UNIVERSIDADE FEDERAL DE MINAS GERAIS

Faculdade de Odontologia

“Análise comparativa do comportamento mecânico dos instrumentos rotatórios de NiTi fabricados com a tecnologia *CM*, *M-Wire* e com o fio de NiTi convencional”

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Belo Horizonte
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Tese apresentada ao Programa de Pós-Graduação em Odontologia da Universidade Federal de Minas Gerais, como requisito parcial à obtenção do grau de Doutor em Odontologia - Área de Concentração: Endodontia

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Dedicatória

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RESUMO

“Análise comparativa do comportamento mecânico dos instrumentos rotatórios de NiTi fabricados com a tecnologia *CM*, *M-Wire* e com o fio de NiTi superelástico convencional”

As propriedades mecânicas dos instrumentos endodônticos são afetadas por fatores como microestrutura, diâmetro, design, composição química, e tratamentos termomecânicos aplicados durante o processo de fabricação. O objetivo deste trabalho é comparar as propriedades físicas, químicas e estruturais e o comportamento mecânico de instrumentos *Hyflex* e *Typhoon*, que possuem memória de forma controlada (CM), em comparação aos instrumentos *ProFile Vortex*, fabricados com o fio de NiTi *M-Wire*, e *EndoWave* e *ProTaper Universal*, fabricados com fios de NiTi convencional. A composição química dos instrumentos foi analisada por espectroscopia de energia de raios X (EDS) e as temperaturas de transformação determinadas por calorimetria exploratória diferencial (DSC). Imagens das seções longitudinais e transversais foram empregadas para determinar o diâmetro e a área a 3 mm da ponta (D3 e A3), posição onde as solicitações mecânicas se concentram durante o uso clínico e os instrumentos são submetidos à maiores amplitudes de tensão ou são apreendidos nos testes. O comportamento mecânico foi avaliado através de ensaios de fadiga flexural até fratura e testes de flexão a 45° e torção até a ruptura, de acordo com a especificação ISO 3630-1. Os instrumentos *Hyflex CM*, *Typhoon CM*, *ProFile Vortex*, *ProTaper Universal* e *EndoWave* apresentaram composição química semelhante. Entretanto, os valores médios das temperaturas de transformação dos instrumentos *Hyflex CM*, *Typhoon CM* e *ProFile Vortex* foram estatisticamente superiores aos demais sistemas testados. Nos ensaios de fadiga, os instrumentos *Hyflex (CM)* e *Typhoon (CM)* apresentaram uma vida em fadiga significativamente superior ($p = 0,000$) aos instrumentos *ProFile Vortex*, fabricados com o fio de NiTi *M-Wire*, e estes foram significativamente superiores ($p = 0,000$) aos sistemas *ProTaper Universal* e *EndoWave*, fabricados com liga NiTi convencional. Entre os instrumentos *CM*, observou-se que o sistema *Hyflex* foi significativamente superior ($p = 0,000$) ao *Typhoon.* Os instrumentos *Hyflex CM e Typhoon CM* foram significativamente mais flexíveis aos demais instrumentos avaliados, possivelmente, devido aos tratamentos térmicos a que foram submetidos durante a fabricação, resultando em amaciamento da liga. Os instrumentos *ProTaper Universal F2 (PTU F2)* apresentaram valores de torque máximo significativamente superiores ($p = 0,034$) àqueles dos instrumentos *Typhoon CM*, de seção transversal semelhante, apesar do primeiro possuir A3 significativamente superior ($p = 0,000$). Já os instrumentos *Hyflex CM* e *Endowave*, apresentaram valores de torque máximo semelhantes ($p = 0,228$) e também valores médios de A3 (p =

0,276). Considerando que os instrumentos apresentam geometria e diâmetro semelhantes, o tratamento térmico, a que os instrumentos *CM* foram submetidos, representou importante melhora na vida em fadiga destes instrumentos, quando comparados com sistemas fabricados com fio *M-Wire* e com NiTi superelástico convencional. Em relação à resistência torcional, a aplicação de tratamentos térmicos pareceu não influenciar o comportamento mecânico dos instrumentos *CM* estudados.

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

ABSTRACT

"Comparative analysis of the mechanical behavior of NiTi rotary instruments manufactured with the CM technology, M-Wire and NiTi superelastic conventional"

The mechanical properties of the endodontic instruments are affected by factors such as diameter, design, chemical composition and thermo-mechanical treatments applied during the manufacturing process. The objective of this study is to compare the physical, chemical and structural properties and mechanical behavior of Typhoon and Hyflex instruments, which have a controlled memory (CM) compared to ProFile Vortex instruments, manufactured with M-Wire, and EndoWave and ProTaper Universal, manufactured with conventional NiTi wires. The chemical components have all been analyzed with X-Ray energy spectroscopy (EDS) and the transformation temperatures obtained with differential scanning calorimeter (DSC). Images from the longitudinal and transversal sections were used for determining the diameter and the area 3 mm away from the tip (D3 and A3), the position where the mechanical requests are focused on during the clinical use and the instruments are apprehended in the tests. The mechanical behavior has been evaluated through simulation of flexion and torsion until rupture accordingly to the ISO 3630-1, and flexural fatigue test until fracture. The instruments Hyflex CM, Typhoon CM, ProFile Vortex, ProTaper Universal e EndoWave showed similar chemical composition. However, the average values of transformation temperatures of the CM instruments and ProFile Vortex were significantly higher than other systems tested. In the fatigue tests, the Hyflex and Typhoon CM instruments showed a fatigue life significantly higher ($p = 0.000$) than ProFile Vortex instruments, fabricated with M-Wire, and these were significantly higher ($p = 0.000$) than ProTaper Universal and EndoWave systems, manufactured with conventional NiTi alloy. Among the CM instruments, it was observed that the Hyflex system was significantly higher ($p = 0.000$) than Typhoon. The Hyflex CM and Typhoon CM instruments were significantly more flexible, possibly due to thermal they have been subjected during manufacture, resulting in softening of the alloy treatments. The ProTaper Universal F2 instruments showed maximum torque values significantly higher ($p = 0.034$) of those Typhoon CM instruments, similar cross section, although the former have A3 significantly higher ($p = 0.000$). The Hyflex CM and Endowave instruments showed similar maximum torque values ($p = 0.228$) and also mean values of A3 ($p = 0.276$). Whereas instruments have similar diameters and geometry, the thermomechanical treatment performed in CM instruments represented an important improvement in the mechanical properties of the instruments, when compared with rotary systems manufactured with M-Wire and conventional NiTi wires. Regarding torsional strength, application of heat treatments seemed not influence the mechanical behavior of the instruments CM studied.

Keywords: NiTi rotary instruments, M-Wire, CM instruments, torsion resistance, flexural fatigue.

INTRODUÇÃO

1. INTRODUÇÃO

Os instrumentos rotatórios fabricados a partir de ligas níquel-titânio (NiTi) superelásticas foram introduzidos na prática endodôntica com o intuito de aumentar a segurança na formatação de canais radiculares curvos, minimizando os erros durante este procedimento. Esses têm apresentado bons resultados na formatação de canais curvos, mantendo a trajetória original do canal com características de fluxo contínuo, permitindo a obturação tridimensional do sistema de canais radiculares, além de diminuir o tempo de trabalho (PETTIETTE *et al.*, 2001; PETERS, 2004).

As ligas NiTi passam por uma mudança de fase adifusional no estado sólido denominada transformação martensítica (TM), a qual ocorre em função de variações de temperatura e de aplicação de tensão. Essas ligas possuem a capacidade de alterar sua estrutura cristalina, levando a mudanças em suas propriedades mecânicas. Devido à TM duas propriedades diferenciadas são observadas nas ligas NiTi: o efeito memória de forma (EMF) e a superelasticidade (SE). O EMF é uma propriedade encontrada em um grupo de materiais metálicos que, após deformações relativamente elevadas, são capazes de recuperar sua forma e/ou dimensões originais, através de um aquecimento moderado. A SE é um caso particular do EMF em que a recuperação de forma se dá apenas com a retirada da tensão, sem necessidade de aquecimento (OTSUKA & WAYMAN, 1998). Nos instrumentos endodônticos, a TM ocorre em função da tensão gerada pela curvatura no interior do canal radicular. Assim que a tensão cessa a transformação reversa ocorre restaurando a forma original do instrumento (THOMPSON, 2000).

