INFLUENCE OF CYCLIC TORSIONAL LOADING ON THE FATIGUE RESISTANCE OF K3XF INSTRUMENTS

by

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BDS, King Saud University 2009

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE OF

MASTER OF SCIENCE

in

THE FACULTY OF GRADUATE AND POSTDOCTORAL STUDIES

(Craniofacial Science)

THE UNIVERSITY OF BRITISH COLUMBIA

(Vancouver)

May 2015

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Abstract

Objective: To evaluate the torsional and cyclic fatigue behavior of post-machining heat-treated K3XF and conventional K3 nickel titanium (NiTi) instruments.

Methodology: New K3XF and K3 files size 25/0.04 (n = 15) were tested in torsion and fatigue tests until fracture to determine the mean number of cycles to failure (NCF) and torque to failure. The cyclic torsional loading experiment was conducted; K3XF and K3 files (n = 30 in each group) were programmed to repeatedly rotate from zero angular deflection to 180° and then return to zero torque. Each rotation was defined as one cycle. Each file was subjected to 10 cycles of torsional loading. Fifteen files from each group were subsequently tested in torsion until fracture. Also, fifteen files subjected to cyclic torsional loading were examined using a three-point bending apparatus to obtain the mean number of cycles to failure. The fracture surface was examined with a scanning electron microscope. The crack-initiation sites and the percentage of dimple area of the whole fracture cross-sectional area were recorded.

Results: There was no statistically significant difference between the new K3 and K3XF instruments in the maximum torque or maximum angular deflection. For the previously cycled files, K3XF demonstrated higher torque at fracture values than K3 Instruments ($P < .05$). The fatigue resistance of K3XF was significantly higher than K3 in both the new and previously cycled groups ($P < .05$). The NCF value of K3XF with torsional loading was even higher than that of K3 files without torsional loading, although there was no significant difference. New K3XF files demonstrated a significantly higher NCF than previously cycled files ($P < .05$).

Conclusions: Cyclic torsional loading decreased the cyclic fatigue resistance of K3XF and K3 instruments although it did not affect their torsional properties. K3XF demonstrated better cyclic fatigue resistance than K3 for both new and previously torqued files.

Preface

This thesis is an original, unpublished, and independent work by Abdulmohsen Alfadley. The project was performed under the supervision and guidance of Dr. Ya Shen and Dr. Markus Haapasalo.

Abdulmohsen Alfadley performed the following aspects of the research project; the fatigue testing, data collection and scanning electron microscopy imaging. The torsion tests were done by Dr. Les Campbell at the calibration department of D&S Dental in Tennessee. Dr. Ya Shen and Dr. Zhejun Wang performed statistical analysis of the research. The relative contribution of the collaborators in this project was as follows; Dr. Abdulmohsen Alfadley 60%, Dr. Markus Haapasalo 10%, and Dr. Ya Shen 30%. Dr. Ya Shen and Dr. Markus Haapasalo reviewed the manuscript and provided an invaluable feedback.

This research was generously supported by The Canadian Academy of Endodontics and the American Association of Endodontists. SybronEndo donated the files used in this study.

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List of Abbreviations

NiTi: Nickel-Titanium

CM: Controlled memory

TF: Twisted File

DSC: Differential scanning calorimetry

SEM: Scanning electron microscope

A_f: Austenite finish temperature

N_f: Total number of revolutions to failure

NCF: Number of cycles to failure

ISO: International Organization for Standardization

Acknowledgements

I would like to express my deepest appreciation and gratitude to Dr. Ya Shen for her continuous support and guidance throughout this project. She was always there to address my questions and concerns and provided crucial assistance in the statistical analysis of this study. It has been an absolute pleasure to have Dr. Shen as a research mentor and teacher.

Thank you to Dr. Markus Haapasalo, as my research co-supervisor. His extensive knowledge and understanding of endodontic research has significantly contributed to my professional and personal growth. I appreciate the time he took to review and reflect on this manuscript.

I was also blessed to have Dr. Jeffery Coil as my program director and as a committee member. I learnt a lot from his endodontic knowledge and experience, as well as from his professional and friendly approach with his patients. I will always remember the days we spent in Haida Gwaii, and the endodontic surgeries which we performed. Thank you for those memories and support.

Thank you to Dr. Jolanta Aleksejuniene for her participation as a research committee member and for the suggestions she made throughout this project. My appreciation also goes out to Dr. Les Campbell for his contribution in the torsional testing and Dr. Tianfeng Du for her help with the SEM imaging.

Finally, a special thank you also goes out to all the endodontic faculty, staff, and residents for their continuous support throughout the past three years.

Dedication

To my wife, Aroob, my parents, brothers, and sisters, and friends who have always been supportive throughout my education.

Chapter 1. Introduction and review of literature

1.1 Background

Thorough chemomechanical debridement of the root canal system is a key to the success of endodontic treatment. While chemical cleaning aims to remove all organic substances and microbial biofilms from the root canals, one of the primary objectives of mechanical shaping is to develop a continuously tapering canal preparation (Schilder 1974) which allows for optimal delivery of disinfecting irrigants, intracanal medicaments, and filling of the root canal system. Traditionally, stainless steel instruments were used to prepare root canals for almost a century. However, stainless steel instruments are stiff and associated with increased operator fatigue. In addition, when used in the preparation of curved root canals, the restoring forces of the instruments tend to return the instrument back to its original shape resulting in canal transportation. These limitations have initiated a need for change. Therefore, a variety of instrumentation techniques and devices have been introduced to minimize the incidence of procedural errors caused by the use of stiff instruments in preparing curved root canals.

Walia et al. (1988) introduced the use of nickel titanium (NiTi) root canal files to overcome the rigidity of stainless steel instruments. This has been a major breakthrough in the field of endodontics as the superelasticity of NiTi alloy allows the instrument to effectively follow the original path of the canal (Thompson 2000). Furthermore, in comparison to stainless steel instruments, NiTi files offer increased flexibility, rapid and centered canal preparation, safer preparation of curved canals, improved cutting efficiency, superior torsional resistance, and

improved treatment outcome (Walia et al. 1988, Glosson et al. 1995, Short et al. 1997, Thompson 2000, Cheung and Liu 2009). In spite of all these advantages, a main limitation in the use of NiTi instruments is the risk of their fracture (Sattapan et al. 2000, Shen et al. 2009).

1.2 Instrument fracture

Rotary NiTi instrument fracture during root canal preparation is a serious concern for both the patient and the dental practioner. Although the sole mechanical retention of the fragment may not predispose the tooth to post-treatment disease, it may, however, impede access for chemomechanical debridement beyond the fractured fragment. According to Lin et al. (2005), the degree of asepsis or cleanliness of the root canal space is the principal factor influencing treatment outcome. Spili et al. (2005) evaluated the impact a fractured instrument may have on the outcome of endodontic treatment. The results of the study suggested that the presence of periapical lesion was the main factor impairing the treatment outcome.

Instrument fracture is a complex, multifactorial event (Parashos et al. 2004, Shen et al. 2009). Factors influencing the fracture risk of NiTi instruments include the skill level of the operator, instrument design and dimension, instrumentation technique, radius and angle of root canal curvature, torque setting, surface condition, and rotation speed (Cheung 2009, Shen et al. 2013, Campbell et al. 2014).

The reported incidence of rotary NiTi instrument fracture in clinical practice for files used multiple times varies from 3% to 21% (Sattapan et al. 2000, Parashos et al. 2004, Peng et al. 2005, Alapati et al. 2005, Shen et al. 2006). However, the results of the previously published studies should be interpreted with caution due to the presence of several uncontrolled variables

such as; number of uses, instrument size and taper, torque setting, rotational speed, and complexity of the cases. Interestingly, Arens et al. (2003) reported that approximately 1% of NiTi instruments fractured during their first use.

In the unfortunate event of instrument fracture, the clinician must determine whether it is feasible or not to attempt removal of the fragment. There is a general agreement in the endodontic literature that the location of the broken segment in relation to root canal curvature is a key determinant of instrument retrieveability. Therefore, injudicious removal of radicular dentin for the sake of instrument removal cannot be justified. In any event, the patient should be informed about the incidence and what impact it may have on treatment outcome.

Fracture of a NiTi instrument may occur from either torsional or cyclic fatigue or both in combination (Sattapan et al. 2000, Cheung et al. 2005, Cheung 2009). Torsional fracture occurs when the instrument tip is locked into the canal while the shaft continues to rotate. In this scenario, the handpiece torque exceeds the torsional strength of the metal and consequently leads to plastic deformation and ultimately instrument fracture (Sattapan et al. 2000, Cheung 2007). Cyclic fatigue failure occurs as a result of rotating an instrument in a root canal curvature generating tension and compression cycles at the point of maximum flexure causing work hardening of the metal and subsequently fracture (Pruett et al. 2007, Plotino et al. 2009). Therefore, a rotary NiTi instrument needs to have sufficient shear strength in order to prevent torsional failure and have a high resistance to cyclic fatigue, allowing optimal preparation of curved systems (Shen and Cheung 2013).

Several studies have attributed the cause of rotary NiTi instrument fracture to metal fatigue (Parashos et al. 2004, Cheung et al. 2005, Peng et al. 2005, Shen et al. 2006, Shen et al. 2009). For instance, Cheung et al. (2005) reported that 93% of ProTaper S1 instruments failed due to flexural fatigue mode. In contrast, Sattapan et al. (2000) examined the lateral view of fractured Quantec rotary instruments following clinical use and reported that approximately 56% of these instruments failed due to torsional fracture. Furthermore, a scanning electron microscopy (SEM) study suggested that the fracture of rotary NiTi instruments was caused by a single overload incidence during canal preparation rather than accumulated metal fatigue (Alapati et al. 2005). Of note is that rotary NiTi instruments are subjected to a combination of flexural fatigue and torsional stress during root canal preparation which leads to instrument separation due to mixed failure mode (Gambarini 2001, Cheung 2009).

As of today, there is no specification or international standard to test the cyclic fatigue resistance of endodontic rotary instruments (Gambarini et al. 2009). This presents a dilemma to the endodontic discipline, as the comparison of fatigue behavior between different studies is difficult and may have some inherent problems, although it may allow us to rank the performance of different rotary systems. Ideally, the device for flexural fatigue testing should confine the rotatory NiTi file into a precise trajectory, in terms of the radius and angle of the curvature and the location of the maximum curvature (Shen and Cheung 2009). Four methods of imposing a curvature on a rotating NiTi instrument have been described: (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).

An obvious limitation of the most currently available devices is that the handpiece is held in a static position during the test which does not mimic the clinical situation. In fact, it has been suggested that incorporating a pecking motion seems to enhance fatigue life (Li et al. 2002, Yao et al. 2006) as the cyclic load is distributed over some length of the rotating instrument instead of acting at just one particular location. Cheung introduced a three-point bending apparatus to study the fatigue behavior of rotary NiTi instrument (Cheung and Darval 2007, Cheung et al. 2007). The V-shaped groove on the lowest pin of the apparatus maintains the position of the test instrument during rotation. An additional advantage of this device is that it allows the experiment to be conducted in both dry and wet environments (Shen et al. 2012, Shen et al. 2014).

