UNIVERSIDADE FEDERAL DE MINAS GERAIS

School of Dentistry

# **“Influence of cyclic flexural deformation on the torsional resistance of *CM* files in comparison to conventional NiTi instruments”**

Eufemia Carolina Peláez Acosta

Belo Horizonte
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# "Influence of cyclic flexural deformation on the torsional resistance of *CM* files in comparison to conventional NITI instruments"

Research project presented to the Post-Graduation Program in Dentistry of the Universidade Federal de Minas Gerais, as a requirement for the degree of Master in Science.

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**UNIVERSIDADE FEDERAL DE MINAS GERAIS**

**PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA**

# FOLHA DE APROVAÇÃO

**Influence of cyclic flexural deformation on the torsional resistance of CM instruments in comparison to conventional NiTi instruments.**

**EUFEMIA CAROLINA PELAEZ ACOSTA**

Dissertação submetida à Banca Examinadora designada pelo Colegiado do Programa de Pós-Graduação em ODONTOLOGIA, como requisito para obtenção do grau de Mestre em ODONTOLOGIA, área de concentração ENDODONTIA.

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Belo Horizonte, 7 de julho de 2016.

**UNIVERSIDADE FEDERAL DE MINAS GERAIS**

**PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA**

# ATA DA DEFESA DE DISSERTAÇÃO DA ALUNA **EUFEMIA CAROLINA PELAEZ ACOSTA**

Aos 07 dias de julho de 2016, às 08:00 horas, na sala 3403 da Faculdade de Odontologia da Universidade Federal de Minas Gerais, reuniu-se a Comissão Examinadora composta pelos professores Maria Guiomar de Azevedo Bahia (Orientadora) – FO/UFMG, Renata de Castro Martins – FO/UFMG e Isabella Faria da Cunha Peixoto – PUC-Minas, para julgamento da dissertação intitulada: **Influence of cyclic flexural deformation on the torsional resistance of CM instruments in comparison to conventional NiTi instruments**. A Presidente da Banca, abriu os trabalhos e apresentou a Comissão Examinadora. Após a exposição oral do trabalho pela aluna e arguição pelos membros da banca, a Comissão Examinadora considerou a candidata:

(X) Aprovada

( ) Reprovada

Finalizados os trabalhos, lavrou-se a presente ata que, lida e aprovada, vai assinada por mim e pelos demais membros da Comissão. Belo Horizonte, 07 de julho de 2016.

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## Dedication

To ***God,*** my parents ***Norca and Gilberto*****,** my sisters ***Pely and Betty*****,** my brother ***Gilbert,*** and my uncle ***Tio Papito*****.**

I dedicate my dissertation work to my family. A special feeling of gratitude to my loving parents, ***Norca and Gilberto*****,** whose words of encouragement and push for tenacity will forever ring in my ears. To my sisters ***Pely, Betty***, and brother ***Gilbert*** whom have been greatly supportive, helping me achieve my cherished goal. Moreover, to my uncle, ***Tio Papito*** my second father, who has always been unconditional.

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## SUMMARY

# Contents

# RESUMO

## "Influência do carregamento cíclico flexural na resistencia torcional dos instrumentos com memória controlada (CM), em comparção com instrumentos convencionais de NiTi"

As propriedades mecânicas dos instrumentos endodônticos são afetadas por fatores como microestrutura, diâmetro, geometria, composição química, e tratamentos termomecânicos aplicados durante o processo de fabricação. Os instrumentos de NiTi com Memória Controlada (CM) sofrem um tratamento térmico especial que os tornam mais flexíveis e resistentes à fadiga que instrumentos de NiTi convencional. O objetivo deste estudo é avaliar como a fadiga influencia nas propriedades torcionais dos instrumentos CM, quando comparados com instrumentos superelásticos (SE) com geometria e características dimensionais similares. Instrumentos novos Hyflex CM (HF; Coltene/ Whaladent Inc.), Typhoon CM (TYP; Clinician´s Choice Dental Products), RaCe (RC; FKG, La-Chaux De Fonds, Switzerland) e Protaper Universal  F2 (F2; Dentsply Maillefer, Ballaigues, Switzerland), todos de calibre e conicidade 30/.06, foram avaliados. Características geométricas e dimensionais como o diâmetro, comprimento de pitch e ângulo helicoidal foram medidos ao longo da parte ativa dos instrumentos. A fadiga flexural (Nf) e a resistência torcional para cada instrumento (n=10) foram determinadas de acordo com a especificação ISO-3630. Dez novos instrumentos de cada sistema foram fadigados a 3/4 de Nf e depois submetidos à torção até a ruptura. Os dados foram analisados utilizando o teste ANOVA ($\alpha$=.05). A superfície longitudinal, bem como a superfície de fratura após a fadiga foi examinada com um microscópio eletrônico de varredura (MEV). Os resultados mostram que os instrumentos CM tiveram valores de Nf significativamente maiores quando comparados aos instrumentos SE, com HF exibindo o maior Nf (*P* = .001). Os valores de torque médio para F2 foram os mais elevados (*P* = .001), seguido de TYP, HF e RC. Instrumentos CM pré-carregados a 3/4 de Nf e submetidos à torção até a ruptura tiveram valores de torque menores quando comparados aos instrumentos não carregados (HF *P* = .003; TYP *P* =.001), enquanto os instrumentos SE não apresentaram diferenças significativas (F2 *P* = .059; RC *P* =.079).

A superfície de fratura dos instrumentos CM mostraram maiores superfícies lisas de fraturas e menores regiões de dimples, em relação aos instrumentos SE. Conclui-se que a vida em fadiga dos instrumentos CM foi significativamente maior quando comparada aos instrumentos SE, com o instrumento HF apresentando o valor de Nf maior. Os valores de torque para F2 foram maiores entre todos os instrumentos. Portanto a resistência torcional dos instrumentos CM pode ser comprometida após o pré carregamento em fadiga.