É possível variar a composição química da liga para obter fios com características de EMF e SE. As diferenças entre as ligas estarão no seu conteúdo de níquel e na faixa das temperaturas da transformação martensítica e reversa. As temperaturas de transformação são muito dependentes da concentração de níquel na liga e da sua história termomecânica. Um aumento no teor de níquel leva a uma diminuição drástica nas temperaturas de transformação (OTSUKA & REN, 2005). Portanto, existe um interesse pelas ligas NiTi ricas em níquel devido ao controle das temperaturas de transformação através do teor de Ni. Em geral, as temperaturas de transformação martensítica e reversa, determinadas em amostras de instrumentos endodônticos de NiTi, são em média: 18,2ºC para martensita (B19’) inicial (Ms); -2,3ºC para martensita final (Mf); 3,4ºC para austenita inicial (As) e 22,9ºC para austenita final (Af). Dessa forma, pode ser verificado que a liga encontra-se totalmente austenítica (fase β) à temperatura ambiente, apresentando características de SE (BAHIA *et al*., 2005).

Ainda, com o aumento do conteúdo de Ni, podem ser formados precipitados de Ti_3Ni_4 finamente dispersos que são muito efetivos em melhorar as características de EMF e SE. A precipitação de Ti_3Ni_4 endurece a matriz da liga, melhorando, assim, a capacidade de recuperação do EMF e SE (MIYAZAKI *et al*., 1982; SABURI *et al*.,1982). Estes precipitados são coerentes com a matriz, possuem uma forma lenticular e dão origem a campos de tensão ao seu redor (ALLAFI *et al*., 2002).

Na liga NiTi, os tratamentos térmicos mais comumente empregados são o recozimento, a solubilização, a têmpera e o envelhecimento. Tais tratamentos podem implicar em três reações diferentes no estado sólido: (1) mudança local na composição química (precipitação); (2) redução dos defeitos (recristalização); e (3) transformação estrutural de fase. A solubilização utiliza temperatura mais alta para se dissolver os precipitados, a têmpera constitui-se no resfriamento do material e o recozimento é o tratamento em temperatura baixa ou intermediária, realizado geralmente após solubilização, para que ocorra a precipitação em condições controladas formando precipitados finos e coerentes capazes de aumentar a resistência da matriz à deformação por escorregamento (HUANG & LIU, 2001; CHENG *et al*., 2003).

Os precipitados de Ti_3Ni_4 são conhecidos por promoverem melhorias nas características de memória de forma e superelasticidade das ligas NiTi. Eles também afetam as características da transformação martensítica, podendo atuar como centros de nucleação para a formação de fase R. A introdução de finos precipitados de Ti_3Ni_4 no recozimento ou a introdução de células de deslocações através de ciclos de deformação/aquecimento são capazes de mudar a TM de B2-B19’ para B2-R-B19’. Este comportamento é explicado pelo fato de os precipitados e células de deslocações induzirem o aparecimento de campos de tensões na matriz circundante (ZHANG & SEHITOGLU, 2004; OTSUKA & REN, 2005). Estes campos de tensões produzem uma forte resistência às grandes deformações associadas com a deformação de B19’. A fase R produz uma deformação na rede cristalina significativamente menor (SOMSEN *et al*., 1999; ALLAFI *et al*., 2002).

Entretanto, apesar dessas propriedades desejáveis, esses instrumentos podem sofrer fraturas ocasionais durante a instrumentação dos canais. Dois mecanismos distintos de fraturas foram descritos por SATTAPAN *et al.* (2000): sobrecarga em torção e fadiga por flexão. Os instrumentos fraturados por torção geralmente apresentam características macroscópicas de deformação plástica, enquanto aqueles que falham por fadiga não exibem tais padrões de deformação. Quando o instrumento é girado no interior de um canal curvo, ele sofre tensões alternadas de tração e compressão levando à nucleação de trincas, que se propagam e coalescem até a fratura final do instrumento. Os níveis de tensão durante o carregamento cíclico são geralmente dependentes da geometria da curvatura e das cargas aplicadas, com um nível maior de tensão na área de curvatura máxima do canal radicular (SERENE *et al.*, 1995, PRUETT *et al.*, 1997, BAHIA & BUONO 2005). A ruptura por torção de um metal dúctil ocorre por cisalhamento ao longo dos planos submetidos às tensões máximas de cisalhamento, resultando em um plano de fratura normal ao eixo longitudinal (DIETER 1986).

Limiares de fadiga mais altos e taxas de propagação de trinca mais lentas têm sido encontrados na martensita comparada à austenita estável e superelástica. A austenita superelástica apresenta piores propriedades em relação ao crescimento de trincas, embora seja a microestrutura mais utilizada para aplicações comerciais (MCKELVEY & RITCHIE 2001, FIGUEIREDO *et al.* 2009, SHEN *et al*., 2013).

Já a fratura por torção ocorre quando parte do instrumento se prende no canal, enquanto sua haste continua a girar. Se o limite elástico do instrumento é excedido, ele sofre deformação plástica, que pode ser seguida de fratura. A fratura decorrente de sobrecarga torcional tem sido relatada como a causa mais comum de falha em instrumentos rotatórios de NiTi (SATTAPAN *et al*., 2000).

O torque aplicado em instrumento é um entre muitos parâmetros que influenciam a incidência de travamento, deformação e fratura por torção. O torque é geralmente expresso em gf.cm ou N.cm: o produto entre uma unidade de força e uma unidade de distância. Durante a preparação do canal radicular cada instrumento pode ser submetido a níveis diferentes de torque. Os valores de torque a que o instrumento é submetido durante a formatação dos canais radiculares é determinada, em parte, pela extensão da área de contato entre o instrumento e as paredes do canal. Quanto maior este contato, maior o torque gerado (BLUM *et al.*, 1999; SCHRADER & PETERS 2005).

As propriedades mecânicas dos instrumentos endodônticos e, portanto, sua flexibilidade, resistência à fadiga e à torção são afetadas por uma variedade de fatores como calibre, conicidade, geometria, composição química da liga e tratamentos termomecânicos aplicados durante o processo de fabricação. O aumento da resistência à fratura de instrumentos rotatórios de NiTi tem sido o foco das indústrias na área endodôntica. Possíveis soluções estão relacionadas com alterações no processo de fabricação, com o intuito de se obter propriedades mecânicas superiores (SHEN *et al*., 2009, PEREIRA *et al*., 2012, BRAGA *et al*., 2013). Três novos sistemas diferenciados de NiTi são atualmente comercializados: *ProFile Vortex* (*PV*) (*Dentsply Tulsa Dental Specialities, Tulsa, OK*), *Hyflex CM* (*HF*) (*Coltene/Whaledent, INC, Cuyahoga Falls, OH*) e *Typhoon CM (TYP)* (*Clinician's Choice Dental Products, New Miliford, CT*).

A tecnologia do fio *M-Wire*, que é usado para produzir os instrumentos *ProFile Vortex*, baseia-se num processamento termomecânico (BERENDT, 2007), que resulta na associação de duas estruturas: austenítica e martensítica (ALAPATI *et al*., 2009). O fio *M-Wire* é composto de NiTinol 508 submetido a um tratamento próprio, sob tensões específicas e tratamentos térmicos a várias temperaturas, resultando em um material que inclui martensita e Fase R. Especificamente, o fio é submetido a uma tensão (não informada pelo fabricante), enquanto ocorrem no mínimo cinco ciclos térmicos entre banhos frios (de aproximadamente 0°C a 10°C) e banhos quentes (de aproximadamente 100°C a 180°C) (BERENDT, 2007). Isto melhora a flexibilidade e a resistência à fadiga do instrumento (JOHNSON *et al*., 2008, PEIXOTO *et al*., 2010, PEREIRA *et al*., 2012) de modo a permitir um resultado mais previsível na instrumentação de canais radiculares curvos.

