress is influenced by the tip and taper of the instrument, as well as the size of the canal, and an increase in instrument diameter, as well as a corresponding increase in cross-sectional area, may result in increased torsional

resistance⁴⁰. The clinician, on the other hand, has control over endodontic instrument manipulation and thus has the ability to reduce the effects of torsional stress fractures⁴⁸.

1.3.8.2 Flexural/Cyclic fatigue

Cyclic fatigue occurs when an instrument freely rotates in a curved canal, resulting in tension/compression loops and, eventually, a fracture. While the instrument was in a static position, it was rotated. The shaft's outer half was tensed, while the inner half was flattened. The instrument's cyclic fatigue increases with time as a result of the repetitive^{9,40}. When the diameter of a rotary file is increased, the tolerance of the file to cyclic fatigue increases proportionately. This is related to the instrument's metal mass at the point of maximum tension. There is a correlation between the severity of the angle and radius of the canal curvature and the life span of the instrument. This correlation existed because the instrument's lifespan is correlated with the severity of the angle and radius of the canal curvature. According to the findings of both in vitro and clinical studies, increasing the severity of the angle and radius of the curvature can result in a shorter life span for the instrument⁴³.

1.3.8.3 Other factors

The fracture of NiTi instruments has been linked to several factors, including:

1. Operator skill/experience: Barbakow and Lutz (1997) investigated the effects of operator experience⁴³, and Mandel et al. (1999)³⁵ discovered that proper experience and training is needed to reduce the occurrence of instrument separation³⁸.
2. A crown-down instrumentation technique and the development of a manual glide path have been recommended to minimize the risk of instrument fracture. These strategies may help to minimize instrument 'taper lock' which is related to torsional fracturing^{51,52}.

3. Torque: This may affect the likelihood of instrument locking, deformation, and separation. A high-torque instrument is potentially very active, which increases the risk of instrument locking. On the other hand, a low torque would decrease the instrument's cutting efficiency and make progress in the canal difficult, and the operator would be tempted to force the instrument, potentially resulting in instrument locking, deformation, and separation⁵⁰. According to Kobayashi et al., the torque for ProFiles should be set between 40 and 80 Ncm to avoid instrument failure⁵³.
4. The number of times a NiTi rotary file is used reduces its flexural fatigue resistance. As a result, they fail at a higher rate than new instruments⁵⁴.
5. Tooth anatomy: The majority of NiTi rotary instrument fractures occur in the mesial canals of mandibular and maxillary molars. In retreatment cases, fractures were also normal compared to the initial endodontic treatment^{42,55}.
6. Instrument separation was less likely when the rotational speed was lower⁵⁰. Manufacturers, on the other hand, recommend a specific number of rotations per minute (rpm) for safe use of rotary NiTi instruments, which is typically between 250 and 600 rpm⁵⁶.
7. Instrument size and radius of curvature: In vitro studies show that smaller NiTi instruments have a higher frequency of fracture and distortion⁵⁷. It has been demonstrated that as the radius of curvature and angle of curvature decrease, so does the number of cycles required for the file to fracture⁴⁷.
8. Surface condition and instrument inspection: The manufacturer of rotary NiTi instruments recommends that instruments be inspected for defects and microcracks on a regular basis to avoid instrument fracture^{57,58}.
9. Intracanal temperature

Clinical steps to reduce torsional stress and flexural/cyclic fatigue are recommended (A-G)⁴⁰

- A. Reduce the touch surface area between the files and dentine walls by using a crown-down instrumentation sequence or a hybrid instrumentation protocol.
- B. Slowly advance files into a canal until resistance is met, then withdraw gently without placing excessive pressure on the file.
- C. Check for finger rests to compensate for patient movement and avoid taper lock.
- D. Using a torque control motor with the manufacturer's recommended settings for each instrument.
- E. To minimize friction, use chelators and lubricants.
- F. Recapitulate, rinse, and wipe instrument blades often to prevent dentine debris from clogging file blades and to minimize friction.
- G. Prepare a working glide path by manually pre-flare and planning a glide path.
- H. The radius of curvature is increased while the number of curves is decreased by having straight-line access to the apical half of the canal.
- I. Avoid using a rotary file with a 6 percent taper or higher in canals with mid-root curvature.
- J. To delay the onset of fatigue, reduce the rotation speed.
- K. When a file is inserted into the canal, the concentration of the bending stress decreases at any point along the length of the file owing to continuous in-out axial movement.
- L. Limiting the number of times NiTi files are used, particularly after they have been used in many curved canals.

1.4 Cyclic vs Dynamic cyclic fatigue testing

In some studies, employing a dynamic design, a vertical motion has been added to better simulate the clinical environment. In both the static and dynamic test models, a standardized procedure is lacking. One of the earliest studies utilizing a dynamic test design (Dederich & Zakariasen, 1986) compared static and dynamic cyclic fatigue testing with an axial amplitude of 8 mm, revealing a 230–530 percent increase in time until failure in the dynamic test⁵⁹. Unfortunately, only a few nickel titanium rotary instruments have been tested in both static and dynamic fatigue^{60,61}. Li et al. (2002) investigated the static and dynamic cyclic fatigue resistance of ProFile size 25/.04 taper instruments with 1–3 mm vertical amplitudes. The time until fracture in dynamic tests was approximately 20%–40% longer than in static tests⁶². Several subsequent studies have confirmed the finding that the time until fracture was longer in dynamic tests than in static tests^{63,64}. In general, a given instrument's dynamic cyclic fatigue resistance is significantly greater than its static fatigue resistance; for some instruments, the difference was up to 150%. This increased longevity of NiTi instruments in dynamic cyclic fatigue tests is primarily explained by the distribution of the bending load along the instrument, which avoids a localized load on a single point of the file⁶¹. The phase transformation of NiTi alloys distributed along the instrument during the dynamic test prevents microcrack formation at a specific point of the instrument, resulting in a longer lifetime of NiTi instruments in dynamic tests¹².

1.5 Effect of temperature on fracture resistance of the instrument

Endodontic instruments are made of NiTi alloy because it has unique properties such as super elasticity and shape memory that outperform stainless steel alloys¹⁸. These distinct properties influence the mechanical properties of NiTi instruments, where a phase transition from austenite to martensite occurs as a result of a temperature decrease and/or the application of stress^{18,21}. The super elastic NiTi alloy is composed primarily of austenite

and exhibits significant hardness and stiffness values above the transformation temperature range, whereas at lower temperatures it is composed primarily of martensite and exhibits reduced hardness and higher flexibility^{65,66}. Previous studies used room temperature for in vitro fracture testing^{67,68}, which is much lower than body temperature and should not be clinically relevant because the NiTi instrument is used inside the root, which is surrounded by periodontium and thus has an environment that is close to body temperature. Clinical studies have shown that the temperature inside the canal is ~35 °C. Even if the irrigation solutions are introduced at higher or lower temperature levels (20.7°C – 66°C), they will be buffered to the original canal temperature after 240 seconds. ⁶⁹. Performing the experiment at a controlled temperature close to body temperature may be more relevant. Early cyclic fatigue studies focused on testing NiTi instruments at room temperature^{51,67}, which did not reflect the actual clinical situation in which the NiTi instrument is used at body temperature. According to de Vasconcelos, endodontic instruments' cyclic fatigue is much lower at body temperature than at room temperature when tested in air⁷⁰.

1.6 ProTaper Universal and ProTaper Gold

ProTaper Universal (PTU, Dentsply Maillefer, Ballaigues, Switzerland) is a well-researched NiTi rotary system with variable taper across the length of the cutting blades, convex triangular cross sections, and noncutting tips. ProTaper Gold (PTG, Dentsply Maillefer, Ballaigues, Switzerland) instruments were recently introduced. The PTG files have the same geometries as the PTU files but are more flexible and have been developed using proprietary advanced metallurgy. According to the manufacturer, these instruments have fatigue resistance that is superior to PTU. These new manufacturing methods and materials for NiTi instruments have the potential to advance the science of endodontic rotary instrumentation.

