THE EFFECT OF USAGE AND AUTOCLAVING ON THE CYCLIC FATIGUE RESISTANCE OF HEAT-TREATED NICKEL-TITANIUM INSTRUMENT

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ABSTRACT

Effect of usage and autoclaving on the cyclic fatigue resistance of heat-treated nickel-titanium instrument

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Background: Nickel-titanium (NiTi) instruments are often reused in endodontic treatment for economic reasons. Thus, cleaning, disinfection, and sterilization of these endodontic instruments are essential. Several studies have shown that additional heat treatment during the sterilization of NiTi instruments affects their physical properties. Some researchers concluded that sterilization has a detrimental impact on the physical properties of NiTi files. However, others found that sterilization for up to 10 cycles does not increase the risk of NiTi failure.

Aim: To evaluate the effect of usage and sterilization on the cyclic fatigue resistance of a heat-treated NiTi instrument (ProTaper Gold).

Materials and Methods: Thirty ProTaper Gold NiTi F2 files (length 25 mm) were used in this study. The files were assigned to three groups: Group I, new files (control group); Group II, files that underwent autoclave sterilization at 134 °C for 30 mins; and Group III, files that were prepared in a standardized J-shaped endo block followed by autoclave sterilization. Each NiTi file was rotated in an artificial curved metal block using a custom-made device in a repetitive up and down motion in a dynamic mode until fracture occur. The duration taken for the fracture to occur was recorded and translated into a number of cycles to failure (NCF). A digital micro-caliper was used to measure the length of the broken fragment. Following the cyclic fatigue test, all fractured fragments were examined under a scanning electron microscope to determine the topographic characteristics of the damaged surfaces. The data was analyzed using SPSS. The NCF was compared across the three groups using ANOVA, and fragment length using the Kruskal-Wallis test. A *p*-value of 0.05, was considered significant in all experiments.

Results: There was no statistically significant difference in the number of cycles to failure (NCF) among the three classes (P = 0.292.). Scanning electron microscopy revealed the typical features of cyclic fatigue failure of each fracture surface.

Conclusion: Under the limitations of this analysis, the findings provide precursory evidence that single canal file use and a single autoclave sterilization cycle do not affect the cyclic fatigue resistance of heat-treated nickel-titanium endodontic instrument (ProTaper Gold).

DEDICATION

This dissertation is dedicated to my beloved parents (Abdalla and Alya) and my dear husband (Abdalla) 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.

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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.

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Signature:

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1. INTRODUCTION

Cleaning and shaping root canal systems is critical stage in root canal therapy [1]. Nickel-titanium (NiTi) rotary instruments have been a centerpiece in therapeutic endodontics since their invention in 1988 owing to their remarkable ability to form root canals with possibly fewer procedural problems [2]. They have many advantages over manual stainless steel files, including faster set-up time, higher cutting performance, and clinical centering capability [3]. Despite being more stable and resilient than stainless steel files, NiTi rotary instruments are prone to deformation and fracture [4]. The two main causes of NiTi instrument fracturing, according to Sattapan et al., are cyclic fatigue, which occurs most widely in curved root canals when the instrument rotates freely throughout the curvature without bending, concentrating compression and tensile stress at the point of maximum flexure before a fracture occurs, and torsional stress caused when the instrument's tip is locked in the canal and the shaft continues to rotate [5]. A new thermomechanical treated alloy was recently introduced to the market. The primary benefit of heat treatment is that it increases the austenite finish (Af) temperature of the alloy. If the value for "Af" in intracanal temperature is greater than body temperature, the file would have a martensitic, R-phase, and austenitic composition. As a result, the heat-treated instruments demonstrated significantly increased flexibility and flexural fatigue resistance [6]. In general, previous research explored the design properties of files and the alloy composition, as well as their effect on cyclic fatigue resistance [3,7,8]. For economic reasons, NiTi files are usually reused during clinical procedures, followed by repeated autoclave sterilization[9]. According to

Yared et al., rotary files can be used safely in up to ten curved canals. As a result, it is critical to determine how repeated sterilization procedures affect the NiTi instrument's stability, cutting performance, and cyclic fatigue resistance [10].

2. REVIEW OF THE LITERATURE

2.1. Objectives of cleaning and shaping root canal system

Cleaning and shaping the root canal system, which is considered one of the most critical steps of root canal treatment, is essential for the effectiveness of root canal treatment [1]. It entails enlarging and forming the intricate endodontic space, as well as disinfecting it [1]. Endodontic failure is thought to be caused by a persistent microbial infection within the root canal system, according to Kakehashi 1965 and Siqueira (2001). As a result, extensive chemo-mechanical preparation is needed for effective root canal treatment [11,12.]

The aim of Schilder's root canal cleaning and shaping concept was to construct a three-dimensional continuous funnel tapering in multiple planes with adequate apical enlargement while maintaining foramen position and size [13]. These objectives were divided into mechanical and biological objectives. The mechanical objectives were to create a continuous tapering funnel from the access cavity to the apical foramen, to maintain initial course of the main canal during root canal preparation, the apical foramen should remain in its original position, and the apical opening should be kept as small as practical. The biological objectives were to confine instrumentation to the root canals only, ensure that no necrotic or instrumentation debris pushed beyond the apical foramina, optimum root canal debridement, and eventually the creation of a sufficient space for intracanal medications [13].

Young et al. explored the concept of chemo-mechanical planning in 2007. Biological objectives include removing microorganisms from the root canal system, removing pulp tissue that may promote microbial growth, and ensuring that debris does not pass the apical foramen, as this may induce inflammation [2]. The technical aims are to structure the canal in order to meet the biological goals and in order to place a high-quality root filling simpler [2].

2.2. Development of endodontic strategies

Regardless of the technique used in cleaning and shaping, the use of traditional steel instruments causes a variety of iatrogenic root damage. Among these are some preparation errors including elbow, zip, ledge, and perforation [14,15]. Several canal preparation techniques have been developed for this purpose. The step-back technique involves preparing the apical area of the root canal with smaller files first, followed by coronal flaring using larger files to allow obturation [16]. Crown-down techniques include using Gates-Glidden drills to widen the canal orifices, followed by manual files to remove organic canal material from the canal orifice to the apical part [16]. The crown-down method yields less apically extruded debris than the step-back method [18]. Using modified stainless-steel files, the balanced force technique allows the preparation of larger curved canals. NiTi rotary instruments have almost eliminated iatrogenic instrumentation issues that are often associated with endodontic steel instruments [17]. Peters et al. examined micro-computed tomography (μCT) scans before and after mechanical instrumentation in 2001. They found that 35% or more of root canal surfaces remained uninstrumented, independent of instrumentation technique [4]. In 2002, Tan and Messer reported that using rotary NiTi instrumentation resulted in a significantly cleaner canal in the apical 3 mm to a larger file size than hand instrumentation [18]. Paqué and Peter used μCT to compare the preparation efficacy of

ProTaper files and stainless-steel hand-files in the distal oval canals of lower molar teeth in an in vitro study conducted in 2011. Regardless of the files or methods used, they indicated that the sample's unprepared areas ranged from 59.6% to 79.9%, and they found no statistically significant difference between the groups. Furthermore, the increased volume observed in the root canal was consistent for all groups ($P < 0.001$)[19]. Hilal et al. (2015) used μCT to evaluate 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%[20]. Schäfer and Zapke (2018), on the other hand, identified an uninstrumented region with residual debris in all canals regardless of the preparation technique in a curved root canal of extracted human teeth using either rotary NiTi or stainless-steel hand files [21].

2.3. Endodontic instruments

2.3.1. Stainless steel instrument

The root canal instruments were made of carbon steel until 1960. Stainless steel alloys are now commonly used [22]. Canal shaping has traditionally been accomplished by hand using ISO-normed 0.02 tapered stainless steel tools [23]. Initial canal negotiation, defining an endodontic glide path, assessing working length radiographically or with the assistance of electronic apex locators, and checking patency are all still performed with stainless-steel manual files [24]. These instruments lack versatility, particularly in larger sizes, which can often result in procedural errors, resulting in a lower rate of endodontic treatment success [25]. Briseno and Sonnabend, in 1991, in an in vitro study, announced that no hand stainless-steel instrument produced ideal results, despite the results indicating that the nine instruments compared made clinically appropriate canal shapes [26].

2.3.2. Nickel-titanium alloy (NiTi)

In the early 1960s, W. F. Buehler worked at the Naval Ordnance Laboratory in Silver Springs, Maryland, USA, produced nickel-titanium alloys for space programs [27]. Andreasen and Hilleman were the first to use NiTi wires in orthodontics, noticing differences in the physical properties of Nitinol and stainless steel orthodontic wires, which enabled them to use lighter forces [28]. Walia, Brantley, and Gerstein developed endodontic files from nitinol orthodontic wires. As opposed to similar stainless-steel files, these files exhibited two to three times the elastic strength in bending and torsion, as well as superior resistance to torsional fracturing [29].