Os instrumentos rotatórios de NiTi *CM* são fabricados utilizando um processo térmico especial, que controla a memória do material (*Coltene-Endo* 2012). Além disso, estes instrumentos são comercializados como possuindo maior flexibilidade e melhor resistência tanto à fadiga quanto à torção devido à presença da fase martensítica (*CM Wire* 2010). Esta fase do NiTi possui algumas propriedades únicas que a fizeram ideal para muitas aplicações (DAVIS, 2000). A fase martensítica da liga NiTi apresenta notável resistência à fadiga. Instrumentos endodônticos na fase martensítica podem ser facilmente deformados, mas recuperarão suas formas no aquecimento acima das temperaturas de transformação (FIGUEIREDO *et al.* 2009, SHEN *et al*., 2013). Provavelmente esses instrumentos são submetidos a um tratamento térmico, do qual se desconhece o tempo e a temperatura utilizados, resultando na fabricação de instrumentos com memória de forma controlada. Ambos os sistemas *HyFlex* e *Typhoon* são instrumentos fabricados com tecnologia *CM*.

Até o momento, pouca informação foi disponibilizada na literatura sobre o comportamento mecânico dos instrumentos *Hyflex*, *Typhoon* e *ProFile Vortex*, portanto é necessário aprofundar o conhecimento a respeito de suas características geométricas, dimensionais, propriedades físicas, estruturais e mecânicas, no sentido de aumentar a previsibilidade e segurança no uso clínico.

Este trabalho tem como objetivo avaliar e comparar entre si as propriedades mecânicas de instrumentos *Hyflex* e *Typhoon*, fabricados com a tecnologia *CM*, com os instrumentos *ProFile Vortex*, fabricados com o fio de NiTi *M-Wire*, e com os instrumentos *ProTaper Universal (PTU) (Dentsply Maillefer, Ballaigues, SUI)* e *EndoWave (EW) (J. Morita Corporation, Osaka, JP)*, fabricados com o fio de NiTi superelástico convencional. A comparação será feita baseando-se nas características físicas, químicas e estruturais, em termos de composição química, temperaturas de transformação de fase e microdureza; características geométricas e dimensionais; resistência à fadiga e à torção em ensaios de fadiga e torção até a ruptura, respectivamente, bem como a flexibilidade. Os instrumentos *EW* e *PTU* foram escolhidos, pois possuem seção transversal semelhante aos instrumentos *HF* e *TYP*, respectivamente.

OBJETIVOS

3. OBJETIVOS

3.1. Objetivo Geral

Avaliar as propriedades físicas, químicas e estruturais e o comportamento mecânico de instrumentos *Hyflex* e *Typhoon*, que possuem memória de forma controlada (*CM*), em comparação aos instrumentos *ProFile Vortex*, fabricados com o fio de NiTi *M-Wire*, e *EndoWave* e *ProTaper* Universal, fabricados com fios de NiTi convencional.

3.2. Objetivos Específicos

Analisar as características físicas, químicas e estruturais de instrumentos rotatórios de NiTi *Hyflex*, *Typhoon*, *ProFile Vortex*, *EndoWave* e *ProTaper Universal*, em termos de composição química, temperaturas de transformação de fase e microdureza;

Avaliar as características geométricas e dimensionais de instrumentos rotatórios de NiTi *Hyflex*, *Typhoon*, *ProFile Vortex* e *EndoWave*, de mesmo diâmetro (30/0,06), e *ProTaper Universal F2*;

Avaliar a resistência à fadiga dos instrumentos rotatórios de NiTi acima citados em ensaios de fadiga flexural até a ruptura, através do número de ciclos até a fratura;

Avaliar a flexibilidade dos instrumentos rotatórios de NiTi *Hyflex*, *Typhoon*, e *EndoWave*, de mesmo diâmetro (30/0,06), e *ProTaper Universal F2* em ensaios de flexão a 45°, realizados de acordo com a norma ISO 3630-1;

Avaliar a resistência à torção dos instrumentos endodônticos de NiTi acima citados em ensaios de torção até a ruptura, realizados de acordo com a norma ISO 3630-1;

ARTIGOS

3. ARTIGOS CIENTÍFICOS

3.1 Artigo 1

Impact of Heat Treatments on the Fatigue Resistance of Different Rotary Nickel-titanium Instruments

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Keywords: Controlled memory, fatigue resistance, heat treatment, M-Wire, nickel-titanium endodontic instruments, transformation temperatures.

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Impact of Heat Treatments on the Fatigue Resistance of Different Rotary Nickel-titanium Instruments

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Abstract

Introduction: The aim of this study was to assess the influence of M-Wire (Dentsply Tulsa Dental Specialties, Tulsa, OK) and controlled memory technologies on the fatigue resistance of rotary nickel-titanium (NiTi) files by comparing files made using these 2 technologies with conventional NiTi files. **Methods:** Files with a similar cross-sectional design and diameter were chosen for the study: new 30/.06 files of the EndoWave (EW; J. Morita Corp, Osaka, Japan), HyFlex (HF; Coltene/Whaledent, Inc, Cuyahoga Falls, OH), ProFile Vortex (PV; Dentsply Tulsa Dental Specialties, Tulsa, OK), and Typhoon (TYP; Clinician's Choice Dental Products, New Milford, CT) systems together with ProTaper Universal F2 instruments (PTU F2; Dentsply Maillefer, Ballaigues, Switzerland). The compositions and transformation temperatures of the instruments were analyzed using x-ray energy-dispersive spectroscopy and differential scanning calorimetry, whereas the mean file diameter values at 3 mm from the tip (D3) were measured using image analysis software. The average number of cycles to failure was determined using a fatigue test device. **Results:** X-ray energy-dispersive spectroscopy analysis showed that, on average, all the instruments exhibited the same chemical composition, namely, 51% Ni–49% Ti. The PV, TYP, and HF files exhibited increased transformation temperatures. The PTU F2, PV, and TYP files had similar D3 values, which were less than those of the EW and HF files. The average number of cycles to failure values were 150% higher for the TYP files compared with the PV files and 390% higher for the HF files compared with the EW files. **Conclusions:** M-Wire and controlled memory technologies increase the fatigue resistance of rotary NiTi files. *(J Endod 2014;■:1–4)*

Key Words

Controlled memory, fatigue resistance, heat treatment, M-Wire, nickel-titanium endodontic instruments, transformation temperatures

Because of their mechanical properties, rotary instruments made of nickel-titanium (NiTi) alloys are commonly used in endodontic treatments to facilitate the shaping of root canals that exhibit complex anatomies (1). Despite the favorable properties of these alloys, a high risk of fracture continues to be a problem during endodontic therapy; metal fatigue represents a predominant reason for file separation (2). Flexural fatigue fractures occur as a result of repeated compressive and tensile stresses that accumulate at the point of maximum flexure when instrument rotates within a curved canal (3, 4). The strain levels attained by rotary endodontic instruments during clinical use depend on the root canal geometry and the applied loads that are concentrated on the region of maximum curvature within the root canal, which can vary with the instrument diameter (3, 5).

Increasing resistance to instrument fracture has been a focus in the advancement of rotary NiTi instrument technology. Over the past 10 years, significant improvements in instrument design, control of raw material properties, and manufacturing processes have been achieved (6, 7). An example of a recent advancement involving the improvement of raw material properties is the proprietary thermomechanical processing procedure that is applied to conventional NiTi wire (8), which led to the development of the M-Wire (MW) (Dentsply Tulsa Dental Specialties, Tulsa, OK) that is used in the ProFile Vortex (PV) and GTX instruments (Dentsply Tulsa Dental Specialties). This material exhibits a more efficient superelastic behavior with reduced generation and accumulation of lattice defects during each load-unload cycle (9), increasing the fatigue resistance of these instruments (10). Another example is controlled memory (CM) technology in which endodontic instruments are subjected to a special thermal process after being machined from conventional NiTi wire to increase their fatigue resistance (11). The HyFlex (HF; Coltene/Whaledent, Inc, Cuyahoga Falls, OH) and Typhoon (TYP; Clinician's Choice Dental Products, New Milford, CT) files were recently developed using CM technology.