According to the American Dental Association specification (ANSI/ADA) no. 28 (ANSI/ADA 1988), the torsional properties of endodontic instruments can be evaluated as the torque and angle of rotation required to cause instrument fracture. Several studies have classified the mechanism of instrument breakage into torsional fracture when macroscopic, plastic deformation is present (Sattapan et al. 2000, Parashos et al. 2004, Peng et al. 2005). However, it should be noted that such plastic deformation may not always appear when the instrument undergoes shear failure (Cheung et al. 2005, Wei et al. 2007).

The measurement of the torsional strength of root canal instruments is typically performed in a torsiometer, according to the procedure described by the ANSI/ADA no. 28 (ANSI/ADA, 2002). The International Organization for Standardization (ISO/ANSI) specifications have prescribed a test method for stainless-steel root canal reamers and files where 3 mm of the tip of the instrument is rigidly fixed and subjected to twisting in a clockwise or a counterclockwise

direction. A wide variety of rotary NiTi instruments have been tested in this method. However, torsional failure due to such a monotonic condition rarely occurs clinically. Both the ADA method (ANSI/ADA Specification No. 28, 2002) and ISO protocol #3630-1 (ISO 2008) only simulate torsional bending in straight canals, as the torque is measured in relation to the rotation axis. Rotary instruments are subjected to varying loads in actual clinical situations (Blum et al. 1999), and as a consequence fractures are likely to be a result of both repetitive flexural and torsional stresses.

1.3 Heat treatment of NiTi instruments

K3 (SybronEndo, Orange, CA) is a second generation NiTi rotary instrument designed by Dr. John McSpadden in 2003. In contrast to the first generation rotary systems, K3 demonstrates a unique cross-sectional design; a slightly positive rake angle for greater cutting efficiency, wide radial lands, and a peripheral blade relief for reduced friction. In the lateral aspect, the K3 has a variable pitch and variable core diameter, which makes the instrument stronger close to the tip of the file (Haapasalo and Shen 2013).

Changes in the production may increase efficiency and safety of NiTi rotary instruments, such as an improvement in the manufacturing process or the use of new alloys with superior mechanical properties (Gambarini et al. 2011). At the beginning of this century, a series of studies (Kuhn et al. 2001, Kuhn et al. 2002, Hayashi et al. 2007, and Yahata et al. 2009) found that changes in the transformation behaviour, via heat treatment, increased the flexibility of NiTi endodontic instruments. Since this discovery, heat-induced manipulations have been used to influence or alter the properties of NiTi endodontic instruments. Today, thermomechanical processing is the

most fundamentally used procedure to adjust the transition temperatures of NiTi alloys (Frick et al. 2005, Gutmann and Gao 2012, Shen et al. 2013), and optimize the fatigue resistance of NiTi endodontic files.

In the last few years, a wide variety of thermomechanical processing and manufacturing technologies have been developed in order to optimize the microstructure and improve the mechanical properties of NiTi alloys. Recently, several new thermomechanically processed endodontic NiTi files have been introduced to the endodontic market, including HyFlex CM (HyFlex; Coltene Whaledent, Cuyahoga Falls, OH), K3XF (SybronEndo, Orange, CA), ProFile GT Series X (GTX; Dentsply Tulsa Dental Specialties, Tulsa, OK), ProFile Vortex, Vortex Blue, ProTaper Next, and ProTaper Gold (Dentsply Tulsa), TYPHOON™ Infinite Flex NiTi (TYP CM; Clinician's Choice Dental Products, New Milford, CT), and Twisted Files (TFs; SybronEndo).

M-Wire (Dentsply Tulsa Dental Specialties) was introduced in 2007, and is produced by applying a series of heat treatments to NiTi wire blanks. M-Wire instruments include Dentsply's ProFile GT Series X, ProFile Vortex, Vortex Blue, and ProTaper Next. In 2008, a new manufacturing process was also developed by SybronEndo (Orange, CA, USA), creating a NiTi endodontic instrument known as Twisted File. According to the manufacturer, Twisted File instruments were developed by transforming a raw NiTi wire in the austenite phase into the R-phase through a thermal process.

The design of K3 instruments was recently updated by SybronEndo, and the new system, K3XF, has been available since 2011. K3 and K3XF instruments are identical in shape and differ only in production, where K3XF instruments undergo post-machining heat treatment (Gutmann and Gao

2012). The main advantage gained by this specific heat treatment is not only does it improve the flexibility and strength of the file, but also it modifies the crystalline structure of the alloy. This structural modification enables the file to accommodate some of the internal stress caused by the grinding process (Gambarini et al. 2011, Shen et al. 2013b). Therefore, this new development may eliminate many of the drawbacks experienced by older generation files during the grinding process, and produce an instrument with superior mechanical properties. The manufacturer claims that K3XF provides clinicians with the basic features of the original K3, with an enhanced level of flexibility and resistance to cyclic fatigue with the proprietary R-phase technology (Ha et al. 2013). Several studies (Gambarini et al. 2011, Plotino et al. 2012, Ha et al. 2013, Shen et al. 2014) have shown that K3XF instruments have a fatigue resistance superior to K3 NiTi instruments. However, there is no statistically significant difference between K3 and K3XF in the torsional test (Ha et al. 2013, Shen et al. 2013b)

1.4 Metallurgical characterization

NiTi is known as an exotic metal because it does not conform to the standard rules of metallurgy. The NiTi alloys used in root canal treatment contain approximately 56% (wt) nickel and 44% (wt) titanium (Walia et al. 1988). The resultant material is an alloy with a 1:1 atomic ratio (equiatomic ratio) of the two elements (Lagoudas 2008). It is worth noting that only the NiTi alloys with nearly equiatomic ratio possess the unique superelasticity and shape memory effect. This is due to the narrow solubility range of the "TiNi" phase at 500°C or below, which becomes negligible at about 500°C (Otsuka and Ren 2005). Near equiatomic NiTi alloys contain three microstructural phases; austenite – a high temperature or parent phase with a B2 cubic crystal structure, martensite – a low-temperature phase with a monoclinic B190 structure, and R-phase

(Hodson et al. 1990, Bidaux et al. 1993, Torrisi 1999, Zhang and Sehitoglu 2004, Shen et al. 2013a). The transformation temperatures, the temperature where one phase begins to transform into another, determines the relative proportions of the various phases within the material, and hence the behavior of the final material (Brantly et al. 2001). The transformation temperatures can be altered by small changes in composition, impurities, and heat treatment during the manufacturing process (Cheung 2006, Lagoudas 2008). Various metallurgical techniques have been used to investigate the microstructure and phase transformation of different makes of NiTi instruments including differential scanning calorimetry, X-ray diffraction, optical microscopy, scanning electron microscopy, and qualitative energy-dispersive spectrophotometrics (Brantly et al. 2002, Otsuka and Ren 2005, Lagoudas 2008, Alapati et al. 2009, Shen and Cheung 2013).

Brantley et al. (2002) were the first ones in the endodontic literature to show that the structure of the NiTi rotary instruments can be conveniently investigated by differential scanning calorimetry (DSC). In this analytical modality, the difference in thermal energy supplied to a test specimen and an inert control is precisely measured and compared, when they are heated at the same rate. Phase transformations, of the test material, reveal endothermic peaks on the heating curves, and exothermic peaks on the cooling curves. The difference between the material's transformation temperatures and the ambient/working temperature determines the stability of the various NiTi phases (Lagoudas 2008). DSC results (Figure 1) showed that K3XF and K3 instruments have an Af temperature of about 25°C and 18°C, respectively (Shen et al. 2013b). This suggests both instruments have an austenite structure at body temperature and would exhibit a superelastic property during clinical application. However, the phase transformation peaks of K3XF were significantly higher than K3 instruments. On the heating plot for K3XF, two of the endothermic

peaks overlapped, which indicates that the reverse transformation of the alloy passes through the intermediate R-phase (Shen et al. 2013b). This reflects the complex relationship between phase transformation behavior and the manufacturing process.

Superelasticity is the main reason for using NiTi alloys in endodontic instruments, because it provides NiTi rotary files with superior flexibility and allows them to follow the complex anatomy of root canals, therefore reducing the risk of ledging and perforations (Kenneth 2001). In near equiatomic NiTi alloys, superelasicity is attributed to the reversible, stress-induced martensitic transformation. This transformation is driven by stress and influenced by the temperature difference between the working temperature and Af, the critical finishing temperature for the reverse transformation of martensite upon heating (McCormick et al. 1993). The superelasticity of NiTi allows the recovery of deformations formed from strain exerted up to 8%, compared with a maximum of less than 1% with other alloys such as stainless steel (Thompson 2000).

The R-phase is an intermediate phase with a rhombohedral structure which forms during forward transformation from martensite to austenite on heating and during reverse transformation from austenite to martensite on cooling. It occurs within a very narrow temperature range. (Otsuka and Ren 2005, Shen et al. 2013a). Two NiTi instruments produced by Sybron Endo, which uses R-phase technology are the Twisted File and K3XF. The Twisted File is manufactured with the R-phase alloy using a twisting method and research has reported that it exhibits a higher fatigue fracture resistance than some ground files (Gambarini et al. 2008, Kim et al. 2010, Bhagabati et al. 2012, Ha et al. 2013). In contrast, K3XF is manufactured using a grinding process rather than

a twisting process. This proprietary thermomechanical processing is a complex process that integrates hardening and heat treatment into a single process, which is performed on K3XF files after the grinding process. This treatment enhances the flexibility and strength of the material and allows internal stresses, from the grinding processes, to be absorbed into the material (Gambarini et al 2011, Shen et al. 2013b)

A Summary of the metallurgical and mechanical properties of K3 and K3XF instruments is provided in table 1.

