# THE EFFECT OF CYCLIC FATIGUE ON THE HARDNESS OF NEW NITI ENDODONTIC FILES: A NANOINDENTATION STUDY

by

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## Abstract

**Objective**: The purpose of this study was to compare the flexibility and cyclic fatigue of ProTaper Universal (PTU; Dentsply Tulsa Dental Specialties, Tulsa, OK) and ProTaper Gold (PTG; Dentsply Tulsa Dental Specialties, Tulsa, OK) in relation to their phase transformation behavior, as well as to determine the effect of cyclic fatigue on the nanohardness with a nanoindentation method.

Hypotheses: PTG and PTU have similar flexibility and fatigue resistance. Cyclic fatigue has no effect on the hardness of both PTG and PTU NiTi rotary endodontic instruments.

**Methodology**: PTU and PTG instruments were subjected to rotational bending at a curvature of 40° and a radius of 6mm. The number of cycles to fracture (NCF) was recorded. According to the ISO 3630-1 specification, 45° bending tests was used to determine the flexibility. Unused and fractured instruments were studied by differential scanning calorimetry. The hardness and modulus of elasticity of new files, fractured instruments and instruments stressed to 50% of the NCF for sizes S1, F1 and F2 were measured with the use of a nanoindenter.

**Results**: PTG had a cyclic fatigue resistance superior to PTU in all sizes ($P < .001$). The fractured files of both PTU and PTG showed the typical fracture pattern of fatigue failure. Bending load results for PTG were significantly lower than that for PTU ($P < .05$). The differential scanning calorimetry analyses showed that PTG instruments had a higher austenite finish temperature (50.1°C ± 1.7°C) than the PTU instruments (21.2°C ± 1.9°C) ($P < .001$). There were no significant differences in the austenite finish between unused files and instruments subjected to the fatigue process. There were statistically significant differences in nanohardness and elastic modulus between PTU and PTG groups ($P < 0.05$).

**Conclusions**: Within the limitation of this study, PTG files were significantly more flexible and resistant to fatigue than PTU files. PTG exhibited different phase transformation behavior than PTU, which may be attributed to the special heat treatment history of PTG instruments. The fatigue process had no significant effect on the hardness and elastic modulus of both NiTi instrument. PTG may be more suitable for preparing canals with sudden curvature.

## Preface

Some of the material included in this thesis has been previously published in the following paper:

Hieawy, A., Haapasalo, M., Zhou, H., Wang, Z. & Shen, Y. 2015, "Phase Transformation Behavior and Resistance to Bending and Cyclic Fatigue of ProTaper Gold and ProTaper Universal Instruments", Journal of Endodontics, vol. 41, no. 7, pp. 1134.

This publication as well as this thesis is the principal work of the candidate, Ahmed Hieawy. The project was performed under the guidance and supervision of Dr. Y. Shen. Ahmed Hieawy was responsible for all parts of the research, including the fatigue testing, flexibility testing, hardness testing, data gathered from such tests, and microscopic image photography. The relative contribution of the collaborators in this project was: Dr. Ahmed Hieawy 80% and Dr. Ya Shen 20%. Dr. Ya Shen did manuscript editing.

This research was supported in part by the Canadian Academy of Endodontics.

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## List of Abbreviations

NiTi: Nickel-Titanium

PTU: ProTaper Universal

PTG: ProTaper Gold

DSC: Differential scanning calorimetry

SEM: Scanning electron microscope

$A_f$: Austenite finish temperature

MOE: Modulus of elasticity

NCF: Number of cycles to failure

ISO: International Organization for Standardization

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Special thanks are owed to my father, who has supported me throughout my years of education both morally and financially, to my wife and children for being so patient and supportive all these years and finally I truly owe everything to my late mother.

## Dedication

To Dr. Nancy Scott,

The example of your teaching and the spirit of your love will live on this faculty, in the hearts of those whom you've touched and inspired, those who will continue caring about their students as you did, forever.

This is for you, with love!

Ahmed Hieawy

* Dr. Nancy L. Scott (1955-2014) An outstanding Clinical Educator, UBC alumnus and a Clinical Assistant Professor at the Faculty of Dentistry, University of British Columbia.

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# Chapter 1: Introduction

## 1.1 Background

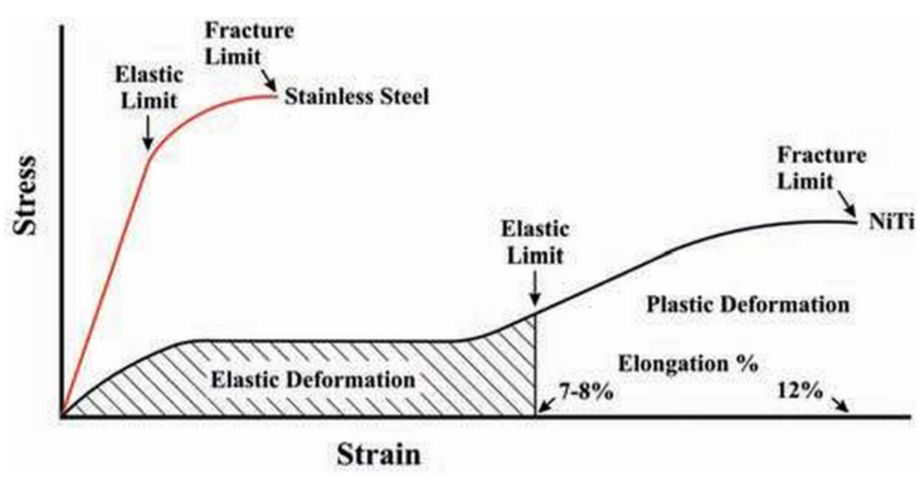
The main goal of endodontics is to avoid and treat apical periodontitis (Ørstavik & Pitt Ford 2008, Ricucci 2009, Siqueira et al., 2014). Understanding the etiology and the pathological process of endodontic infections provides a foundation on which preventive measures and different treatment technique can be integrated to meet this goal (Haapasalo et al., 2003). Both mechanical instrumentation and antimicrobial irrigation of the root canal i.e. chemo-mechanical preparation, are deemed to be the critical stage in canal disinfection. The root filling and the coronal seal will prevent the ingress of bacteria into the root canal, as well as help to entomb of the remaining ones inside the root canal. Biologically, the goals of chemomechanical preparation are to remove the contents of the root canal that may support microbial growth as well as the microorganisms carefully to prevent pushing debris beyond the foramen (Vianna et al., 2006, Young et al., 2007).

Biologically, the principal goal is to reduce the bacterial load as it is stated that manual instrumentation and physiological saline irrigation will result in a 100 to 1000 fold reduction (Byström & Sundqvist 1981). While technically, the foremost goal of canal preparation is the shaping the canal so as to simplify the placement of a good root filling (Harrison 1984, Young et al., 2007).

Up until 20 years ago, endodontic files were made out of stainless steel. These files have an intrinsic stiffness that increases with the instrument size. That’s why preparing a curved canal, the spring action will try to push the instrument back to its original shape, and this is more evident when the instrument used in a filing action. And that's the reason why pre-curving the steel instruments is a must prevent them from being used with a rotary motion (Haapasalo & Shen 2013). To overcome this shortcoming of stainless steel files (Figure 1), Walia et al. began the use of nickel titanium (NiTi) instrument (Walia et al. 1988). This was a major development in the discipline of endodontics as the superelasticity of NiTi alloy helps the instrument to trail the original path of the canal effectively (Thompson 2000, Cheung et al., 2011).

The stress-induced phase transformation at the crystal level of the alloy is related to the superelasticity of NiTi instruments. Only light stressing force for bending is required for the austenitic phase transforms into the martensitic one (Hülsmann et al., 2005).

**Figure 1:** "Stress-strain curve of stainless steel (red line) and NiTi (black line). Elastic limit = maximum stress without permanent deformation; fracture limit = stress at which fracture occurs; Elongation % refers to the deformation that results from application of a tensile stress, calculated as (change in length/ original length) x 100%". (Young et al., 2007).



## 1.2 Instrument Fractures

Root canal preparation with NiTi rotary instruments can maintain the appropriate centrality of the canal and provide a predictable outcome more than stainless steel files (Schäfer et al., 2004). However, no material is immune against fracture, as it will break if the ultimate strength is surpassed, or if the residual intact cross-section of material is incapable to withstand the running stress after crack extension. The mishap of file separation is so distressful to the patient and the dentist (Cheung 2009). Poor management of the incident might be expected to result in legal implications. The possible difficulty in retrieving the fractured instrument fragments (Ward et al., 2003) and the expected prognostic effect were challenging the implementation of the NiTi instruments. Accordingly, large number of research has been carried out in order to understand and hopefully prevent the fracture instruments (Parashos & Messer 2004).