Accurate information concerning the fatigue behavior of these new NiTi rotary instruments remains limited, particularly because instrument dimensions and cross-sectional designs must be considered when comparing fatigue properties. The aim of this study was to evaluate the effects of these new technologies on the physical properties and fatigue behavior of rotary NiTi instruments. Care was taken in choosing similar file dimensions and geometries. The representative technologies investigated to identify potential advantages and limitations were conventional NiTi (C-NiTi), MW, and CM.

Materials and Methods

We evaluated new 30/.06 files of the EndoWave (EW; J. Morita Corp, Osaka, Japan), HF, PV, and TYP systems together with ProTaper Universal F2 instruments (PTU F2; Dentsply Maillefer, Ballaigues, Switzerland). These instruments were chosen

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because they have similar geometric designs; EW and HF exhibit triangular cross-sections, whereas PTU F2, PV, and TYP exhibit convex triangular cross-sections. However, these instruments use the following different technologies: EW and F2 are made of C-NiTi, PV is made with MW, and HF and TYP are CM files.

X-ray energy-dispersive spectroscopy (Noran TN-M3055, Middleton, WI) was used to determine the average amounts of nickel and titanium contained within the files. Five small areas were analyzed within each type of system. The HF and TYP instruments were analyzed before and after removal of their oxide surface layer by grinding. Transformation temperatures were determined as the beginning and end of exothermic/endothermic peaks on the heating and cooling curves recorded by differential scanning calorimetry (DSC; Shimadzu DSC 60, Kyoto, Japan). Three tests were performed using the different samples of each system; in each test, the sample was heated to 80°C and then cooled to −80°C at a rate of 10°C/min.

Ten instruments of each system were photographed using a high-resolution digital camera (20D; Canon, Tokyo, Japan) to assess their dimensional characteristics based on the criteria of American National Standards Institute/American Dental Association Specification No. 101. Lines were drawn on either side of the images, and the outermost diameters at each millimeter from the tip were measured using Image Pro Plus 6.0 (Media Cybernetics, Silver Spring, MD).

The instruments (N = 10) were then fatigue tested at room temperature using a bench device with an artificial canal that was made of quenched tool steel consisting of an arch with a radius of 5 mm, an angle of curvature of 45°, and a guide cylinder 10 mm in diameter made of the same material. The device was described in detail elsewhere (12). The chosen geometry placed the area of maximum canal curvature at 3 mm from the tip of the instruments. The instruments were allowed to rotate freely until breakage inside 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 determined by measuring the fractured file using an endodontic ruler. The fracture surfaces of 3 randomly selected instruments of each system were observed using a scanning electron microscope (JSM 6360; JEOL, Tokyo, Japan) to evaluate the features associated with the failure process.

The statistical significance of the differences in the measured parameters among the different types of instruments was assessed by using 1-way analysis of variance at 95% confidence level.

Results

The results of the semiquantitative x-ray energy-dispersive spectroscopy analysis showed that, on average, all of the instruments exhibited the same chemical composition, namely, 51% Ni–49% Ti. High amounts of oxygen were initially found on the HF and TYP files because of their thick oxide surface layers. When this layer was properly ground and the analysis was repeated, the average values of Ni and Ti were the same as those of the other instruments.

The transformation temperatures determined by DSC are shown in Table 1 along with the mean diameter values at 3 mm from the tip (D3). The Ms and Mf temperatures correspond to the start and finishing of the formation of martensite during cooling, whereas As and Af represent the corresponding temperatures for the reverse transformation that occurred upon heating. The value of the material's Af temperature in relation to test or clinical use temperatures is important to define the phases present in that condition. For EW and PTU F2 files, Af temperatures were close to room temperature; in the other systems, Af was well above room temperature, with the TYP instruments exhibiting the highest Af value, indicating that a reasonable amount of martensite should be present in these instruments at the test temperature. On the other hand, the cooling curve of the HF files, in contrast to the others, revealed that those instruments first transformed to the R-phase and then to B19′ martensite, thus presenting 2 peaks, Rs and Ms (Table 1).

TABLE 1. Mean Values of Martensitic and Reverse Transformation Temperatures (standard deviations <3°C) and Diameter at 3 mm from the Tip (D3) (standard deviations <0.02 mm) of the Instruments Studied

	Transformation temperatures						
Instruments	Rs	Rf	Ms	Mf	As	Af	D3 (mm)
EW	—	—	15.3	−16.0	−7.3	23.3	0.53
HF	14.3	3.3	−22.0	−47.3	15.8	31.3	0.52
PV	—	—	36.3	24.1	29.0	43.4	0.51
TYP	—	—	22.2	4.0	16.8	43.8	0.50
PTU F2	—	—	18.8	−11.0	−3.7	26.4	0.50

Af, austenite finishing temperature; As, austenite start temperature; EW, EndoWave; HF, Hyflex; Mf, martensite finishing temperature; Ms, martensite start temperature ; PTU F2, ProTaper Universal F2 instruments; PV, ProFile Vortex; Rf, R-phase finishing temperature; Rs, R-phase start temperature; TYP, Typhoon.

Statistical analysis of the D3 values revealed that the instruments with triangular cross-sections (ie, HF and EW) exhibited similar diameters at 3 mm from the tip (P = .426) as those with convex triangular cross-sections (ie, PTU F2, PV, and TYP [P>.050]). On the other hand, the D3 values of PTU F2 and TYP were significantly smaller than those of HF and EW (P<.05). However, although they were different from EW (P = .027), the PV instruments exhibited no statistically significant difference in D3 compared with HF (P = .133).

The results of the fatigue tests are summarized in Figure 1. The Nf values exhibited statistically significant differences (P<.001) among all of the instruments, with higher values for CM instruments. The average Nf value for the HF instruments was significantly increased compared with that of all of the other systems. For all of the instruments, the average point of fracture was 3.0 mm from the tip with a standard deviation of 0.2 mm.

The fracture surfaces of fatigue-tested instruments observed by scanning electron microscopy exhibited the typical features of this fracture mode. The secondary electron images in Figure 2 exemplify the larger areas of nucleation and slow crack propagation found in the CM instruments compared with the other instruments. Fatigue striations and secondary cracks were observed within the smooth regions of the

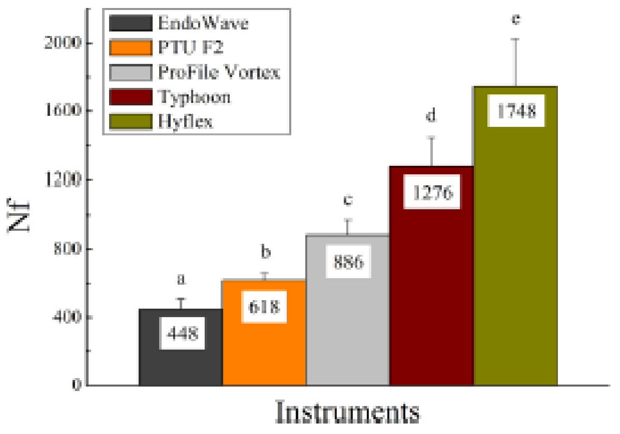

Figure 1. The mean values of Nf measured in the flexural fatigue tests. Bar values marked with different letters were statistically different (P≤.05).