**Palavras-chave:** Instrumentos rotatórios de NiTi, instrumentos *CM*, resistência torcional, resistência à fadiga.

## ABSTRACT

**"Influence of cyclic flexural deformation on the torsional resistance of CM instruments in comparison to conventional NiTi instruments"**

Introduction: To evaluate the influence of cyclic deformation on the torsional resistance of NiTi controlled memory (CM) files, when compared to superelastic (SE) instruments with similar geometric and dimensional characteristics. Methods: New 30/.06 HyFlex (HF; Coltene/Whaledent,Inc, Cuyahoga Falls, OH), Typhoon (TYP; Clinician's Choice Dental Products, New Milford, CT), RaCe (RC; FKG, La-Chaux De Fonds, Switzerland), and ProTaper Universal F2 instruments (F2; Dentsply Maillefer, Ballaigues, Switzerland) were assessed. Diameter, pitch length and helical angle were measure along the active part of the instruments. Flexural fatigue and torsion to failure tests for each file (n = 10) were carried out. Ten new instruments of each type of file were fatigued to 3/4 of their fatigue life and then submitted to torsion until rupture. The longitudinal surface, as well as the fracture surface after fatigue was examined with a scanning electron microscope. Data were analyzed using one-way analysis of variance ($\alpha = .05$). Results: CM files had a significantly higher mean number of cycles to fracture (Nf) when compared to SE instruments, HF exhibiting the highest Nf ($P = .001$). The mean torque value for F2 was the highest ($P = .001$). CM files pre-cycled to 3/4 Nf had a significantly lower torque than the new files (HF $P = .003$, TYP P = .001), whereas the SE instruments displayed no significant differences (F2 $P = .059$, RC $P = .079$). The surface fracture of CM files displayed larger smooth areas and smaller dimple region than SE instruments. Conclusion: Fatigue life of CM instruments was significantly higher than SE instruments, with HF displaying the highest mean Nf values. Torque value for F2 was significantly higher among all of the instruments. Cyclic flexural loading significantly reduced the torsional resistance of CM instruments.

**Key words:** Controlled memory, fatigue resistance, torsional resistance, nickel-titanium endodontic instruments.

# INTRODUCTION

## INTRODUCTION

Nickel-Titanium alloy offers special mechanical properties like shape memory effect (SME) and superelasticity (SE) (WALLIA, 1988). Because of these particular properties, the alloy was introduced to the endodontic practice with the outlook of decreasing procedural errors during the preparation of curved canals. Endodontic rotary NiTi instruments are efficient to prepare complex curved root canals, maintaining the original path, thus allowing proper three-dimensional obturation (PETTIETTE *et al.,* 2001; PETERS, 2004).

Nickel-Titanium (NiTi) alloy, in the solid state, experiments transformation of phases called Martensitic Transformation (MT). This transformation takes place in a seizing course. The atoms of the alloy move in cooperative way in the presence of low temperature or when submitted to tension, although the chemical composition of the matrix during transformation is not altered. Two crystalline structures are present in the MT: austenite (B2) or parent phase, which is a cubic body-centered structure stable at high temperature; and martensite (B19') that is a monocyclic structure stable at low temperatures. NiTi alloy has the ability to alter the crystalline structure due to its mechanical properties, superelasticity (SE) and shape memory effect (SME). The SME is the capacity of the alloy to recover great non-linear deformations by moderate increase of temperature, despite of the material apparently suffered permanent deformation. On the other hand, SE is a special form of SME where the recovery of the original shape happens right after the removal of tension (OTSUKA & WAYMAN, 1998). For endodontic instruments, the MT occurs due to the tension generated by the curvature and small diameter of the root canal. Once this tension ceases, the reverse transformation develops, restoring the original shape of the instrument (THOMPSON, 2000).

The differences in the alloys will be given by the content of Nickel (Ni) and the temperature where the martensitic transformation occur. The transformation temperatures are highly conditioned to the Ni concentration and the thermomechanical treatment, implicating that an increase of Ni will severely decrease the transformation temperatures (OTSUKA & REN, 2005).

The most commonly used thermal treatments for NiTi alloys are annealing, solubilization, quenching and aging. Three different reactions could result after one of these treatments: 1- change in the chemical composition (precipitation); 2- reduction of defects (re-crystallization); 3- structural transformation of phase.  Solubilization uses a higher temperature to dissolve the precipitates, the quenching is the cooling of the material, and the annealing is the treatment at a lower or intermediate temperature which usually happens after solubilization, allowing the precipitates to form under controlled conditions. (HUANG & LIU, 2001; CHENG *et al*., 2003).

NiTi alloys with a small excess of Ni respond well to heat treatment at lower temperatures because coherent, submicroscopic Ti3Ni4 precipitates can form within the β-phase matrix which are considerably effective to improve the properties of SME and SE (SABURI *et al.,* 1982). This precipitates have a lenticular form which correspond with the matrix, giving origin to stress fields around them (ALLAFI *et al.,* 2002).  These stress fields can be points of nucleation for the formation of R phase. The introduction of fine Ti3Ni4 precipitates in the annealing process, or the insertion of dislocations cells through cycles of deformation/heating, are able to change the MT from B2-B19' to B2-R-B19'.  The precipitates and dislocations induce the formation of stress fields in the matrix around them (OTSUKA & REN, 2005), which produce high resistance to great deformations associated to the deformation of B19'. The R phase creates a deformation in the crystalline structure significantly lower (ALLAFI *et al*., 2002).

Despite of the particular properties of NiTi alloy, NiTi rotary endodontic instruments can undergo unexpected fracture. Fracture of NiTi rotary instruments has been classified into torsional failure and flexural fatigue (SATTAPAN *et al.,* 2000). An instrument suffers flexural fatigue when is rotated inside of a curved canal, undergoing stresses of compression and tension causing crack nucleation that propagate and coalesce until fracture takes place. These stresses are usually related to the geometry of the canal, with higher tensions of cyclic loads in the area of maximum curvature (PRUETT *et al*., 1997; BAHIA & BUONO 2005). Whilst in the case of fracture by torsional loads, the instrument is clamped in one of the walls of the root canal, if it exceeds the elastic limit, it may suffer plastic deformation followed by fracture. Torque is one of the parameters that influence the torsional failure of an instrument. During the instrumentation it may be affected by the extension of contact between the instrument and the canal walls, larger the extension higher the torque. SATTAPAN *et al*. (2000) reported a higher prevalence of torsional fracture for multiple use NiTi rotary files; however, other researchers have suggested that flexural fatigue is probably the most prevalent cause of unexpected fracture encountered clinically (SHEN *et al*., 2006, PRUETT *et al.,* 1997).

Different manufacturing processes have been developed to improve the mechanical properties of the alloy through different thermal treatments, thus enhancing the instruments' flexibility and fatigue resistance (JOHNSON *et al.,* 2008; LARSEN *et al.,* 2009; KRAMKOWSKI & BAHCALL, 2009; SHEN *et al*., 2009; GAO *et al.,* 2010, PEIXOTO *et al.,* 2010; PEREIRA *et al.,* 2012; BRAGA *et al.,* 2014). Higher fatigue resistance and slower propagation of cracks have been observed in instruments with certain content of martensite at room temperature compared to fully austenite or superelastic files (PEIXOTO *et al*., 2010; SHEN *et al*., 2013; PEREIRA *et al.,* 2013; BRAGA *et al*., 2014). Heat treatment is one of the most fundamental approaches favoring the adjusting transition temperatures of these alloys, with the purpose of controlling the alloy microstructure and consequently influencing its mechanical behavior (SABURI *et al*., 1982).