Figure 1. (A) Differential scanning calorimetry curves of K3XF and K3 instruments. Heating (upper) and cooling (lower) curves are shown. (B) X-ray diffraction patterns for K3XF and K3 at 25 °C, showing the 3 major peaks for the (110), (200), and (211) atomic planes in austenite (Shen et al. 2013)

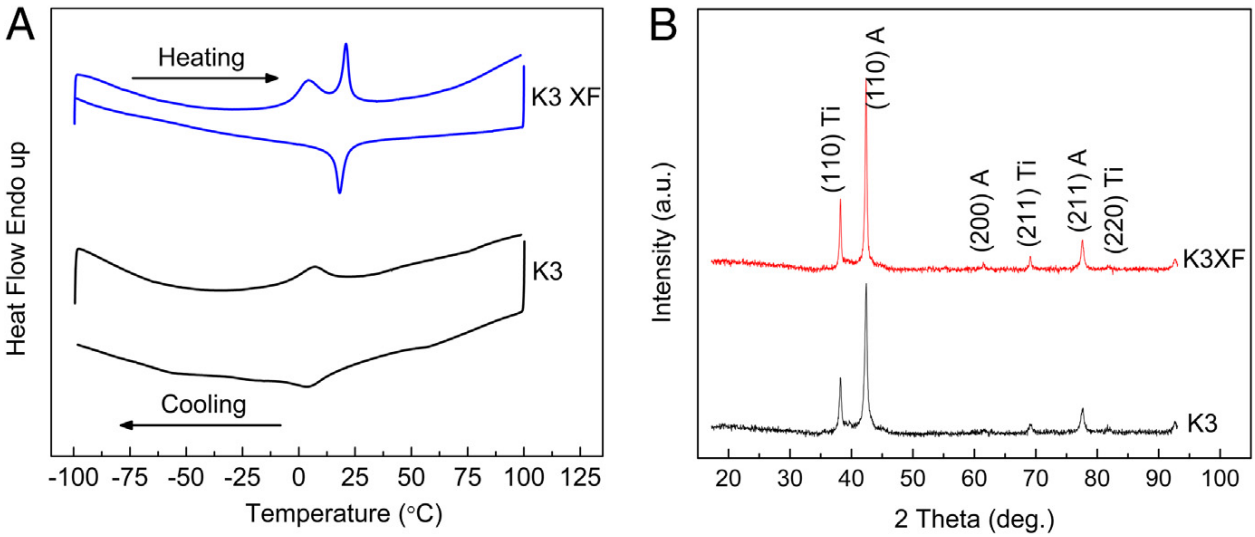


Table 1. Summary of the metallurgical and mechanical properties of K3 and K3XF instruments.

Properties	K3	K3XF
Ni Content (wt %)	54.7 (Zinelis et al. 2010) 54.2 (Shen et al. 2013)	54.0 (Shen et al. 2013)
Af (°C)	17.63 ± 1.76 (Shen et al. 2013b) 5.4 ± 6.8 (Miyai et al. 2006) 3.88 ± 3.21 (Hou et al. 2013)	24.89 ± 1.98 (Shen et al. 2013b)
Hardness (GPa)	3.47 ± 0.49 (Shen et al. 2014) 3.82 ± 0.22 (Shen et al. 2014)	2.99 ± 0.12 (Shen et al. 2014) 2.97 ± 0.16 (Shen et al. 2014)
Elastic modulus (GPa)	50.83 ± 4.54 (Shen et al. 2014) 54.51 ± 4.66 (Shen et al. 2014)	51.10 ± 4.54 (Shen et al. 2014) 50.63 ± 4.69 (Shen et al. 2014)
Torque at fracture (Nmm)	7.29~25.03 (Shen et al. 2013b) 26~140 (Ninan & Berzins 2013)	7.88~23.51 (Shen et al. 2013b)
Angle of rotation at fracture (°)	581.49~656.34 (Shen et al. 2013b) 1187~1412 (Ninan & Berzins 2013) 797 ± 147 (Shen et al. 2015)	710 ~ 747 (Shen et al. 2013) 810 ± 130 (Shen et al. 2015)
NCF	552.5 ± 91.54 (Gambarini et al. 2008) 579 ± 60.1 (Gambarini et al. 2008) 439 ± 64 (Hayashi et al. 2007) 542 ± 81 (Gambarini et al. 2011) 544 ± 122 (Shen et al. 2014) 489 ± 75 (Shen et al. 2014) 647 ± 139 (Shen et al. 2015)	651 ± 149 (Plotino et al. 2012) 1198 ± 279 (Gambarini et al. 2011) 977 ± 252 (Shen et al. 2014) 1146 ± 192 (Shen et al. 2014) 1507 ± 52 (Shen et al. 2015)

1.5 Existing literature on the influence of cyclic torsional loading on the fatigue resistance of K3 and K3XF instruments

The effect of previous torsional angular deformation on the flexural fatigue life of K3 and K3XF instruments has been previously studied (Barbosa et al. 2007, Bahia et al. 2008, Shen et al. 2015). Barbosa et al. (2007) subjected new K3 instruments to a pre-defined rotation (90°, 180°, or 420° angular deformation) before flexural fatigue testing in a small glass tube with a curvature of 45° and a 5 mm radius. They reported that as the prior angular deformation increases, the number of cycles attained under flexural fatigue decreases. Interestingly, a reduction in the fatigue life was recorded even when the initial torsional loads were below the elastic limit of the material, even at a minimum angular deformation of 90°. Hence, it is plausible that transient torsional overloads will act in tandem with flexural fatigue and reduce the resistance of NiTi files causing their failure in clinical situations.

Recently, Shen et al. (2015) evaluated the influence of previous angular deformation on cyclic fatigue resistance of K3XF instruments. In this study, new K3 and K3XF files were precycled to four conditions (0%, 25%, 50%, and 75% of the angular deflection) and fatigue resistance tests were subsequently performed. After torsional preloading, the total number of revolutions to failure (Nf) was measured for each file. It was noted that with both the K3 and K3XF instruments, the 25%, 50% and 75% torsional preloading groups, had significantly lower Nf than the no preloading group. Furthermore, the K3XF instruments had significantly higher Nf than the K3 in all preloading groups.

The contribution of cyclic torsional loading, instead of a single monotonic overload, to instrument fracture has been reported by Bahia et al. (2008). The authors also investigated the influence of cyclic torsional loading on the flexural fatigue resistance (45°angle and 5 mm radius of curvature) and torsional property (ISO 3630-1 protocol) of K3 instruments. The results showed that torsional preloading did not affect torsional resistance, but reduced the flexural fatigue resistance of K3 instruments. This behavior is probably associated with the generation of surface defects developing during the loading cycles, which serve to act as crack nucleation sites for flexural fatigue.

The bending, torsional, and fatigue properties of K3 and K3XF instruments are summarized in Tables 2-4.

Table 2. Bending properties of K3 and K3XF instruments.

Author	Rotary systems	Findings
Lopes et al. 2013	K3, Revo-S SU, ProFile Vortex, K3XF	Flexibility decreased in the following order: K3XF > Revo-S SU > Profile Vortex > K3.
Shen et al. 2013b	K3, K3XF	The bending load values were significantly lower for K3XF than for K3 in the superelastic ranges. ($P < .05$)
Ninan & Berzins 2013	HyFlex CM, Phoenix Flex, ProFile ISO, K3, GT Series X, and ProFile Vortex	The shape memory files were more flexible.
Gambarini et al. 2011	K3, K4 prototypes	K4 prototypes instruments showed a significant increase in flexibility when compared to K3 instruments.
Hou et al. 2011	K3 and Twisted Files	The bending load values in elastic and super-elastic ranges were lower for Twisted Files than those of K3 at 37 °C. Twisted Files appear to be more flexible than K3.
Melo et al. 2008	K3	The bending moment of the tested instruments increased significantly with diameter and cross-sectional area at 3 mm from the instrument tip.
Miyai et al. 2006	EndoWave, HERO 642, K3, ProFile.06, and ProTaper)	The bending load values of HERO and K3 were significantly higher than those of EndoWave, ProFile, ProTaper and K-file.

Table 3. Torsional properties of K3 and K3XF instruments.

Author	Rotary systems	Findings
Shen et al. 2015	K3, K3XF	The angles of rotation at fracture of K3XF instruments were similar to those of K3 instruments.
Lopes et al. 2013	K3, Revo-S SU, ProFile Vortex, K3XF	No statistically significant difference in maximum torque at fracture between K3 and K3XF. K3XF did demonstrate a significantly higher angular deflection at fracture compared with the other 3 test instruments.
Ha et al. 2013	K3, K3XF	No statistically significant difference in torsional resistance in terms of ultimate strength, fracture angle, and toughness between K3XF and K3.
Shen et al. 2013b	K3, K3XF	No statistically significant difference between K3 and K3XF in the maximum torque or maximum angular deflection before failure. The torque at fracture values of K3 and K3XF increased significantly with the diameter.
Ninan & Berzins 2013	HyFlex CM, Phoenix Flex, ProFile ISO, K3, GT Series X, and ProFile Vortex	The shape memory files showed a high angle of rotation before fracture but were not statistically different from some of the other files.
Bahia et al. 2008	K3	Cyclic torsional loading caused no significant differences in maximum torque or in maximum angular deflection of the instruments analyzed.
Barbosa et al. 2008 b	K3	Electrochemical polishing has no influence on torsional resistance to fracture of K3.
Melo et al. 2008	K3	Torque increased significantly with diameter and cross-sectional area at 3 mm from the instrument tip.

Author	Rotary systems	Findings
Miyai et al. 2006	EndoWave, HERO 642, K3, ProFile.06, and ProTaper)	The maximum torsional torque values of HERO, K3 and ProTaper were significantly higher than those of EndoWave, ProFile and K-file.
Yared et al. 2003	K3	Torque at fracture of the new instruments increased significantly with the diameter. Torque and angle of rotation at fracture were also significantly affected by the repeated use of .06 taper K3 instruments in resin blocks.

Table 4. Fatigue properties of K3 and K3XF instruments

Author	Rotary systems	Findings
Shen et al. 2015	K3, K3XF	The fatigue resistance of K3XF instruments was two times higher than that of K3 instruments
Shen et al. 2014	K3, K3XF	The K3XF instruments had a fatigue resistance superior to K3 instruments under dry and aqueous environments. The fatigue life of K3 instruments was similar under both conditions, whereas the Nf of K3XF was greater under water than in air, especially at the size 40, 0.04 taper.
Lopes et al. 2013	K3, Revo-S SU, ProFile Vortex, K3XF	Ranking in the fatigue resistance test: K3XF > K3 > ProFile Vortex, Revo-S SU.
Gambarini et al. 2013	K3XF, ProFile Vortex	No significant difference in resistance to cyclic fatigue when rotary NiTi instruments are used in continuous clockwise or counterclockwise rotation. In both directions of rotation, K3XF showed a significant increase ($P < 0.05$) in the mean number of cycles to failure when compared to ProFile Vortex of the same size (25/0.04).
Ha et al. 2013	K3, K3XF	K3XF showed superior cyclic fatigue resistance compared to K3.
Pérez-Higueras et al. 2013	K3, K3XF, and Twisted Files.	The probability of a longer mean life, for all files, was greatest under a reciprocating motion. K3XF was more resistant than K3 and Twisted Files under continuous rotation. Twisted File lasted significantly longer than K3 and was more resistant to cyclic fatigue at 300 rpm, compared to 500 rpm. Under reciprocating motion, there were no statistically significant difference in the mean life of K3XF and Twisted File, but both showed a significantly longer than the K3 mean life.

Author	Rotary systems	Findings
Plotino et al. 2012	K3, Mtwo, Vortex, and K3XF	Repeated cycles of autoclave sterilization did not influence the mechanical properties of 3 of the test instruments. K3XF prototypes demonstrated a statistically significant increase in cyclic fatigue resistance.
Gambarini et al. 2012	K3XF	Movement kinematics (reciprocating movements in various angles) had a significant influence on the cyclic fatigue life of the tested NiTi instruments.
Gambarini et al. 2011	K3, K4 prototypes	K4 prototype instruments showed a significant increase in the mean NCF when compared to K3 instruments
Wealleans et al. 2011	K3, Twisted Files, and EndoSequence.	The K3 and Twisted Files performed significantly better than the EndoSequence files
Melo et al. 2008	K3	The fatigue resistance of the instruments showed a tendency to decrease as the diameter of the instruments increased.
Barbosa et al. 2007	K3	As the previous angular deformation increases, the number of cycles to fracture in flexural fatigue test decreases.
Barbosa et al. 2008 b	K3	Electrochemical polishing has no influence on fatigue resistance to fracture of K3 instruments.
Bahia et al. 2008	K3	Cyclic torsional loading decreased the flexural fatigue resistance of K3 instruments.
Gambarini et al. 2008	K3, GTX, and Twisted Files.	Twisted Files had significantly more resistance to fatigue than K3. No significant difference between K3 and GTX.