2.3.3. Basic metallurgy of nickel-titanium (NiTi)

The nickel-titanium alloy used in root canal therapy is composed of approximately 56% (wt.) nickel and 44% (wt.) titanium. As with other metallic systems, the resulting mixture has a one-to-one atomic ratio (equiatomic) of the significant components, and the alloy will exist in various crystallographic forms [27]. A common term used for these alloys is 55-Nitinol. It exists in two temperature-dependent crystal structures. At higher temperatures, Nitinol is in an austenitic state. At lower temperatures, Nitinol has a martensitic crystalline structure. These two distinct characteristics result from the transformation of austenite to martensite in the NiTi alloy. They are known as super-elasticity and shape memory effects. These are caused by temperature and stress [27,30].

Because of the super-elasticity of NiTi, deformations of up to 8% strain can be recovered. On the other hand, stainless steel can only tolerate a maximum pressure of less than 1% before permanent deformation occurs.

Copper-zinc alloys, copper-aluminum alloys, gold-cadmium alloys, and nickel-niobium alloys are examples of super-elastic alloys. However, none of these have the extent of strain or heat recovery, general corrosion resistance, human tissue compatibility, or the fluid body compatibility of nitinol [25, 27, 31].

2.3.4. 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 complications [2]. Several experiments have shown that rotary NiTi instruments retain the original canal curvature better than stainless steel hand instruments when used on an extracted human tooth [32]. In terms of NiTi rotary files' ability to shape, Short and Gluskin et al. reported that rotary NiTi instruments, particularly in the apical region of the root canal, maintain the original canal curvature better than stainless-steel hand instruments [33,34]. Esposito and Cunningham found that NiTi files were substantially more potent than stainless steel hand files in saving the initial canal course when the apical preparation was extended beyond ISO size 30 [35]. According to in vitro research, NiTi instruments yield considerably less straightening and more oriented practices than stainless steel hand files, reducing the likelihood of iatrogenic errors. Petiette et al. prepared 40 teeth with NiTi hand files or stainless-steel K-files and discovered that NiTi instrumentation preserved the initial canal shape better. When the investigators compared the two groups one year after the endodontic surgery, they found that teeth prepared with NiTi files had a slightly higher healing rate (as assessed by changes in the densitometric ratio) [36]. Tan and Messer discovered that using rotary NiTi instruments to instrument greater file sizes produced considerably cleaner canals in the apical 3 mm than hand instrumentation [18]. When root canals of extracted teeth are prepared with rotary NiTi instruments, many procedural errors are minimized, including lack of working volume, instrument divergence, canal transportation, zip or elbow shape, strip perforation, and unnecessary root weakening [37, 38].

Furthermore, when rotary nickel-titanium endodontic instruments are used, they have a higher success rate than when solely stainless-steel hand instruments are used [39]. In two recent studies, undergraduate dental students at the University of Jordan used NiTi rotary files to improve the overall technical performance of root canal fillings in posterior molar teeth. Their initial experience was more reliable and successful than doing it by hand [40]. Moreover, because of the high level of acceptance of the new technique, undergraduate students' knowledge and satisfaction were excellent, indicating the need for systematic incorporation of rotary NiTi instruments and methods for undergraduate teaching and future clinical practice [41].

2.3.5. Different generations of NiTi alloy.

The first commercially available nickel-titanium rotary files appeared in the market in the 1990s. The mechanical classification of each file system generation was addressed. [42].

2.3.5.1 First-generation files

Dr. John McSpadden produced the first rotary NiTi instrument with a 0.02 taper, which was released to the market in 1992 [42]. These instruments are in the austenitic phase with austenite finish (Af) (temperature below body temperature (16-31 ºC) and exhibit super-elastic properties [43]. By 1994, he developed the Profile Line with two different tapers, 0.04 0.06. They have fixed tapers and passively cut radial lands over the full working length, allowing the file to remain balanced in canal curvatures. Subsequently, LightSpeed rotary files were developed [42]. This generation of NiTi rotary instruments' main limitation is that it requires multiple files to achieve these goals and complexity[44].

2.3.5.2 Second-generation files

In 2001, the generation of NiTi rotary files hit the market [48]. 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 canal [42]. Under clinical conditions, they create super-elastic wire blocks with a solid martensite phase. Thermal processing in alloy manufacturing increases the crystal structure arrangement and changes the number of phases in the alloy. Heat treatment usually results in finely distributed NiTi particles in the matrix and an upward change in the Af of the alloy (austenite finish), resulting in a different crystallographic percentage of martensite and/or austenite near body temperature [43]. This generation of NiTi files includes the ProTaper rotary files, which have a tweaked guiding tip and multiple rising and decreasing taper [42].

2.3.5.3 Third-generation files

In the third generation, NiTi metallurgy has advanced. Heat treatment is one of the most effective methods for modifying the transition temperatures of NiTi alloys, thereby influencing the fatigue tolerance of NiTi endodontic files. Three different wires were fabricated depending on the thermodynamic treatment of the wire before or during manufacturing [43]. They are the M-wire, R-phase, and CM wire (controlled memory).

2.3.5.4 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 process [45]. 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 size [42]. This concept has spawned the fourth generation of canal-shaping instruments. In 2010, a self-adjusting file was introduced. WaveOne and Reciproc (VDW) were added as single file forming techniques. In both files, the M-wire was used [42].

2.3.5.5 Fifth-generation files

The fifth-generation files were constructed with a balanced center of mass or center of rotation [46]. These offset-designed files generate motion waves along the active section of the file. The offset design has the advantage of eliminating the taper lock or screwing effect between the file and dentin, which causes instrument separation [44].

The asymmetrical cross-section of the Revo-S NiTi rotary mechanism increases the usable volume for upward debris removal [45]. The Revo-S asymmetric cross-section allows for snake-like penetration and root canal shaping that is suited to biological and ergonomic requirements [44].

ProTaper Next (PTN) files feature progressive percentage tapers on a single file, as well as the 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.

2.3.6. Types of heat treatment wires

2.3.6.1 M-Wire

Dentsply Tusla Dental specialist launched the M-Wire file in 2007. It goes through a sequence of heat and cooling cycles, and this cycling mechanism helps to preserve the crystalline structure of Nitinol in its more martensitic state at body temperature. The M-wire included austenite, martensite, and R-phase [43]. M-Wire has an austenite finish temperature of 45–50 °C at room temperature; the temperature range for the phase transition means that M-Wire instruments are martensitic. The ProFile GT sequence X, ProFile Vortex, ProTaper Next files (PTN), PathFiles, WaveOne (WO), and Reciproc are all M-Wire instruments [43].

2.3.6.2 R Phase

SybronEndo developed the first fluted NiTi file in 2008, using a plastic deformation process similar to that used in manufacturing most stainless steel K-files and reamers

[42]. The aim of this new manufacturing process is to transform raw NiTi wire from the austenitic phase to the R-phase and to stabilize it at higher temperatures [43]. The R-phase has a smaller shear modulus than martensite and austenite, and at body temperature, it is entirely austensite [43].

2.3.6.3 CM Wire (Controlled memory NiTi wire)

The CM wire was first used in 2010. The thermomechanical method aimed to improve stability, increase transformation temperatures to approximately 50 °C, minimize shape memory, and achieve stable martensite at a body temperature [43]. According to Testarelli et al., CM contains less nickel (52 percent Ni wt.) than the more widely recognized NiTi [47]. A recent study by De Vasconcelos (2016) discovered that the austenite finish (Af) temperature of Hyflex CM and Typhoon CM are approximately 47 °C and 55 °C, respectively, leading him to the believe that this instrument at body temperature is a mixture of martensitic, R-phase, and an austenitic structure. This result supports a previous study, which discovered that instruments made of super-elastic NiTi show an austenitic process at room temperature. On the other hand, the MW and CM files are in the martensite and R-phase, as well as austenite [48].

2.3.6.4 Gold and Blue heat-treated wire

Dentsply International Inc. is a dental supply company in the United States. Vortex Blue, the first blue-colored endodontic instrument, was introduced by Tulsa Denta in 2011. There are two gold-heat-treated NiTi systems (ProTaper Gold and WaveOne Gold) and two blue-heat-treated NiTi systems (Vortex Blue and Reciproc Blue). These instruments have a controlled memory effect and can be deformed [56]. The main difference between the CM wire and the heat-treated gold and blue instruments is that these files are ground before being heat-treated after machining [48]. Compared to standard NiTi and M-Wire instruments, both gold and blue heat-treated files demonstrated improved flexibility and fatigue resistance, which could be attributed to their martensitic state. In terms of cyclic fatigue tolerance, only Hyflex EDM files outperformed ProTaper Gold, WaveOne Gold, and Reciproc Blue [21].

2.3.6.5 MaxWire

FKG Dentaire recently launched MaxWire (Martensite-Austensite-electropolish-flex), a new thermomechanical treated NiTi alloy. In clinical use, it possesses both shape memory and super-elasticity. Two of these techniques are the XP-endo Shaper and XP-endo Finisher. These instruments are straight in their martensitic state at room temperature, but they curve when subjected to intracanal temperature due to a phase change to an austenitic state. [21].