2. AIM

To compare the difference in the cyclic fatigue resistance between heat treated and non-heat treated NiTi rotary instruments in static and dynamic modes.

2.1 Null hypotheses

There is no difference in the cyclic fatigue resistance between ProTaper Gold and ProTaper Universal in either static or dynamic modes of testing.

3. MATERIALS AND METHODS

3.1 Sample size

Sample size calculation for comparison means between groups, continuous outcome

The formula

$$n = 2 \left(\frac{z_{1-\alpha/2} + z_{1-\beta}}{SE} \right)^2$$

Where

$$SE = \frac{\mu_d}{\sigma_d}$$

Power	α	$2(z_{1-\alpha/2} + z_{1-\beta})^2$	Sample size
0.95	0.01	31.5	25
	0.05	26	21
0.90	0.01	17.1	14
	0.05	21	17
0.80	0.01	20.1	16
	0.05	12.4	10

Sample size calculation was based on a pilot study. Considering a test power of 0.80 (G*Power 3.1.9.2 software, Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany) with $\alpha = 0.05$, the minimum sample size was established at 10 (12.4/1.24) instruments for each group (n=10).

Sixty NiTi rotary endodontic files will be used for each of the 2 experimental groups: ProTaper Universal (PTU) F2 and ProTaper Gold (PTG) F2. (at 37°C). A total of 30 new files of each system will be used for static cyclic fatigue test (n=15) and dynamic cyclic test (n= 15).

Files that meet the following criteria will be included in the study. The files in the pack were brand new and had never been used before; and the files will be examined under a dental operating microscope, with any defective instruments discarded.

Files were distributed as follow:

ProTaper Universal group: (n= 15) for static cyclic fatigue test & (n= 15) for dynamic cyclic test.

ProTaper Gold group: (n= 15) for static cyclic fatigue test & (n= 15) for dynamic cyclic test.



Figure 1: ProTaper Universal NiTi file (PTG; Dentsply Sirona, Endodontics, Switzerland). Size F2 length 21 mm



Figure 2: ProTaper gold NiTi file (PTG; Dentsply Sirona, Endodontics, Switzerland). Size F2 length 21 mm

3.2 Investigation design

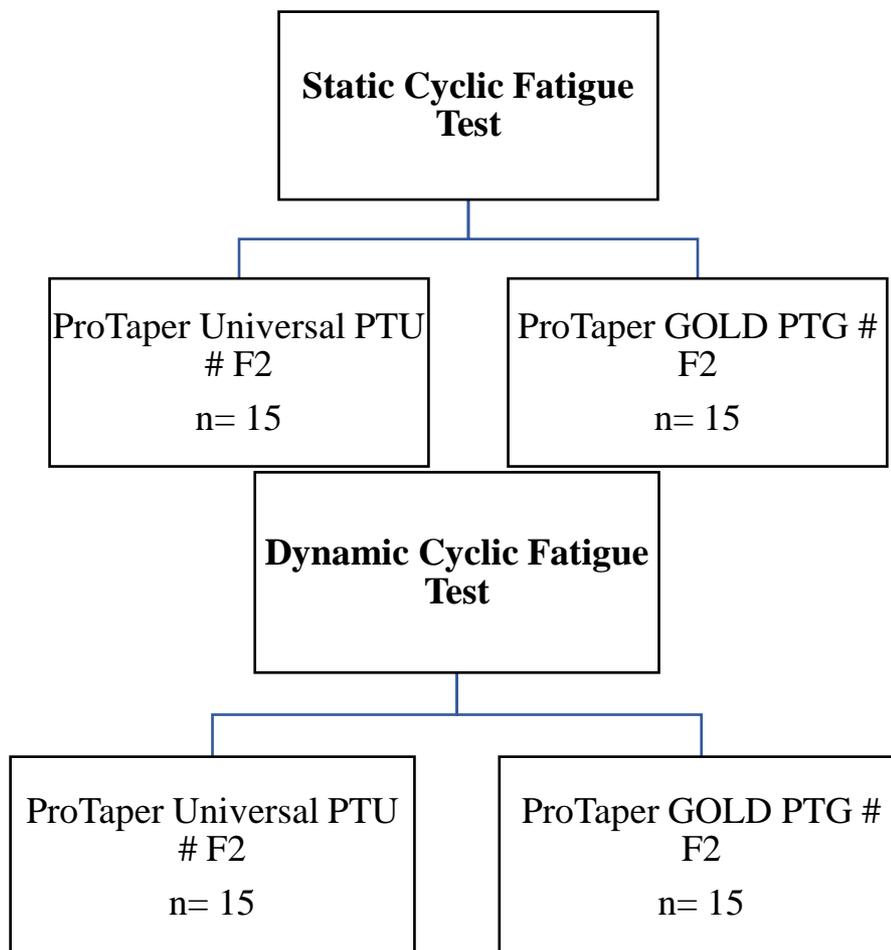
The sixty files were assigned to four groups:

Group (PTU-S): (n= 15) PTU F2 static motion

Group (PTG-S): (n= 15) PTG F2. Static motion

Group (PTU-D): (n= 15) PTU F2 dynamic motion

Group (PTG-D): (n= 15) PTG F2 dynamic motion



3.3 Cyclic fatigue testing:

The cyclic fatigue resistance was tested by a customized device A: (EndoC: DMJ system, Busan, Korea) (Fig. 3) by using simulated canal made of temporary steel as described similarly in Ha et al.

An artificial canal block will be made of tempered steel with a 7.82mm radius of canal curvature and 35° angle of curvature [17,18].

Synthetic oil (WD- 40; WD-40 Company, San Diego, CA) will be sprayed into the canal space to reduce the friction between the files and metal canal walls. The files will be freely rotated in the canal at a speed of 300 rpm in both static and dynamic mode. When the file will fracture, the time elapsed until the fracture will be recorded using a chronometer. The number of cycles to failure (NCF) for each instrument will be calculated by multiplying the total time(s) to failure by the rotation rate.