1.6 Study rationale

Rotary endodontic instruments used to clean and shape root canals can fail through fatigue and torsional modes. Such failure will result in a fractured instrument and will impede the chemomechanical debridement of the apical part of the root canal system and hence compromising the prognosis of endodontic treatment. The majority of fatigue experiments have been performed under uniaxial or rotational bending conditions (Plotino et al. 2009, Shen et al. 2011). NiTi rotary instruments, however, are generally subjected to multiaxial loading. The repeated torsional loading and unloading applied to rotary NiTi instruments during clinical use can lead to torsional fatigue. Recently, a unique thermal process has been developed by SybronEndo (Orange, CA, USA) and is applied to NiTi files after completion of the grinding process. Theoretically, the main advantages gained by this heat treatment is not only an improvement in the flexibility and strength of the file, but it also modifies the alloy's crystalline structure, allowing the material to absorb some of the internal stresses caused by the grinding process (Gambarini et al 2011, Shen et al. 2013b). SybronEndo claims that their R-Phase technology, used in the production of K3XF instruments, provides clinicians with the original features of K3 instruments, but with an additional level of flexibility and resistance to cyclic fatigue (Ha et al. 2013). Several studies (Gambarini et al. 2011, Plotino et al. 2012, Ha et al. 2013, Shen et al. 2014, Shen et al. 2015) have shown that K3XF instruments have fatigue resistance superior to K3 NiTi instruments. There was no statistically significant difference between K3 and K3XF in the torsional test (Ha et al. 2013, Shen et al. 2013b). However, there is currently little information available on torsional fatigue of K3XF instruments.

1.7 Aim

To evaluate the torsional and cyclic fatigue behavior of post-machining heat-treated K3XF and conventional K3 NiTi instruments.

1.8 Hypothesis

Post-machining heat-treated K3XF NiTi instruments have a better torsional fatigue resistance than K3 instruments.

Chapter 2. Methods

K3XF and K3 files have an identical geometric design but different metal alloy; K3 is made from conventional NiTi, and K3XF is made from a novel R-phase heat-treated metal alloy. All instruments used in this study were size 25/0.04.

New K3XF and K3 instruments (n=15 in each group) were torsion tested until rupture to establish their mean values of torque to failure and maximum angular deflection. The torsion tests were performed according to ISO 3630-1 (ISO 2008) using a torsion machine (Figure 2). The rotation speed was set clockwise to 2 rpm. Before testing, each instrument handle was removed at the point where the handle is attached to the shaft. The end of the shaft was clamped into a chuck connected to a reversible geared motor. In order to prevent sliding, three millimeters of the instrument's tip were clamped in another chuck with brass jaws. The torsional loads and distortion angles were recorded until the file broke.

In order to evaluate the effect of torsional loading on the torsional resistance, the cyclic torsional loading testing was performed; the file was programmed to repeatedly rotate from zero angular deflection to 180° and then return to zero applied torque. Each rotation was defined as one cycle. K3XF and K3 instruments (n=15 in each group) were subjected to 10 cycles of torsional loading. Subsequently, files subjected to cyclic torsional loading were examined using a torsion machine to obtain the maximum torque and angle of rotation at fracture.

The fatigue testing protocol has been described previously and was reproduced throughout the experimental period (Shen et al. 2011, Shen et al. 2012). Briefly, each NiTi instrument was constrained to a curvature by three rigid, stainless steel pins (Figure 3), and a calibrated digital

photograph of the curvature was taken (Figure 4). The instrument was then allowed to rotate at 500 rpm until fracture. The fatigue life, or the total number of revolutions to failure (Nf), was recorded. NiTi rotary K3XF and K3 NiTi instruments (n=15 in each group) were subjected to rotational bending at a curvature of 45° with a 7 mm radius in deionized water at a temperature of 23° ± 2°C. Only a 16 mm length from the tip of the instrument was immersed in the liquid medium during the test (Shen et al. 2012).

In order to evaluate the effect of torsional loading on the cyclic fatigue resistance, the cyclic torsional loading testing was performed as described above, and the files were examined using a three-point bending apparatus to obtain the mean number of cycles to failure.

The fracture surfaces of all fragments were examined under a scanning electron microscope (Stereoscan 260; Cambridge Instruments, Cambridge, UK). The region in which the dimple area could be found was outlined on the photomicrograph for fatigue failure groups and measured with ImageJ 1.4 g software (National Institutes of Health, Bethesda, MD) on each photomicrograph. The results were analyzed using one-way analysis of variance with post hoc analysis using software (SPSS for Windows 11.0; SPSS, Chicago, IL) at a significance level of $P < .05$.

Figure 2. Demonstration of torsional machine used for torsional tests. The end of the shaft is clamped into the chuck in the right-hand side while the three millimeters of the instrument tip are clamped into the chuck in the left-hand side. (Courtesy of Dr. Ya Shen)

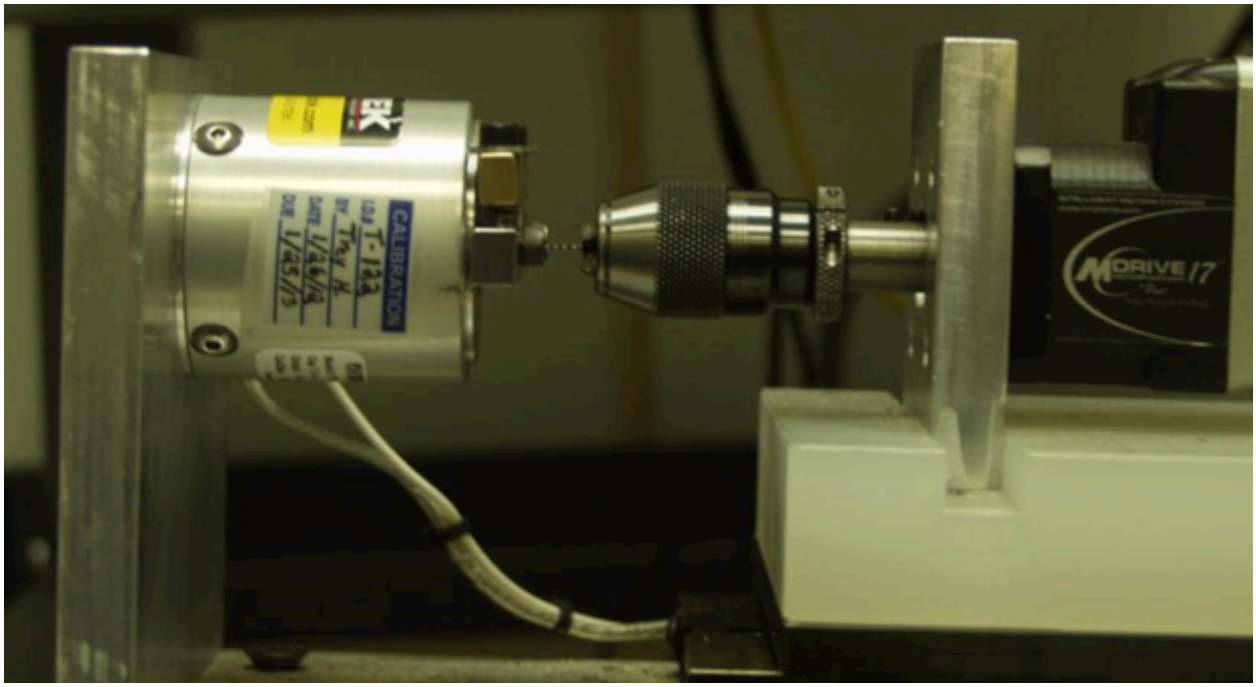
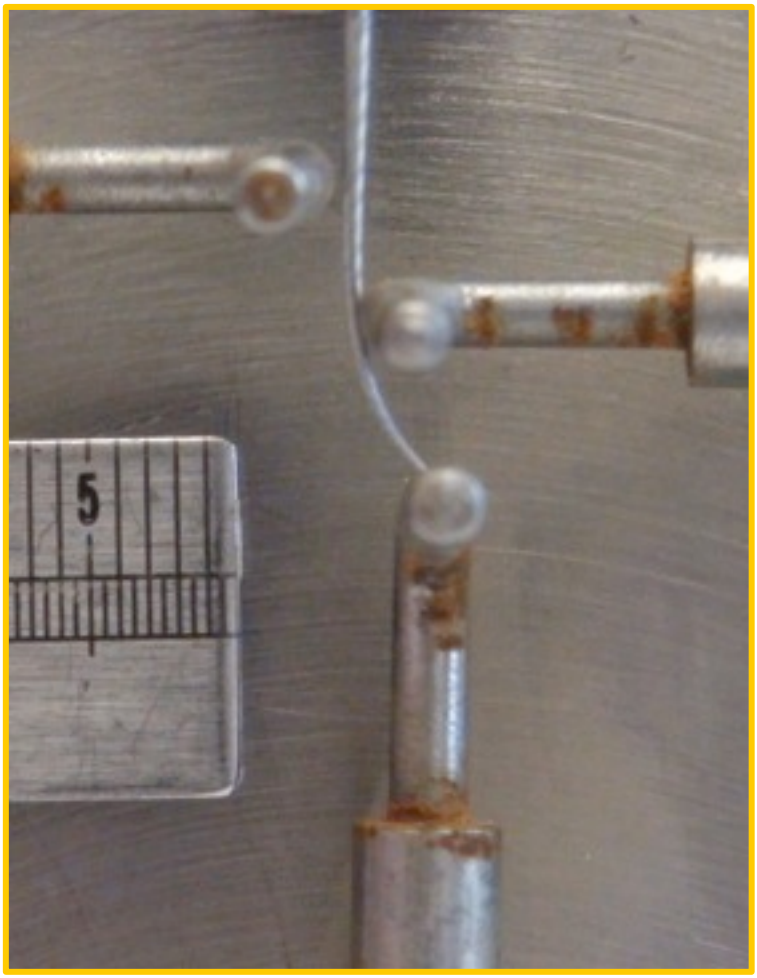


Figure 3. Demonstration of a three-point bending apparatus.

Figure 4. K3XF file during flexural fatigue test in a three point bending apparatus.



Chapter 3: Results

The mean values of maximum torque and angle of rotation at fracture of the new instruments and of those previously submitted to 10 cycles of torsional loading are presented in Table 5. The maximum torque of new K3XF instruments was similar to that of K3 instruments. However, comparative statistical analysis between maximum torque values of previously cycled K3XF and K3 instruments showed a significantly higher torsional resistance for K3XF instruments ($P < 0.05$). The angle of rotation at fracture of K3XF was similar to that of K3 instruments in both the new and previously cycled groups.