The inappropriate shaping, disinfection and/or sealing of the root canal can adversely affect the success of endodontic treatment. And sometimes it's really hard for the patient to understand the relationship between the treatment which had been done a long time ago and the current failure, as it may take a really long time to get these objective findings like the radiographic evidence (Sabeti et al., 2006, Simon et al., 2008).

Sattapan et al., in (2000) documented a 21% separation frequency from 378 used Quantec file gathered from endodontic practices over a six-month period. A much lower frequency of 5% was recorded in 2004 by Parashos & Messer, who studied large number of discarded rotary NiTi instruments (Parashos & Messer. 2004). Alapati et al., in 2005 also reported 5.1% of 822 rotary files collected from graduate students. Other reviews reported a median range of 0.4%–3.7% for the separation frequency of NiTi files (Parashos & Messer 2006).

There are complex and multifactorial reasons for fracture behind the fracture of the rotary NiTi, like instrumentation method, use of ‘torque-controlled’ motors, instrument size and radius of curvature, surface situation, rotation rate, effect of sterilization as will as many other variables like operator’s skill and experience which may help to explain the variation in the prevalence among different studies (Cheung 2009, McGuigan et al., 2013).

Based on the microscopical existence or not of plastic deformation adjacent to the separation site, the fracture can be considered ‘torsional’ or ‘flexural’, respectively (Sattapan et al., 2000). Efficient, high magnification fractographic examination came out with two different mechanisms by which separation of NiTi rotary files: shear and fatigue (Peng et al., 2005).

Torsional fracture happens when the instrument tip getting locked in the canal while the rest of the file continues to rotate until the elastic limit is exceeded that’s when the fracture happens. Signs of plastic deformation can be seen when the instrument fractured due to torsional strain (Parashos et al., 2004).

Cyclic fatigue, on the other hand can be seen due to the repeated tension/compression of the instrument when rotating in a curved canal, which eventually ended with fracture. Repeated tension-compression rotations inside the curved canals may increase the cyclic fatigue of the file (Peters 2004). Therefore, optimum curved canal system preparation requires a satisfactory shear strength to prevent torsional failure of the NiTi rotary file and a high resistance to cyclic fatigue (Shen and Cheung 2013).

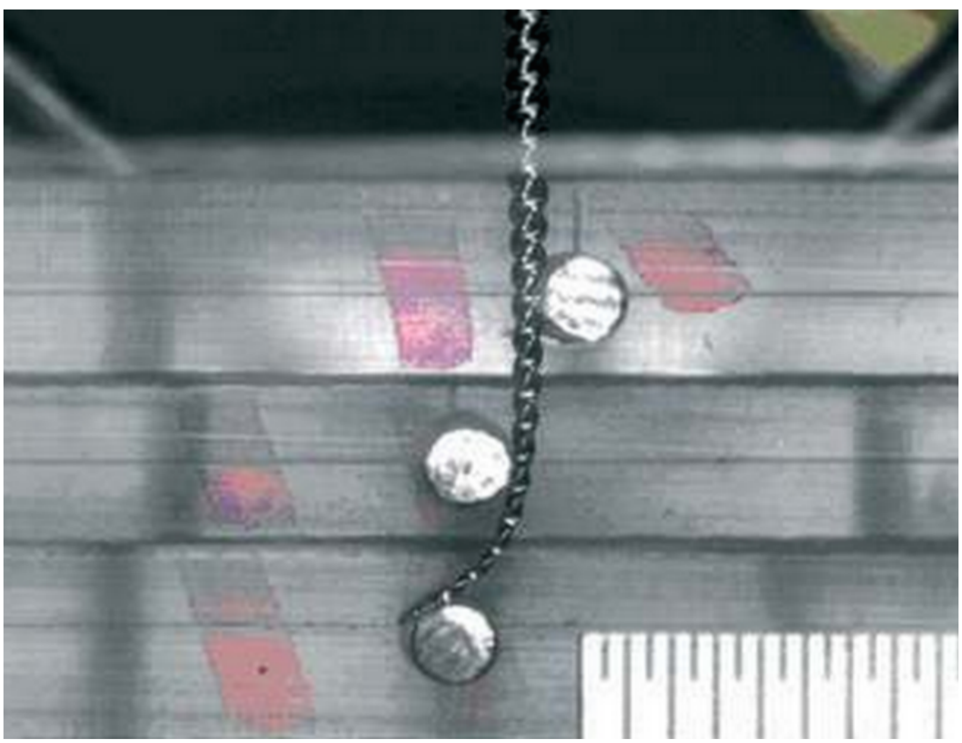
Peng et al., (2005) considered fatigue as the main mechanism for material failure when they categorized most of the instruments failure as flexural. In a related study, Cheung et al., (2005) pointed out that more than 90% of instruments failed because of flexural fatigue. Which might indicate that in NiTi alloys the fatigue-crack growth rates are significantly greater than in other metals of similar strength (Dauskardt et al., 1989). Consequently, catastrophic failure will be the abrupt end once a micro crack is started (Plotino et al., 2009)

### 1.3 Methods for Fatigue Test

Based on the fact that root canal systems are rarely straight, then having fatigue resistant file is of great benefit. That's why so much research was done in the 'fatigue resistance' area (generally defined as the number of revolutions sustained before breakage) for different types of NiTi. All of which tried to imitate the file rotation inside the curved canal to find the time or number of revolutions before fatigue fracture happened. Four methods used to simulate canal curvature were the NiTi instrument can be rotated "(i) curved metal tube (or a hypodermic needle); (ii) grooved block-and-rod assembly; (iii) rotation against an inclined plane; and (iv) three-point bend of a rotating instrument" (Cheung 2009). Each technique has its own problems, for example the reproducibility of the actual file trajectory is really hard by the tube-like devices, while in the grooved block and the inclined surface the physical shape of each file will affect the location where the change in the long axis will happen therefor it will be hard to expect or scheme the angle (Plotino et al., 2009).

The rotation of the 3-point bend was put forward by Cheung et al., (2007) to overcome the previously mentioned shortcomings. Three smooth cylinder-shaped 2mm diameter hardened stainless steel pins attached to acrylic shims, with a changeable horizontal direction; the curve of the file will be determined by the position of the pins. A tiny indentation prepared on the lower pin will help to keep the position of the tip during the spin, the approximation should be reasonable as stated by the author. Such restraints in a three-point bending test of NiTi file will produce a circular curvature (Wick 1995). (Figure 2)

**Figure 2:** "Rotation of the 3-point bend. profile 06 instrument constrained into a curvature by three rigid pins set in acrylic shims" (Cheung et al., 2007).



An important issue must be taken in consideration here, which is the absence of international standardization in testing the cyclic fatigue for endodontic instruments. Some reports mentioned that ISO and ADA are working on that. It's definitely important for the companies, researchers, and dentists to be able to characterize the properties of the NiTi instrument for safe use of their new productions (Plotino et al., 2009).

### 1.4 Metallurgy and Mechanical Properties

NiTi alloys used to produce endodontic files contain about 56% (wt) nickel and 44% (wt) titanium. Some types may have cobalt on the expense of the nickel percentage and its usually called 55- Nitinol. At high temperature, the structure of NiTi alloy is a stable, body-centered cubic lattice, called the austenite phase. On the other hand, martensitic transformation will happen when the temperature reduced under the conversion temperature range and this will change the elastic modulus. The start and finish temperature will control the extent of this change (Thompson 2000).

A reversible atomic process called twinning is needed for the martensitic transformation to allow reduction of strain (Ōtsuka & Wayman 1998). However, unwanted and unanticipated files fracture during rotation is real problem in clinical use (Zhou et al., 2013). During the last decade, thermal and mechanical procedures seem to provide alloys with higher cyclic fatigue resistance in relation to the usual NiTi files (Gambarini et al., 2011, Zinelis et al., 2007).

The properties of NiTi alloys from a mechanical point of view, depends on their chemical composition, phase constitution, and manufacturing procedures as internal factors while the cold working, annealing, and aging are considered as the external factors (Zhou et al., 2013).

NiTi alloy mechanical properties can be optimized by "precise control of the composition, cold work, and continuous strain age annealing" (Pelton et al., 2000). The exact thermomechanical process is unknown for the protection of intellectual rights, however indirect analysis of the phase transformation behavior may give us an idea to assess the effect of thermomechanical treatments on the mechanical properties (Zhou et al., 2013). Regulating the chemical composition and the environment of heat treatment can affect the reverse transformation temperatures of austenite start (As) and finish ($A_f$) and therefore play a major role in controlling the mechanical properties of the alloy (Viana et al., 2010).