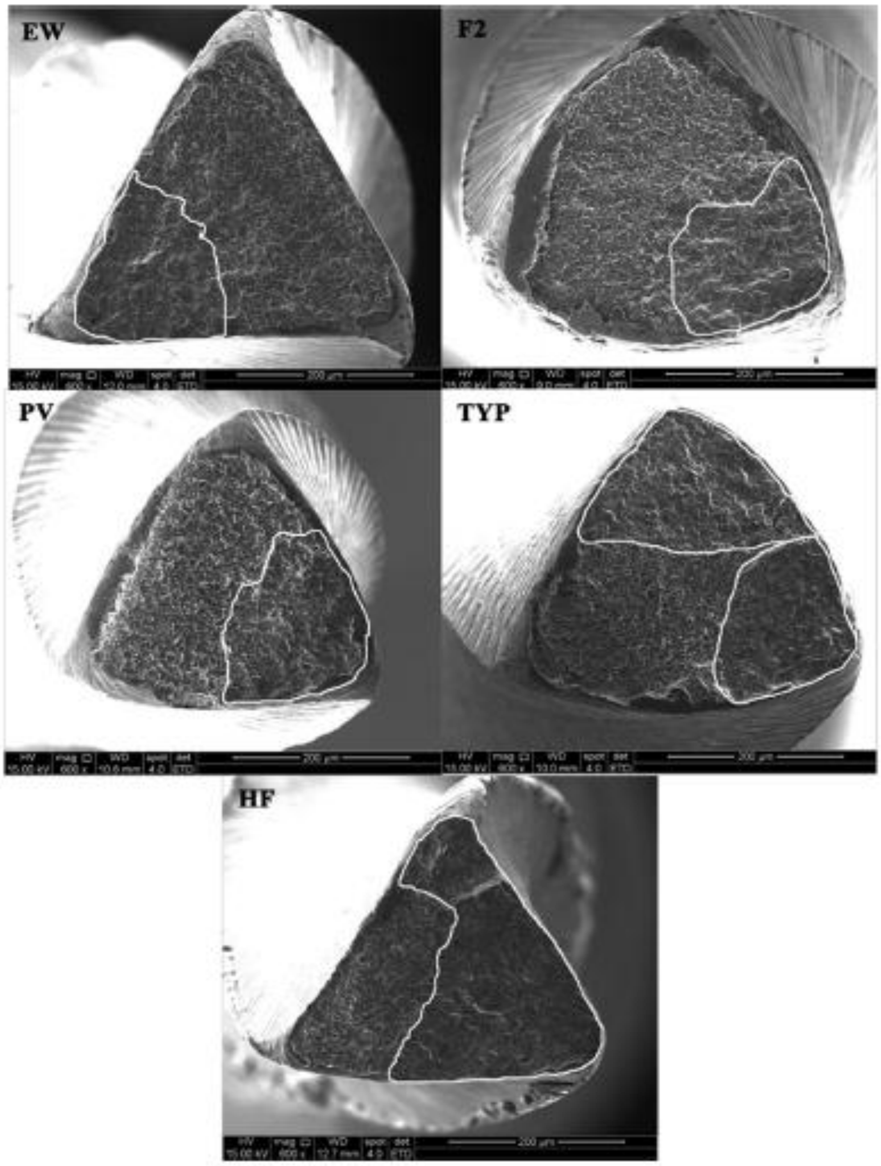

Figure 2. Secondary electron images of typical fracture surfaces of conventional NiTi, M-Wire, and CM instruments. Outlined areas correspond to the region of fatigue crack nucleation and propagation (smooth areas).

fracture surface at a higher magnification, confirming that the instruments failed because of fatigue (5).

Discussion

This study evaluated the fatigue resistance of endodontic instruments that are manufactured using conventional NiTi, MW, and CM technology, considering their structural and geometric characteristics. As with other alloys, the mechanical behavior of NiTi alloys is determined by the relative proportions and characteristics of their microstructural phases. Thermal processing is 1 of the most fundamental approaches used to adjust the Ni content of the β-phase and, consequently, the transition temperatures of NiTi alloys (11, 13). However, despite the strong influence that these parameters exert on the fatigue resistance of NiTi rotary files, information concerning the thermal treatment of raw materials and instruments currently remains limited because such heat treatments are generally proprietary and are not disclosed by manufacturers.

The semiquantitative chemical analysis of the instruments examined in this study indicated that they contain appropriate amounts of the alloying elements (51% Ni–49% Ti) to be sensitive to the low-temperature heat treatments. NiTi alloys with a small excess of Ni respond well to heat treatment at lower temperatures because coherent, submicroscopic Ti_3Ni_4 precipitates can form within the β-phase matrix (14). Because the Ti_3Ni_4 precipitates contain more Ni than Ti, their formation decreases the amount of Ni in solid solution and increases the transformation temperatures, which are sensitive to the Ni content (15).

The DSC results (Table 1) showed that the PV, HF, and TYP files exhibited Af temperatures above 30°C, whereas the NiTi instruments that were made from conventional NiTi exhibited Af temperatures of approximately room temperature. These data are consistent with previous studies, which showed that instruments made from C-NiTi alloys exhibit an austenitic phase at room temperature during clinical applications (10, 16, 17), whereas MW and CM instruments, in addition to the austenite, also contained martensite B19′ and R-phase (9, 18, 19).

The instrument diameter at the point of the maximum canal curvature has been identified as the most important factor in the control of fatigue resistance of endodontic files (3, 5, 20). The critical parameter is the maximum tensile strain amplitude, ε_T, which is determined using the expression $\varepsilon_T = D/(2R–D)$, where the canal radius is measured at the outer canal wall, as in this study. Considering that the mean diameters at 3 mm from the tip (D3) of the EW and HF, PV, and PTU F2 and TYP files were 0.52, 0.51, and 0.50 mm, respectively, a maximum tensile strain amplitude between 5.5% 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 wire.

A similar type of comparison of PTU F2, PV, and TYP instruments, which helped to determine the influence of MW, showed that the PV files exhibited a significantly higher Nf compared with PTU F2 files. A similar result was reported comparing PV files with others made of conventional NiTi alloy (21). In contrast, the fatigue resistance exhibited by the PV files was significantly lower than that of the TYP and HF files, as previously mentioned. Furthermore, although the TYP files exhibited a reduced 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.

The results of this study indicated that the NiTi instruments that were made using CM technology were approximately 150% and 390% more resistant to fatigue compared with the instruments that were made from MW and conventional NiTi wire, respectively. These data are consistent with previous studies (7, 10, 11,22–24) and with the view that the martensitic phase in near equiatomic NiTi alloys exhibits remarkable fatigue resistance (25, 26). It appears that a hybrid (austenite-plus-martensite) microstructure exhibiting a certain proportion of martensite, such as that observed in the PV, HF, and TYP files, is more likely to exhibit favorable fatigue resistance compared with a fully austenitic microstructure because of the significant number of interfaces present. These interfaces are known to cause the formation of complex arrays of secondary cracks, which can dissipate the energy required for crack propagation (27). In addition, the presence of these martensitic structures has the potential of hardening the heat-treated alloys, as recently reported (24, 28), ruling out the possibility that the treatments involved in MW and CM technologies may compromise the torsional resistance of endodontic instruments.

In summary, under the conditions of this study, it has been shown that, along with the trend of continually improving the microstructure and transformation behavior of NiTi alloys, endodontic files have been developed to present significantly increased fatigue resistance as follows: CM technology > MW > C-NiTi. These findings provide clinicians with an important understanding of the properties and differences of these materials so that they may take advantage of the latest technology.

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3.2 Artigo 2

Comparison of the flexibility and torsional behavior of instruments made with the CM technology and superelastic nickel-titanium wire

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Keywords: Controlled memory, torsional behaviour, heat treatment, flexibility, nickel-titanium endodontic instruments.

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Comparison of the flexibility and torsional behavior of instruments made with the CM technology and superelastic nickel-titanium wire

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Abstract

Aim To assess the impact of the CM technology in relation to conventional superelastic NiTi on flexibility and torsional resistance of rotary files with similar geometric and dimensional characteristics. **Methodology** New 30/.06 HyFlex (HF), Typhoon (TYP), EndoWave (EW), and ProTaper Universal F2 instruments (F2) were evaluated. Instrument diameter and pitch length at each millimeter, besides cross sectional area at 3 mm from the tip were the dimensional parameters measured. Transformation temperatures and Vickers microhardness were also determined to collaborate in a better understanding of the mechanical behavior. Flexibility and torsional strength of each instrument (n = 10) were assessed according to specification ISO 3630-1. Data were analyzed using analysis of variance ($\alpha = 0.05$). **Results** M_B values for HF instruments were significantly lower than those for EW instruments ($p = 0.000$) and TYP results were also significantly lower than those of PTU instruments ($p = 0.000$). Torsional resistance of HF and EW instruments, measured in terms of the maximum torque, was similar ($p = 0.228$) while the torsional resistance of the TYP instruments was significantly lower than that of PTU instruments ($p = 0.034$). **Conclusions** HY and TYP instruments, more flexible, would be suitable for shaping root canals with strong curvature. The comparative information provided by this research faced with the existing literature was intended to help clinicians to better understand differences in raw materials maximizing the benefits from the selection and application of NiTi rotary instruments.