The cyclic fatigue life of new K3XF was two times higher than that of K3 instruments (Table 6). Also, the fatigue resistance of previously cycled K3XF was significantly higher than K3 instruments ($P < 0.05$). Comparative statistical analysis between the measured NCF values of new and previously cycled K3XF instruments showed significant differences ($P< 0.05$), indicating that torsional loading decreased the cyclic fatigue resistance of K3XF instruments. In contrast, the cyclic fatigue behavior of K3 instruments was comparable for both the new and previously cycled groups.

The lateral view of the new K3 and K3XF instruments which fractured due to torsional failure showed evidence of macroscopic plastic deformation (Fig 5&6). There was minimal difference in appearance, longitudinal or lateral aspect, between the new and previously cycled K3 and K3XF files (Fig 7&8); the lateral aspect of the preloaded files did not show any specific topographic features. However, after 10 cycles of torsional loading, three of the K3XF

instruments and six K3 files had plastic deformation, approximately 3 mm away from the tip as evidenced under the lateral view (Fig. 9&10). Furthermore, longitudinal cracks were detected in some files following cyclic torsional loading experiment (Fig. 9&10). Interestingly, the micro-cracks did not seem to follow the machining grooves on the instrument surface, but rather ran in irregular patterns (Fig. 10). The K3XF instruments demonstrated unique surface characteristics with numerous irregular micropores (Fig 10).

Fractographic features of the fracture surface were similar for both K3XF and K3 instruments. In instruments which failed by fatigue only or fatigue after torsional loading showed crack origins, areas of microscopic fatigue-striations, and dimple rupture on all fracture surfaces (Fig. 11-13). Most of the specimens displayed fatigue crack initiations at one or more cutting edges of the fracture cross section (Fig. 11). The area of the fracture surface occupied by the dimple region was significantly smaller in K3XF instruments than in K3 instruments. The fracture pattern of K3 and K3XF instruments with and without preloading was similar. The fractography corresponding to the torsional failure showed a torsional fracture pattern with circular abrasion marks and skewed dimples near the center of rotation (Fig. 12).

Table 5. Maximum torque and angle of rotation at fracture of new and previously cycled K3 and K3XF instruments.

Size 25/.04	New files		10 cycles*	
	Torque (Nmm)	Angle (°)	Torque (Nmm)	Angle (°)
K3	6.3 ± 1.4[a]	830.8 ± 206.8[c]	6.4 ± 0.8[a]	824.5 ± 113.9[c]
K3XF	6.8 ± 0.6[a,b]	720.9 ± 132.8[c]	7.0 ± 0.8[b]	759.4 ± 126.5[c]

*Each cycle of torsional loading is comprised of automatically rotating the file from zero angular deflection to 180° and then returning to zero torque. K3 and K3XF instruments in the preloading group were subjected to 10 cycles of torsional loading.

Different superscript letters indicate statistically significant difference between groups ($P<0.05$).

Method: one-way ANOVA with Turkey post hoc analysis.

Table 6. Number of cycles obtained in cyclic fatigue tests (45°, 7 mm radius) of K3 and K3XF instruments with and without 10 cycles of torsional loading.

	K3	**K3XF**
New files	625 ± 112[acd]	1128 ± 202[b]
10 cycles	576 ± 107[d]	794 ± 182[c]

Different superscript letters indicate statistically significant difference between groups ($P<0.05$).

Method: one-way ANOVA with Turkey post hoc analysis.

Figure 5. Lateral-view scanning electron microphraph of new K3 files which fractured due to torsional failure (A); B, C&D are high magnification views of A. Macroscopic plastic deformation is clearly evident in (D).

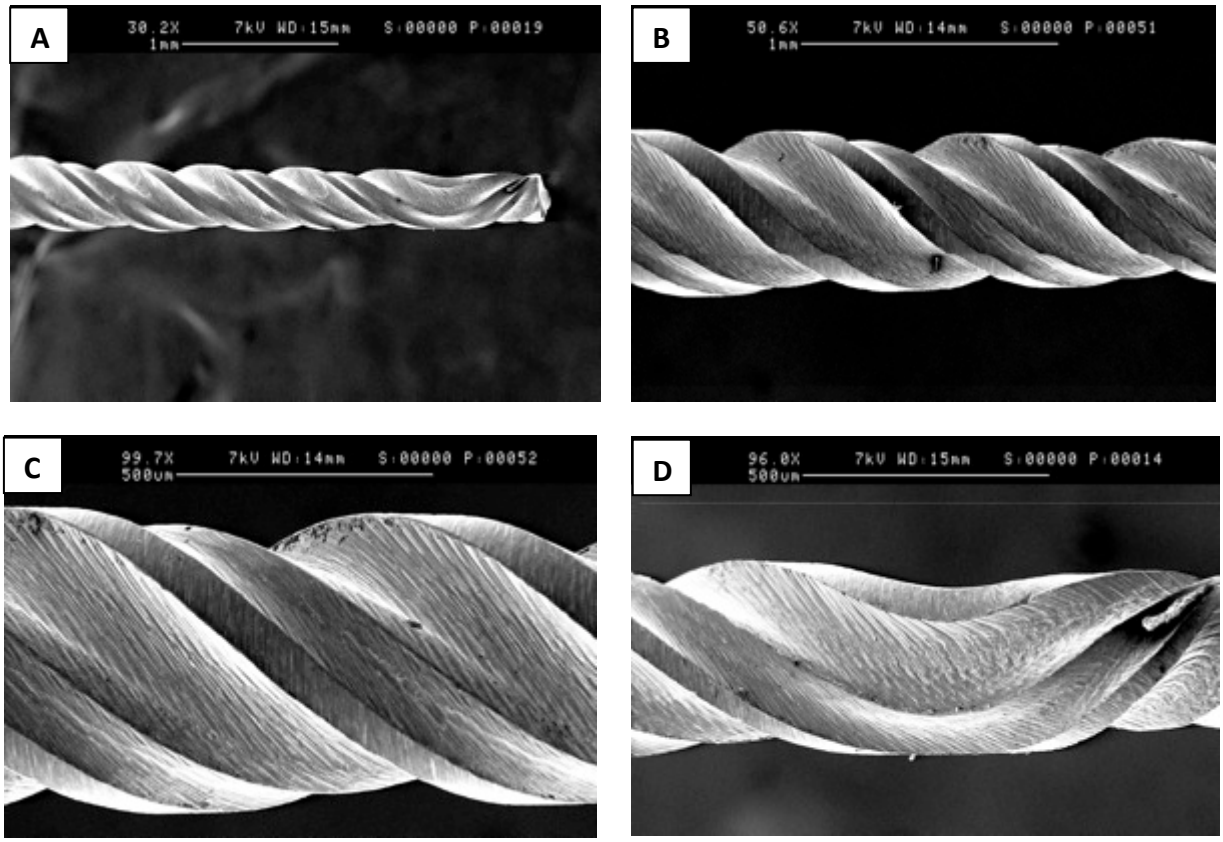


Figure 6. Lateral-view scanning electron microphraph of new K3XF files which fractured due to torsional failure (A); B&C are high magnification views of A. Macroscopic plastic deformation is evident at all magnifications.

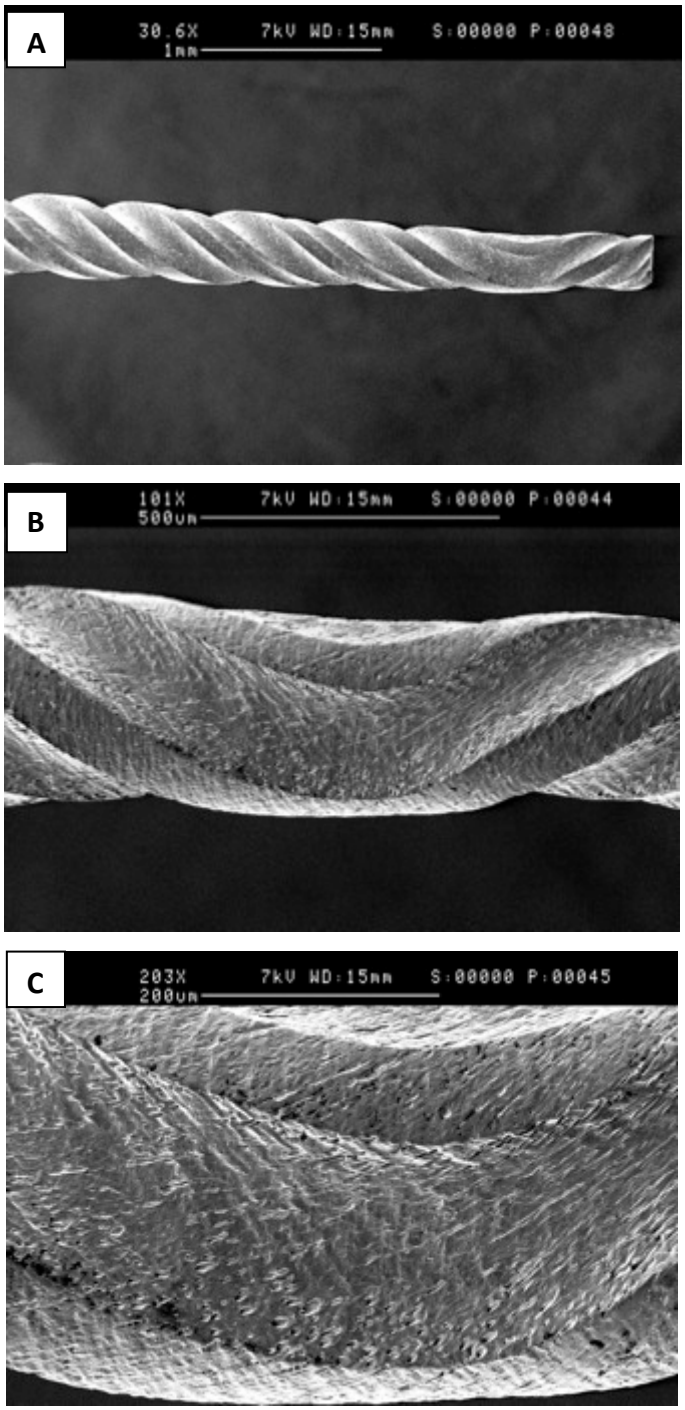


Figure 7. Lateral-view scanning electron microphraph of new K3 files after 10 cycles of torsional loading (A&C). B&D are high magnification of A&C, respectively, shows surface micropitting around the cutting edge and no other specific topographic features can be seen.