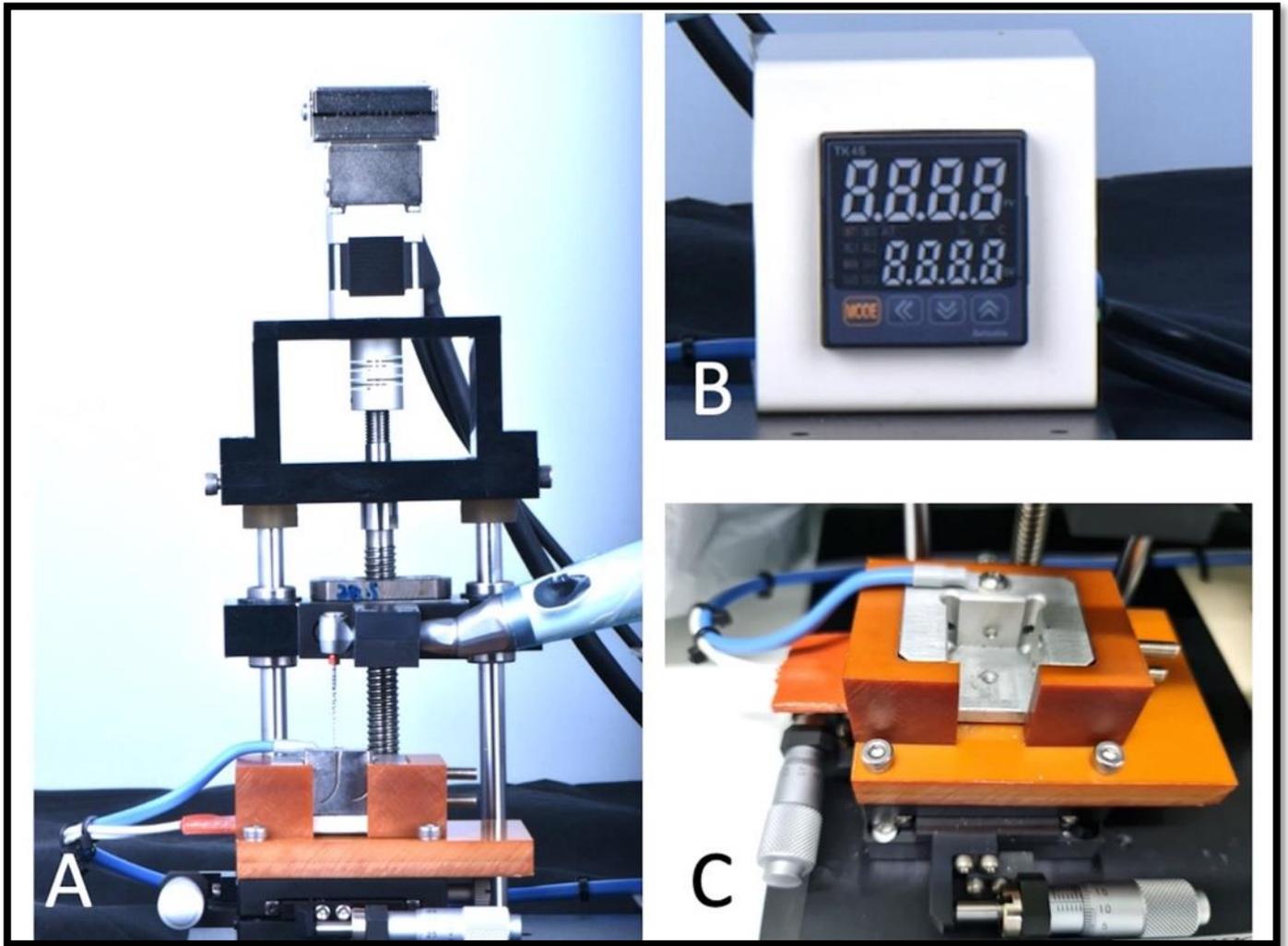


Figure 3. Experimental setup for the cyclic fatigue fracture test. (A) EndoC: DMJ mechanism, Busan, Korea), (B) Heat control panel, and (C) Heat pad assembly.

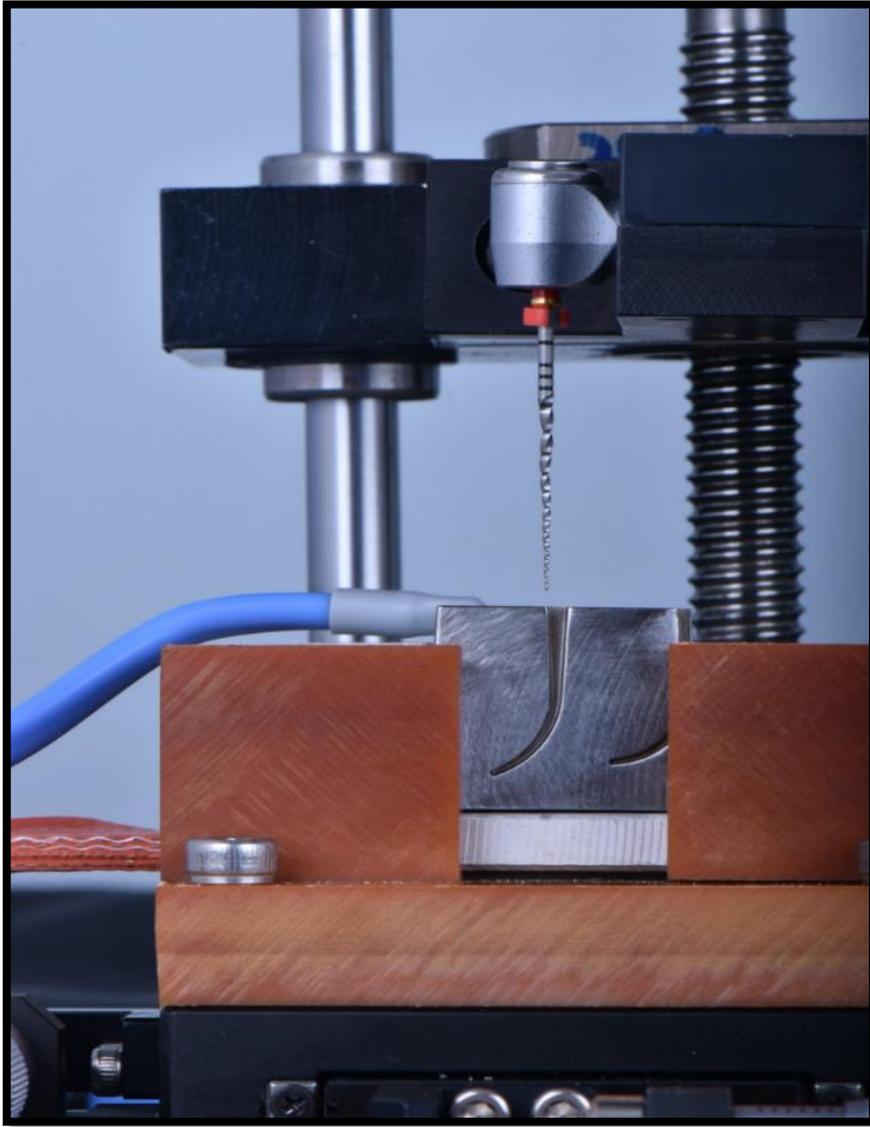


Figure 4: A tempered steel canal with a length of 17mm, a radius of 7.82 mm, and a curvature of 35°

3.4 Statistical analysis

SPSS version 25.0 was used to enter the data (SPSS Inc., Chicago, IL, USA). Means and standard deviations were used to characterize the continuous results. The Kolmogorov–Smirnov test was used to evaluate the normality of the continuous results (NCF and fragment length). The NCF and fragment length was compared using analysis of variance (TWO WAY ANOVA) between the four groups. In all experiments, a p-value of less than 0.05 was considered significant.

3.5 SEM Evaluation

Scanning electron microscopy was used to examine the topographic characteristics of the damaged surfaces during the cyclic fatigue examination (SU8220; Hitachi High Technologies, Tokyo, Japan).

4. RESULTS

Table 1. shows the results of the cyclic fatigue resistance of the ProTaper Universal and ProTaper Gold F2 NiTi instruments. These results show a significant statistical difference in the number of cycles to failure (NCF) between the four groups with a P-value of 0.001.

Furthermore, the results show insignificant difference in fragment length fracture between all tested groups of PTU and PTG, with a P-value of 0.534. The ProTaper Universal and ProTaper Gold F2 have different fatigue resistance in both static and dynamic motion. PTG was significantly more resistant to cyclic fatigue fracture compared to PTU.

In figure 5, Among PTU Files PTU-D was significantly better in average of cyclic fatigue resistance than PTU-S files. While for PTG files, PTG-D was significantly better in average of cyclic fatigue resistance than PTG-S. This indicates that heat treatment of NiTi files improves cyclic fatigue resistance in both static and dynamic motions, as shown in figures and 2, comparing PTU with PTG.

The SEM topographic examination revealed the typical cyclic failure mode appearances. Cross-sectional views of specimens from cyclic fatigue tests revealed crack initiation area(s), crack propagation, and overloaded fast fracture zone(s).

However, in longitudinal views, PTG showed less microcrack near the fracture line than PTU, which could be attributed to the surface treatment.