Key Words: CM technology, shape memory instruments, conventional superelastic NiTi instruments, flexibility, torsional resistance

Introduction

Rotary instruments manufactured from nickel-titanium (NiTi) alloys are a major breakthrough because of their unique properties of superelasticity and shape memory effect (Walia *et al.* 1988). For the last 10 years, there have been significant enhancements in the design and control of the raw materials relative to microstructure, material properties, and manufacturing processes for NiTi endodontic instruments (Thompson, 2000; Alapati *et al.* 2009, Johnson *et al.* 2008, Shen *et al.* 2013). Recently, a new NiTi wire termed M-Wire (Sportswire LLC, Langley, OK, USA) has been developed through a proprietary thermomechanical processing procedure with improved flexibility and fatigue resistance of rotary instruments (Larsen *et al.* 2009, Peixoto *et al.* 2010, Pereira *et al.* 2012). Although these newly developed instruments have been clinically successful, manufacturers are constantly attempting to improve files. Following this trend, another recent development is the Controlled Memory (CM) technology, by means of which NiTi instruments can be made even more flexible for negotiating complex canal pathways, while minimizing the unwanted lateral forces that might transport, and straight canals (Pettiette *et al.* 2001). Instruments made using CM technology were reported to be between 300% and 800% more resistant to fatigue compared to those made of conventional superelastic NiTi wire (Shen *et al.* 2011a, Shen *et al.* 2011b, Shen *et al.* 2012, Peters *et al.* 2012, Braga *et al.* 2014).

As more advanced NiTi endodontic instruments are being developed, understanding the nature of different raw materials and post-machining heat-treatments, as well as their impact on instrument performance, has become more imperative for clinicians as they relate to instrument choice and achieving the desired outcome. Bending and torsional properties of CM instruments have been reported in a few studies showing they had an increased flexibility over those made with conventional superelastic NiTi (Testarelli *et al.* 2011, Ninan *et al.* 2013), in addition to a greater torsional strength and higher deformation before failure (Casper et al. 2011). However, these studies did not take into account the dimensional characteristics of the instruments tested.

Comparison of clinical-related properties among instruments made with different raw materials and manufacture technologies requires standardization of geometric and dimensional parameters. Thus the purpose of the present study was to evaluate the impact of the CM technology in relation to conventional superelastic NiTi on flexibility and torsional resistance of rotary files with similar geometric and dimensional characteristics.

Materials and Methods

New 30/.06 HyFlex (HF; Coltene/Whaledent, Inc., Cuyahoga Falls, OH) and Typhoon (TYP; Clinician's Choice Dental Products, New Milford, CT), representing the CM technology instruments, together with EndoWave (EW; J. Morita Corporation, Osaka, JP) and ProTaper Universal F2 instruments (F2; Dentsply Maillefer, Ballaigues, Switzerland), made with conventional SE NiTi, were evaluated. These instruments were chosen because they have similar designs: EW and HF exhibit triangular cross-sections, whereas F2 and TYP exhibit convex triangular cross-sections.

Before mechanical tests, ten instruments of each system were photographed using a high-resolution digital camera (20D; Canon, Tokyo, Japan) to assess their dimensional characteristics based on American National Standards Institute/American Dental Association Specification No. 101. Lines were drawn on the instrument images, and the pitch length was measured by using Image Pro Plus 6.0 (Media Cybernetics, Silver Spring, MD).

Other 3 instruments of each type were cut to approximately 2.7 mm from the tip using a metallographic cutter (Isomet 1000; Buehler, Lake Bluff, IL, USA). The cross-sectional surfaces were polished with sandpaper to reach 3.0 mm from the tip and then imaged under a scanning electron microscope (JSM 6360; Jeol, Tokyo, Japan) at 150x magnification. The cross-sectional areas were determined using the same software already mentioned.

Vickers microhardness measurements (Durimet 2; Leica, Wetzlar, Germany) with a 100-gram force load were performed in another 3 specimens of each type of instrument previously mounted with a metallographic resin, ground, and polished with diamond paste. Ten indentations were made on each specimen, totaling 30 measurements per instrument type.

Ten instruments of each type were submitted to bending tests according to specification ISO 3630-1. The test conditions were described in detail by Câmara et al (2009) and included fixing the instruments at 3 mm from the tip and bending them perpendicularly to the long axis by 45° with a rotation speed of 0.5 rpm. The bending moment (M_B) was automatically recorded during the shaft's movement by a load cell. After the tests, each instrument was photographed using a high-resolution digital camera (20D; Canon, Tokyo, Japan). The residual deformation at the tip of the instrument was measured by the angle formed from a line parallel to its longitudinal axis with another straight line from the tip of the instrument and the encounter with the longitudinal axis. The same software used for pitch length and cross-sectional areas performed the angle measurements.

Another group of instruments (n = 10 each) was tested in torsion until fracture based on specification ISO 3630-1 using a torsion test device described in Câmara et al (2009). The rotation speed was set clockwise to 2 rpm. The end of the shaft was clamped into a chuck

Another group of instruments (n = 10 each) was tested in torsion until fracture based on specification ISO 3630-1 using a torsion test device described in Câmara et al (2009). The rotation speed was set clockwise to 2 rpm. The end of the shaft was clamped into a chuck with brass jaws to prevent sliding and connected to the reversible geared motor of the test apparatus. Three millimeters of the instrument's tip was clamped in another chuck with brass jaws. Continuous torque recordings were provided by a specifically designed computer program.

The statistical significance of differences in the measured parameters among different types of instruments was determined using one-way analysis of variance at a 95% confidence level.

Results

Pitch length increased along the active part of EW, PTU and TYP instruments, with a steeper increase recorded in EW instruments (Fig.1). In contrast, the HF instruments showed constant pitch length. In general, EW presented larger pitch lengths than the other instruments. The mean values and standard deviations (SD) of cross-sectional area at 3 mm from the tip (A3) of the analyzed instruments are shown in Table 1. HF instruments presented mean A3 values similar to EW instruments ($p = 0.276$), while TYP presented significant smaller values of A3 than PTU instruments ($p = 0.000$).

Mean Vickers microhardness (standard deviation), expressed in Kgf/mm^2, were 316 ± 12 and 445 ± 20 for HF and EW instruments, respectively, the former being statistically softer ($p = 0.000$) than the latter. Regarding TYP and PTU groups, the results obtained were 271 ± 10 those for EW instruments (p = 0.000) and TYP results were also significantly lower than those of PTU instruments (p = 0.000). The results of residual deformation after the bending test (deformation angle) are also shown in Table 1. Contrary to what was found for EW and PTU instruments, HF and TYP files were deformed after the test, with TYP showing significantly higher mean values of deformation angle than HF instruments (p = 0.000).

The results of the torsion tests are shown in Figure 2 and indicate that the torsional resistance (Fig. 2a) of HF and EW instruments, measured in terms of the maximum torque, was similar (p = 0.228). The torsional resistance of the TYP instruments was significantly lower than that of PTU instruments (p = 0.034). Typical torque curves versus angular deflection for these instruments are shown in Figure 2b.

Discussion

Geometrical and dimensional characteristics of NiTi instruments might have a crucial effect on their flexibility and torsional resistance (Vieira *et al.* 2009; Braga *et al.* 2013). Therefore, instruments with identical geometric features but different raw materials were used to eliminate this influence in the present study.