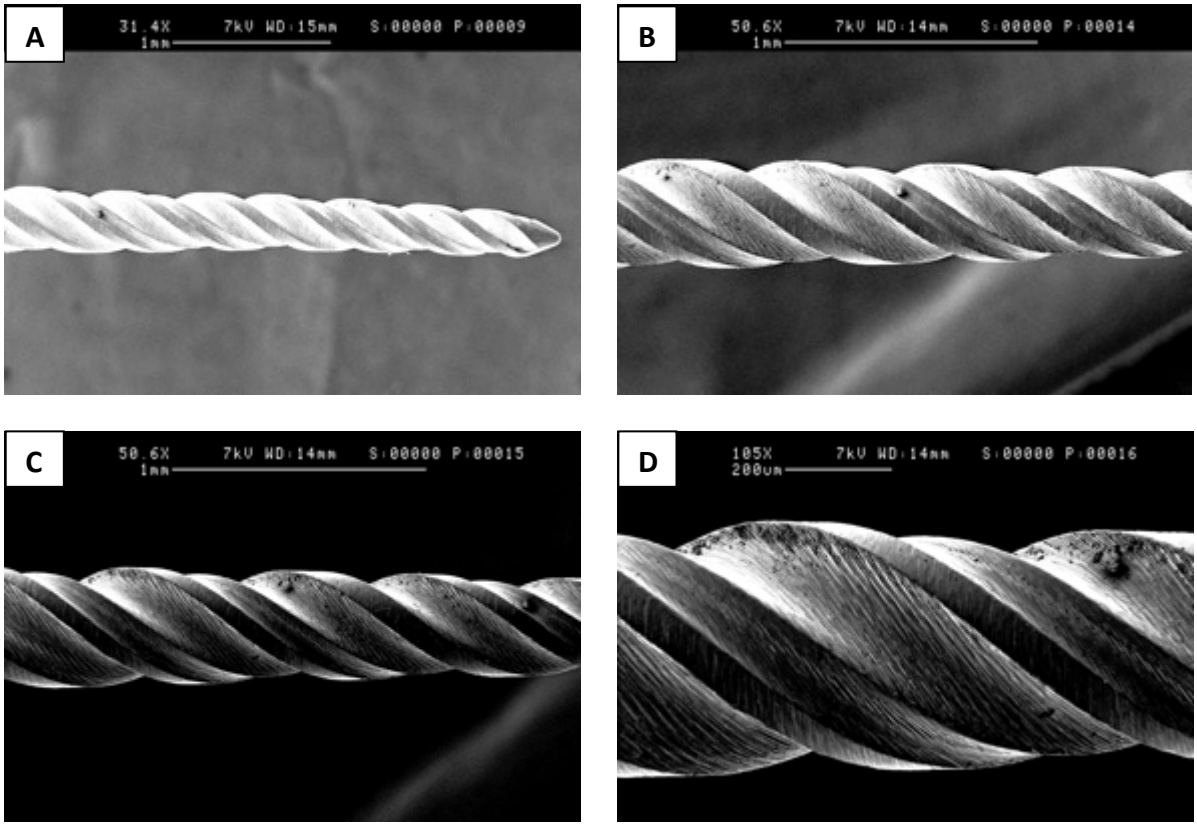


Figure 8. Lateral-view scanning electron microphraph of new K3XF files after 10 cycles of torsional loading (A). B, C &D are high magnification views of the instruments shows surface micropitting around the cutting edge and no other specific topographic features.

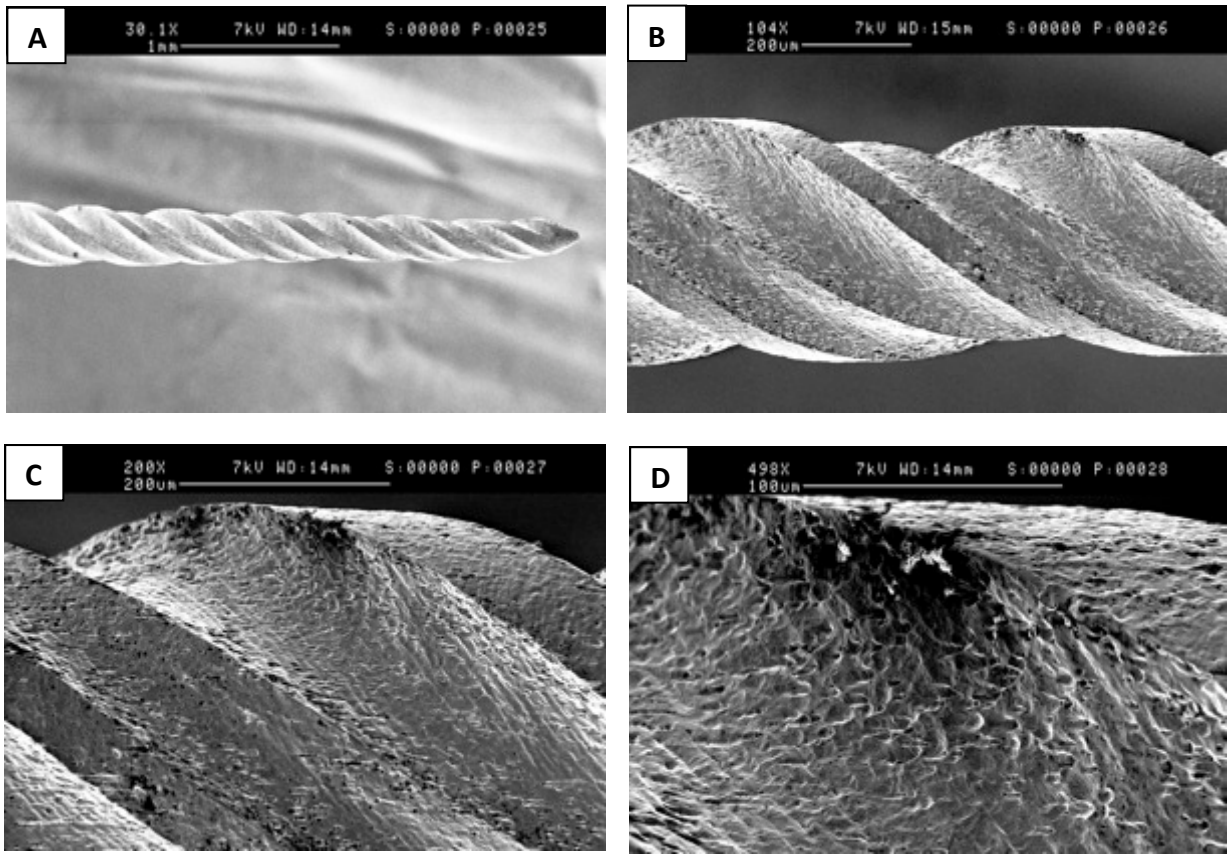


Figure 9. Lateral-view scanning electron microphraph of new K3 files after 10 cycles of torsional loading (A). B&C are higher magnification views of A demonstrating surface defects and longitudinal microcracks. Plastic deformation of the flutes can be noted at all magnifications.

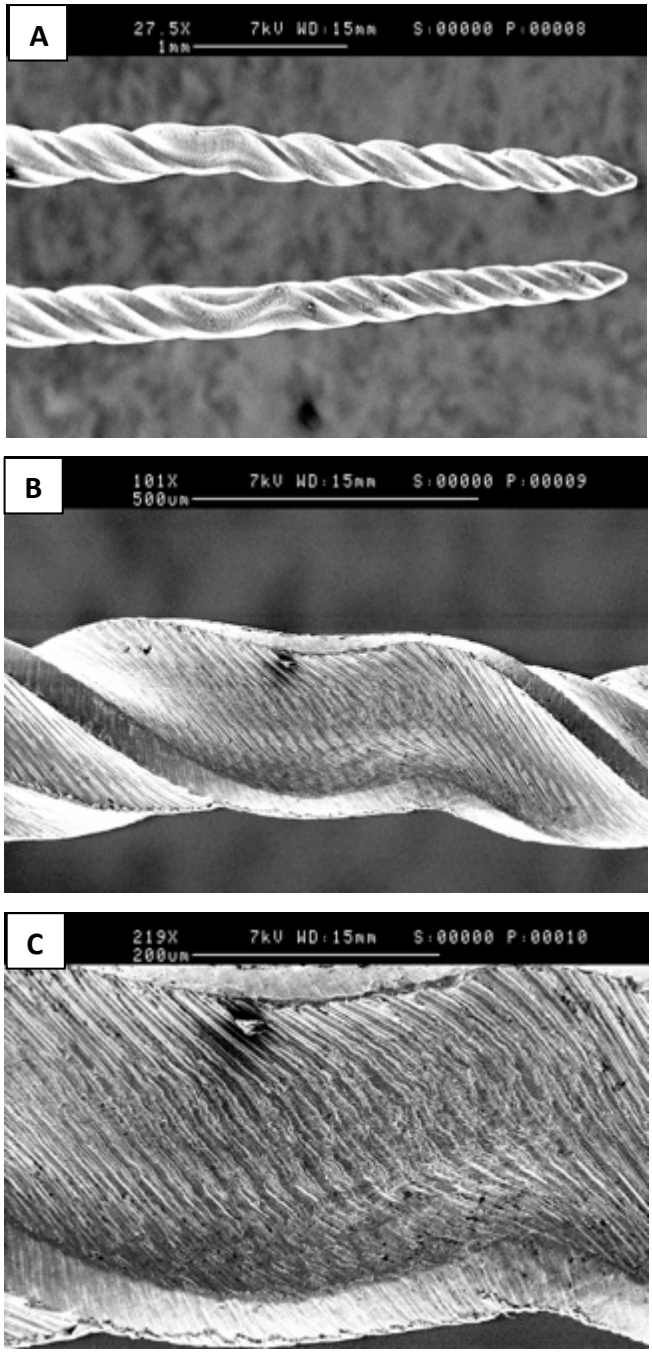


Figure 10. Lateral-view scanning electron microphraph of new K3 files after 10 cycles of torsional loading. Plastic deformation of the flutes is clearly evident in A&B. C&D are high magnification views of B&C, respectively, showing micropores and microcracks.