### 1.5 The Differential Scanning Calorimetry (DSC)

A minute change in temperature will accompany phase transformation (Lagoudas DC 2008). To clarify, the austenite to martensitic transformation is slightly exothermic, while the opposite change will absorb heat and DSC can be used to study the structure of the NiTi alloys, since the fatigue and fracture behaviors are basically related on the $A_f$ transition temperature by supplying thermal energy to a test sample and a passive control specimen heated at the same rate is measured very accurately (Brantley et al., 2001). DSC indicates the existed phases at that particular temperature (martensitic, R-phase, or austenitic), the phase transformations will be shown as endothermic peaks on the heating curves and as exothermic peaks on the cooling one

(Thayer et al., 1996). These variations of $A_f$ considered being critical to the file performance and life expectancy (Wu et al., 2012).

### 1.6 Nanoindentation

Nanoindentation "is a novel method to characterize material mechanical properties on a very small scale. Recently, use of nanoindentation technology has been suggested to show more precisely the micro structural changes in conventional superelastic NiTi" (Jamleh et al. 2012; Shen et al. 2014). According to Kim et al., in 2005 and Alapati et al., in 2006, the available data on Vickers hardness values for the NiTi ranging between 313–481. While the stainless steel values were between 546-673 (Darabara et al., 2004). However, these values may only represent the innermost part of the material and not the surface area where the residual stresses prepared in the course of the manufacturing processes (Zinelis et al., 2008).

Alapati et al., recommended the use of a nanoindentor to verify the structural changes in NiTi, as the low force and reduced dints significantly decrease the effect of the material volume on readings if we compare it with microhardness (Alapati et al., 2006). Traditional hardness tests apply a single static force with a specific tip form and material, causing a hardness dint that is measured by millimeters to give a single hardness value (penetration depth of the indentation tip into the sample) in contrast to the nanoindentor where force and the displacement are measured concurrently and constantly over a complete cycle. Moreover, the exceptionally low force and dislodgment let this device characterize almost any alloy. It's mostly machine controlled measurement uses active swinging to enhance sensitivity. Numerous developments in materials science, predominantly regarding essential physical performance at micron or less level were the results of the high levels of control, sensitivity, and data record obtained by the indenter (Van Landingham 2003). That's why the mechanical properties of different areas in the petite size endodontic files can be accurately determined using the nanoindentation technology, and this will help us to better understand the performance of rotary NiTi file (Sadr et al., 2009).

### 1.7 New Generation NiTi files

Endodontic files were produced from raw NiTi alloy for about 20 years. Interest in NiTi instrument research has not waned with time (Shen & Cheung 2013). Safe design of the super elastic metal was the main feature of the earliest generation, after which the attention was directed to the more recent thermally and mechanically modified instruments with special design characters like the alternating cross section of the cutting part (Haapasalo & Shen 2013).

This thermo-mechanical manufacturing will help to refine the structure and transformation performance of the file, and this will influence the physical characteristics of the rotary instrument (Gambarini et al., 2008, Bardsley et al., 2011, Gao et al., 2012, Shen etal.;2012). ProTaper Universal (PTU, Dentsply Tulsa Dental Specialties) is one of the most widely used rotary instruments (Lee et al. 2012). PTU is manufactured with a variable taper along the length of the cutting blades, convex triangular cross sections, and non-cutting tips. Recently, ProTaper Gold (PTG, Dentsply Tulsa Dental Specialties) instruments were introduced. The PTG files have a design that features identical geometries as PTU but is more flexible and have been made with exclusive advanced metallurgy. The company states that these files have higher fatigue resistance than PTU.

## 1.8 Rational

The PTU and PTG have identical geometries and design features, and the company claims that the PTG, which was developed with proprietary advanced metallurgy, is more flexible and more fatigue resistance, However, the properties of the PTG files have not been examined by independent research.

The association between the metallurgical properties and the physical properties is of prime importance for the clinician to decide the appropriate file for that particular root canal, however it doesn't get that much of attention from researchers (Zhou et al., 2013). The association between thermal behavior and fatigue properties of new PTG endodontic files has not been examined yet.

## 1.9 Aims

- To examine the flexibility and fatigue behavior of the PTG and compare it with its predecessor the PTU.
- To evaluate the phase transformation behavior of PTG and PTU files using DSC analysis.
- To investigate the effect of cyclic fatigue on local nanohardness of both PTG and PTU using a nanoindentation hardness technique.

## 1.10 Hypothesis

PTG and PTU have similar flexibility and fatigue resistance. Cyclic fatigue has no effect on the hardness of both PTG and PTU NiTi rotary endodontic instruments.

# Chapter 2: Methods

## 2.1 Cyclic Fatigue Life

During the cyclic fatigue test (Shen et al., 2011a) the NiTi rotary instruments of PTG and PTU sizes S1, S2, F1, F2 and F3 were subjected to 3-point bending using a device which included a reduction handpiece (W&H 8:1) attached firmly on a stainless steel framework (Figure 3). The handpiece was connected to an torque control motor (AEU-20T Endodontic System) with a curvature of 40° with a 6-mm radius which was determined using a calibrated digital photograph (Figure 4). The test was done under deionized water at room temperature (23°C ± 2°C) in the laboratory. The file was then rotated at 300 rpm with torque of 150 - 520 g-cm (as recommended by the manufacturer) until it fractured and the fatigue life, or the number of cycles to fracture (NCF), was recorded. Each group included 15 instruments.

Separated pieces were measured for length. The fractured instrument was further cleansed in an ultrasonic bath in absolute alcohol, and the fractured surface was faced upward for a fractographic examination using a scanning electron microscope (SEM) (Helios Nano Lab 650; FEI, Eindhoven, Netherlands) operating at 3 kV (Cheung et al., 2005).

**Figure 3**: Hand piece mounted on a three-point bending apparatus.

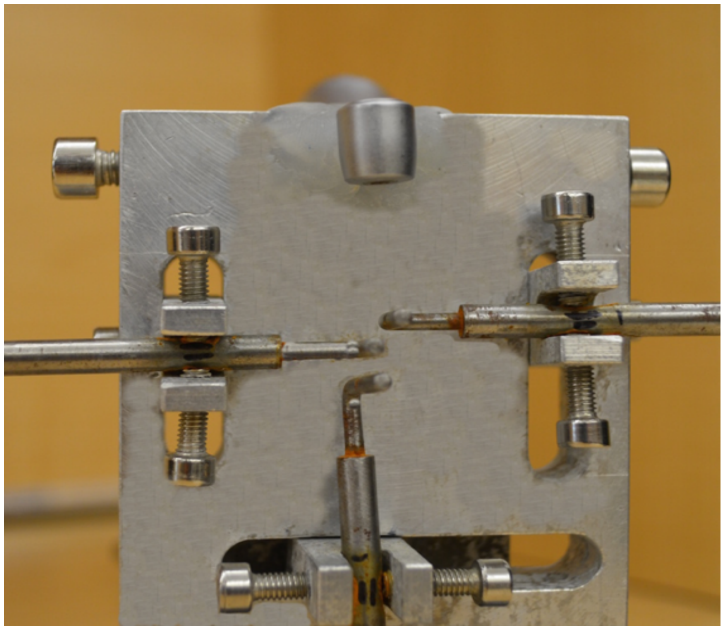
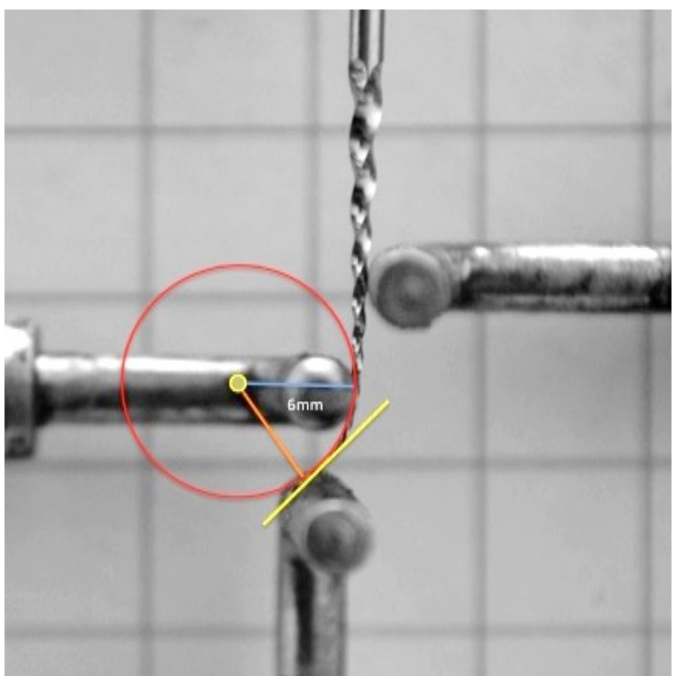


**Figure 4**: F2 protaper universal rotary file mounted with 6mm radius and 40° curvature.



## 2.2 Bending Test

Flexibility was measured via a bending test, which was done using a torsiometer (Sabri Dental Enterprises, Downers Grove, IL) (Figure 5) at room temperature as per to the International Organization for Standardization specification number 3630-1 (ISO 2008). The files were secured at about 3mm from the tip and then bent 45° around their long axis, and the moment of bending at an angular deflection of 45° was documented. Twelve files were checked for each group for S1, S2, F1, F2 and F3 for PTG and PTU.