In general, a smaller pitch length tend to provide greater torsional resistance to the instrument and reduced cutting efficiency, whereas a longer pitch length permits higher cutting efficiency and more efficient debris removal, in addiction to preventing the screw-in effect (Mounce 2003, Diemer & Calas 2004). HF instruments showed significantly smaller bending moment than EW files even though their pitch lengths were smaller. Considering that parameter, higher mean torque values would be expected to be found in HF instruments when efficiency and more efficient debris removal, in addiction to preventing the screw-in effect (Mounce 2003, Diemer & Calas 2004). HF instruments showed significantly smaller bending moment than EW files even though their pitch lengths were smaller. Considering that parameter, higher mean torque values would be expected to be found in HF instruments when compared to EW files, but that could not be observed and can be probably explained due to its manufacturing technology.

When the mechanical properties of CM and SE NiTi instruments of similar diameters and cross-sectional areas were compared, such as HF and EW instruments, the influence of the manufacturing method could be better appreciated. No significant differences in D3 (Braga *et al.* 2014) and A3 were found for HF and EW instruments, therefore these parameters had no influence on the significantly higher flexibility of the HF files. TYP files had significantly smaller A3 compared to PTU, which may have influenced the lower bending moment displayed by the first, but as this difference was considerably large (Table 1), the CM technology applied to TYP instruments might have been responsible for that.

It is important to understand the differences in the raw materials and processing technology to make the most of what their differential properties have to offer. Instruments in the martensite phase can easily be deformed recovering their shape on heating above the transformation temperatures. Conventional SE NiTi instruments have an austenitic structure (Brantley *et al.* 2002, Shen *et al.* 2013), whereas NiTi instruments with thermal processing present high austenite transformation temperatures being essentially in the martensitic condition at room temperature (Alapati *et al.* 2009, Shen *et al.* 2011a, Ye *et al.* 2012, Zhou *et al.* 2012, Braga *et al.* 2014). In this research CM technology instruments showed significantly lower hardness compared with those produced using conventional SE NiTi alloy. HF instruments presented a low hardness value (316 VHN) with similar results found by Zinelis *et al.* (2010), and Shen *et al.* (2013). The thermal treatments may be responsible for the difference in microhardness between the TYP, HF, PTU and EW instruments, being the first significantly softer compared to the other groups and the latter the hardest among the groups. Relating Microhardness values with DSC results it was possible to observe that in the HF cooling curve, unlike the others instruments analyzed, two transformation peaks were verified suggesting a first transformation to the R-phase and then a second to B19' martensite. The presence of R-phase probably would be due to precipitation of Ti_3Ni_4, resulting in matrix hardening (Braga *et al.* 2014). Therefore, microhardness tests results indicate a significantly superior hardness to EW, compared with the HF instruments, opposite to the expected of an R-phase NiTi alloy. This was possibly due to the heat treatment to which HF instruments are subjected to during the manufacturing process, which may soften the material. This means that the dislocations created during the low temperature thermal treatment to manufacture the wires may cause strain hardening of the austenite matrix, rearrange themselves into dislocation cells, decreasing the material's hardness (Read-Hill *et al.* 2008).

CM instruments demonstrated greater flexibility than other tested instruments (Tab. 1). Despite the fact that the flexibility is influenced by instrument's design, such improvement is probably related to their proprietary manufacturing process. Similar results have been reported by other authors (Testarelli *et al.* 2011; Zhou *et al.* 2012; Ninan *et al.* 2013) who found CM instruments more flexible than other NiTi rotary instruments. However, D3 and A3 values were not considered in these researches and may have influenced the results. This superior flexibility of CM instruments is consistent with a previous study wherein tensile results showed that CM Wires had a higher maximum strain before fracture and lower critical stress values than did conventional superelastic NiTi Wire. This behavior may be attributed to stress-induced R-phase and B19′ martensite (Pereira *et al.* 2012, Braga *et al.* 2013, 2014), thus reducing the apparent elastic modulus of austenite and the stress at the transformation plateau. The deformation angle measurement is related to the bending moment, with TYP and HF instruments presenting the highest deformation angle thereby being the most flexible instruments.

There is a strong relationship between maximum torque and instrument diameter (Braga *et al.* 2013) and/or cross-sectional area (Melo *et al.* 2008). In the present study, the fact of the instrument pairs EW; HF, and PTU; TYP presenting similar cross section design, area and diameter at 3 mm from the tip (Braga *et al.* 2014) (except A3 for PTU and TYP) allows not consider these variables, emphasizing the manufacturing process. According to Shen *et al.* (2013) it is expected that thermomechanically treated NiTi instruments maintain the same torsional properties as conventional SE NiTi instruments. The torsional resistance of 10 Series files (CM Wire), Twisted File and ProFile Vortex before and after autoclaving was studied by Casper *et al.* (2011) showing that 10 Series Files (CM Wire) and ProFile Vortex displayed significantly greater resistance to torsional load than Twisted Files. Other study found variability in maximum torque values among the shape memory instruments, sometimes exhibiting the highest or lowest torque depending on brand, size and taper (Ninan *et al.* 2013). Peters *et al.* (2012) showed that HF instruments presented similar torsional resistance as instruments made from SE NiTi. A recent study showed that TYP instruments without cyclic preloading had similar torque values than instruments made of conventional NiTi (Campbell *et al.* 2014). The conclusions of Peters *et al.* (2012), Shen *et al.* (2013), and Campbell *et al.* (2014) are consistent with the results presented for EW and HF. In the case of PTU herein analyzed, their higher torsional resistance in relation to TYP instruments could be attributed to their significantly higher cross-sectional area at 3 mm from the tip.

It is known that the critical stress for martensite reorientation of CM Wires is in the range of 128-251 MPa at room temperature being lower than the critical stress for stress-induced martensite transformation of the SE wires (490-582 MPa) (Zhou *et al.* 2012). Also, the ultimate tensile strength for the CM Wires (about 1094 MPa) is lower than that of SE Wires (about 1415 MPa). However, the maximum strain of CM Wires before fracture is much higher than that of the SE wires (Shen *et al.* 2013). Therefore, it would be clinically relevant the indication of HF and TYP instruments, more flexible, for shaping root canals with strong curvature. The comparative information provided by this research faced with the existing literature was intended to help clinicians to better understand differences in raw materials maximizing the benefits from the selection and application of NiTi rotary instruments for root canal treatment.

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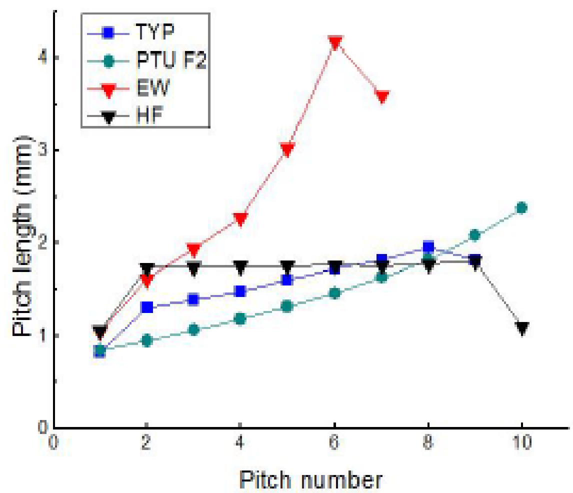
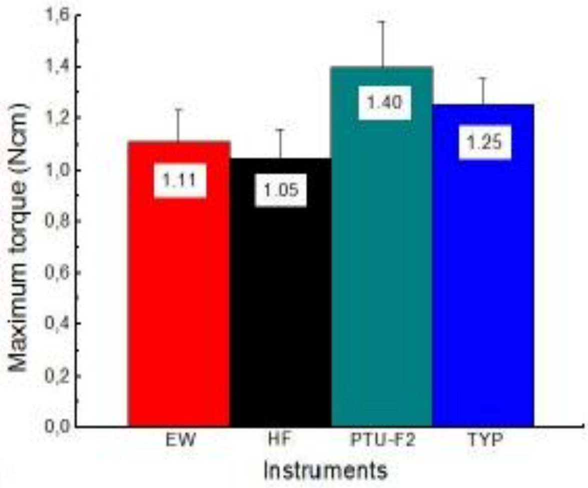
Figure Legends

Figure 1 Mean values of pitch length (mm) at each millimeter from the tip.

Figure 2 Mean values of maximum torque (a) and typical torque curves versus angular deflection (b) for the analyzed instruments.