Among the first thermo-mechanical treatments applied to the conventional NiTi alloy is the M-Wire (MW) (Dentsply Tulsa Dental Specialties, Tulsa, OK), introduced in 2007. The 508 Nitinol wire (Nitinol Devices & Components, Inc.) is draw back and submitted to different thermal treatments (BERENDT, 2007), resulting in a material that includes some amount of R-Phase and B19' martensitic phases (PEREIRA *et al*., 2012). This material exhibits a more efficient superelastic behavior with reduced generation and accumulation of lattice defects during each load-unload cycle, increasing the flexibility and fatigue resistance of these instruments (JOHNSON *et al.,* 2008; PEIXOTO *et al.,* 2010; PEREIRA *et al.,* 2012). Besides, accumulation of latticework defect is considerably diminished, due to improved superelastic behavior (PEREIRA *et al*., 2012).

Later in 2010, NiTi rotary instruments made from a NiTi wire were subjected to a particular proprietary thermal processing named the control memory technology (CM). These instruments content a considerable amount of stable martensite at room temperature, displaying superior flexibility and a controlled memory effect. They can be easily deformed and recovered to their original shape under heating above their transformation temperatures (SHEN *et al.,* 2013; BRAGA *et al*., 2014). Studies have shown that these instruments have superior flexibility and fatigue resistance than conventional NiTi rotary instruments made from superelastic wire (SHEN *et al.,* 2011; BRAGA *et al.,* 2014; PLOTINO *et al.,* 2014, CAMPBELL *et al*., 2014). Hyflex (HF) (Coltene Whaladent, Cuyahoga Falls, OH) and Typhoon (TYP) (Clinician's Choice Dental Products, New Milford, CT) are manufactured from CM technology.

The etiology of instrument fracture during root canal preparation is complex; however flexural fatigue and torsional failure have been frequently implicated (BAHIA e*t al*., 2008, PEIXOTO *et al.,* 2010). These stresses may take place simultaneously but literature commonly reports both factors separately. Previous studies have observed that flexural preloading cycles significantly affect the torsional resistance of conventional NiTi rotary instruments (ULLMANN & PETERS, 2005, BAHIA *et al*., 2006, KIM *et al*., 2012). However, the literature still remains limited in evaluate the influence of cyclic loads on the fracture resistance of CM instruments (CAMPBELL *et al*., 2014, PEDULLA *et al.,* 2014). Campbell et al., 2014 reported that a TYP CM instrument after been submitted to 75% of the fatigue life presented similar results as observed in this study, considerably reducing their resistance to torsional loads. The aim of this study was to assess the influence of flexural cyclic preloads on the torsional resistance of the instruments HF and TYP manufactured with CM technology,

# OBJECTIVES

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## General Objectives

To assess the influence of cyclic flexural loads over the torsional resistance of CM instruments Hyflex and Typhoon, when compared to conventional NiTi instruments RaCe and ProTaper Universal F2, after the instruments been submitted to three fourths of their resistance to fatigue.

## Specific Objectives

To assess the dimensional and geometrical characterization of the CM instruments Hyflex and Typhoon, as well as conventional NiTi instruments Race and PTU F2. Diameters at three mm from the tip (D3), pitch length and helical angle measurements.

To evaluate the flexural fatigue resistance of these rotary instruments until fracture to determine the number of cycles until failure (Nf).

To evaluate the torsional resistance until fracture, to determine the maximum torque of the rotary NiTi instruments previously mentioned, performed according to the ISO 3630-1 specification.

To assess the influence of the flexural fatigue over torsional resistance when the NiTi rotary instruments have been fatigued to three fourths of their Nf.

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# ARTICLE

## SCIENTIFIC ARTICLE

### ARTICLE

**Influence of cyclic flexural deformation on the torsional resistance of CM instruments in comparison to conventional NiTi instruments**.

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**Keywords:** Controlled memory, fatigue resistance, torsional resistance, nickel-titanium endodontic instruments.

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## Abstract

**Aim:** To evaluate the influence of cyclic deformation on the torsional resistance of controlled memory (CM) NiTi files, when compared to superelastic (SE) instruments with similar geometric and dimensional characteristics. **Methods**: New 30/.06 HyFlex (HF; Coltene/Whaledent,Inc, Cuyahoga Falls, OH), Typhoon (TYP; Clinician's Choice Dental Products, New Milford, CT), RaCe (RC; FKG, La-Chaux De Fonds, Switzerland ), and ProTaper Universal F2 instruments (F2; Dentsply Maillefer, Ballaigues, Switzerland) were assessed. Diameter, pitch length and helical angle were measure along the active part of the instruments. Flexural fatigue and torsion to failure tests for each file (n = 10) were carried out. Ten new instruments of each type of file were fatigued to 3/4 of their fatigue life and then submitted to torsion until rupture. The lateral surface, as well as the fracture surface after fatigue waere examined with a scanning electron microscope. Data were analyzed using one-way analysis of variance ($\alpha = .05$). **Results:** CM files had a significantly higher mean number of cycles to fracture (Nf) when compared to SE instruments, HF exhibiting the highest Nf (*P* = .001). The mean torque value for F2 was the highest (*P* = .001). CM files pre-cycled to 3/4 Nf had a significantly lower torque than the new files (HF *P* = .003, TYP *P* = .001), whereas the SE instruments displayed no significant differences (F2 *P* = .059, RC *P* = .079). The surface fracture of CM files displayed larger smooth areas and smaller dimple region than SE instruments. **Conclusion:** Fatigue life of CM instruments was significantly higher than SE instruments, with HF displaying the highest mean Nf values. Torque value for F2 was significantly higher among all of the instruments. Cyclic flexural loading significantly reduced the torsional resistance of CM instruments.

**Key words:** Controlled memory, fatigue resistance, torsional resistance, nickel-titanium endodontic instruments.

## Introduction

The endodontic field encountered an important breakthrough with the introduction of Nitinol alloy. It offers special mechanical properties like shape memory effect (SME) and superelasticity (SE) (1). Notwithstanding of the particular properties of NiTi instruments, they could suffer unexpected fractures during root canal preparation.

The performance of endodontic NiTi instruments under cyclic loading relies on material strength, microstructure, surface quality and type of loading. It is associated to structural and functional fatigue, which are damages generated either to the microstructure, or to the functional properties of the material, respectively (2).

According to some researchers, flexural fatigue is probably the most prevalent cause of unexpected fracture encountered clinically (3, 4). Though, Sattapan *et al*. (5) reported a higher prevalence of torsional fracture for multiple use of NiTi rotary files. Despite these remarks, it is difficult to avoid that both flexural and torsional stresses occur concomitantly, but until now just a few reports associate them together. It has been reported that cyclic flexural straining significantly reduced the torsional resistance, particularly of the instruments preloaded at three fourths of their lifespan (6-8).

In the need to improve the resistance of the NiTi files, different thermo-mechanical treatments have been applied to the NiTi alloy to enhance their endurance (9-12). Heat treatment is one of the most fundamental approaches favoring the adjusting of the transition temperatures, with the purpose of controlling the alloy microstructure and consequently improving its mechanical behavior (13).