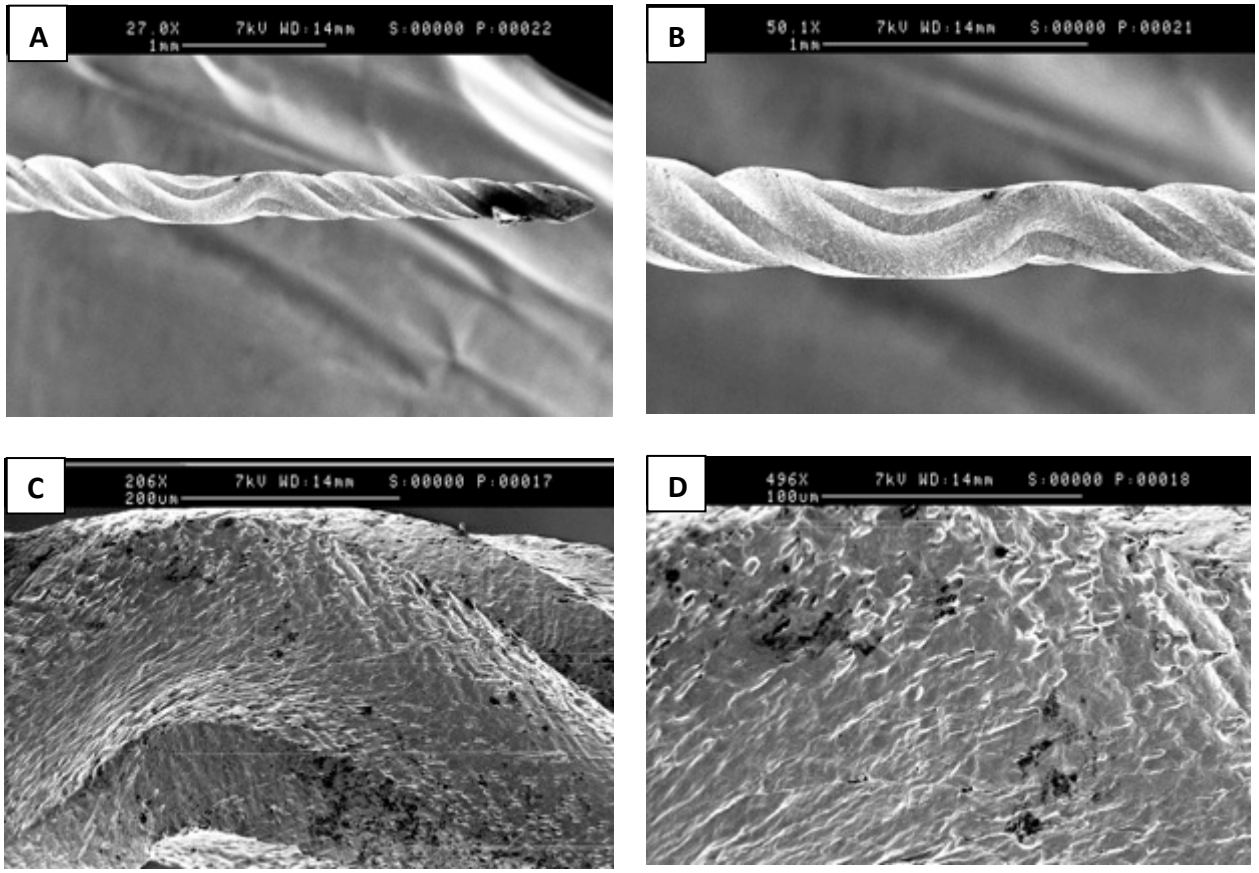


Figure 11. Fracture surfaces of new K3 (A) and K3XF (B) instruments after separation due to fatigue. Fatigue crack initiation areas can be seen at the cutting edges of both instruments.

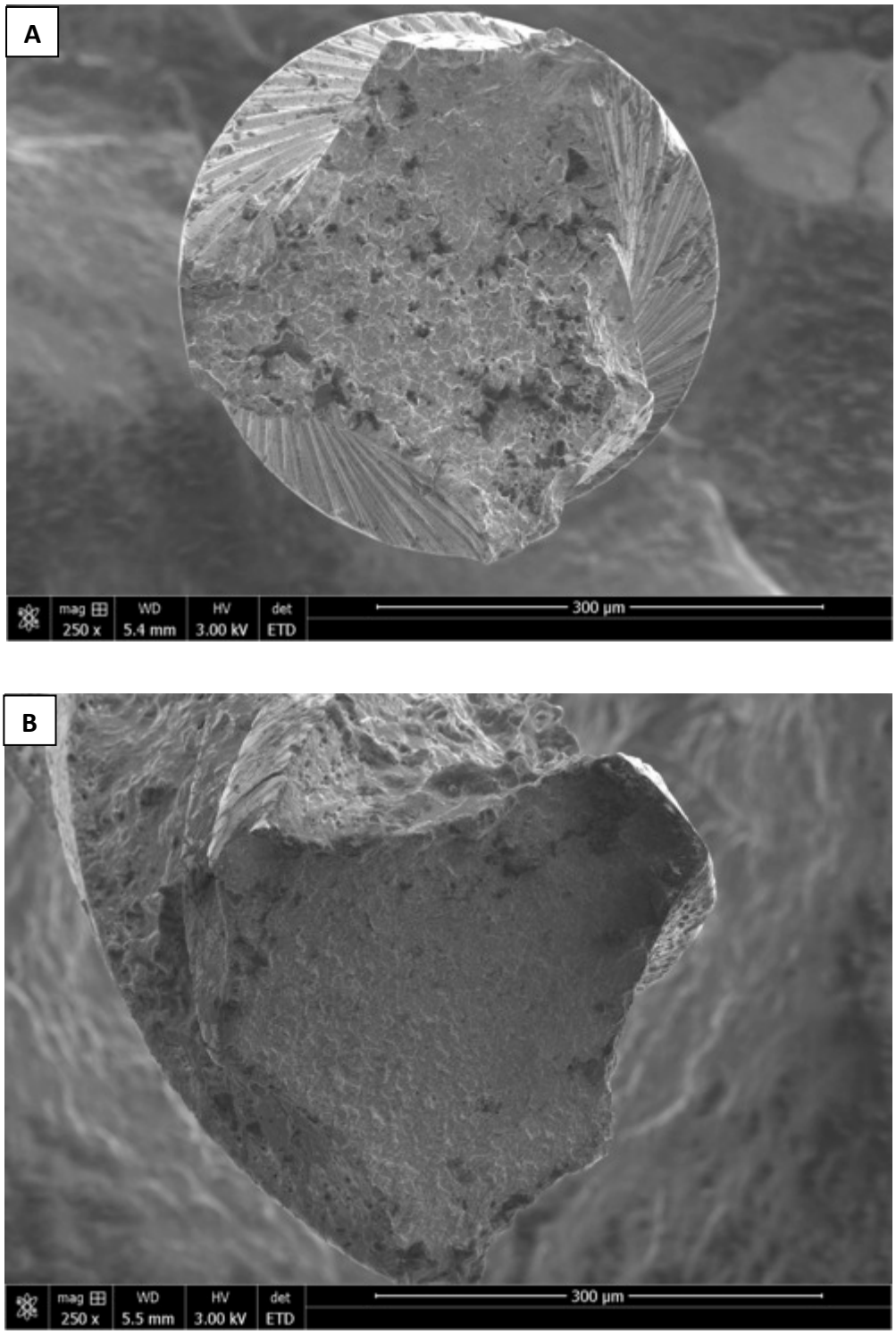


Figure 12. Fracture surfaces after instrument separation due to torque of K3 (A) and K3XF (B) instruments, clearly shows circular abrasion marks and skewed dimples near the center of rotation. Corresponding surfaces after fracture by fatigue of K3 (C) and K3XF (D) show fatigue striations running through the cross-section of both instruments indicating where the fracture originate.

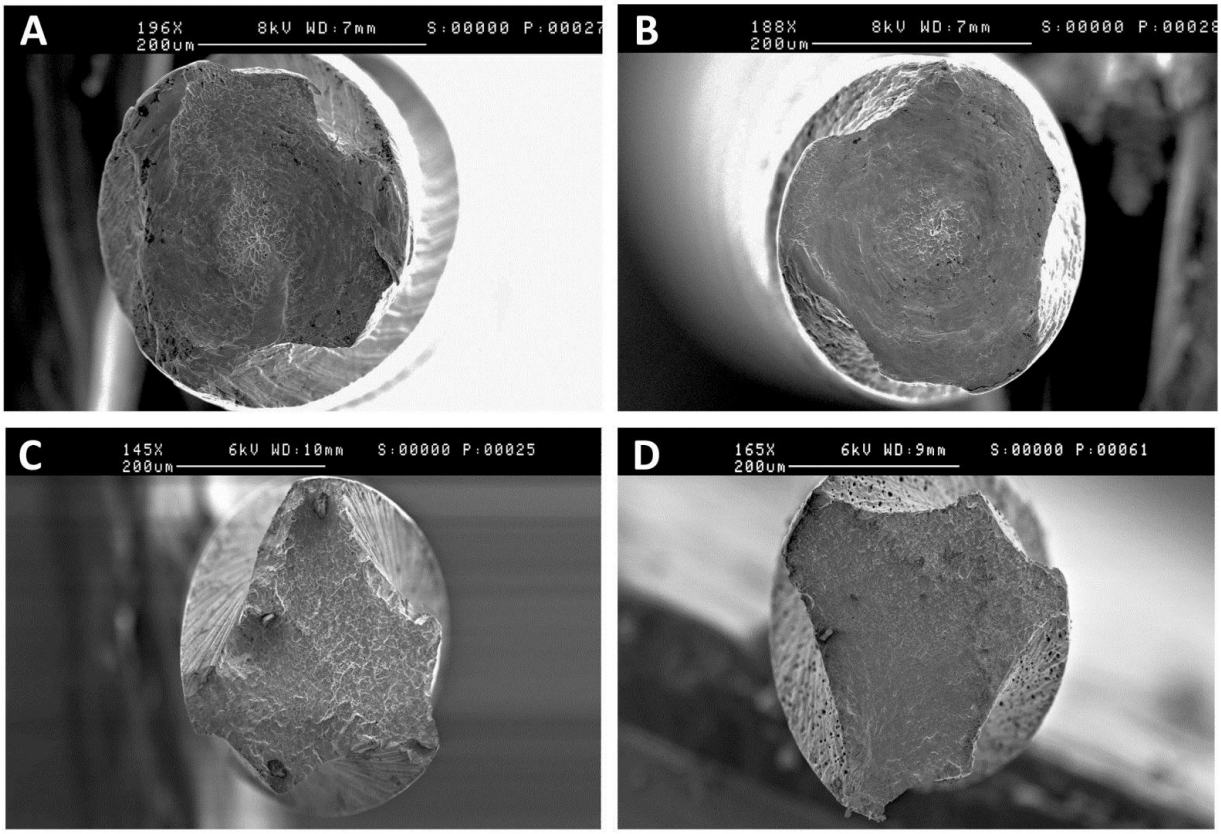
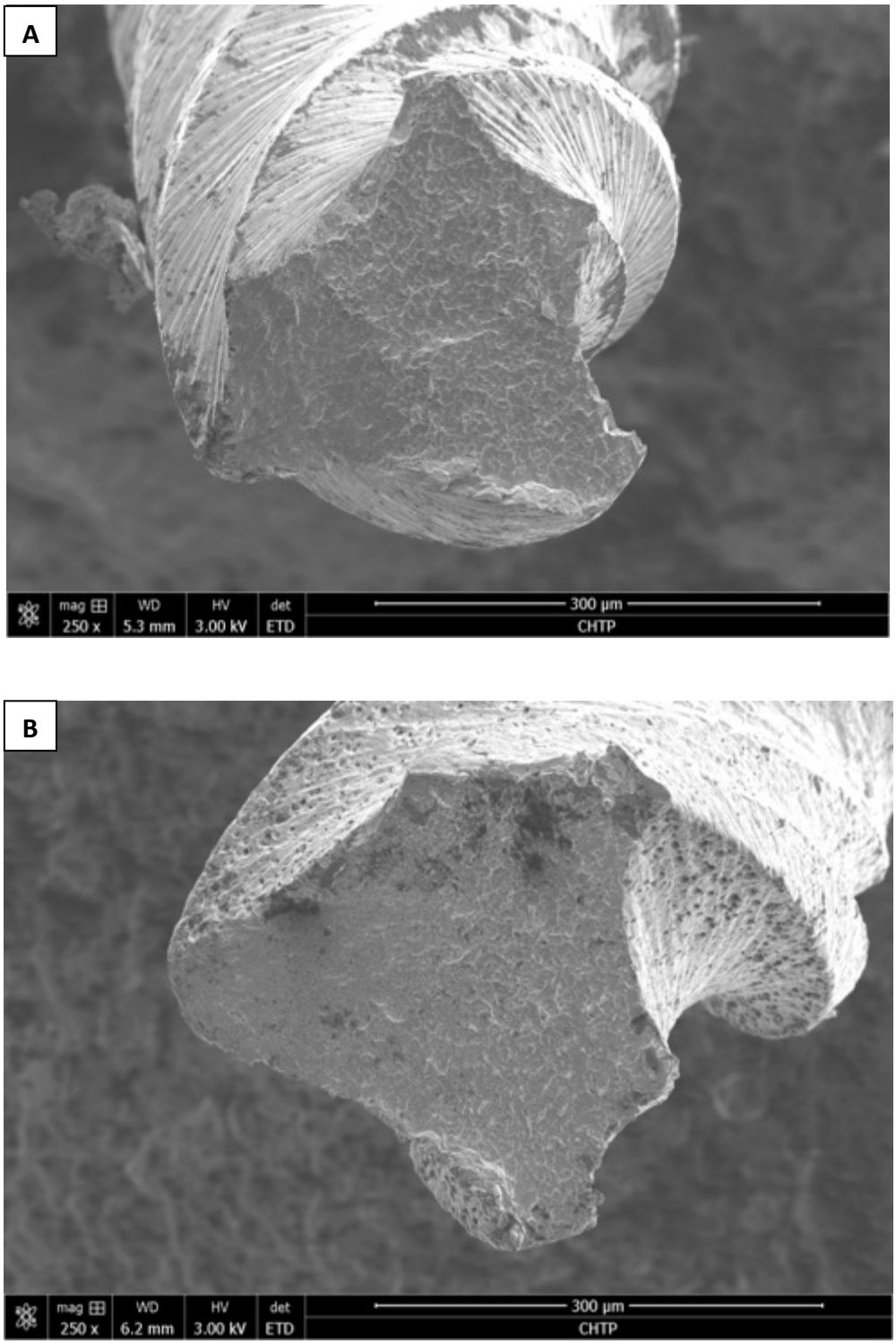