**Figure 5**: Torsiometer from Sabri dental enterprises, IL. The file will be clamped into the chuck in the left side and the data will be recorded in the computer.

### 2.3 DSC Analysis

DSC analyses were carried out for unused and fractured S1, F1and F2 for both PTG and PTU files. Five specimens from each group were analyzed. Each sample contained 2 segments 3 to 4mm in length. "DSC analyses of full cycles were conducted (PYRIS Diamond Series DSC; PerkinElmer, Shelton, CT) over a temperature range from -80° C to 80° C using liquid nitrogen cooling to achieve sub-ambient temperatures. The transformation temperatures were available from the intersection between the extrapolation of the baseline and the maximum gradient line of the lambda-type DSC curve. The $A_f$ was determined" (Hou et al., 2011, Shen et al., 2015).

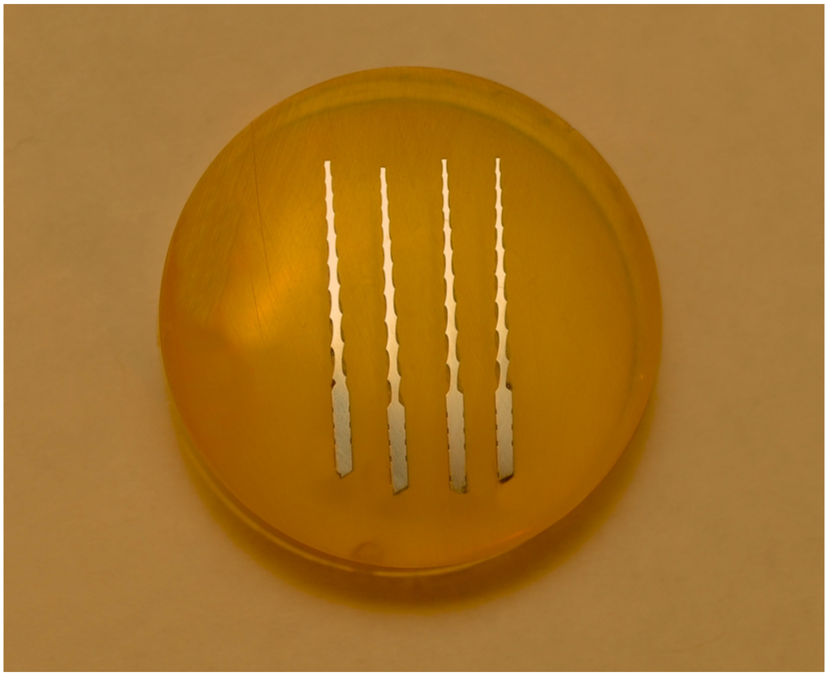
The data for bending moment, NCF, and $A_f$ were analyzed statistically using 2-way analysis of variance (SPSS for Windows 11.0; SPSS, Chicago, IL). Post hoc multiple comparison (Tukey test) was used to isolate and compare the means of the results at a significance level of $P < .05$.

### 2.4 Nanoindentation

#### 2.4.1 Sample preparation

After removing the handle, the files were fixed with a small adhesive tape horizontally at the base of special round plastic mold before pouring the vacuum mixed acrylic resin, leaving it to fully polymerize for 24h as recommended by the manufacturer (Cold Cure, System Three Resins, USA). The rounded blocks with the samples were subjected to metallographic preparation (Figure 6). The surfaces of the mounted specimens were ground with sand paper (150 -1200 grit size SiC) and were followed by polishing using 6μm and then 1μm size diamond paste. Polished samples were submerged in a purified water bath and cleaned ultrasonically for 10 minutes.

**Figure 6**: Image of the file specimen embedded in acrylic resin after metallographic preparation.



### 2.4.2 Nanoindentation test

To assess the effect of cyclic fatigue on hardness, files size S1, F1 and F2 were preloaded to 50% of their respective mean NCF (Table 1). The nanohardness and modulus of elasticity of the of the new, fractured and the 50% preloaded files size S1, F1 and F2 were then measured through a nanoindentation test using a nanoindentation device (Nano Indenter® XP system, Oak Ridge, Tennessee, USA) (Figures 7 and 8) using a calibrated Berkovich indenter at a constant room temperature. 6 specimens for each group for both PTU and PTG were tested for 12 indentations each, and the areas were selected (Figure 9) as 4 points close to the fatigue fractured edge and at a distance of 5 mm from the tip of the new files group and the 50% preloaded group, corresponding to the same regions as for the fractured group. Four points on each side of both fractured-shank and the non fractured-shank for both PTU and PTG.

Indentation areas were selected using an optical microscope and camera connected to the nanoindentation test device. The loading rate was 10 mN/s with load increasing up to 100 mN, with incremental increase of 0.2 mN to the current load per 20ms interval. This loading was followed by a holding section; after which the load was gradually reduced in the unloading section. By analyzing the displacement data during the loading–unloading sequence, the hardness and the modulus of elasticity the can be calculated (Oliver & Pharr 2004). All the results of the examined groups were statistically analyzed using 2-way analysis of variance (SPSS for Windows 11.0; SPSS, Chicago, IL). Post hoc multiple comparison (Tukey test) was used to isolate and compare the means of the results at a significance level of $P < .05$.