Table 1: Univariate analysis to explain NCF by Method and type of Alloy

Tests of Between-Subjects Effects

Dependent Variable: NCFG

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	20136881.250 ^a	3	6712293.750	179.328	<.001
Intercept	65552853.750	1	65552853.750	1751.337	<.001
Method	8077670.417	1	8077670.417	215.806	<.001
Alloy	11012450.417	1	11012450.417	294.213	<.001
Method * Groups	1046760.417	1	1046760.417	27.966	<.001
Error	2096090.000	56	37430.179		
Total	87785825.000	60			
Corrected Total	22232971.250	59			

a. R Squared = .906 (Adjusted R Squared = .901)

The fitness of the model was approved by the correction of the model ($F = 179.328$, $df = 3$ and p -value <0.001). The interpretation of the variation of NCF is 90.6%. The Model revealed that there is a significant difference between method and type of Alloy.

Table 2 Cyclic fatigue resistance and Fracture fragment length (mm) of ProTaper Universal and ProTaper Gold F2 files (Mean \pm SD).

Testing method	Groups	Number of cycles to failure (NCF)	Fracture fragment length (mm)
Static cyclic fatigue	PTU-S	382 \pm 43	3.35 \pm 0.43
	PTG-S	974.66 \pm 93	3.24 \pm 0.18
Dynamic Cyclic fatigue	PTU-D	852 \pm 149	3.13 \pm 0.67
	PTG-D	1973 \pm 614	3.29 \pm 0.24
	P-Value	.001	.534

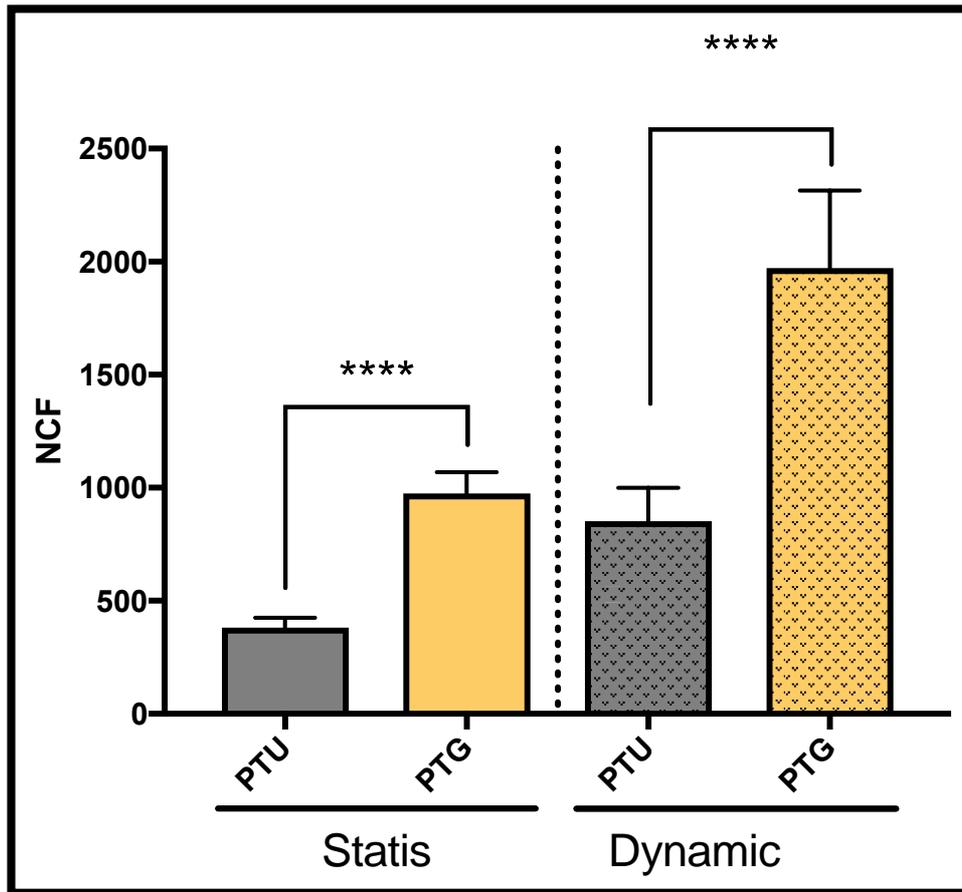


Figure 5. Show comparison between ProTaper Universal and instrument in number of cycles to failure (NCF) in static and dynamic motion.

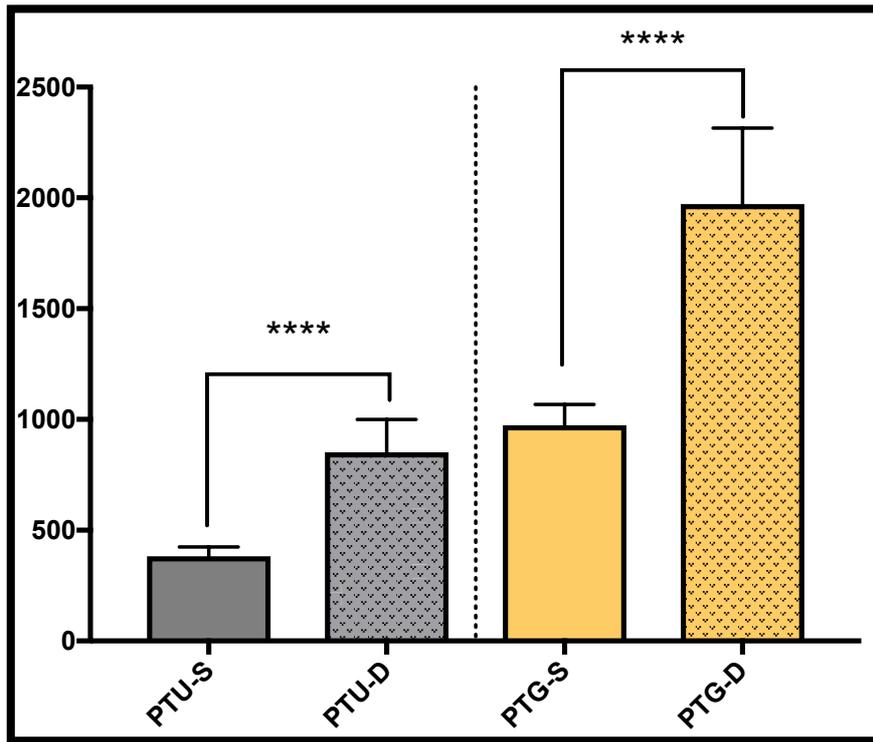


Figure 6. Show comparison between ProTaper Universal and ProTaper Gold F2 NiTi instrument in fractured fragment length (mm) in static and dynamic motion.