2.3.6.6 Summary of the new thermomechanical treatment for the NiTi alloy

A recent thermomechanical therapy for the NiTi alloy allows for a change in the phase structure under clinical conditions, resulting in the appearance of the martensite state. The martensitic instruments are more lightweight, have a higher cyclic fatigue resistance, and a larger angle of rotation. However, they have a lower torque at fracture and a larger angle of rotation than the ferrite instruments. These devices can be used for canals that are severely inclined or have a double curvature. They can be prebendable, which is advantageous for avoiding ledges [21].

2.3.7. Fracture of the NiTi instrument

Instrument fracture is a common problem in NiTi rotary files. However, NiTi rotary files are more versatile and durable than stainless steel files [4]. Separation can occur with no apparent evidence of prior irreversible deformation, simply beyond the elastic boundary of the file [49, 50], lowering the likelihood of achieving the ultimate goal of the targeted treatment [13]. The average clinical fracture frequency of rotary NiTi instruments, according to a literature review, is approximately 1.0%, with a range of 0.4–3.7%. The average prevalence of retained broken endodontic hand instruments (mostly stainless steel files) is approximately 1.6%, with a range of 0.7%–7.4% [49]. According to one study [58], NiTi rotary instrument fractures were seven times more common than hand instrument fractures in an endodontic residency program. Unfortunately, the literature presents conflicting results regarding the therapeutic value of preserving fracture files inside treated root canals. Nonetheless, evidence suggests that if the instrument fragments in some situations (teeth with necrotic pulps or teeth with periapical lesions), it would be more painful and unfortunate, as healing chances would be diminished [51]. The prognosis of a tooth undergoing root canal treatment is poor if the endodontic instrument is split in the apical third of the root canal or beyond the root canal curvature. Removal of the fragment becomes more difficult. The more curvature there is in the root canal, the more difficult it is to extract the fractured segment [52, 53].

2.3.8. Factors influencing the mechanism of NiTi instruments

For NiTi instruments to be used safely in clinics, it is essential to understand the basic fracture mechanisms and how they relate to canal anatomy. They are prone to structural fatigue when they are used in a rotary motion, which can lead to fracture if the file's resistance surpasses [54]. In 2000, Sattapan et al. identified two forms of fracture for rotary NiTi instruments: cyclic fatigue (when repeated compressive and tensile stresses work on the outer fibers of a file revolving in a curved canal) and torsional failure (when the tip of the instrument binds). Even then, the shank of the file continues to spin. In clinical trials, he found that torsional failure (56 percent vs. 44 percent) was more common than cyclic fatigue for a variety of applications of NiTi-based rotary files [5].

2.3.8.1 Torsional stress

Torsional stress fractures occur as the instrument tip or another part gets stuck in a canal when the shaft starts to rotate, and the elastic limit of the instrument is breached, resulting in plastic deformation (unwinding, reverse winding) and fracture [5]. It normally occurs when the user adds excessive apical force to the rotating instrument in the root canal, or when smaller diameter root canal instrumentation creates more torsional tension than larger diameter root canal instrumentation during the cleaning and shaping procedure [5, 55]. Torsional stress fractures may occur when the apex of a spinning instrument thrust into a narrow root canal. Due to the increased friction, a greater torque is required to rotate the file, putting excessive pressure on the fragile instrument tip [56]. Since it may occur with equally tapered instruments having different tip diameters rather than variably tapered instruments, this phenomenon is known as ‘taper lock’ [10]. The tip and taper of the instrument, as well as the size of the canal, influence torsional stress, and an increase in instrument diameter, as well as a corresponding increase in cross-sectional area, may result in increased resistance to torsional failure [49]. On the other hand, the clinician has power over endodontic instrument manipulation and therefore has the potential to mitigate the effects of torsional stress fractures [5].

2.3.8.2 Flexural/Cyclic fatigue

Cyclic fatigue occurs when an instrument rotates freely in a curved canal, causing tension/compression loops and ultimately a fracture. The instrument was rotated while it was in a static position. The outer half of the shaft was tensed, while the inner half was flattened. Due to the repetitive tension-compression period induced by rotating inside curved canals, the instrument's cyclic fatigue increases with time and can be a contributing factor to instrument fracturing [4, 57]. When the diameter of a rotary file increases, its tolerance to cyclic fatigue also increases. This is related to the metal mass of the instrument at the point of high tension. In vitro and scientifically, increasing the degree of the curve's angle and radius, around which the device rotates, reduces the instrument’s lifespan [58].

2.3.8.3 Other factors

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

A. Operator skill/experience: Barbakow and Lutz (1997) investigated the effects of operator experience [58], and Mandel et al. (1999) [59] discovered that proper experience and training is needed to reduce the occurrence of instrument separation [10].

B. 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 [60, 61].

C. 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 separation [10]. According to Kobayashi et al., the torque for ProFiles should be set between 40 and 80 Ncm to avoid instrument failure [55].

D. The number of times a NiTi rotary file is used decreases its resistance to flexural fatigue. As a result, they have a higher failure rate than new instruments [62].

E. Tooth anatomy: The mesial canals of mandibular and maxillary molars are where the majority of NiTi rotary instrument fractures occur. In comparison to the initial endodontic treatment, fractures were also normal in retreatment cases [50, 63].

F. Rotational speed: Instrument separation was less likely with a lower rotational speed [10]. On the other hand, manufacturers suggest a specific number of rotations per minute (rpm) for the safe use of rotary NiTi instruments, which is normally in the range of 250-600 rpm [64].

G. Instrument size and radius of curvature: Smaller NiTi instruments have a higher frequency of fracture and distortion, according to in vitro studies [65]. It has been shown that as the radius of curvature and angle of curvature decrease, the number of cycles required for the file to fracture also decreases [54].

H. Surface condition and instrument inspection: To avoid instrument fracture, the manufacturer of rotary NiTi instruments recommends that instruments be inspected for defects and microcracks on a regular basis [65, 66].

I. Sterilization: Several studies have found that after several sterilization/autoclave cycles, NiTi instruments reveal crack initiation and extension, as well as an increase in the depth of surface irregularities. There is also evidence of a decrease in cutting quality [67, 68, 69]. The negative effects of heat sterilization on the mechanical properties of NiTi files have been questioned, with other studies concluding that it has no impact on the incidence of NiTi instrument fractures [1,70, 9.]

Clinical steps to reduce torsional stress and flexural/cyclic fatigue are recommended (A-G) [49,39].

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.

2.4. Effect of usage and sterilization on fracture resistance of the instrument

Instrument reuse is common in dentistry, and endodontic treatment often requires reused NiTi instruments for economic reasons [71]. During endodontic instrumentation, different types of debris are encountered, such as vital tissue, dentin shaving, bacteria, blood, and necrotic tissue, along with the accumulation of debris on the instruments' flutes [72]. Transferring debris from one patient to another or the dental staff is unacceptable as they act as infecting agents, potentially some serious diseases, such as hepatitis. Efficient cleaning, disinfection, and sterilization of endodontic instruments are important [73]. Sterilization is a method of removing all viable microorganisms from an object, including viruses and bacterial spores. Sterilization of endodontic instruments is needed to protect patients and healthcare providers from cross-contamination. Endodontic devices may be sterilized using sponge and brush sterilization, ultrasonic cleaning, glass bead sterilization, dry heat sterilization, or autoclave sterilization [73]. Several studies have shown that additional heat treatment during NiTi instrument sterilization affects the physical properties of the instruments[74]. Some researchers concluded that sterilization has a detrimental impact on the physical properties of NiTi files and allows files to fail prematurely [75]. Other researchers have found that heat sterilization for up to 10 cycles does not increase the risk of NiTi failure risk [9]. Before reusing an old file or using a new one, NiTi files are sterilized, usually in an autoclave at temperatures above 100 °C [76]. although their properties remained unchanged, Plotino et al. discovered that repeated sterilization increased the cyclic fatigue resistance of only one of the four checked files (K3XF) [77]. Zhao et al. discovered that the HyFlex CM and K3XF files had a longer cyclic fatigue life than the other three files tested [79]. According to a recent analysis, autoclave sterilization had no impact on the physical and mechanical properties of most NiTi endodontic files [78].

2.5. ProTaper Gold

ProTaper Gold rotary files (Dentsply Sirona, Malliefer, Ballaigues, Switzerland) have the same geometries as ProTaper Universal, but have a 24% improvement in flexibility and up to 2.6 times greater resistance to cyclic fatigue, both of which are significant advantages that reduce the risk of separation. The technology behind these files involves a series of "Shaping" and "Finishing" files that produce ProTaper's predicted shape and a method that has been proved by science and is trusted by clinicians all over the world. The shaping files are designed to be used with the same familiar outstroke brushing technique to pre-enlarge canals. On the other hand, the finishing flies provide trusted deep shapes that facilitate 3D root canal cleaning and filling. Its shorter handle makes it easier to reach the teeth. The gradual taper and convex triangular cross-section increase the cutting action by reducing the rotational friction between the file and the dentin blade. The non-cutting tip of each instrument allows it to follow the protected portion of the canal securely, while the small flat area on the tip enhances its ability to pass through soft tissue and debris.