Among the first thermomechanical treatments applied to the conventional NiTi alloy is the M-Wire (MW) introduced in 2007 by Berendt (14). The 508 Nitinol wire (Nitinol Devices & Components, Inc) is draw back and submitted to different thermomechanical treatments resulting in a material that includes some amount of the R-phase and B19' martensite (15), thus improving the flexibility and fatigue resistance of the instruments (9, 10, 16). The accumulation of lattice defects is considerably lowered due to a more competent superelastic behavior (15). Later in 2010, NiTi rotary instruments made from a NiTi wire were subjected to a particular proprietary thermal processing named the Controlled Memory technology (CM Wire; DS Dental, Johnson City, TN). Studies have shown that instruments made with the CM technology have superior flexibility and fatigue resistance than instruments of conventional NiTi. (17, 18, 19). The aim of this *in vitro* study was thus to assess the influence of flexural fatigue loading on the torsional resistance of the CM rotary files, when compared to conventional NiTi files with similar dimensions and geometries.

## Materials and Methods

New 30/.06 instruments type Typhoon (TYP; Clinician's Choice Dental Products, New Milford, CT) and HyFlex CM (HF; ColteneEndo/Whaledent, Cuyahoga Falls, OH), along with RaCe (RC; FKG Dentaire, La-Chaux de Fonds, Switzerland) and ProTaper Universal F2 (F2; Dentsply Maillefer, Ballaigues, Switzerland) made of conventional NiTi (C-NiTi) were analyzed. These instruments were chosen due to their similar geometric design, RC and HF with triangular cross-sections, F2 and TYP with convex triangular cross-sections.

A total of 120 files were used in this study. The instruments were divided into three groups: (i) Control Group 1 (CG1), comprising of 10 new instruments of each system, that were fatigued until fracture to determine the number of cycles to failure (Nf); (ii) Control Group 2 (CG2) (n=10 each system) submitted to torsion tests to failure to determine the maximum torque; and (iii) Experimental Group (EG), where new instruments (n = 10 each system) were fatigued to three fourths of their Nf and then submitted to torsion tests to failure.

To assess the dimensional characteristics based on the American National Standards Institute/American Dental Association Specification No. 101, ten instruments of each system were photographed using a high-resolution digital camera (20D; Canon; Tokyo; Japan). Lines were drawn on the instrument images, and the outermost diameter at each mm from the tip, together with the distance between each pitch, were determined using ImageJ 1.49 V (NIH, Bethesda, MD).

Ten instruments (n=10) of each system were tested in fatigue until failure at room temperature, using a bench device with an artificial canal made of quenched steel. This device consists of an arch with a curvature radius of 5 mm, and an angle of 45° in a guide cylinder of 10 mm in diameter, all of them made of the same material (20). The chosen geometry placed the area of maximum tensile strain amplitude at about 3 mm from the tip of the instrument. The instruments, attached to handpiece with a reduction of 16:1 and a torque of 4 N.cm, were allowed to rotate freely until fracture inside of the artificial canal, aligned between the arch and the guide cylinder, and the number of cycles to failure (Nf) was obtained by multiplying the rotation speed (300 rpm) by the test time registered using a digital chronometer. The point of fracture relative to the tip of the instrument was estimated by measuring the fractured file with an endodontic ruler. Once the mean Nf was determine, new files (n = 10) were tested in the fatigue test bench up to 3/4 of their previously determined fatigue life, and subsequently tested in torsion to fracture, corresponding to the experimental group for each system.

The torsion tests were performed according to the ISO 3630-1 in a bench machine (AN8050; Analógica, Belo Horizonte, MG, Brazil). The rotation speed was set clockwise to 2rpm. The end of the shaft was clamped into a chuck connected to a reversible geared motor. Three millimeters of the instrument's tip were clamped in another chuck with soft brass jaws to prevent sliding. Continuous recording of torque and angular deflection was provided by a specifically designed computer program. The statistical significance of the differences of the measurements among the four types of files was assessed by one-way analysis of variance ANOVA and post hoc Tukey's test at a significance level of $P > .05$.

The fracture surfaces of three randomly selected instruments of each system were examined under a scanning electron microscope (SEM-FEI Inspect S50, Hillsboro, OR) to access the main characteristics of the fracture process.

**Results**

The mean values and standard deviation (±SD) of the diameter at 3 millimeters from the tip (D3) for the instruments exhibiting a triangular cross section, revealed similar values (HF: .48 ± .01 mm, RC: .49 ± .01 mm), with no statistical significant differences between them ($P$ = .180). On the other hand, D3 values for convex triangular cross section instruments (TYP: .48 ± .01 mm, PTU F2: .50 ± .01 mm) displayed PTU F2 instruments with significantly larger D3 ($P$ = .002).

The main geometric characteristics and the mechanical properties of the new files are summarized in Figure 1. PTU F2 instruments exhibited smaller values (1.04 ± .02 mm) of the pitch lengths of all instruments evaluated (Fig. 1a), followed by TYP (1.26 ± .04 mm), HF (1.52 ± .02 mm) and RC (1.66 ± .14 mm) instruments. The pitch lengths increased along the active part for all of instruments, except for HF instruments, that exhibited a constant value of this geometric parameter. The RC instruments exhibited the largest pitch lengths among all of the instruments evaluated. The mean values of the Nf obtained in the fatigue tests of new instruments (CG1) are shown in Fig. 1b (standard deviations shown as error bars). The results indicated that HF instruments presented a significantly higher number of cycles to failure compared to RC instruments ($P$ = .001). The same pattern was observed for TYP and PTU F2 instruments, where TYP showed significantly higher mean Nf values (*P* = .001), confirming the higher fatigue resistance of CM instruments. Amongst all of instruments, the mean point of fracture from the tip was 3.0 mm (± .01 mm).

Average torsion curves for the new instruments (CG2) are shown in Fig. 1c. The results indicated that instruments with triangular convex cross-section (TYP and PTU F2) exhibited higher mean torque values in comparison with instruments with triangular cross-section (HF and RC). The statistical analysis showed that the maximum torque of PTU F2 was significantly higher than TYP files (*P* = .002), with similar cross-section geometry. Differently, HF and RC presented similar results for the maximum torque (*P* = .200). Moreover, PTU F2 presented the highest torsional resistance amongst all of instruments evaluated (*P* = .001).

The mean values of maximum torque of the new instruments (CG2) and those previously fatigued to 3/4 of their fatigue life and then tested in torsion to rupture (EG) are shown in Figure 2. Cyclic flexural preloading caused a statistically significant reduction of maximum torque for the CM instruments of up to 25% (HF, *P* = .003, and TYP, *P* = .001). However, no such difference was noticed between CG2 and EG superelastic instruments: PTU F2 (*P* = .059), and RC (*P* = .079).