**Figure 7:** The MTS nanoindentor XP

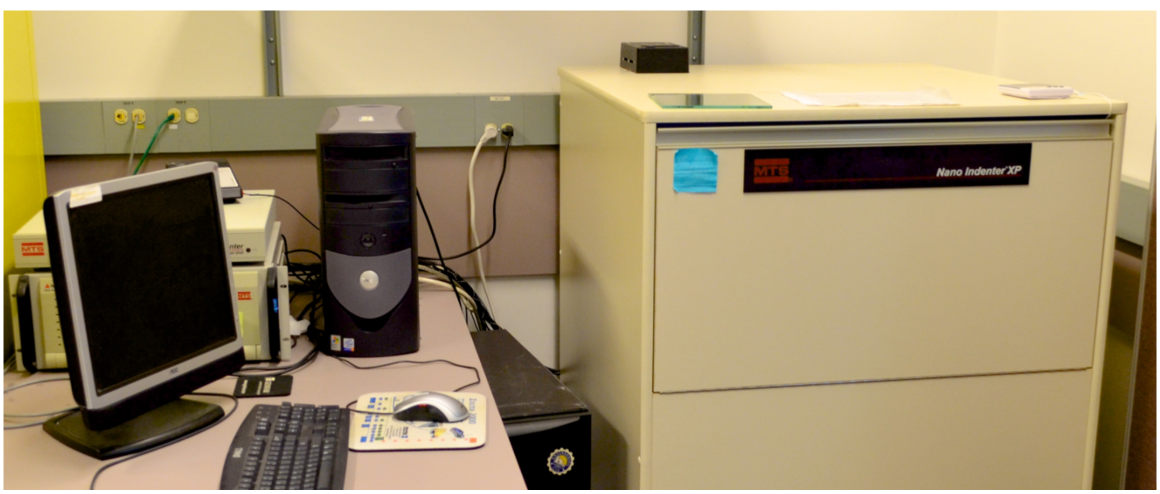


**Figure 8:** Schematic of the nanoindenter" XP (MTS nanoindentor user manual)

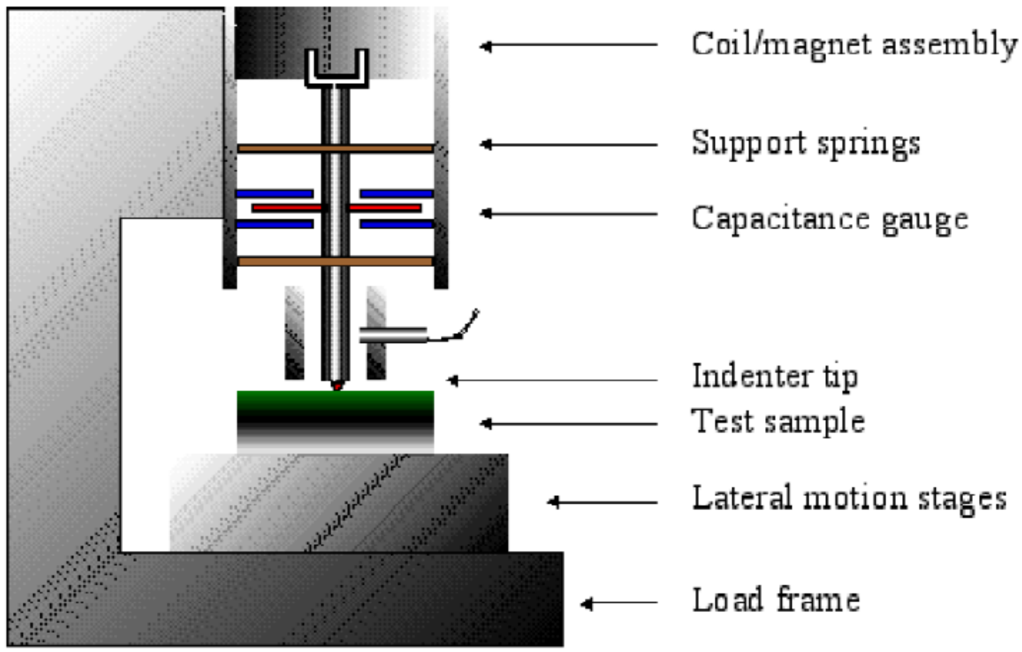
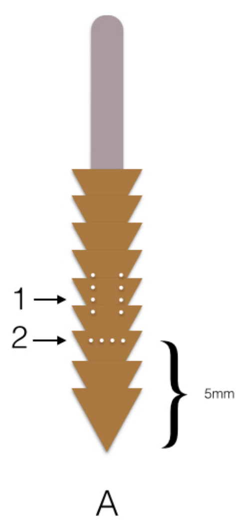



**Figure 9:** Schematic drawings of endodontic instruments showing the tested areas for (A) the new and (B) the fractured file. 1-points in the shank area. 2- point at the proposed edge of the non fractured specimens. 3- The fractured edge.



# Chapter 3: Results

The PTG file had a significantly higher NCF than the PTU file (P <.001) (Table 1). S1 file showed higher resistance to fatigue failure compared with F1 and F2 files in both PTG and PTU systems (P <.001). PTG S1 had the highest NCF among all files (P <.001). Whereas PTU F2 had the lowest NCF.

The fragment length ranged from 3.7–4.8mm. The SEM topographic appearance of the fracture surfaces of PTG and PTU showed classic features of cyclic fatigue, including 1 or more crack initiation areas, the presence of fatigue striations, and a fast fracture zone with dimples (Fig. 10 and 11).

The bending moments of the files tested are shown in table 2. The bending load results were significantly lower for PTG than for PTU (P <.05). There was a significant difference among files (P <.0001) within each file system.

DSC schemes for both the heating and cooling rounds of different sizes of unused instruments and instruments subjected to the fatigue process are shown in Figures 12, 13 and 14. In all DSC plots, the heating curve is shown at the top of the figure, and the cooling curve is shown at the bottom of the figure. The characteristic DSC curve for PTU instruments exhibited a distinct and defined peak on cooling and heating, respectively. $A_f$ temperatures for unused PTU files were

21.2°C ± 1.9°C. Two endothermic peaks (1 weak and 1 intensive peak) were observed on the heating curve of PTG files. $A_f$ temperatures for unused PTG files (50.1°C ± 1.7°C) were significantly higher than those for PTU files ($P < .001$). There was no difference in $A_f$ temperatures between unused and fractured instruments ($P > .05$).

There were statistically significant differences in nanohardness (Table 3) and elastic modulus (Table 4) between PTU and PTG groups ($P < 0.05$). PTU showed higher nanohardness and elastic modulus than the PTG for all the groups, however, there was no significant difference among the fractured, 50% fatigue pre-stressed and new files for both PTG and PTU. There was no significant differences between different file sizes on both PTG and PTU.

There were no significant differences in nanohardness (Table 5) and elastic modulus (Table 6) between the edge and the shank of the file for both PTU and PTG.

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**Table 1:** Number of cycles to fracture for protaper gold and protaper universal in a curvature of 40º with 6mm radius.

| Fatigue test (NCF) | | |
|---|---|---|
| Files | $PTU^{j}$ | $PTG^{i}$ |
| S1 | $1074.2 \pm 168.7^{lm}$ | $1750.4 \pm 129.1^{k}$ |
| S2 | $813.3 \pm 112.8^{nqt}$ | $1388.8 \pm 166.5^{s}$ |
| F1 | $744.0 \pm 151.9^{n}$ | $1168.2 \pm 126.1^{l}$ |
| F2 | $677.6 \pm 172.5^{n}$ | $985.2 \pm 135.5^{m}$ |
| F3 | $564.8 \pm 90.7^{r}$ | $835.5 \pm 119.3^{q}$ |

*Different superscript letters indicate statistically significant difference. ($p < .05$)*

**Table 2:** Bending moment for protaper gold and protaper universal.

| Bending moment (g-cm) | | |
|---|---|---|
| Files | $PTU^{b}$ | $PTG^{a}$ |
| S1 | $9.0 \pm 1.9^{d}$ | $4.8 \pm 0.8^{c}$ |
| S2 | $21.7 \pm 2.3^{fu}$ | $8.8 \pm 1.3^{d}$ |
| F1 | $24.4 \pm 2.6^{f}$ | $14.8 \pm 2.9^{e}$ |
| F2 | $47.1 \pm 4.0^{h}$ | $34.0 \pm 5.0^{g}$ |
| F3 | $57.2 \pm 5.3^{p}$ | $39.5 \pm 4.3^{go}$ |

*Different superscript letters indicate statistically significant difference. ($p < .05$)*

**Figure 10:** The scanning electron micrograph of the fractured surface showing fatigue failure. (A) PTG F1 with two crack origins at the cutting edge (arrows). (B) A higher magnification view of one crack origin (arrow). (C) PTU F1with one crack origin (arrow). (D) A higher magnification view of (C).

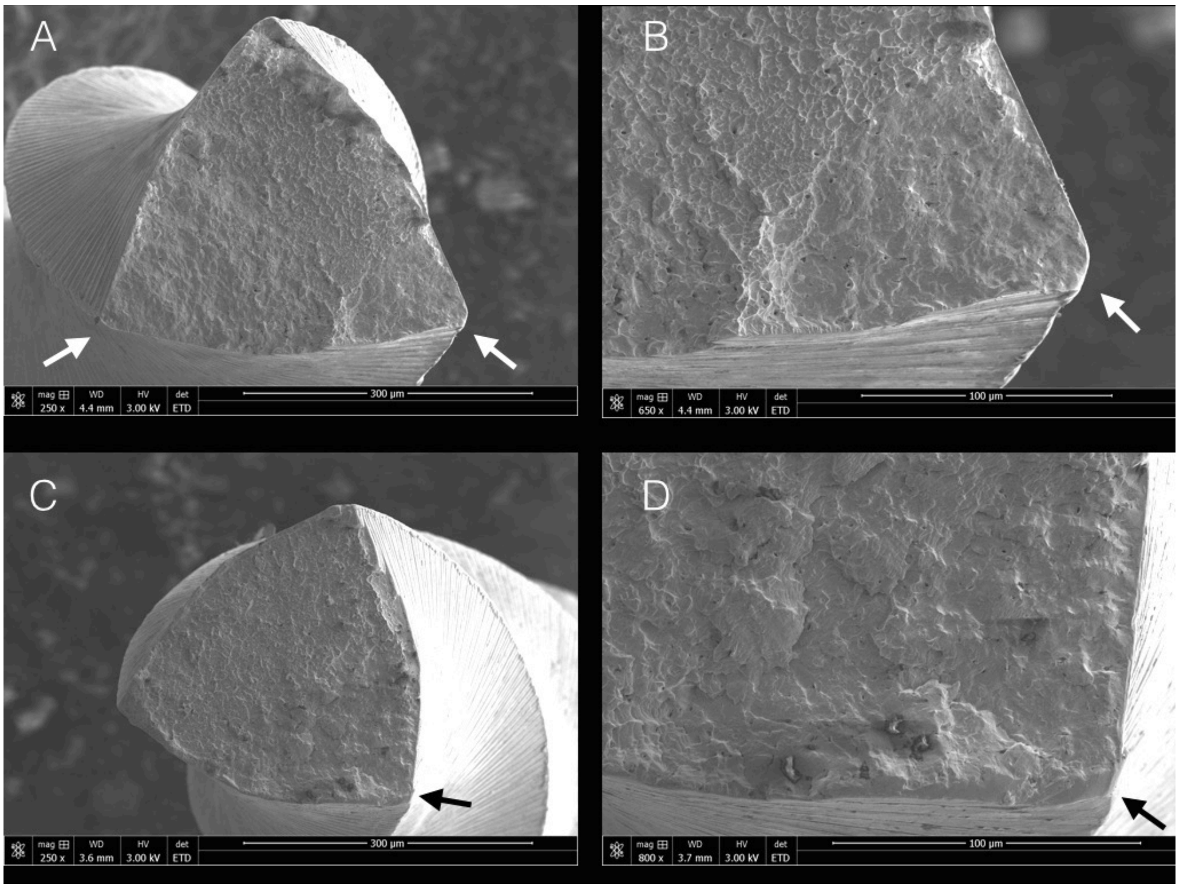

**Figure 11:**: The scanning electron micrograph of the fractured surface showing fatigue failure. (E) PTG F2 with one crack origin (arrow). (F) A higher magnification view of (F). (G) PTU F2 with one crack origin (arrow). (H) A higher magnification view of (G).