3. AIM

The aim of this study was to determine the effect of use and sterilization on the cyclic fatigue resistance of heat-treated nickel-titanium instrument (ProTaper Gold).

3.1. Null hypotheses

Heat-treated nickel-titanium instrument (ProTaper Gold) would have less cyclic fatigue resistance if used and sterilized.

4. MATERIALS AND METHODS

4.1. 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

$$SE = 1.24$$

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 α = 0.05, the minimum sample size was established at 10 (12.4/1.24) instruments for each group (n=10).

Thirty (n=30) ProTaper Gold NiTi files (PTG; Dentsply Sirona, Endodontics, Switzerland) (Figure 1) were used in this study. A size F2 length of 25 mm was selected—new files from the pack and never used. The files were examined under a dental operating microscope, and any defective instruments were discarded.

Figure 1: ProTaper Gold NiTi file (PTG; Dentsply Sirona, Endodontics, Switzerland). Size F2 length 25 mm

4.2. Investigation design

The thirty files were assigned to three groups:

Group I: New files (Control group).

Group II: Files to undergo autoclave sterilization only.

Autoclave sterilization using the STERIS Amsco® Century™ machine (STERIS Corporation, OH, USA) (Figure 2) at 134 oC for 30 minutes.

Group III: Files to undergo preparation in a standardized J-shaped endo block (Dentsply Maillefer; taper = 0.02, apical diameter = 0.15, length = 16.5 mm, angle of curvature = 35°) (Figure 3). Manual preparation using size 10, 15, and 20 K-files. This was followed by autoclave sterilization at 134 °C for 30 min.

They will be distributed as follow:

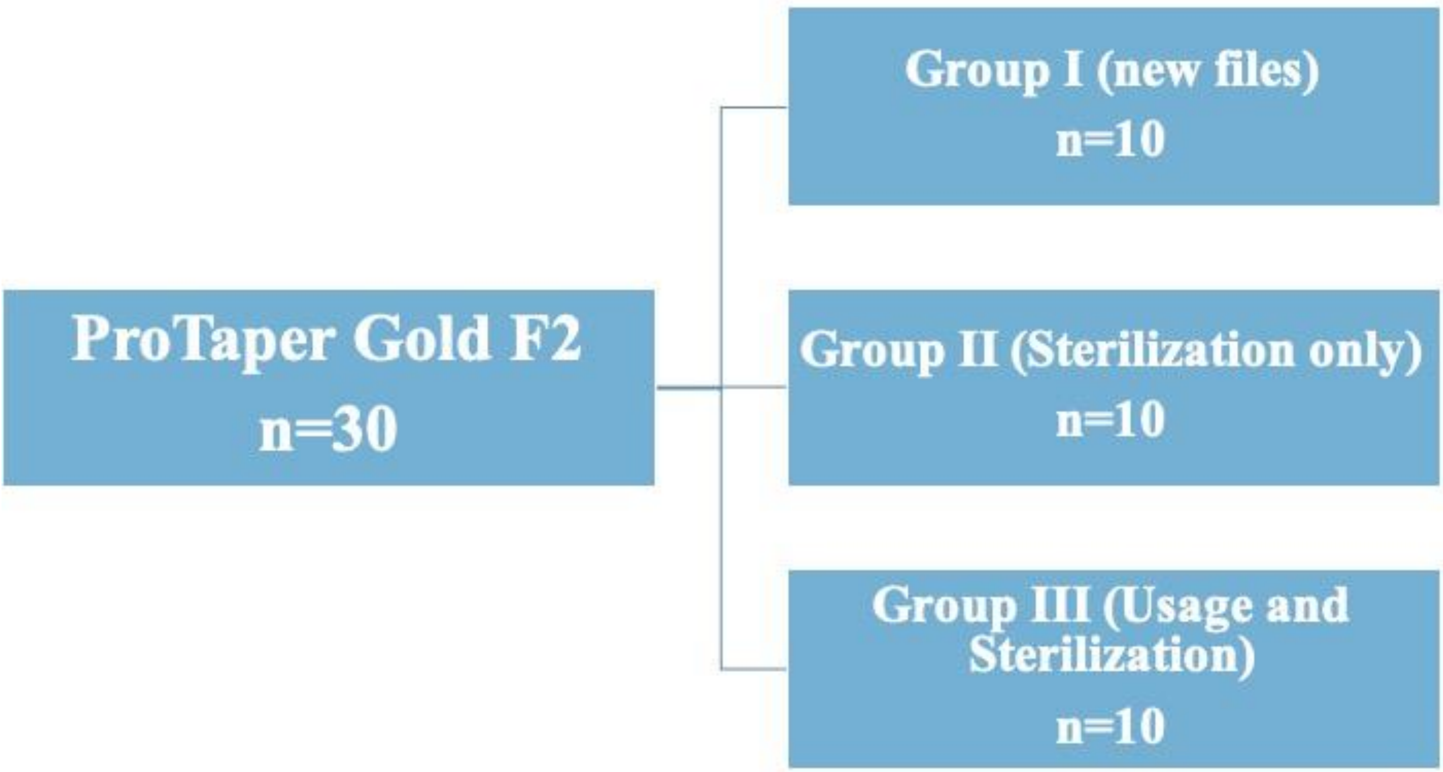

Figure 2: STERIS Amsco ® Century™ machine for autoclave sterilization.

Figure 3: J shaped endo block (Dentsply Sirona)

4.3. Experimental evaluation of cyclic fatigue resistance

Each NiTi file was rotated with a repetitive up-and-down movement in a curved canal using a custom-made device (EndoC, DMJ mechanism, Busan, Korea) with heat element device to maintain body temperature 37^{o}C (Figure 4).

The artificial tempered steel canal measured 17 mm in length, 6 mm in radius, and 35° in curvature (Figure 5). To reduce frictional tension between the canal wall and the NiTi file, synthetic oil (WD-40, WD-40 Company, San Diego, CA, USA) was sprayed into the canal before each drill. To mimic a clinical situation, a cyclic fatigue test was performed in the dynamic mode. The test consisted of a 4 mm displacement in each direction every 0.5 seconds, with a dwell time of 50 ms. Using a torque-controlled engine Dentsply Maillefer (X-smartTM), the file was freely rotated in the canal at a constant speed of 300 rpm. The duration of the fracture was recorded, and the duration was translated into the number of cycles to failure (NCF). A digital micro-caliper was used to determine the length of the fractured fragment (INSIZE Co, DKD, Germany).