The secondary electron images of the fracture surfaces obtained by SEM are shown in Figure 3 and are typical of fatigue fracture of metals. Outlined areas correspond to the region of slow fatigue crack nucleation and propagation (smooth areas), which are considerably larger in

CM instruments.

**Discussion**

The NiTi rotary instruments have improved the quality and time of preparations of root canals; even so their fracture incidence still remains a concern. During the instrumentation, the file undergoes flexural and torsional stresses or a combination of both. These may be influenced by different factors such as: operator experience, instrument design, curvature radius of the canal, alloy treatment, among others. Hitherto, the literature remains limited when assessing these factors together in CM instruments (19, 21). Therefore, considering that these stresses may take place concomitantly, the proposal of this study was to assess the influence of cyclic flexural deformation on the torsional resistance of CM in comparison to SE instruments.

It is generally accepted that the high degree of cyclic deformation to which NiTi rotary instruments are submitted during the preparation of curved root canals can lead to mechanical fatigue (3, 20, 22). The fatigue resistance of a rotary endodontic instrument is related to their diameter and to the degree to which it is flexed when placed in a curved root canal, with greater angles of curvature and smaller radii leading to a shorter life expectation (3, 18, 20). The maximum tensile strain amplitude, $\varepsilon T$, is used to establish the influence of the factors involved, and it is expressed in the formula $\varepsilon T = D/(2R-D)$, where R is the canal radius and D is the instrument's diameter at the point of maximum canal curvature. Considering that the mean diameters at 3 mm from the tip (D3) of the RC, HF and TYP, and PTU F2 files were

.49, .48, .48 and .50 mm, respectively, a maximum tensile strain amplitude between 5.0% and 5.3% would be imposed on the instruments introduced into the artificial canal used here (with a 5-mm radius of curvature). Thus, the significantly increased fatigue resistance exhibited by the HF and TYP files cannot be attributed to the small difference in strain amplitude and is most likely caused by the superior mechanical properties of CM instruments. Results of the present study showed, in addition, that although the TYP files exhibited a similar D3 value compared with the HF files, the latter exhibited significantly increased fatigue resistance, suggesting that the proprietary heat treatments applied to the HF and TYP files are not equivalent.

When assessing the torsional resistance of the control group instruments (CG2), higher mean torque values would be expected for the instruments with triangular convex cross-section, higher D3 values, shorter pitch lengths, and greater helical angles (16, 23). These parameters were determinant in the significantly higher torsional resistance of PTU F2 instruments. They displayed the higher D3 values, shortest pitch lengths along the active part, corresponding with their greater helical angle, justifying a higher torque than TYP and the rest of the files. On the other hand, when HF and RC were compared, the mean torque values were similar, in accordance with their D3 dimensions and triangular cross section. Despite of these values, it would be expected that HF files displayed higher torque values, when compared to RC, because of the larger pitches of the latter; however, the torque values were similar, probably due to the thermal treatment to which CM instruments are submitted, as observed elsewhere (19).

Study reports, in which the properties and behavior of NiTi rotary instruments in flexural fatigue and torsional resistance are assessed separately, are commonly found in the literature. Some studies have described how cyclic flexural loads influenced the torsional resistance of superelastic NiTi instruments (6, 7, 8, 22, 24). Also, it has been reported that lateral pressure may also limit the endurance to torsion of SE NiTi instruments (25), as well as the multiple uses affect the file's ability to undergo torsional loads (22, 26).

The present work showed that the cyclic flexural deformation to 3/4 of their fatigue life significantly reduced the torsional resistance of the CM files (EG) (Fig. 4). These results are supported by Campbell *et al.* (19) study where preloading cycles up to 75% of the fatigue life affected the torsional resistance of CM instruments TYP 40/.04. Even though CM files displayed a significantly higher fatigue resistance than SE instruments, the cyclic deformation to 3/4 of their fatigue life compromised their torsional resistance. This could be explained by the same mechanism thought to render NiTi martensite more fatigue resistant than austenite, the fact that during fatigue loading thermal martensite, because of the large amount of interface variants, a higher amount of crack ramification takes place, dissipating the crack propagation energy (27, 28). Although this mechanism favors fatigue resistance, when the fatigued material undergoes a different type of loading, it is weakened by the presence of these cracks. In addition, the larger amount of smooth areas of slow crack nucleation and propagation found on the fracture surface of CM instruments (Fig. 3) should contribute for diminishing their ability to withstand torsional loads. These findings are in agreement with previous data (13, 17-19, 29).

According to the results found in this research, cyclic flexural preloads may compromise the ability of CM instruments to tolerate torsional loads when submitted to repeated usage. Microstructure often plays an important role in controlling the mechanical behavior of NiTi rotary instruments. Further research will be necessary to analyze how other factors may affect either torsional or fatigue resistance of thermally treated instruments.

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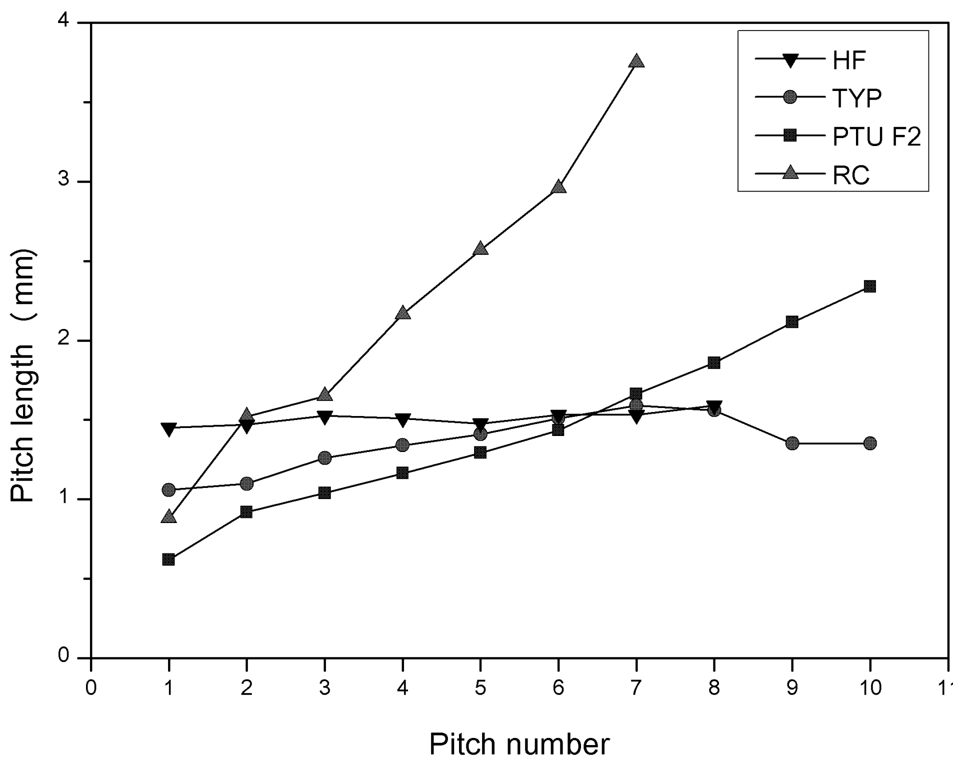
**Figure Legends**

**Figure 1**. (a) Mean values of pitch lengths; (b) mean values of Nf (CG1, standard deviations shown as error bars, differently labeled columns represent statistically significant differences, $p \leq .05$); and (c) average torque curves of the analyzed instruments.