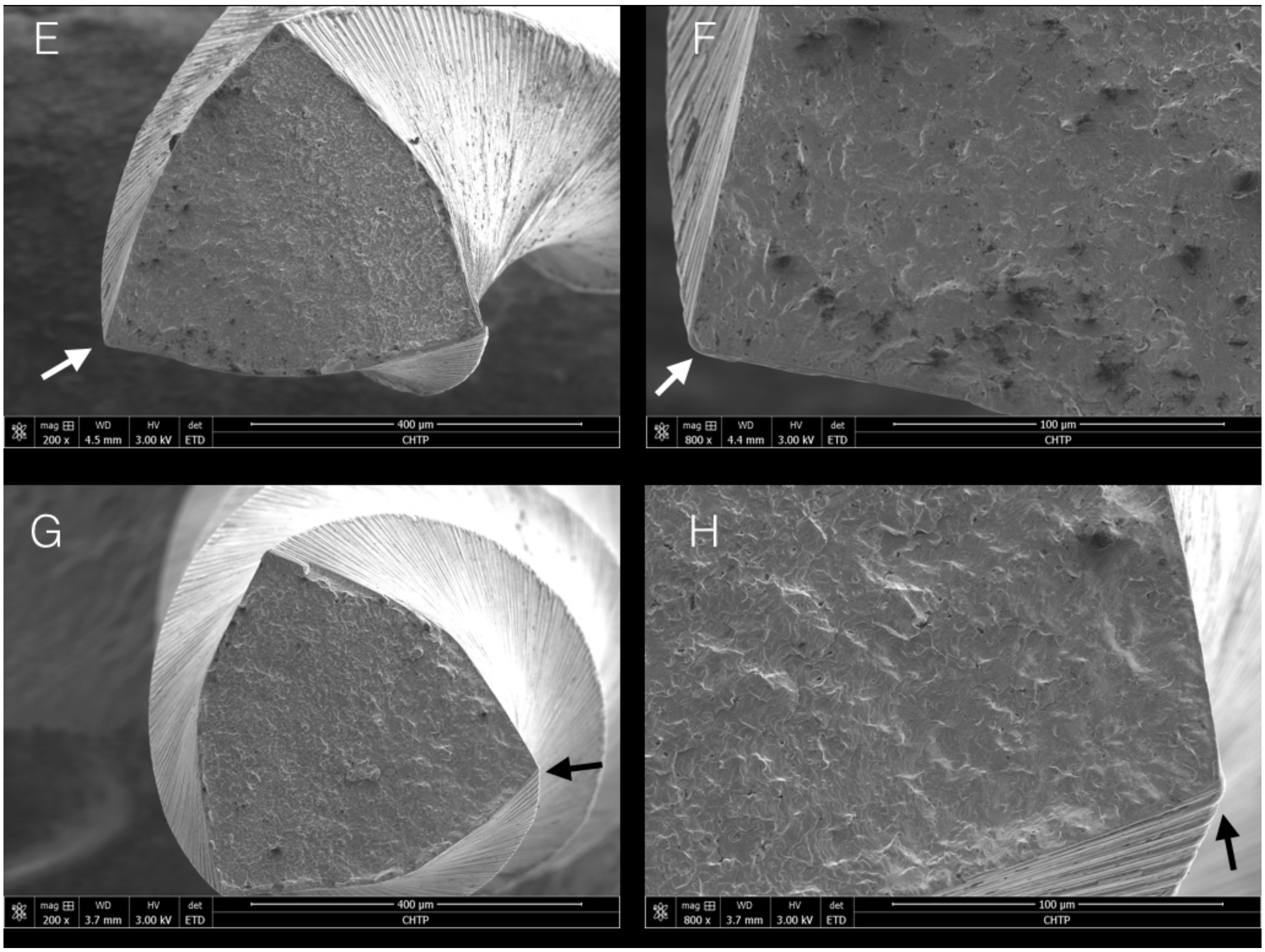

**Figure 12:** Differential scanning calorimetry of unused and fractured S1 for PTG and PTU NiTi instruments.

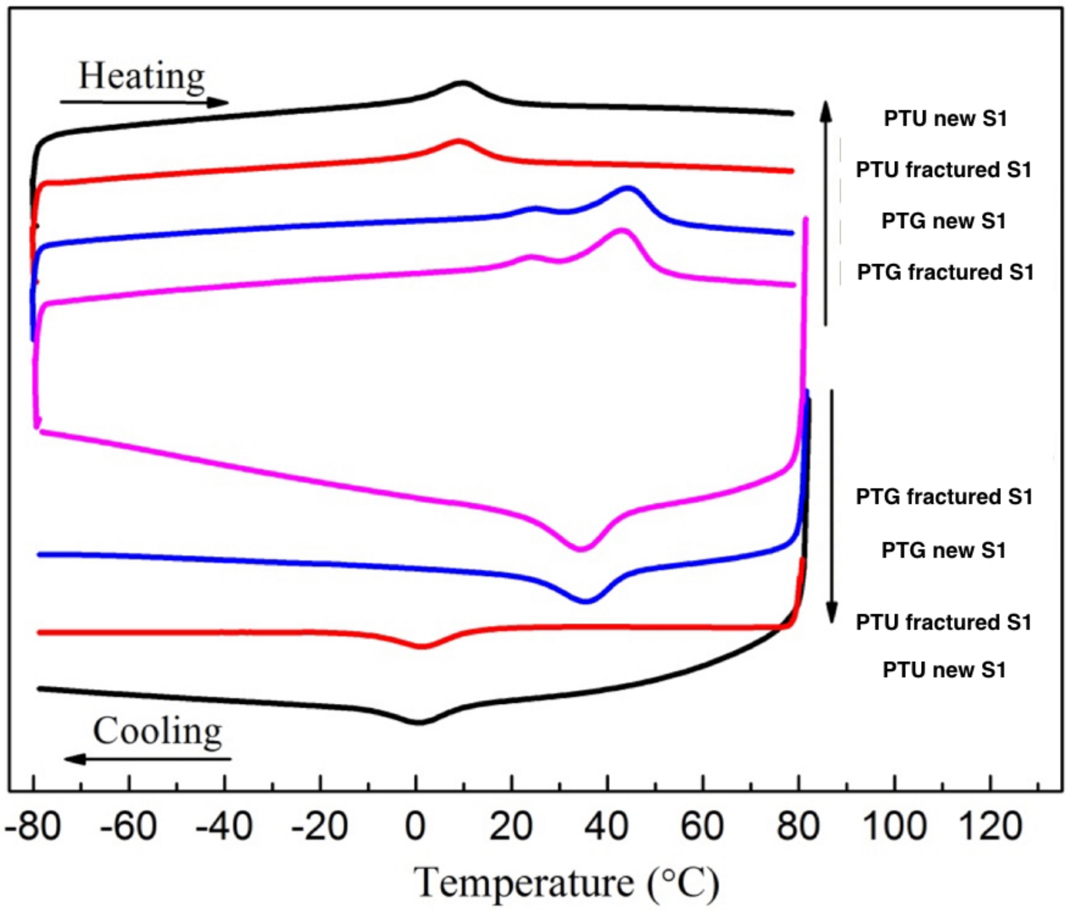

**Figure 13:** Differential scanning calorimetry of unused and fractured F1 for PTG and PTU NiTi instruments.

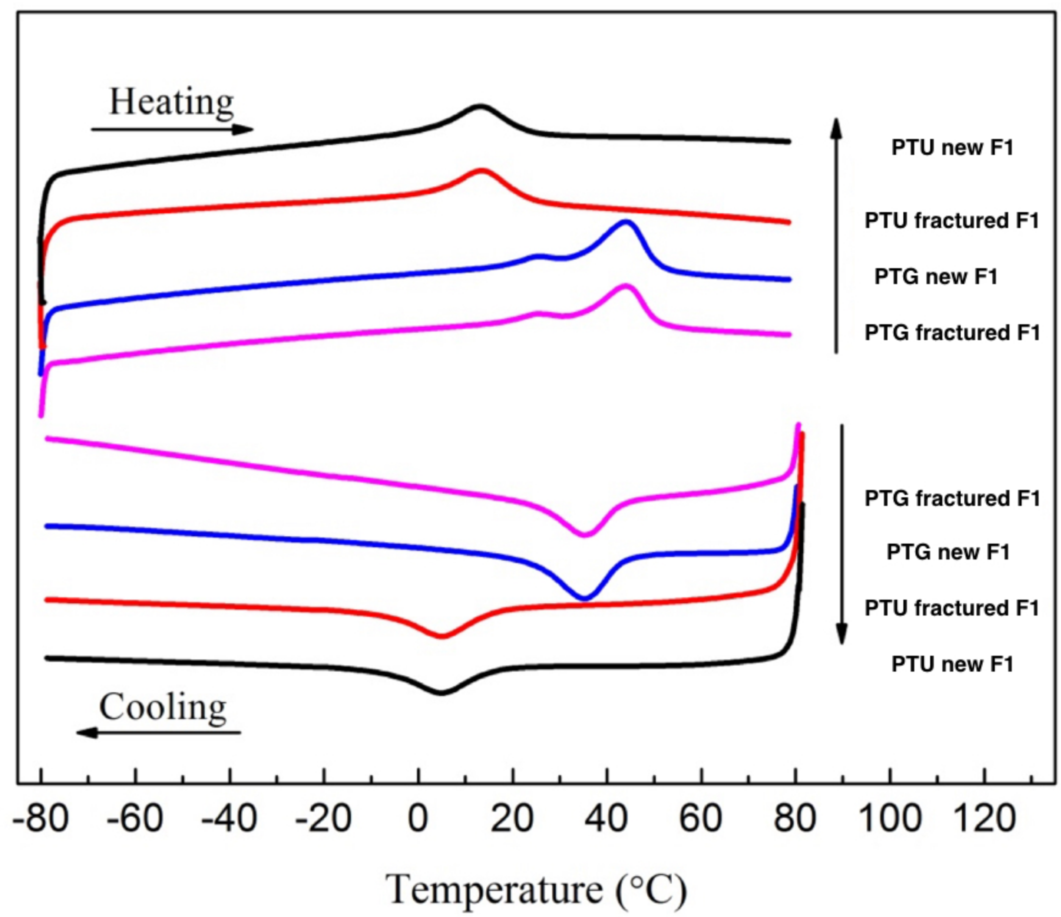

**Figure 14:** Differential scanning calorimetry of unused and fractured F2 for PTG and PTU NiTi instruments.

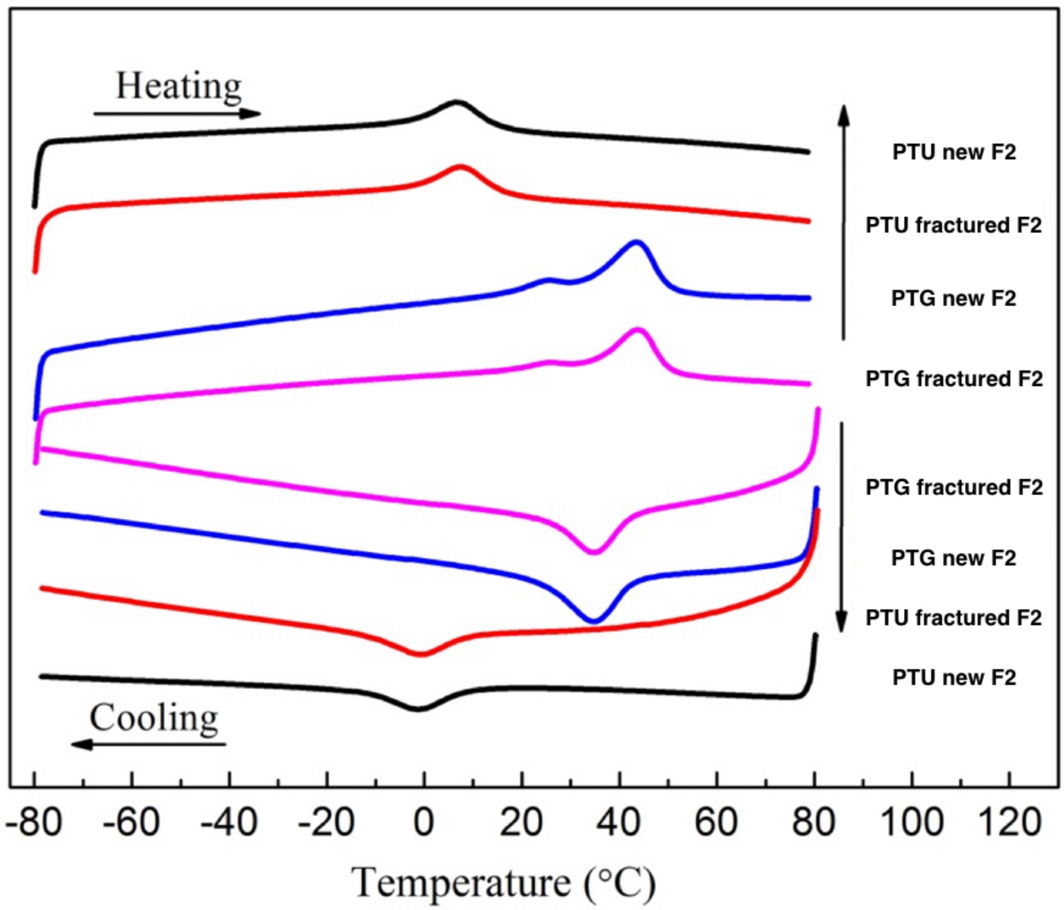

**Table 3:** The mean and standard deviations of nanohardness

| | PTU [a] | | | PTG [b] | | |
|---|---|---|---|---|---|---|
| Files | NEW [c] | 50% fatigued [c] | Fractured [c] | NEW [c] | 50% fatigued [c] | Fractured [c] |
| S1 | 3.60±0.33[df] | 3.61±0.75[df] | 3.63±0.46[df] | 3.30±0.36[df] | 3.30±0.19[df] | 3.35±0.39[df] |
| F1 | 3.49±0.57[df] | 3.60±0.37[df] | 3.65±0.38[ef] | 3.32±0.47[df] | 3.34±0.36[df] | 3.37±0.39[df] |
| F2 | 3.61±0.29[de] | 3.64±0.38[de] | 3.70±0.57[eg] | 3.33±0.21[fh] | 3.32±0.46[fh] | 3.36±0.22[fh] |

Different superscript letters indicate statistically significant difference. ($p<0.05$)

**Table 4:** The mean and standard deviations of MoE

| | PTU [a] | | | PTG [b] | | |
|---|---|---|---|---|---|---|
| Files | NEW [c] | 50% fatigued [c] | Fractured [c] | NEW [c] | 50% fatigued [c] | Fractured [c] |