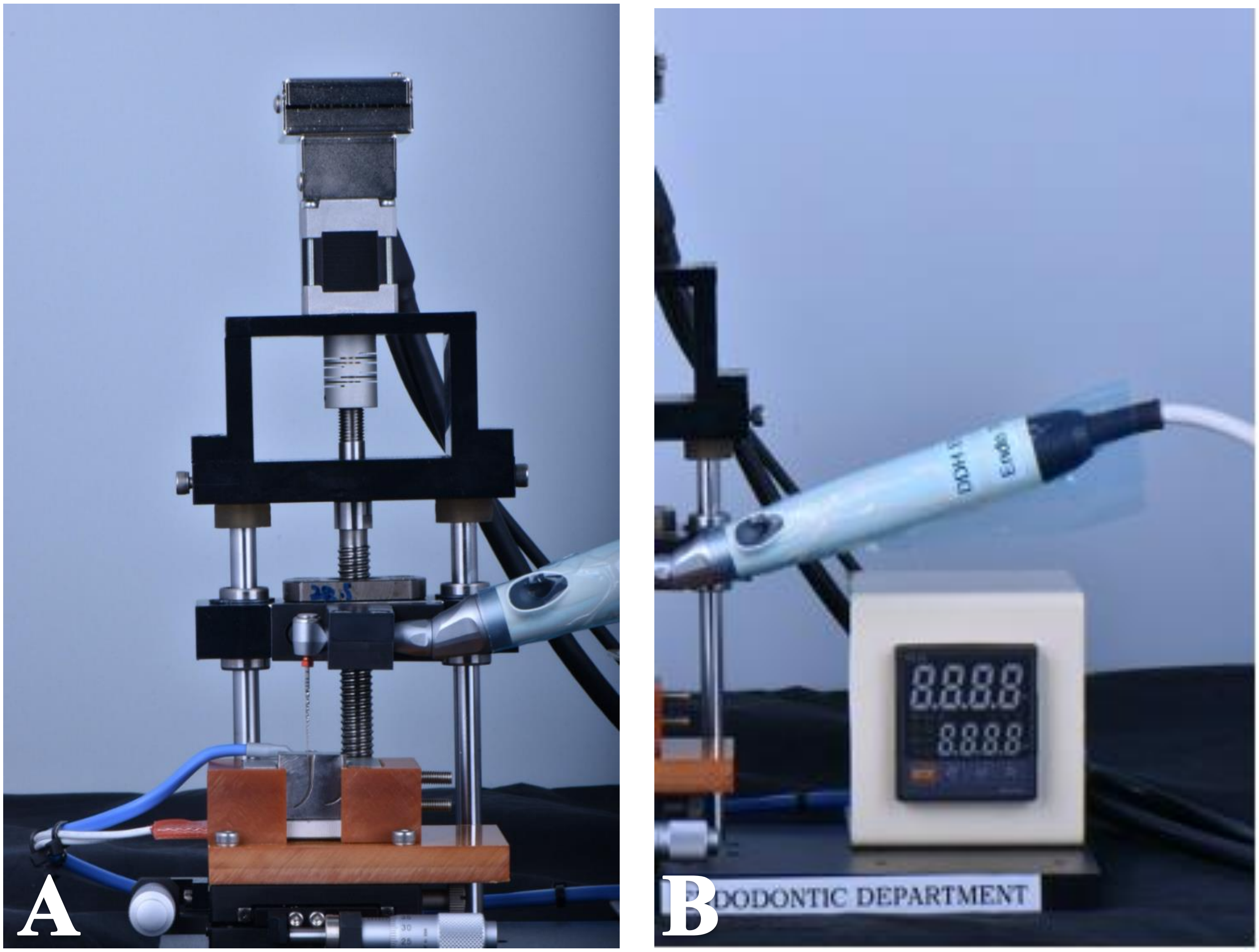

Figure 4: A: (EndoC: DMJ system, Busan, Korea) custom-made device for measuring cyclic fatigue resistance. B: Heat element device

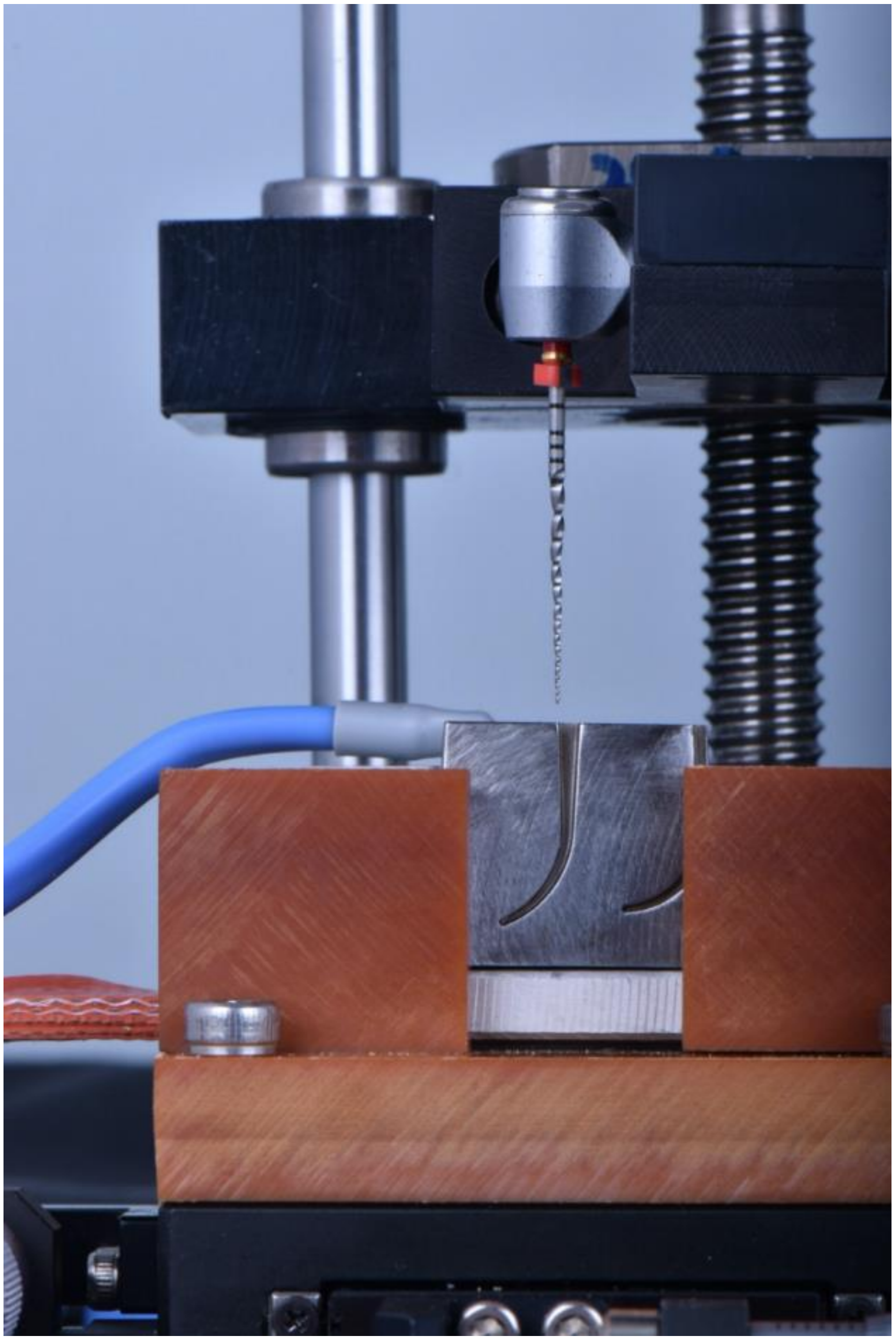

Figure 5: A tempered steel canal with a length of 17mm, a radius of 6 mm, and a curvature of 35°

4.4. 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 was compared using analysis of variance (ANOVA) between the three groups, while fragment length was compared using the Kruskal-Wallis test. In all experiments, a p-value of less than 0.05 was considered significant.

4.5. 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).

5. RESULTS

The cyclic fatigue resistance of the ProTaper Gold F2 NiTi instrument is listed in Table 1.

There is no statistically significant difference in the number of cycles to failure (NCF) between the three groups; the average NCF of group I was 1353.5 (389.6), group II was 1130 (265.7), and group III was 1192.5 (295.6), with a P-value of 0.292. The line chart in (Figure 6) depicts the number of cycles to failure (NCF) of the ProTaper Gold F2.

There is a significant difference in the fractured fragment length (mm) between the three groups; for group I was 3.49 (1.08), group II was 4.79(1.18), and group III was 4.4 (1.20214), with a P-value of .014. The line chart in (Figure 7) depicts the fractured fragment length (mm) of the ProTaper Gold F2.

The SEM topographic images of each fractured surface revealed the typical characteristics of cyclic fatigue failure (Figures 8, 9, and 10). The cross-sectional images in the ProTaper Gold F2 file revealed crack initiation zones, crack propagation, and an overloaded rapid fracture zone in the three classes. The surface of the files showed numerous machining grooves on the longitudinal surfaces of each SEM image.

Table 1: Cyclic fatigue resistance of ProTaper Gold F2 files (Mean ± SD).

Groups	***Number of cycles to failure (NCF)***	***Fracture fragment length (mm)***
Statistical test	*ANOVA*	*Kruskal-Wallis*
Group I	*1353.5 ± 389.6*	*3.49 ± 1.08*
Group II	*1130 ± 265.7*	*4.79 ± 1.18*
Group III	*1192.5 ± 295.6*	*4.4 ± 1.20214*
P-Value	*.292*	*.014*

A significant difference between the groups (P Value ≤ 0.05)

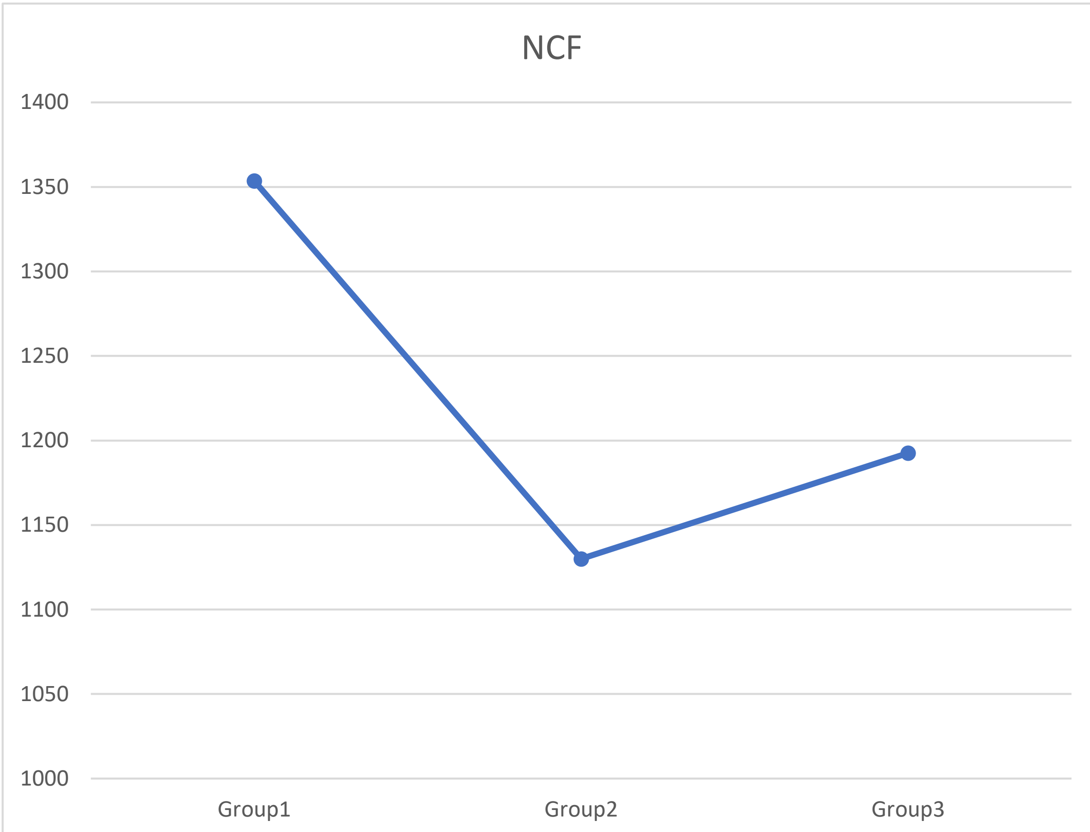

Figure 6: Line graph. The number of cycles to failure (NCF) of ProTaper Gold F2. The horizontal axis (X-axis) represents the groups, and the vertical axis (Y-axis) represents the number of cycles to failure