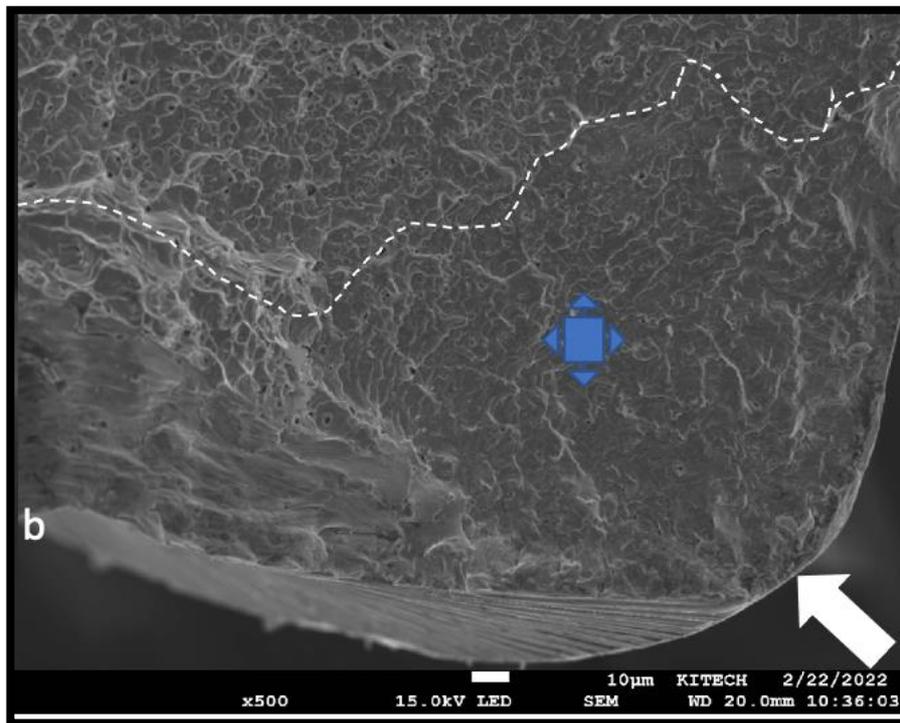
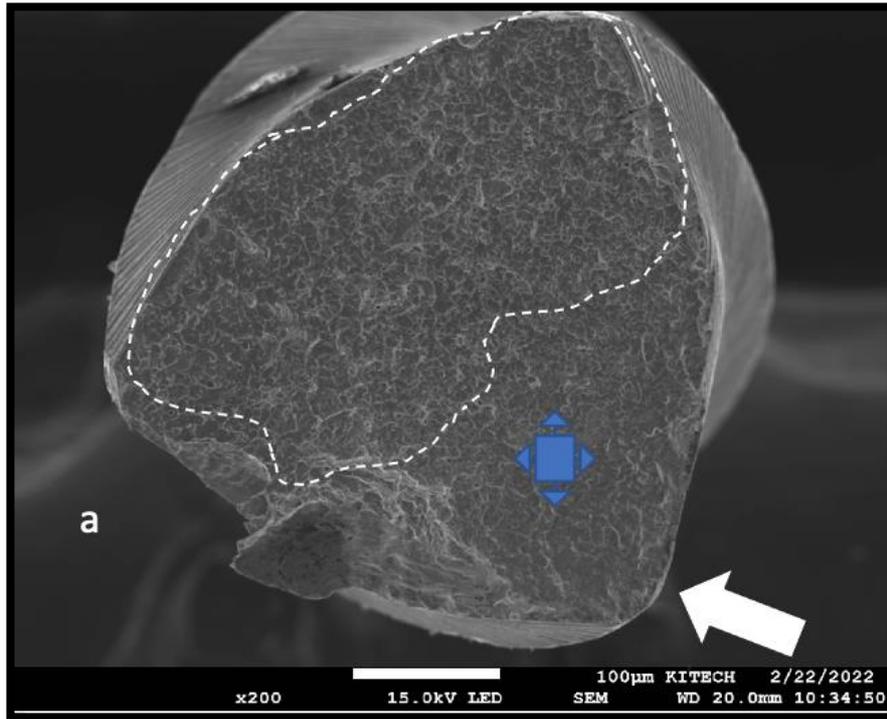


Figure 7: Representative scanning electron microscope pictures of the specimens after cyclic fatigue test. Cross-sectional aspects of PTU (a, b) files showed typical fatigue fracture including crack initiation areas (white arrow indicated) and overloaded fast fracture zone (Fatigue zone; slow fracture area. White dotted area; overloaded fast fracture zone) ◆

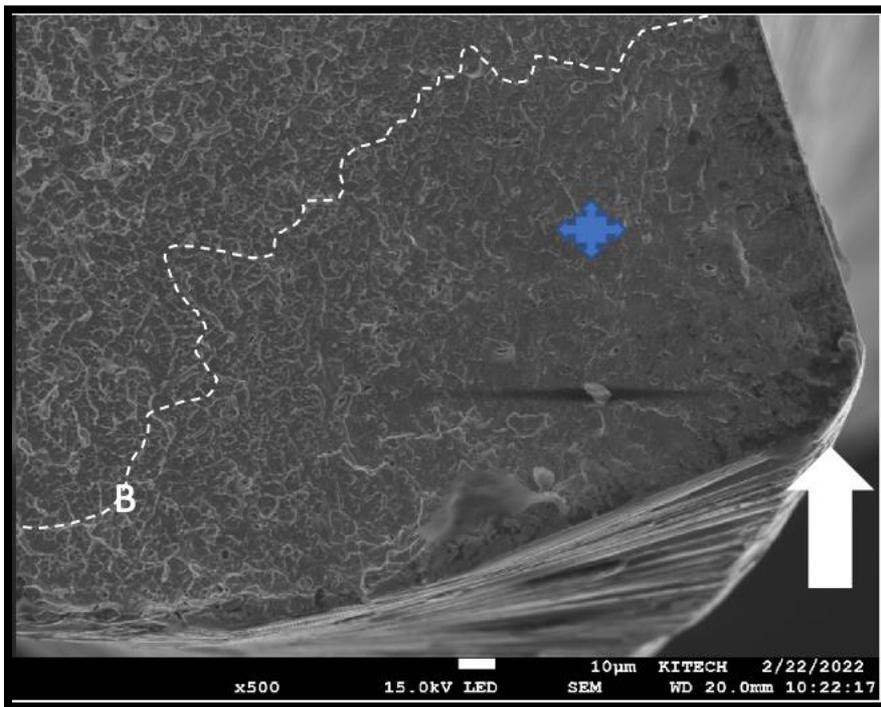
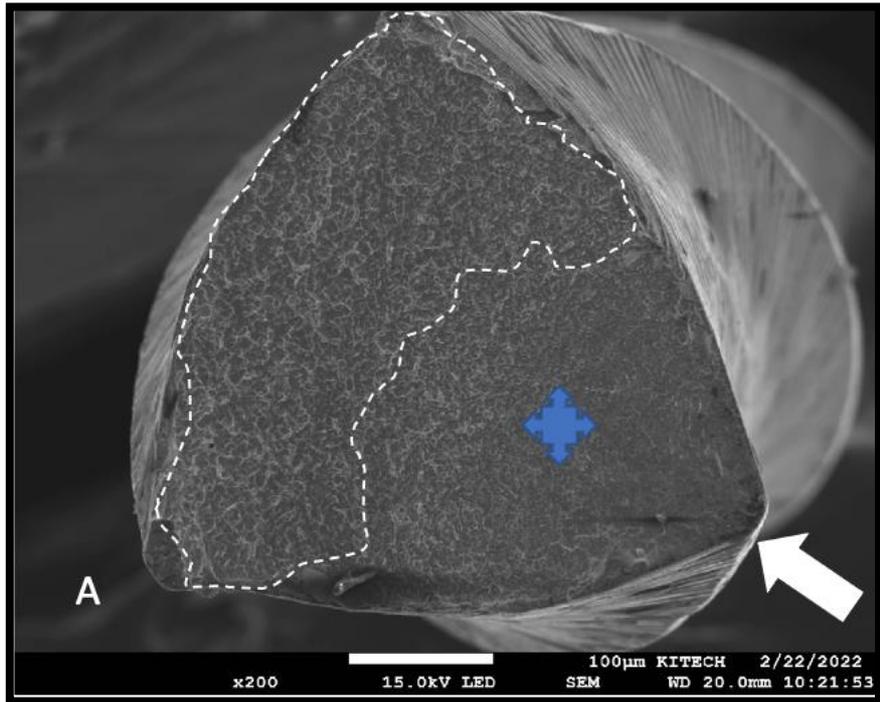


Figure 8: Representative scanning electron microscope pictures of the PTG specimens after cyclic fatigue test. Cross-sectional aspects of PTG (A, B) files showed typical fatigue fracture (B magnified from A) including crack initiation areas (white arrow indicated) and overloaded fast fracture zone (Fatigue zone; slow fracture area . White dotted area; overloaded fast fracture zone)