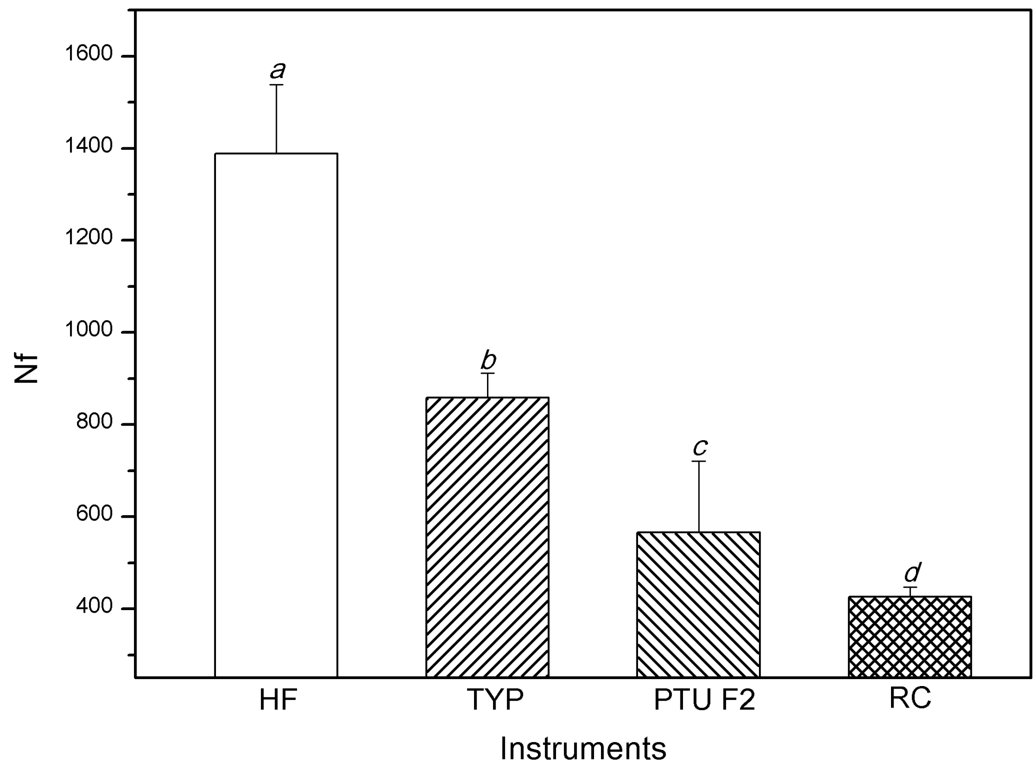
**Figure 2**. Mean values of maximum torque of new instruments (CG2) and of instruments previously fatigued to 3/4 of their fatigue life (EG). Standard deviations shown as error bars (differently labeled columns represent statistically significant differences, $p \leq .05$).

**Figure 3.** Secondary electron images of typical fracture surfaces of superelastic and CM instruments. Outlined areas correspond to the region of fatigue crack nucleation and propagation (smooth areas). (a) RaCe (RC); (b) HyFlex (HF); (c) ProTaper Universal F2 (F2); and (d) Typhoon (TYP).