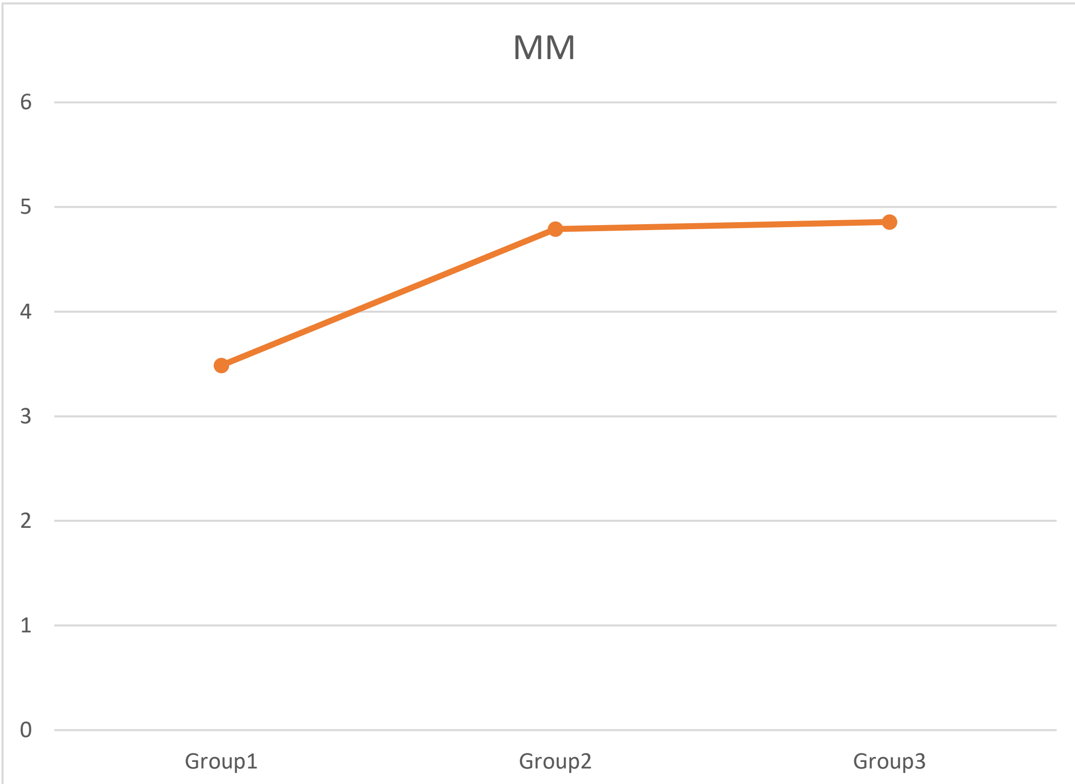

Figure 7: Line graph. The fractured fragment length (mm) of ProTaper Gold F2. The horizontal axis (X-axis) represents the groups, and the vertical axis (Y-axis) represents the fractured fragment length

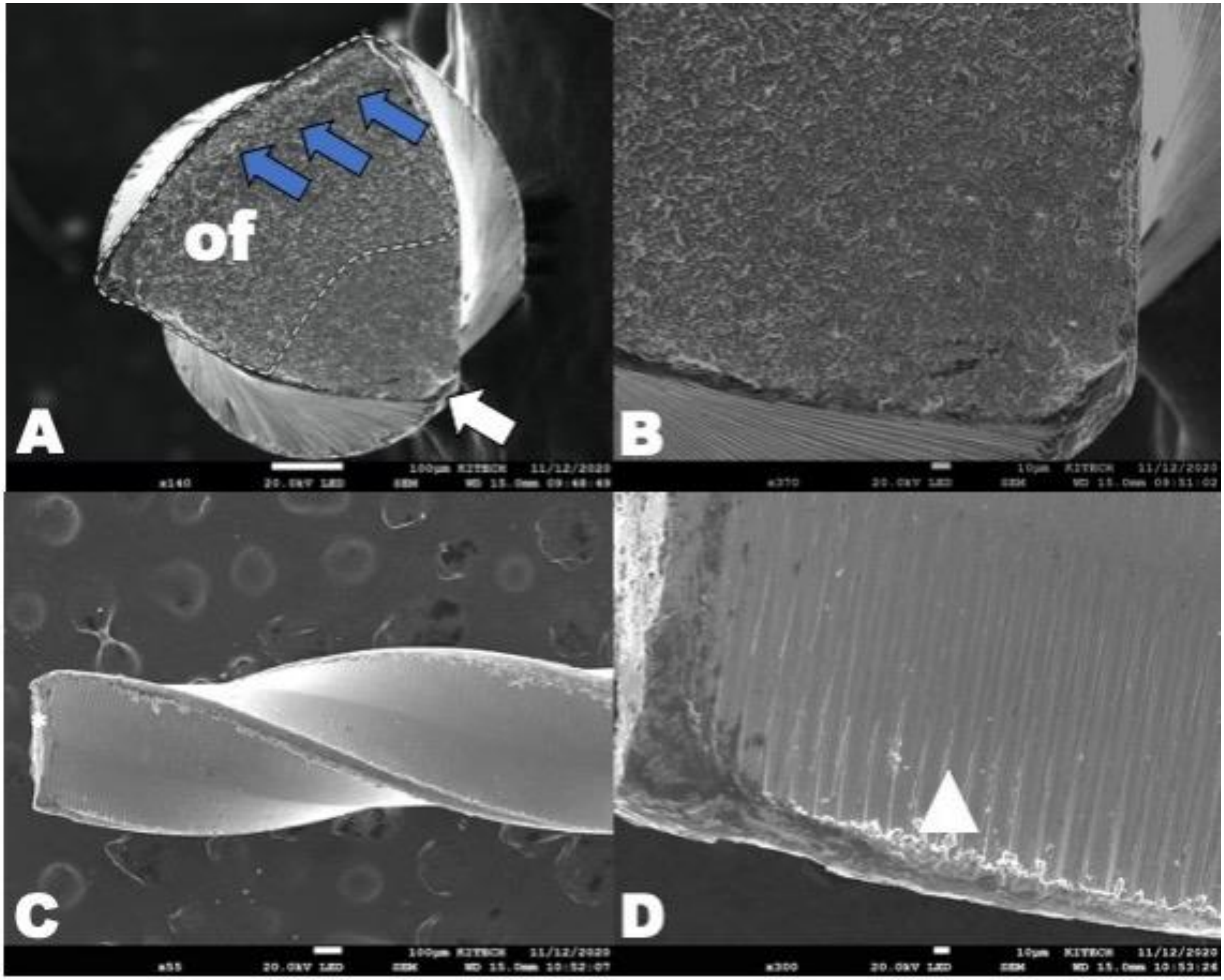

Figure 8: SEM images of the specimens after cyclic fatigue testing. PTG group I, cross-sectional view (A-B). The region of crack initiation is indicated by a white arrow. (Dashed line) The outline of the dimple region shows the fibrous zone of the overload fast fracture zone. Blue arrows denote a wave-like field (Rolled over zone). (B) A magnified view of (A) reveals an overload fast fracture (of) region with several ductile dimples. PTG phase I longitudinal view (C-D). (*) A micro-crack close to the fracture site. (D) A magnified version of (C). Machining grooves have been discovered.