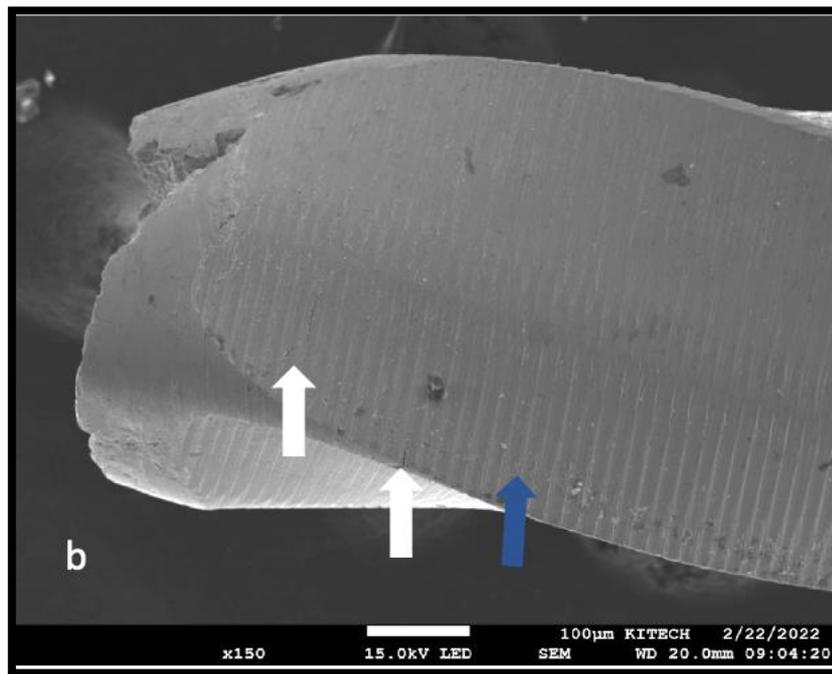
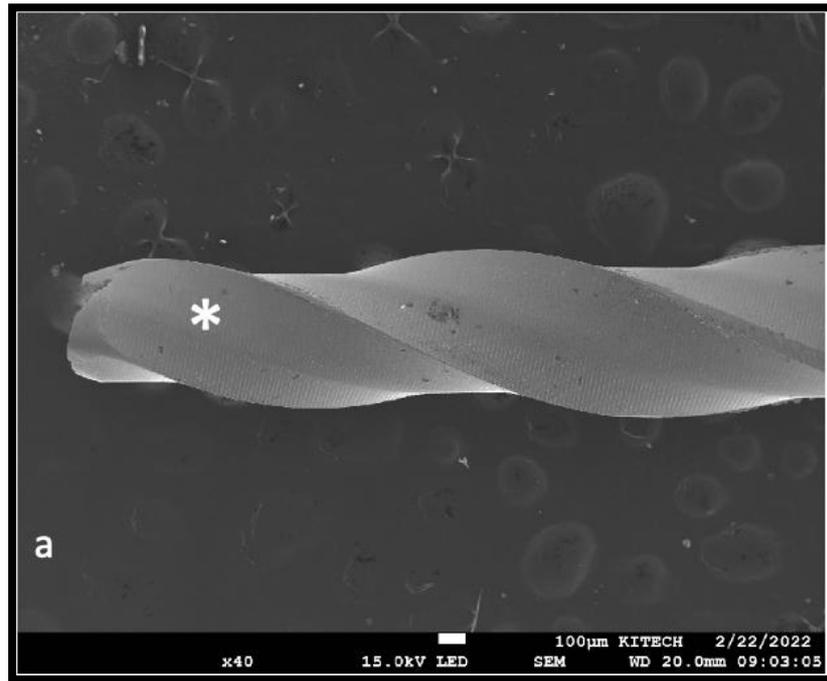


Figure 7: The longitudinal section of SEM micro images of the specimens after cyclic fatigue testing in magnification (X40, X150). PTU (a, b). The white arrows pointed to microcracks. The blue arrow pointed to machining groove.

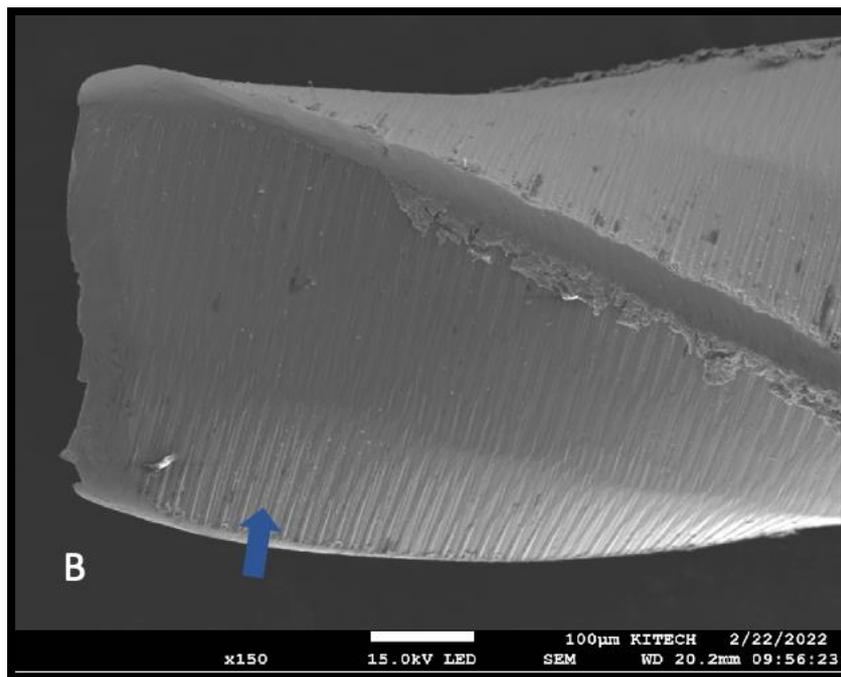
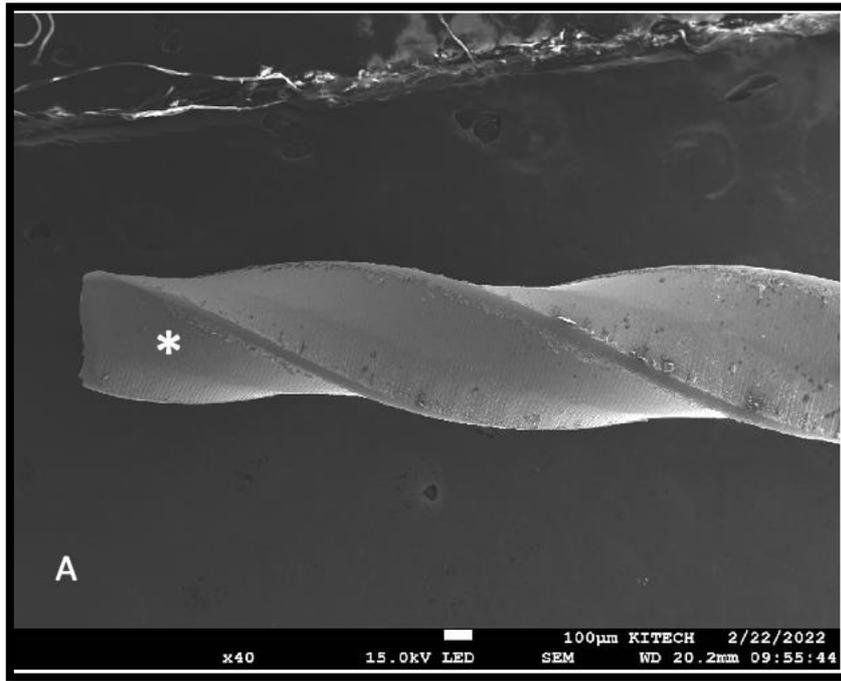