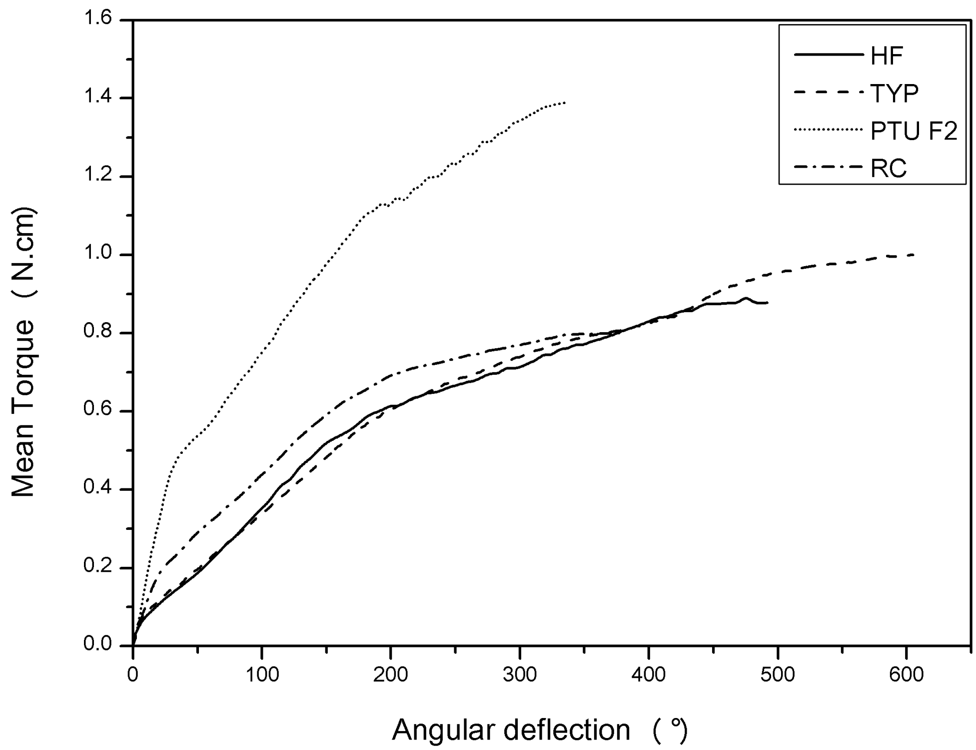
1. **(a)**



**(b)**

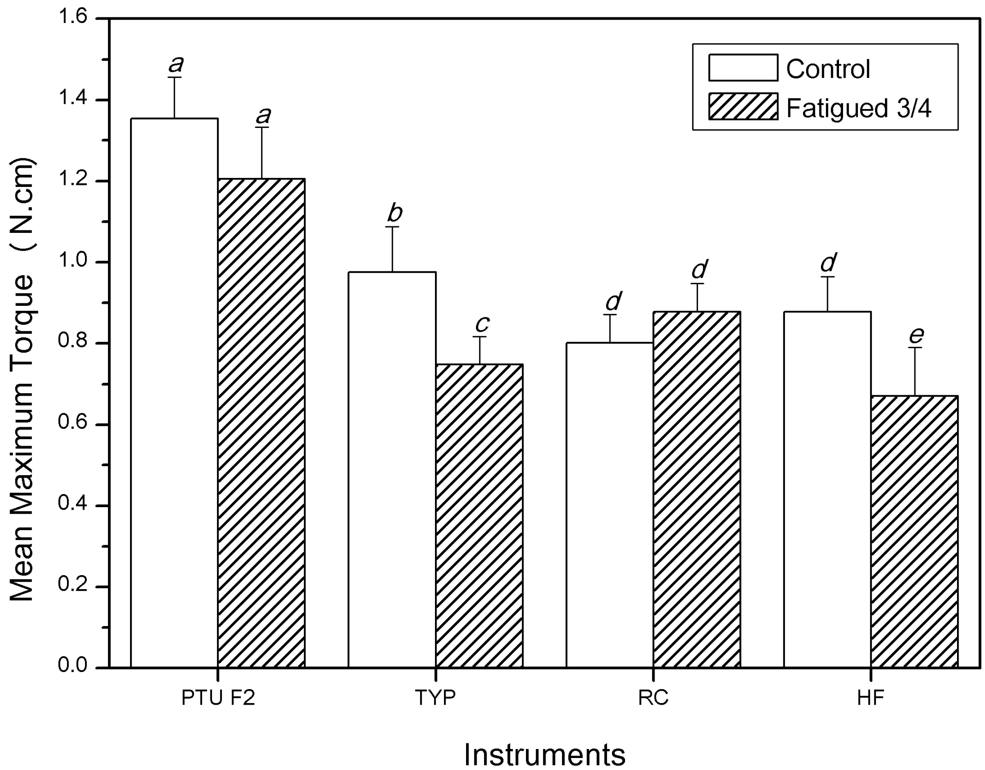

1600
1400
1200
1000
800
600
400
Nf
a
b
c
d
HF
TYP
PTU F2
RC
Instruments

**2. (a)**



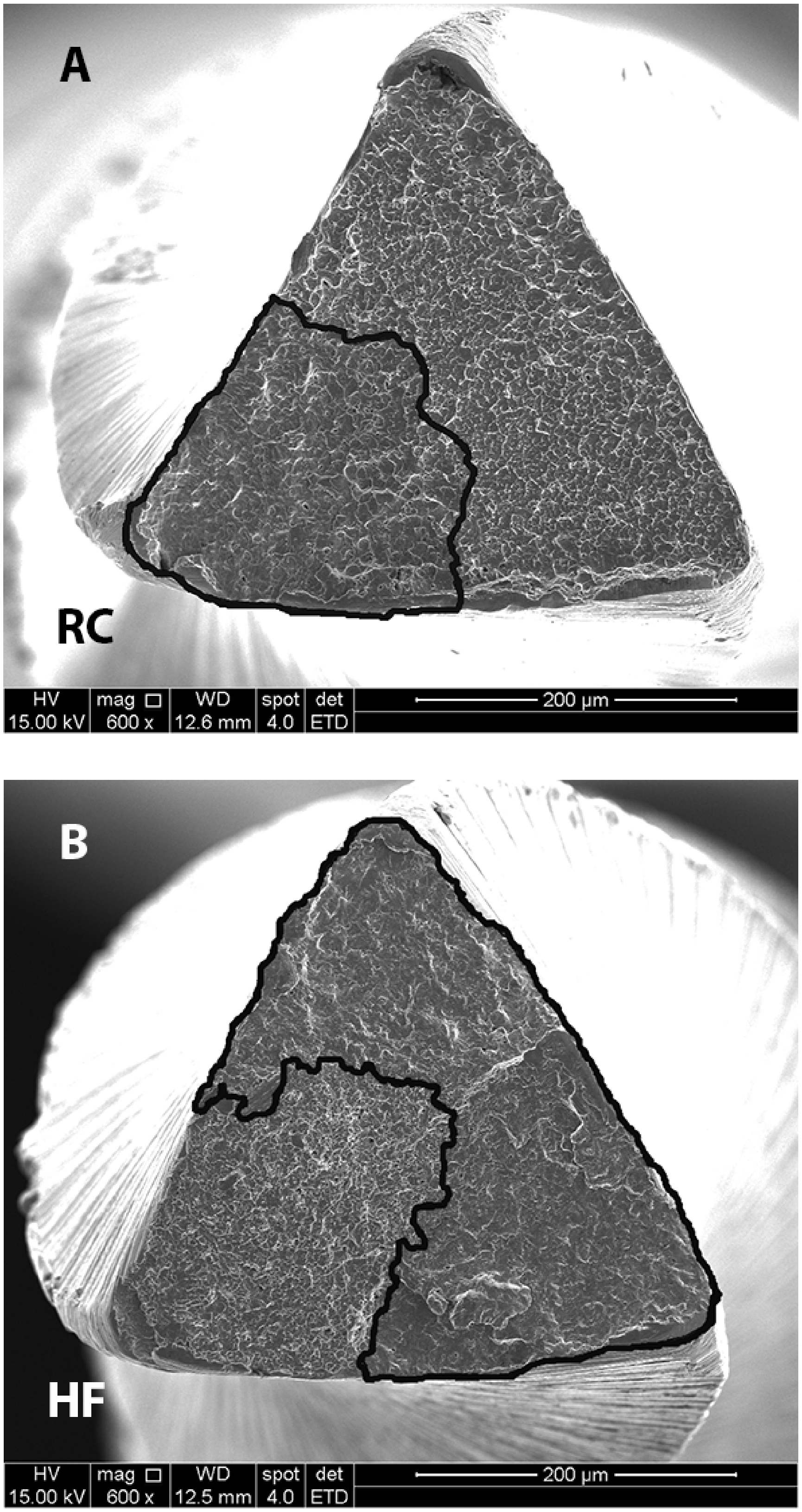
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
Mean Torque (N.cm)
0
100
200
300
400
500
600
Angular deflection (°)
HF
TYP
PTU F2
RC