Figure 13. Fracture surfaces following a combination of cyclic torsional loading and fatigue testing of K3 (A) and K3XF (B) instruments.



Chapter 4: Discussion

The aim of this study was to provide a deep insight into the torsional fatigue behavior of post-machining, heat-treated K3XF instruments and the conventional superelastic K3 NiTi instruments. Currently, there is little information available on torsional fatigue of K3XF instruments (Shen et al. 2013b, Ha et al. 2013, Lopes et al. 2013). A major drawback of the majority of laboratory tests, which investigate the mechanical properties of NiTi rotary instruments, is that confounding factors such as material properties, design, and dimensions of the instrument, are not eliminated. In addition, these factors are also brand specific, making it difficult to quantify the effect a single variable has on the mechanical properties of the instrument. K3 and K3XF instruments are identical in design and differ only in the postmachining heat treatment occurring in the production of K3XF files. Therefore, any differences in the mechanical properties of K3XF instruments can only be explained by this new thermomechanical treatment applied to K3XF.

Although in the oral environment, K3XF and K3 instruments consist mainly of austenite, the phase transformation temperature (Shen et al. 2013b) of the K3XF instruments (Af temperature of 25°C) was significantly higher than in the K3 (Af temperature of 17°C). Differential scanning calorimetric analyses displayed two overlapping endothermic peaks on the heating plot of K3XF instruments. This indicates that reverse transformation of the alloy passes through the intermediated R-phase and thus reflecting the complex phase transformation behavior tracking back to the manufacturing process (Shen et al. 2013b). It was not surprising that K3XF instruments are more flexible and resistant to cyclic fatigue than the K3 instruments (Gambarini et al. 2011, Ha et al. 2013).

It is expected that thermomechanically treated NiTi instruments will maintain the same torsional properties as conventional NiTi instruments (Ha et al. 2013, Shen et al. 2013b). As a general rule, it has been assumed that flexible instruments have a lower resistance to a torsional load compared to stiffer instruments, but are more resistant to cyclic fatigue. The maximum torque and angle of rotation measured at fracture is a sound reflection of the material's fracture resistance and ductility. Wycoff and Berzins (2012) found that post-twisting, Twisted Files (SybronEndo) displayed the least amount of torsional resistance and the highest angular deflection when compared to conventional superelastic NiTi files of a similar cross-sectional design. In the present study, the torque and angle of rotation at fracture of K3XF files were similar to K3 files.

Only one instrument size of both brands (#25/0.04) was used in this study, as it is commonly used for root canal preparation. The maximum angular deflection of 180° was chosen based on previous torsional behavior measurements of K3 instruments. Melo et al. (2008) showed that this chosen angle of deflection is within the range of superelastic straining in torsion. In the present study, the rotational speed of K3XF and K3 instruments was standardized to 500 rpm to permit direct comparisons of the different mechanical properties exhibited by K3 and K3XF caused by the thermomechanical treatment.

The influence of previous torsional angular deformation on the flexural fatigue life of K3 and K3XF instruments has been examined in a few studies (Barbosa et al. 2007, Bahia et al. 2008, Shen et al. 2015). Barbosa et al. (2007) examined new K3 instruments which were subjected to a pre-defined rotation before flexural fatigue testing. The authors concluded, that transient

torsional overloads may act in conjunction with flexural fatigue and reduce failure resistance in NiTi files. The study by Shen et al. (2015) found both the K3 and K3XF instruments had a statistically significantly lower Nf in the torsional preloading groups compared to the no preloading group. In addition, K3XF instruments demonstrated a significantly higher Nf than K3 in the groups where preloading was carried out.

The contribution of cyclic torsional loading, instead of a single monotonic overload, to instrument fracture has been reported by Bahia et al. (2008). The authors reported that cyclic torsional loading did not affect torsional resistance, and in fact reduced the flexural fatigue resistance of K3 instruments. This lends further support to the findings of the present study. Repeated torsional loading is probably associated with the generation of surface defects during the loading cycles, which can serve as crack nucleation sites for flexural fatigue, but with little appreciable change in the cross-sectional area of the instrument that determines the ultimate torsional strength.

Previous studies on instrument separation have been carried out on the lateral view of the instrument under low magnification (Sattapan et al. 2000, Arens et al. 2003, Peng et al. 2005). However, while lateral view examination allows the detection of plastic deformation in the separated instrument, it fails to indicate the actual mechanism involved in the fracture process (Cheung 2009). The goal of fractographic examination is to identify features on the fractured surface which may indicate the origin and the direction of the crack which has led to material's failure (Shen and Cheung 2013). Two mechanisms are involved in the fracture of NiTi rotary instruments in clinical use: (i) fatigue failure, characterized by the presence of fatigue striations;

and (ii) torsional failure, characterized by circular abrasion marks on the fractured surface. Fractographic examination in conjunction with lateral examination is necessary to reveal fracture features that indicate the crack origin and the mode of material failure. The appearance of fracture surface, occurring under various loading conditions was studied by Barbosa et al. (2008a). The authors evaluated the fractured surface of K3 rotary instruments when subjected to different types of mechanical loading. Another two groups were tested with mixed loading modes; torque to failure after transient flexural stresses and flexural fatigue after an incomplete torsional preloadings. In agreement with the current study, the results showed that for the combined loading groups the fractographic pattern exhibited an appearance which was defined by the very last loading regime.

In the present study, the fractographic features of the K3 and K3XF instruments were similar. In most K3 and K3XF instruments, the crack origins were typically found at the cutting edge region. This is not surprising because when a circular beam is bent, its outermost areas are subjected to the greatest stress and strain. Numerous micropores, of various diameters, were detected on the surface of the K3XF instrument flutes. According to the SEM images, these small pores did not appear to contribute to the failure. A similar finding was reported by Ha et al. (2013). The vast majority of K3 and K3XF instruments had a one crack origin. In related studies (Shen et al. 2011, Shen et al. 2012), themomechanically treated controlled memory (CM) instruments displayed a greater number of crack origins compared to conventional superelastic NiTi instruments. Despite the significantly greater number of crack origins, the values of the fraction area occupied by the dimple region seemed to correlate with fatigue life (Shen et al. 2011, Shen et al. 2012). This is in accordance with the present study.

An aquatic medium was chosen to simulate the clinical situation and it was assumed that this would serve as an effective heat sink during the cyclic fatigue testing. The third millimeter from the tip of the instrument (D3) was the chosen point of the file to perform the torsional experiment, where the maximum curvature during flexural fatigue tests was experienced. When flexural fatigue and torsion were combined, they both occurred at the same point on the file. It should be noted that the complexity of the clinical situation cannot be fully reproduced using in-vitro models. However, the methodology employed in the present study tried to address the short falls of previous methodologies and attempted to develop a comprehensive representation of a clinical setting. The torque at fracture values which were sufficient to break the NiTi instrument at D3, when rotated at 2 rpm, were determined according to ISO3630-1. However, clinical torque stresses occurred at higher rotations per minute, and the effects of instrument binding are influenced by factors such as canal dimension and anatomy, magnitude of apical pressure applied, skill level of the operator, and other factors.

Chapter 5: Conclusions

The maximum torque to fracture of heat-treated K3XF was similar to that of conventional superelastic K3 instruments. However, the maximum torque of preloaded K3XF was significantly higher than K3 instruments. The angle of rotation at fracture of K3XF was similar to the K3 instruments in both the new and torsionally preloaded groups. In cyclic fatigue tests, the NCF of the new K3XF files was twice as high compared to the K3 instruments. Furthermore, the cyclic fatigue resistance of the preloaded K3XF was significantly higher than K3 instruments. Repeated cycles of torsional loading decreased the cyclic fatigue resistance in K3XF instruments, although it did not affect the fatigue life of K3 instruments. In contrast, the torsional properties of both NiTi rotary systems remain relatively unaffected following torsional preloading.

Lateral view examination showed evidence of plastic deformation and longitudinal cracks in some of the files following the torsional preloading cycles. Of interest is that the fractographic features of the fracture surface, which were very similar in both K3XF and K3 instruments. The values of the fraction area occupied by the dimple region were significantly smaller in K3XF than in K3 instruments. The fracture patterns of K3 and K3XF with and without preloading were similar. In the combined loading groups, the fractographic pattern corresponded to an appearance which was defined by the very last loading regime.

Since K3 and K3XF files are identical in shape, design, and dimensions, it is plausible to hypothesize that the special thermomechanical treatment performed on the K3XF files is the cause of their superior cyclic fatigue resistance. Therefore, K3XF files should be considered as

an improvement on K3 files with regard to the key characteristics related to fracture risk. The superior performance of K3XF instruments needs to be confirmed by in vivo investigations.

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