Figure10: The longitudinal section of SEM micro images of the specimens after cyclic fatigue testing in magnification (X40, X150). PTG (A, B). The blow arrow pointed the machining groove

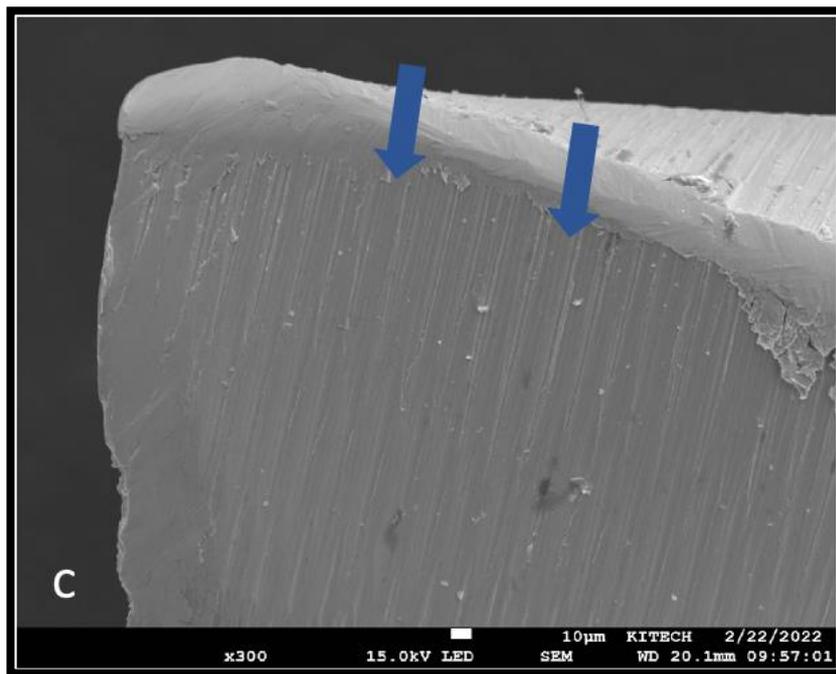
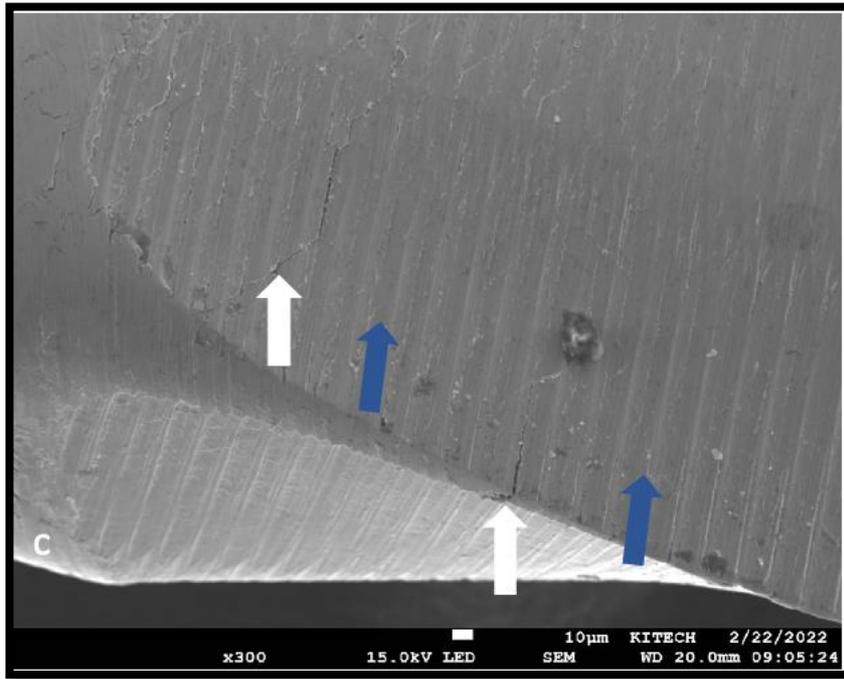


Figure10: The longitudinal section of SEM micro images of the specimens after cyclic fatigue testing in magnification (X300). PTU (c) and PTG(C). The white arrows pointed to microcracks. The blow arrow pointed to machining groove

5. DISCUSSION

Nickel-titanium (NiTi) rotary instruments have been increasingly used in endodontic practice due to their superior capacity to shape root canals with fewer procedural errors compared to stainless steel files⁶². However, a major drawback with these files is their susceptibility to distortion and fracture⁹. The two most common causes of rotary instrument fracture are torsional failure and cyclic fatigue fracture.^{48,71} When a metal is subjected to repeated cycles of tension and compression, its structure breaks down (as a result of stress concentration at the propagating crack front) and the metal eventually fractures⁷². The structural characteristics and geometric designs have a significant impact on the mechanical susceptibility of NiTi instruments⁷³. The main human factor governing the potential for NiTi file fracture is the clinician's handling⁷⁴. Aside from geometric configuration, a number of researchers have emphasized the significance of surface texture, such as machining marks and scratches from manufacturing procedures^{66,75}. These machining scratches on the instrument surface may act as local stress raisers or even crack-like features that may become the origin of a fatigue crack, whereas a smooth surface is less likely to initiate a fatigue crack⁷⁶.

Instrumentation of curved canals in natural teeth would be an ideal model. However, teeth can be only used once making standardization of experimental conditions impossible. Therefore, the use of standardized artificial canals for cyclic fatigue testing can minimize the influence of other variables not related to the tested instrument itself. The bulk of the tests on the cyclic fatigue resistance of NiTi files were conducted in artificial canals^{77,78} to ensure a certain degree of standardization by minimizing the anatomic heterogeneity that could result from natural teeth. In this study, standardization was achieved by testing both file systems using the same artificial canal at the same conditions with the same activation settings to exclude any contributing factors other than the heat treatment of the files.

To simulate the clinical situation, the cyclic fatigue testing was performed in the dynamic rather than static mode using a custom-made device with heat element device to maintain body temperature at 37° C. During cyclic fatigue testing, friction can develop between the instrument and the walls of the canals, which may affect the cyclic fatigue resistance²⁰. Therefore, it was recommended that a lubricant or coolant be used during the testing process¹⁷. The use of synthetic oil prevented the elevation of temperature. According to a study carried out by Nguyen et al., the temperature did not exceed 3 °C when the files were tested in the simulated canal at 300 and 500 rpm⁷⁹. Most of the studies evaluated the cyclic fatigue resistance of NiTi instruments at room temperature^{80,81,82}. Moreover, during clinical preparation of the root canal, the intracanal temperature ranges between 31 °C and 35 °C^{69,83}. In terms of the role of body temperatures in the clinical performance of NiTi files (heat treated and non-heat treated), this study investigated the cyclic fatigue resistance of NiTi instruments using a heating element to simulate the body temperature. Few studies have recently shown that the environmental temperature has a significant impact on the cyclic fatigue resistance of NiTi instruments^{70,84,85}

Despite the fact that the PTG and PTU systems have the same design and operation, the instruments' different manufacturing processes can affect their stress-strain distribution patterns and fatigue resistance behaviors. For rotary ProTaper files, cyclic failure is more common than torque failure. It is critical for clinicians to understand the differences between the PTG and PTU files in order to take advantage of the most recent technology and make the best decisions to meet anatomic challenges.