| S1 | 48.35±5.76[d] | 51.92±10.84[d] | 52.23±6.61[d] | 47.55±5.12[df] | 47.55±2.78[df] | 48.19±5.68[d] |
| F1 | 50.25±8.21[d] | 51.85±5.34[d] | 52.48±5.46[de] | 47.74±6.72[df] | 48.13±5.19[df] | 48.50±5.66[d] |
| F2 | 51.91±4.13[d] | 52.41±5.54[de] | 53.24±8.23[de] | 47.87±3.04[df] | 47.86±6.61[df] | 48.34±3.18[d] |

Different superscript letters indicate statistically significant difference. ($p<0.05$)

**Table 5:** The mean and standard deviations of nanohardness for the edge and shank tested area.

| Files | | PTU [a] | | | PTG [b] | | |
|---|---|---|---|---|---|---|---|
| | | NEW [c] | 50% fatigued [c] | Fractured [c] | NEW [c] | 50% fatigued [c] | Fractured [c] |
| S1 | Shank [d] | 3.60±0.37 | 3.59±0.89 | 3.61±0.51 | 3.30±0.37 | 3.29±0.20 | 3.33±0.26 |
| | Edge [d] | 3.61±0.27 | 3.64±0.49 | 3.66±0.42 | 3.31±0.38 | 3.32±0.20 | 3.35±0.64 |
| F1 | Shank [d] | 3.56±0.57 | 3.60±0.44 | 3.64±0.39 | 3.31±0.53 | 3.34±0.39 | 3.36±0.46 |
| | Edge [d] | 3.61±0.82 | 3.61±0.21 | 3.66±0.41 | 3.32±0.39 | 3.35±0.36 | 3.38±0.25 |
| F2 | Shank [d] | 3.60±0.22 | 3.63±0.45 | 3.68±0.68 | 3.33±0.26 | 3.32±0.53 | 3.35±0.27 |
| | Edge [d] | 3.61±0.43 | 3.65±0.28 | 3.73±0.36 | 3.32±0.11 | 3.34±0.34 | 3.37±0.09 |

Different superscript letters indicate statistically significant difference. ($p<0.05$)