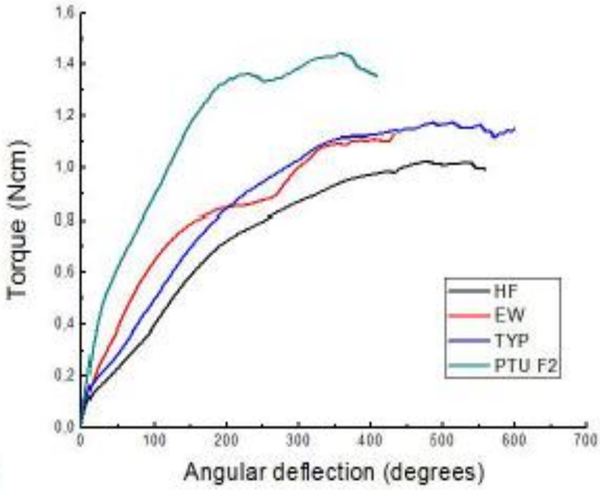
Table 1 Mean values and standard deviations (SD) of cross-sectional area at 3 mm from the tip (A3), bending moment (M_B) and deformation angle (DA) after the bending test of the instruments analyzed.





Maximum torque (Ncm)
1,6
1,4
1,2
1,0
0,8
0,6
0,4
0,2
0,0
1.11
1.05
1.40
1.25
EW
HF
PTU-F2
TYP
Instruments

(a)



Torque (Ncm)
1,6
1,4
1,2
1,0
0,8
0,6
0,4
0,2
0,0
0
100
200
300
400
500
600
700
Angular deflection (degrees)
HF
EW
TYP
PTU F2

(b)

Table 1 – Mean values and standard deviations (SD) of cross-sectional area at 3 mm from the tip (A3), bending moment (M_B) and deformation angle (DA) after the bending test of the instruments analyzed.

Instruments	A3 (mm^2)	M_B (N.cm)	DA (°)
EW	0,082 (0,00)	0,567 (0,04)	-
HF	0,084 (0,01)	0,327 (0,03)	24,84 (1,36)
PTU (F2)	0,121 (0,00)	0,974 (0,04)	-
TYP	0,107 (0,01)	0,315 (0,03)	28,06 (1,40)

CONSIDERAÇÕES FINAIS

4. CONSIDERAÇÕES FINAIS

Os resultados obtidos neste trabalho dão suporte às seguintes considerações:

A análise química semiquantitativa dos instrumentos estudados indicou que as ligas NiTi utilizadas em suas confecções possuem composições químicas similares, com uma razão aproximadamente equiatômica entre os elementos Ni e Ti. Os valores encontrados são compatíveis com os diversos sistemas rotatórios disponíveis no mercado. Em geral, os instrumentos apresentaram um pequeno excesso de Ni, podendo responder positivamente a tratamentos termomecânicos em baixas temperaturas, com possibilidade de melhoria nas propriedades de superelasticidade e efeito memória de forma.

As temperaturas de transformação dos instrumentos *EW* e *PTU*, obtidas por meio da calorimetria exploratória diferencial (DSC), confirmou que a liga utilizada na fabricação desses é austenítica à temperatura ambiente. Já os instrumentos *PV*, *TYP* e *HF* apresentaram um aumento relevante nas temperaturas de transformação, sugerindo a presença das fases R e B19’ coexistindo com a fase austenítica. Neste trabalho, dois picos de transformação da austenita para martensita foram detectados no resfriamento, para os instrumentos *HF*, sugerindo a presença da Fase R. É possível que os maiores valores das temperaturas de transformação dos instrumentos *PV*, *TYP* e *HF* sejam devido à precipitação de Ti_3Ni_4, nos estágios iniciais do recozimento a baixas temperaturas, em que os átomos de Ni se congregam nos precipitados e os átomos de Ti se mantém na matriz da fase β, com consequente redução no conteúdo de Ni na mesma.

É possível observar que a média dos valores de microdureza Vickers apresentados pelos instrumentos *PV*, *EW* e *PTU* foram mais elevados que aqueles obtidos pelos instrumentos *CM*. Uma possível explicação para este resultado são os tratamentos térmicos a que os instrumentos *HF* e *TYP* são submetidos durante a sua fabricação, que podem amaciar o material. Ao se comparar os valores médios de microdureza dos instrumentos *HF* e *TYP*, observa-se que o primeiro apresenta um valor mais elevado, devido, provavelmente, à presença de dois picos encontrados na transformação Martensítica e a formação de precipitados de Ti_3Ni_4.

Em relação à análise dimensional, os instrumentos apresentaram pequena variação entre o D3 nominal e o real. A comparação dos valores de diâmetro a 3 mm da ponta indicou valores semelhantes ($p > 0,05$) para os seguintes instrumentos: *HF* e *EW, PV* e *PTU* e para o trio *PV*, *PTU* e *TYP.*

A análise das imagens dos instrumentos mostrou seções transversais de forma triangular para os instrumentos *HF* e *EW*, e ausência de planos radiais. Por outro lado, nos instrumentos *TYP*, *PV* e *PTU* a imagem da seção transversal é triangular convexa sem planos radiais. Observou-se que os instrumentos *TYP* e *PTU*, apesar de possuírem diâmetros similares, apresentaram áreas a 3 mm da ponta diferentes.

Os ensaios de fadiga indicaram que os instrumentos *CM* (*HF* e *TYP*) apresentaram vida em fadiga significativamente superior aos instrumentos *PV*, fabricados com o fio de NiTi *M-Wire*, e estes foram significativamente superiores aos sistemas *PTU* e *EW*, fabricados com liga NiTi convencional. Pode ser sugerido que os processos térmicos e termomecânicos empregados nos instrumentos *CM* e *M-Wire*, respectivamente, possam ter influenciado o comportamento mecânico, aumentando a resistência à fadiga destes instrumentos.

Os ensaios de flexão a 45° revelaram que os instrumentos *CM* foram significativamente mais flexíveis que os instrumentos *PTU* e *EW*, fabricados com a liga NiTi convencional. É possível que os tratamentos térmicos a que são submetidos os instrumentos *CM* tenham produzido um amaciamento na liga, o que é indicado pela maior flexibilidade e menor microdureza apresentada por esses instrumentos. Apesar de os instrumentos *EW* apresentarem comprimentos de *pitch* maiores do que aqueles apresentados pelos instrumentos de *HF*, o que refletiria em maior flexibilidade, o momento de dobramento encontrado nestes foi significativamente menor do que o encontrado nos EW, provavelmente o resultado de uma maior flexibilidade da tecnologia *CM*.

Em relação à resistência torcional, a aplicação de tratamentos térmicos pareceu não influenciar o comportamento mecânico dos instrumentos *CM* (*HF e TYP*) estudados, visto que os instrumentos *EW* e *HF* apresentaram valores médios de torque máximo semelhantes. Já os instrumentos *PTU* e *TYP* apresentaram valores diferentes, com o primeiro sendo significativamente mais resistente à torção do que o segundo, provavelmente devido a sua maior área a 3 mm da ponta.

Retomando o comprimento de *pitch*, seria de se esperar que os instrumentos *EW* apresentassem valores médios de torque máximo inferiores aos dos instrumentos *HF*, o que não foi confirmado pelo ensaio de torção até a ruptura. Mais uma vez, houve prevalência do tratamento térmico, a que foram submetidos os instrumentos CM, às características dimensionais e geométricas.

Em resumo, nas condições do presente estudo, os instrumentos *CM* parecem ser, dentre os instrumentos rotatórios de NiTi avaliados, a melhor opção na formatação de canais radiculares curvos, uma vez que apresentaram um aumento significativo na flexibilidade e na resistência à fadiga. Pode ser concluído que as novas tecnologias, com abordagens metalúrgicas nas ligas NiTi, têm sido bem sucedidas na obtenção de instrumentos mais flexíveis e resistentes como se segue: tecnologia *CM* > *M-Wire* > NiTi convencional. Esses resultados permitem aos clínicos uma importante compreensão das propriedades estruturais e mecânicas dessas ligas, de forma a permitir a opção mais segura e previsível em cada situação clínica.

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