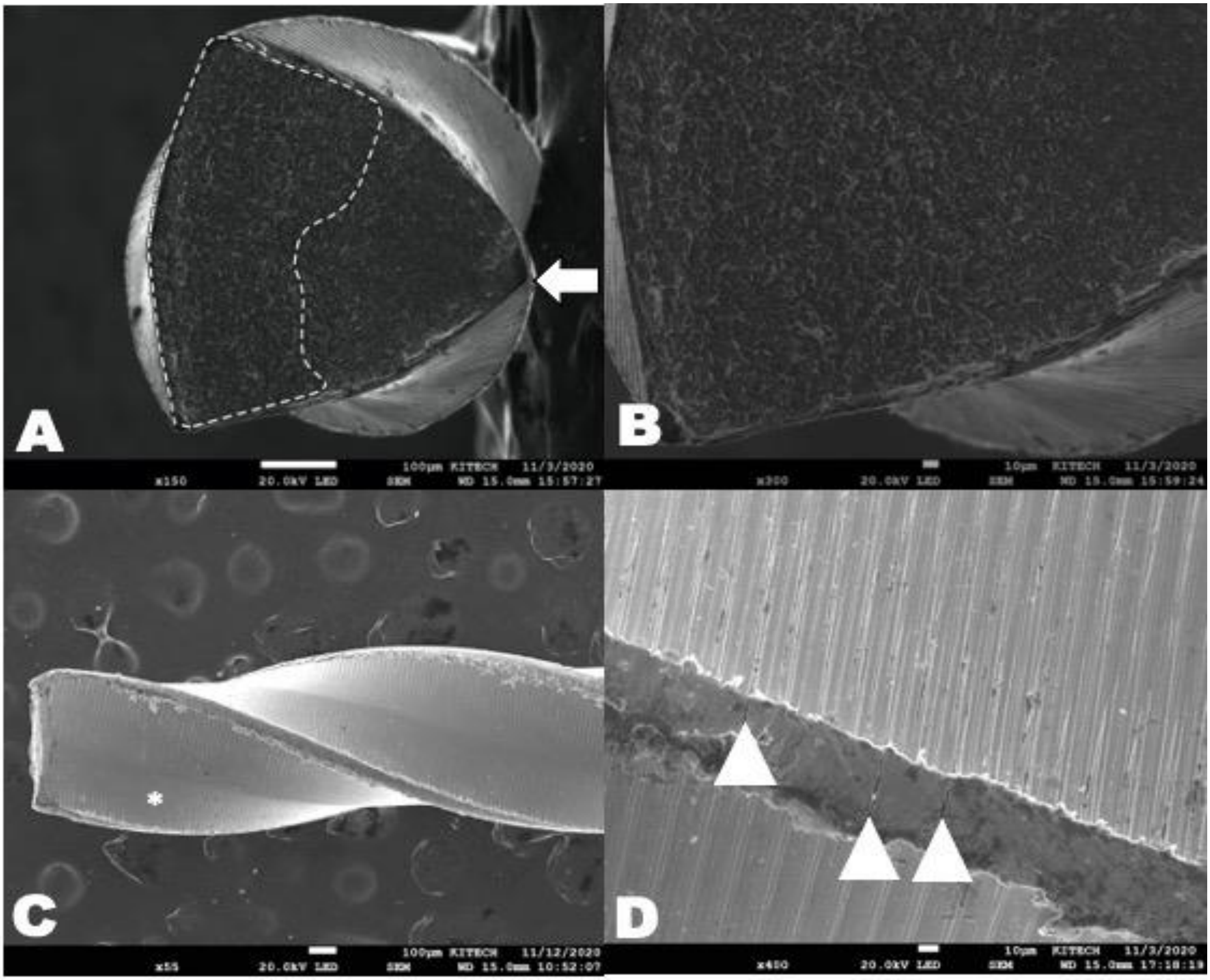

Figure 9: SEM images of the specimens after cyclic fatigue testing. PTG group II, in cross-section (A-B). The region of crack initiation is indicated by a white arrow. (Dashed line) The outline of the dimple region shows the fibrous zone of the overload quick fracture zone. (B) Overload quick fracture (of) area with multiple ductile dimples, as seen in a magnified view from (A). PTG phase II longitudinal view (C-D). (*) A micro-crack close to the fracture site. (D) A magnified version of (C). Multiple machining grooves are indicated by white triangles.

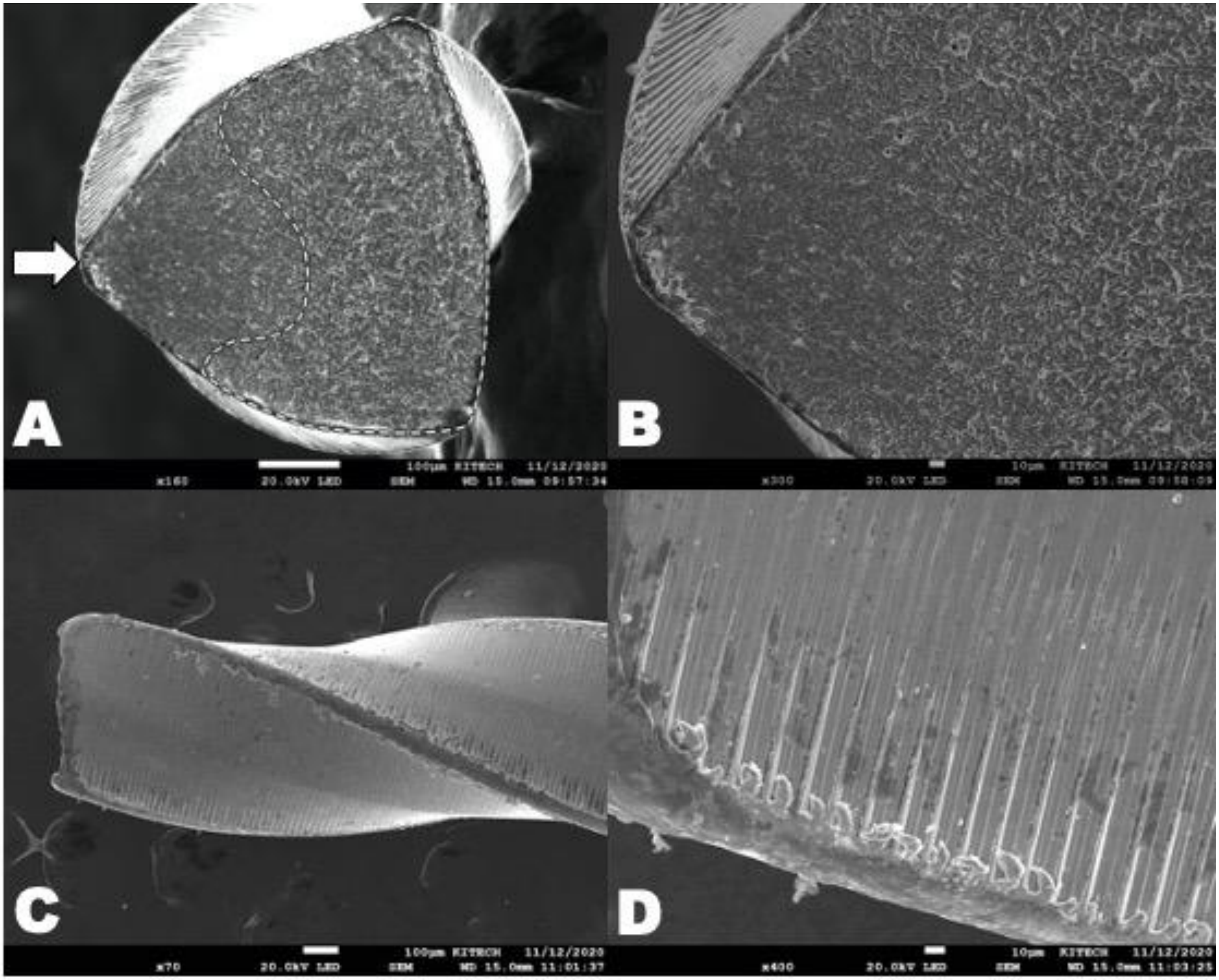

Figure 10: SEM images of the specimens after cyclic fatigue testing. PTG group III, in cross-section (A-B). The region of crack initiation is indicated by a white arrow. (Dashed line) The outline of the dimple region shows the fibrous zone of the overload quick fracture zone. (B) Overload quick fracture (of) area with multiple ductile dimples, as seen in a magnified view from (A). (C-D) PTG phase III longitudinal view. (*) A micro-crack close to the fracture site. (D) A magnified version of (C). Machining grooves have been discovered

6. DISCUSSION

Nickel-titanium rotary instruments (NiTi) have been widely used in endodontic practice since their invention in 1988 because of their ability to shape root canals with fewer procedural errors than stainless steel files [2]. One of the main disadvantages of NiTi rotary instruments is that they are more susceptible to deformation and fracture [4]. Torsional failure and cyclic fatigue fracture are the two leading causes of rotary instrument fracture [5]. Evaluation of the cyclic fatigue resistance of NiTi rotary instruments is a commonly studied subject in endodontics [57]. This type of fracture occurs most widely in curved root canals when the instrument rotates freely throughout the curvature without binding, concentrating compression and tensile stress at the point of maximum flexure before a fracture occurs [5,4,57]. In the literature, several methodologies have been suggested to determine the cyclic fatigue resistance of NiTi instruments using various devices. Most of the studies examining the cyclic fatigue resistance of NiTi files tested instruments in grooved blocks or metal blocks [79].