**Table 6:** The mean and standard deviations of MOE for the edge and shank tested area.

| Files | | PTU [a] | | | PTG [b] | | |
|---|---|---|---|---|---|---|---|
| | | NEW [c] | 50% fatigued [c] | Fractured [c] | NEW [c] | 50% fatigued [c] | Fractured [c] |
| S1 | Shank [d] | 48.53±3.55 | 51.65±12.78 | 52.02±7.28 | 47.50±5.32 | 47.41±2.90 | 47.97±3.80 |
| | Edge [d] | 48.00±9.60 | 52.45±7.02 | 52.65±6.02 | 47.66±5.48 | 47.84±2.91 | 48.65±9.17 |
| F1 | Shank [d] | 51.23±8.15 | 51.77±6.40 | 52.37±5.66 | 47.71±7.58 | 48.06±5.53 | 48.42±6.69 |
| | Edge [d] | 48.29±9.19 | 52.02±2.98 | 52.70±5.87 | 47.80±5.58 | 48.28±5.21 | 48.65±3.62 |
| F2 | Shank [d] | 51.86±3.18 | 52.32±6.42 | 52.99±9.73 | 47.88±3.68 | 47.73±7.63 | 48.22±3.88 |
| | Edge [d] | 52.01±6.24 | 52.58±4.04 | 53.74±5.21 | 47.84±1.55 | 48.11±4.93 | 48.57±1.34 |

Different superscript letters indicate statistically significant difference. ($p<0.05$)

# Chapter 4: Discussion

Stress-strain analysis and fatigue resistance performances of the PTG and PTU systems are strongly influenced by the unique manufacturing processes of the instruments in spite of the undistinguishable design. In rotary ProTaper files, cyclic failure is more predictable than torque failure (Wei et al., 2007). While in manual ProTaper files torsional failure is more evident (Shen et al., 2007). Appreciating the differences between the PTG and PTU files is of a great importance for the practitioner to be in a position to implement this new technology in the daily challenging cases.

The transformation behavior of NiTi alloys is considerably affected by the thermomechanical processing of the files. A one stage transformation of the austenite (A) - martensite (M) or a two stage (A-R-M) martensitic transformation may occur, in reliant to the thermomechanical treatments in the near equimatic NiTi alloy, (Otsuka et al., 2005).

In general, the one stage transformation A-to-M occurs in NiTi alloys with higher Ni percentage, while the supplementary heat treatment will build the two stage transformation A-R-M afterword, which produces the dispersed Ti3Ni4 precipitates in the austenitic matrix (Otsuka et al., 2005, Duerig 1990).

The R-phase can be considered as a prospective martensite phase and the comparative partiality of the R-phase over martensite in the existence of fine particles may help to understand the change form one to the two stage transformation, as the Ti3Ni4 particles are not amenable to the formation of martensite, that is linked to a sizable lattice distortion.

The martensitic transformation occurs in two stages of A-R-M as the R-phase is favoured by the growth of the Ti3Ni4, and the martensite needs additional cooling to be formed (Otsuka et al., 2005). Recently it was documented that ProFile Vortex instruments have only a single definite peak A-to-M on heating and cooling (Shen et al., 2015). While Vortex Blue had a two stage transformations (Tsujimoto et al., 2014) taking in consideration that Vortex Blue files are basically manufactured from M-Wire. It's may be interesting to know that the Af temperatures of Vortex Blue are 38°C which is lower than 50°C of ProFile Vortex instruments (Tsujimoto et al., 2014, Shen et al., 2015). Application of stress above a critical level that happened when the ambient temperature is higher than the $A_f$ temperature of the material is a direct result of the superelasticity of the material.

That's why the pseudo-elasticity will be available only when the working temperature for conventional superelastic NiTi files is higher than the $A_f$. The superelastic ProFile and ProTaper rotary files have their $A_f$ temperatures below 37°C (Alapati et al., 2009, Miyai et al., 2006, Shen et al., 2011b). The presence of stable martensite prevents the rebound effect after unloading of the thermomechanically treated CM Wire files and that's why the working temperature is lower than the $A_f$. In this study, PTG instruments DSC results revealed that a two stage specific transformation behavior, showing that there is an intermediate R-phase in the reverse transformation of the alloy, which might suggest the complex phase transformation behavior related to the manufacturing process. Interesting finding in this metallurgic characterization of PTG files showed that it has a high $A_f$ temperature, similar to CM Wire besides the two stage specific conversion behavior (Shen et al., 2011b). These martensite modifications can be linked to the elevated transformation temperatures found in PTG files and also justifies the variance in fatigue resistance between the PTG and PTU files. Figueiredo et al (2009) showed that the NCF in martensitic NiTi wires may reach 100 times greater than in stable and superelastic austenitic NiTi. In the current study, PTG showed higher flexibility than PTU. Higher flexibility file will experience less stress under a given strain consequently allowing a longer fatigue lifetime taking in consideration that other variables like cross section and design are the same ones. The number of loading cycles needed to start a fatigue fracture and to extend it to a critical size can be applied to express the fatigue life of the element. In martensite, the crack propagation mechanism represented as a very slowly progressing large number and highly branched cracks. In contrary to the superelastic NiTi, where a faster progressing small number of cracks are present (McKelvey & Ritchie 2001). CM Wire NiTi files may show multiple crack origins on the fracture surface (Shen et al., 2011a). However, in this study, thermally modified PTG instruments showed greater cyclic fatigue resistance over the superelastic PTU files, with no significant fractographic differences between them. Normally, the fatigue life of an instrument is a function of the radius of curvature and the size of the instrument.

A widely known fact that the instrument's fatigue life is influenced by the radius of curvature and the size of the instrument (Pruett et al., 1997, Nguyen et al., 2014, Pérez-Higueras et al., 2014). And actually the maximum tension on the surface of the file is determined by these two factors. As different brands of NiTi files showed a connection between the low cycle fatigue life and the surface tension amplitude as a power function relation that agrees with the Coffin-Mason equation (Cheung & Darvell 2007). That's why the greater the ratio of the radius of the file for the breakage point to the radius of curvature i.e. strain the less is the fatigue life. This study also confirmed Nguyen et al. (2014) and Pérez-Higueras et al. (2014) findings that the S1 PTU files had statistically significantly improved cyclic fatigue resistance than the F1, F2, and F3 files which appeared to be the same for PTG files.

The resistance to plastic deformation can be measured using Vickers microhardness test, however the superelastic alloy having an excellent elastic recovery, and that's why the classical Vickers hardness test is not suitable and may lead to uncertainty in hardness (Cheng 2004). Nanoindentation is a sensitive method of mechanical characterization for a broad range of materials in terms of calculating the resistance to elastic and plastic distortion as well as recording the limited phase-changes (Jamleh et al. 2012).

In 2012, a study by Ye and Gao showed that the microhardness of M-Wire instruments amplified as the low cycle fatigue test continued for about 60% or more the NCF, as it was suggested, that local work-hardening of the instrument will eventually lead to cyclic fatigue and the plastic deformation is required to release the residual stresses and the presumed escalation in the resistance to indentation (Ye & Gao 2012). However, Jamleh et al. (2012) showed that stressing the instruments have considerably decreased the nanohardness in the NiTi files. The distortion process controlling the cyclic mechanical behavior of a NiTi files is complex. It may involve processes like micro-twining and gliding dislocation that happen throughout cyclic loading (Gloanec et al. 2013). It's important to follow these changes in hardness during loading (Gloanec et al. 2013). A more recent study by Shen et al. (2014) showed that the nanohardness of K3 files somewhat raised after loading tests, but this was not the case with K3XF files. In the present study, from a longitudinal view, there were no work-hardening effects on both PTG and PTU files during the loading cycle.

The thermal and mechanical treatment of the superelastic file as well as the structure of the alloy seems to affect the hardness levels (Zinelis et al. 2010). And going back to Shen et al. (2014) study, we can see that the mechanical treatment reduced the hardness of the thermally processed NiTi files more than the regular files compared to the non significant difference between the new and loaded files Consequently, it was not surprising that the current study results indicated that thermomechanical treated PTG had slightly lower nanohardness than traditional superelastic PTU files.

Based on this study, we can say PTU and PTG NiTi instruments can be chosen in certain cases based selectively on applications as per the case situations. An example of that would be the use of PTG file in cases where there is a sudden canal curvature as it will resist the cyclic fatigue more efficiently than PTU especially in smaller sizes.

## Conclusion

Under the limitations of this study:

PTG files were significantly more flexible and resistant to fatigue than PTU files. Fatigue life of size S1 and S2 was significantly longer than that of sizes F1–F3 files. PTG exhibited different phase transformation behavior than PTU. The $A_f$ temperature of PTG instruments (50.1°C ±1.7°C) was higher than PTU instruments (21.2°C±1.9°C). Furthermore, PTG instruments showed a two stage transformation behavior. PTU instrument demonstrated higher nanohardness values compared to PTG. The fatigue process had no significant effect on the hardness and the modulus of elasticity of both NiTi instrument.

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