Comparing the cyclic fatigue resistance between non-heat treated PTU NiTi files and heat-treated PTG files in static and dynamic motion showed that PTG had a greater number of cycles to fracture than PTU in both testing modes. The presence of these martensite variants, which can be related to the high transformation temperatures found in PTG files, explains the

differences in fatigue resistance between the PTG and PTU files. Additionally, the cyclic fatigue resistance of both PTU and PTG instruments differed depending on the mode of testing (static vs. dynamic). Our findings demonstrated that the dynamic testing had a significantly higher number of cycles to fracture, regardless to the file system. These findings are similar to those reported by Li et al⁶². Because the endodontic instrument is not moved axially during the static test, the alternating compressive and tensile stresses are concentrated in one area of the instrument. These stresses build up over time, causing microstructural changes in the metal alloy. The current findings supported that concentrating stress in the same area of the instrument shaft reduced the number of cycles required for fracture. Compressive and tensile stresses are distributed along the tapered helical shaft of the instruments during the dynamic test, owing to the axial movement of the instruments within the curved canal. As a result, the fatigue fracture resistance was improved by avoiding stress concentration in the same instrument area. The current findings, as well as those of Li et al⁶², suggest that this principle could be applied to other instruments.

Scanning electron microscopy (SEM) was used in this study to examine the fractured surface of rotary instruments. Crack initiation area(s), crack propagation, and an overloaded fast fracture zone were observed in cross section Cyclic fatigue test specimens(s). In lateral view, however, the majority of PTU specimens had more surface microcracks near the fracture site, whereas PTG specimens had fewer microcracks. It could be because Martensite has a higher fatigue-crack growth resistance than Austenite due to thermal treatment during alloy production, which results in a better arrangement of the crystal structure and changes in the relative percentage of phases present in the alloy. Another reason for fatigue resistance is that the martensitic phase transformation has damping properties due to the energy absorption characteristics of its twinned phase structure, which makes crack propagation more difficult due to the greater number of interfaces present.

6. CONCLUSIONS

1. In dynamic mode, both files (PTU and PTG) demonstrated significantly higher resistance compared to static motion.
2. PTG files were significantly more resistant to cyclic fatigue fracture in static and dynamic motion than PTU files. This means that heat-treated files were significantly more resistant to static and dynamic motions at body temperature. We reject the null hypothesis (no difference in cyclic fatigue resistance between ProTaper Gold and ProTaper Universal in static or dynamic modes of testing) based on our findings, but there was no significant difference in terms of ratio of cyclic fatigue resistance between files in either mode of testing.
3. The SEM images of files' cross sections revealed that there were no significant differences in topographic appearance between PTG files and PTU files. However, longitudinal sections revealed fewer microcracks in PTG, which could be attributed to differences in metallurgy due to heat treatment processes.

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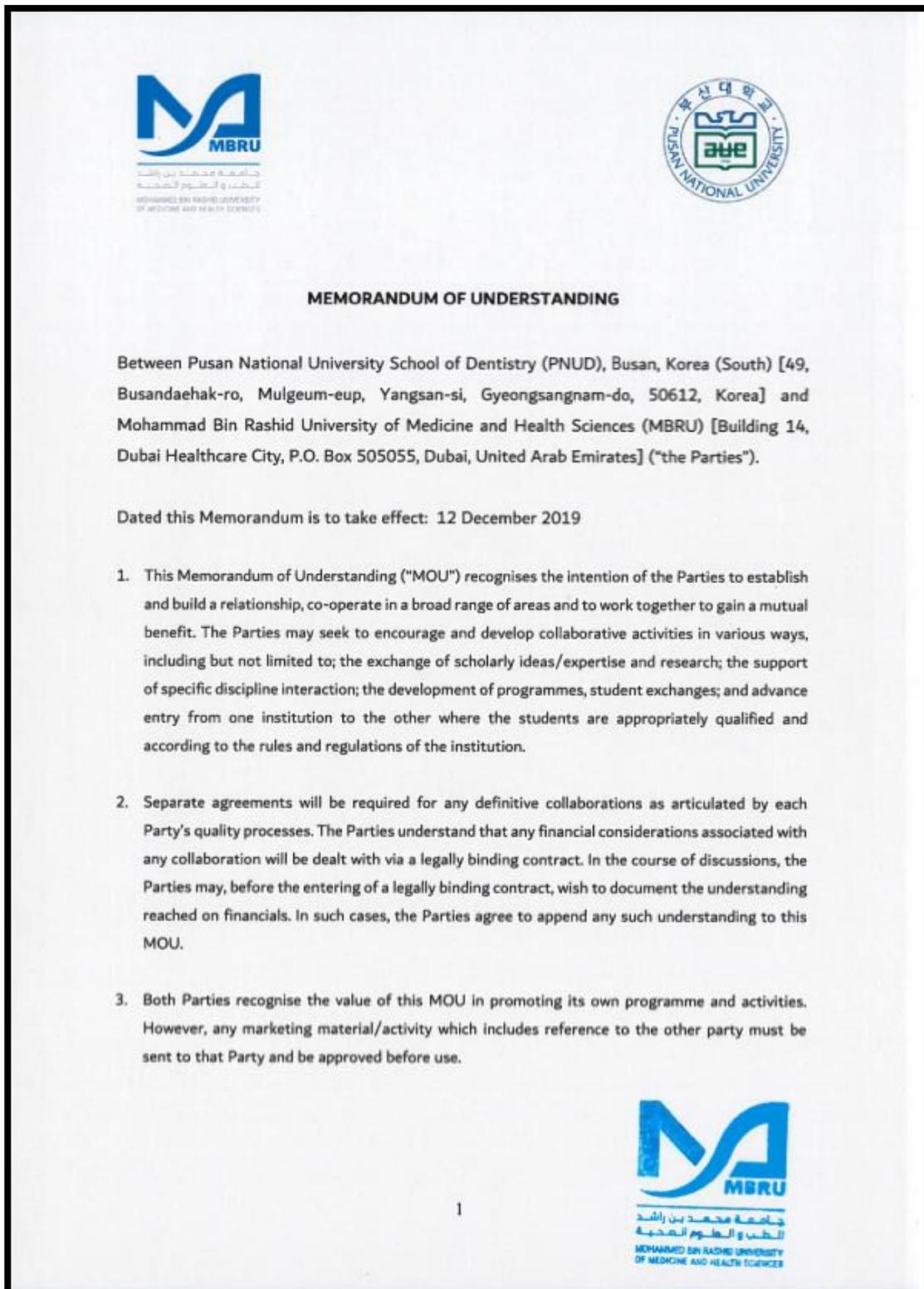
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8. APPENDICES

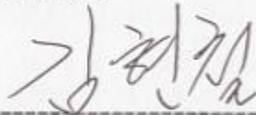
APPENDIX 1



4. This MOU is for 1 year in the first instance and will be reviewed thereafter. Each Party has the right to discontinue the arrangements subject to a period of 3 months' notice to be given. This MoU may also be terminated at any time by mutual consent of both Parties.
5. In the event of termination, the Parties will honour any agreed commitments either via existing agreed arrangements or by suitable negotiated alternatives.
6. The Parties acknowledge that during the term of this MOU, it may be necessary for either Party (the "Disclosing Party") to disclose to the other (the "Receiving Party") certain confidential information including business, marketing, technical, scientific or other information which is disclosed in circumstances of confidence, or would be understood by the Institutions exercising reasonable business judgment, to be confidential in nature ("Confidential Information"). The Receiving Party shall not, during or at any time after the expiry or termination of this Agreement, disclose, transfer, use, comply, or allow access to any such Confidential Information to any third parties, except as authorized by the Disclosing Party, or as required by law.
7. The Parties agree that neither Party will make and claim against the other for any loss or damage including but not limited to any consequential damages or lost profits, arising from any discussions, actions taken in reliance on this MOU or for termination of the negotiations without reaching a comprehensive agreement.

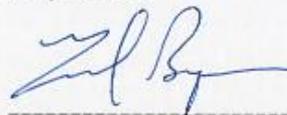
Signatures to the Agreement

Professor Hyeon-Cheol Kim
Dean, PNUD



For and on behalf of Pusan National
University School of Dentistry

Professor Zaid Baqain
Dean, HBMCMDM



For and on behalf of HBMCMDM, MBRU