NiTi rotary instruments are commonly used in clinical procedures. Furthermore, the decision to reuse instruments necessitates a sterilization procedure to avoid cross-infections [80]. The methods for sterilizing instruments in endodontic practice are hot dry air and autoclave sterilization [81]. To improve the microstructure and flexibility of NiTi alloys, many novel thermomechanical processing and proprietary manufacturing technologies have been developed in recent years [44].

The current study investigates the effects of use and sterilization on the cyclic fatigue tolerance of heat-treated nickel-titanium instrument (ProTaper Gold). The files were split into three groups for the current review: unsterilized new files (group I), autoclave sterilization only (group II), and preparation in a standardized J-shaped endo block accompanied by autoclave sterilization (group III). Subsequently, the cyclic fatigue resistance was assessed using a metal block with a simulated canal. The canal had a curve of 35^{o} and a radius of 6 mm. The bulk of the tests on the cyclic fatigue resistance of NiTi files were conducted in artificial canals [82,83] to ensure a certain degree of standardization by minimizing the anatomic heterogeneity that could result from natural teeth. To simulate a clinical situation, the test was performed in the dynamic mode using a custom-made device with heat element device to maintain body temperature at 37^{o}C. During the cyclic fatigue testing process, friction developed between the instrument and the walls of the artificial canals, which could affect the cyclic fatigue resistance of the NiTi rotary instruments [30]. Therefore, it was recommended that a lubricant or coolant be used during the testing process [27]. 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 ^{o}C when the files were tested in the simulated canal at 300 and 500 rpm [84]. Most of the studies evaluated the cyclic fatigue resistance of NiTi instruments at room temperature [85,86,71,87,88]. Moreover, during clinical preparation of the root canal, the intracanal temperature ranges between 31 °C and 35 °C [79,89.] In terms of the role of heat treatment in the clinical performance of NiTi files, this study investigated the cyclic fatigue resistance of NiTi instruments using a heating element to maintain the body temperature. Few studies have recently shown that the environmental temperature has a significant impact on the cyclic fatigue resistance of NiTi instruments [90],[91],[92] .

The SEM investigation in this study reported typical characteristics of cyclic fatigue failure in most cyclic fatigue testing studies. The PTG exhibited a rough surface with multiple machining grooves. In addition, crack initiation occurred on the outer surface of the instrument.

The current findings revealed no statistically significant difference in the number of cycles to failure between the three groups (NCF). In our research, we used a single autoclave sterilization cycle and single canal file use which had little effect on the NCF of ProTaper Gold. On the other hand, there was a statistically significant difference in the fractured fragment length (mm) between the three groups which can be explained that the file move in a dynamic motion within a range of 4mm which is in maximum curvature. This validates the methodology we used to test cyclic fatigue resistance.

A few studies in the literature have examined the impact of autoclave sterilization on the cyclic fatigue tolerance of heat-treated NiTi files [93,88,86,71,87,85,76].

Viana et al. discovered that a five-time autoclave procedure improved ProFile's cyclic fatigue resistance (Dentsply Maillefer) [93]. Hilfer et al. investigated the effect of autoclave sterilization on the cyclic fatigue resistance of twisted files (TF) (25/0.06) and TF (25/0.04) files (sybronEndo). They found that after autoclave sterilization, the cyclic fatigue tolerance of TF (25/0.06) files decreased, but the TF (25/0.04) files remained unchanged [88]. Other researchers found that sterilizing the files in an autoclave at 170°C was inadequate for modifying their mechanical properties [82]. Platino et al. autoclaved K3 (SybronEndo, Green, CA, USA), Mtwo (VDW) and Vortex (Dentsply Maillefer) files ten times each. Although autoclaving files ten times had little effect on cyclic fatigue resistance, it did increase cyclic fatigue resistance in the K3XF

(SybronEndo) file [86]. Zhao et al. examined the impact of autoclave sterilization on the cyclic fatigue resistance of files in artificially curved canals. Sterilization after cyclic stress altered the cyclic fatigue behavior of HyFlex CM and K3XF files, but had no impact on TF, K3, or race files, according to the researchers [71].

Pedullà et al. found no difference in cyclic fatigue between the control and autoclave classes of Hyflex CM. The twisted files that had been autoclaved three times had slightly lower cyclic fatigue resistance than the new ones. There was no difference between the one cycle sterilization and the new ones [85].

Özyürek et al. conducted the only research that examined the effect of autoclave sterilization on the cyclic fatigue resistance of ProTaper Gold. They concluded that ProTaper Next and ProTaper Gold autoclaved ten times had substantially higher cyclic fatigue resistance than new ones [87]. They found that autoclave sterilization affects cyclic fatigue resistance after ten cycles, which coincides with our study's null hypothesis that the usage and sterilization of heat-treated nickel-titanium instruments (ProTaper Gold) will reduce the cyclic fatigue resistance. We cannot equate the findings of this study to those of the previous study because they discovered a discrepancy after ten sterilization cycles. In this analysis, we used only one sterilization cycle.

The clinical relevance of the current study is to know how the latest generation of heat-treated NiTi gold files are affected by usage and sterilization similar to clinical conditions to avoid instrument separation, thus compromising the root canal treatment outcome. The main limitation of this study is that we test cyclic fatigue only, and clinically the files are exposed to both torsional stress and cyclic fatigue as well as single canal file use and single autoclave sterilization cycle.

Finally, further research is required to assess the impact of multiple usages and sterilization cycles and measure torsional stress and cyclic fatigue resistance. The files are exposed to cyclic and torsional pressures in clinical conditions.

7. CONCLUSIONS

Within the limitation of this analysis, the findings provide precursory evidence that single canal file use and a single autoclave sterilization cycle have no effect on the cyclic fatigue resistance of heat-treated nickel-titanium endodontic instrument (ProTaper Gold).

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9. APPENDICES

APPENDIX 1

29 September 2020

Rashid El Abed
Assistant Professor – Endodontics
Hamdan Bin Mohammed College of Dental Medicine

RE: MBRU-IRB-2020-036

Dear Dr Rashid,

Thank you for submitting to the IRB application titled "The Effects of usage and autoclaving on the torsional resistance and cyclic fatigue resistance of Nickel-titanium Instruments" for exempt review. The Board has reviewed the same and has agreed that the study does not require IRB approval as it does not involve human subjects.

To improve the quality of findings, the Board suggests that the following be addressed:

- How will the repeated up and down movement of the cyclic fatigue test be standardized for the study?
- Will one or more artificial steel canal(s) be fabricated.
- Information on how cyclic fatigue will be tested in a dynamic mode

For any questions, please contact the Institutional Review Board irb@mbru.ac.ae.

Thank you for your interest in MBRU's IRB.

Yours Sincerely,

Essa Kazim, FRCS
Chairman, MBRU-IRB

لجنة أخلاقيات البحوث
INSTITUTIONAL REVIEW BOARD

MBRU

جامعة محمد بن راشد للطب والعلوم الصحية

MOHAMMED BIN RASHID UNIVERSITY OF MEDICINE AND HEALTH SCIENCES

PUSAN NATIONAL UNIVERSITY

MEMORANDUM OF UNDERSTANDING

Between Pusan National University School of Dentistry (PNUD), Busan, Korea (South) [49, Busandaehak-ro, Mulgeum-eup, Yangsan-si, Gyeongsangnam-do, 50612, Korea] and Mohammad Bin Rashid University of Medicine and Health Sciences (MBRU) [Building 14, Dubai Healthcare City, P.O. Box 505055, Dubai, United Arab Emirates] ("the Parties").

Dated this Memorandum is to take effect: 12 December 2019

1. This Memorandum of Understanding ("MOU") recognises the intention of the Parties to establish and build a relationship, co-operate in a broad range of areas and to work together to gain a mutual benefit. The Parties may seek to encourage and develop collaborative activities in various ways, including but not limited to; the exchange of scholarly ideas/expertise and research; the support of specific discipline interaction; the development of programmes, student exchanges; and advance entry from one institution to the other where the students are appropriately qualified and according to the rules and regulations of the institution.

2. Separate agreements will be required for any definitive collaborations as articulated by each Party's quality processes. The Parties understand that any financial considerations associated with any collaboration will be dealt with via a legally binding contract. In the course of discussions, the Parties may, before the entering of a legally binding contract, wish to document the understanding reached on financials. In such cases, the Parties agree to append any such understanding to this MOU.

3. Both Parties recognise the value of this MOU in promoting its own programme and activities. However, any marketing material/activity which includes reference to the other party must be sent to that Party and be approved before use.

MBRU

جامعة محمد بن راشد للطب والعلوم الصحية

MOHAMMED BIN RASHID UNIVERSITY OF MEDICINE AND HEALTH SCIENCES

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	Professor Zaid Baqain
Dean, PNUD	Dean, HBMCDM
김현철	Zl By
For and on behalf of Pusan National University School of Dentistry	For and on behalf of HBMCDM, MBRU