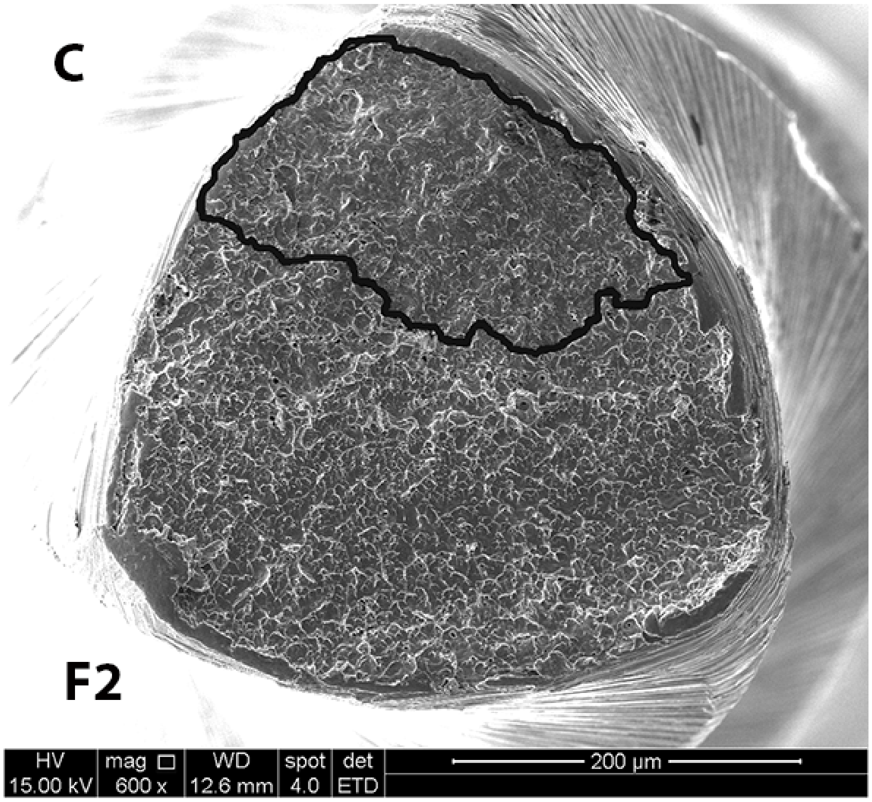
**(b)**

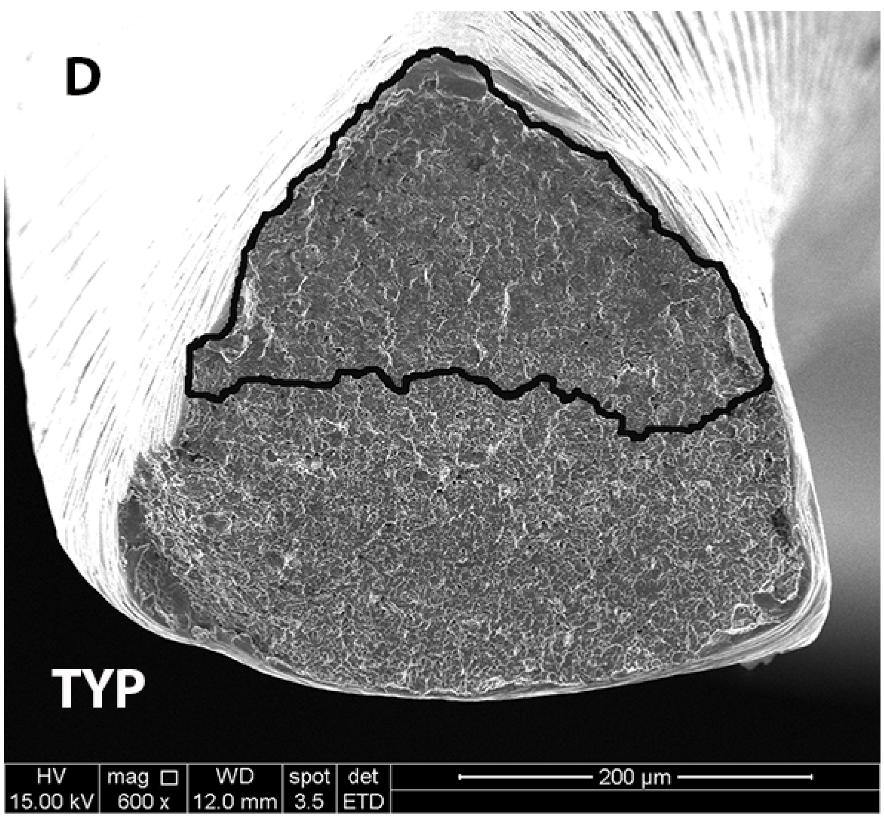


Mean Maximum Torque （N.cm)
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
Control
Fatigued 3/4
a
a
b
c
d
d
d
e
PTU F2
TYP
RC
HF
Instruments

**3.**


A
RC
HV
mag
WD
spot
det
15.00 kV
600 x
12.6 mm
4.0
ETD
200 µm
B
HF
HV
mag
WD
spot
det
15.00 kV
600 x
12.5 mm
4.0
ETD
200 µm


C
F2
HV
15.00 kV
mag □
600 x
WD
12.6 mm
spot
4.0
det
ETD
200 μm


D
TYP
HV
15.00 kV
mag □
600 x
WD
12.0 mm
spot
3.5
det
ETD
200 μm

# FINAL REMARKS

## Final Remarks

The results of this study support the following conclusions:

The comparative analysis of the diameter at 3 mm from the tip exhibited similar values for HF, TYP, and RC instruments, with the exception of F2, which presented larger D3 than the nominal value.

The flexural fatigue tests for new instruments (CG1) indicated that the CM instruments (HF and TYP) had significantly higher mean Nf values when compared to SE instruments (RC and F2) that are made from conventional NiTi alloy. This result suggests that the thermal treatment of the CM instruments improved their mechanical behavior concerning the resistance to flexural fatigue, when compared to rotary conventional NiTi instruments.

Regarding to the torsional resistance of the new instruments (CG2), F2 instruments revealed the highest mean maximum torque values of all of instruments, followed by TYP files. Their convex triangular cross section, larger D3, along with the smaller pitch length values, might have determined their higher resistance to torsion. This result is agreeing with the fact that geometrical and dimensional features may influence the ability of the instruments to withstand torsional loads. However, in the case of HF and RC instruments, it was expected the former to exhibit higher mean torque values because of their smaller pitch length values; however, their mean maximum torque was similar to RC instruments. It is probable that the thermal treatment to which HF instruments were submitted was responsible for the similar torsional resistance to RC instruments.

Despite the fact that CM instruments exhibited higher fatigue resistance than SE instruments, the cyclic flexural preloads up to three-fourths of their previously determined fatigue life (EG) significantly reduced their torsional resistance. A higher amount of cracks nucleation and slow crack propagation, together with smaller dimple areas in the fracture surfaces (SEM) than SE instruments, can be responsible for their reduced ability to withstand torsional loads.

Most of the fracture surfaces of the CM instruments analyzed by SEM showed multiple crack origins, whereas most of SE NiTi instruments had, in general, one crack origin, contributing to a significant reduction of the torsional resistance of the CM files submitted to previous cyclic deformation. These findings suggest that the thermal treatment enhances the fatigue resistance of CM instruments; nevertheless, cyclic flexural preloads affected their torsional resistance.

In summary, within the limitations of this study, it has been observed that CM instruments have a significantly higher resistance to flexural fatigue in comparison to SE instruments, which could be advantageous for the clinical practice. However, cyclic flexural preloads may compromise the torsional resistance of CM instruments. This information may aid clinicians to understand the fracture mechanism of these endodontic instruments, to practice more predictable and safer procedures.

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