UNIVERSIDADE FEDERAL DE MINAS GERAIS
Faculdade de Odontologia

“Efeito do tratamento térmico nas propriedades físicas e mecânicas de fios e instrumentos endodônticos de NiTi”

Érika Sales Joviano Pereira

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.

Orientador(a): Prof^{a}. Dr^{a}. Maria Guiomar de Azevedo Bahia.
Co-Orientador: Prof. Dr. Vicente Tadeu Lopes Buono.
Co-Orientador: Prof. Dr. Ove Andreas Peters.

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PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA

FOLHA DE APROVAÇÃO

Efeito do tratamento térmico nas propriedades físicas e mecânicas de fios e instrumentos endodônticos de NiTi

ÉRIKA SALES JOVIANO PEREIRA

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

Aprovada em 21 de junho de 2013, pela banca constituída pelos membros:

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UNIVERSIDADE FEDERAL DE MINAS GERAIS

PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA

ATA DA DEFESA DE TESE DA ALUNA
ÉRIKA SALES JOVIANO PEREIRA

Realizou-se, no dia 21 de junho de 2013, às 14:00 horas, Faculdade de Odontologia, da Universidade Federal de Minas Gerais, a defesa de tese, intitulada *Efeito do tratamento térmico nas propriedades físicas e mecânicas de fios e instrumentos endodônticos de NiTi* , apresentada por ÉRIKA SALES JOVIANO PEREIRA, graduada no curso de ODONTOLOGIA, como requisito parcial para a obtenção do grau de Doutor em ODONTOLOGIA, à seguinte Comissão Examinadora: Prof(a). Maria Guiomar de Azevedo Bahia - Orientador (UFMG), Prof(a). Vicente Tadeu Lopes Buono (UFMG), Prof(a). José Antonio da Cunha Ponciano Gomes (UFRJ), Prof(a). Ana Cristina Rodrigues Antunes de Souza (Centro Universitário Newton Paiva), Prof(a). Renata de Castro Martins (UFMG), Prof(a). Ana Cecília Diniz Viana Castro (UFMG).

A Comissão considerou a tese:

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( ) Aprovada condicionalmente, sujeita a alterações, conforme folha de modificações, anexa
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Finalizados os trabalhos, lavrei a presente ata que, lida e aprovada, vai assinada por mim e pelos membros da Comissão.
Belo Horizonte, 21 de junho de 2013.

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Dedicatória

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“Quando uma criatura humana desperta para um grande sonho e sobre
ele lança toda a força de sua alma, todo o universo conspira a seu favor”
Johann Goethe

SUMÁRIO

RESUMO

O objetivo deste trabalho foi comparar as propriedades químicas, físicas e mecânicas de fios de níquel-titânio convencionais (CW) e termomecanicamente tratados (MW), utilizados na fabricação de instrumentos endodônticos por dois fornecedores diferentes (Dentsply Tulsa Dental Specialties, Tulsa - OK, USA e Dentsply Maillefer - Ballaigues, Switzerland). Foram ainda avaliados o torque e a força apical requerida pelos novos instrumentos endodônticos ProTaper Next™. A composição química foi determinada por espectroscopia de energia dispersiva de raios-X, a constituição de fase por difratometria de raios-X e as temperaturas de transformação por calorimetria exploratória diferencial. Ensaios de tração em carga e descarga e até a ruptura, flexão em três pontos, flexão rotativa/fadiga e medidas de microdureza Vickers foram realizados para avaliar o comportamento mecânico dos fios. As superfícies de fratura foram observadas por microscopia eletrônica de varredura e as microestruturas não deformadas por microscopia eletrônica de transmissão. Análise de variância, com um nível de confiança de 95%, foi aplicada aos resultados obtidos. Os dois fios apresentaram aproximadamente a mesma composição química, com razão equiatômica de 1:1. As fases austenita (fase β), martensita B19' e fase R foram encontradas no fio MW, em concordância com as temperaturas de transformação mais elevadas encontradas neste espécime. Por outro lado, o fio CW apresentou somente austenita e temperaturas de transformação abaixo da temperatura ambiente. As médias dos valores de microdureza Vickers foram semelhantes para MW e CW ($p = 0,91$) do primeiro fornecedor. Entretanto, os fios MW fornecidos pela Dentsply Maillefer apresentaram maiores valores de microdureza Vickers que o fio CW. A análise das curvas de carga e descarga apresenta menores valores de tensão e maior uniformidade no patamar de transformação para o fio MW, que também apresentou menores histerese de tensão e módulo de elasticidade aparente. Os ensaios de tração até a ruptura resultaram em limite de resistência e alongamento total maiores para o fio MW. O fio MW apresentou flexibilidade e resistência em fadiga significativamente maiores que o CW. Em geral, foi observado que o tratamento termomecânico a que foi submetido o fio MW resultou em melhoria das propriedades físicas e mecânicas. A avaliação do torque e força apical de instrumentos ProTaper Next™ (PTN), fabricados com fio MW,

durante a preparação de canais artificiais, apresentou maiores valores de torque requeridos pelos instrumentos X2. As maiores forças positivas foram obtidas com os instrumentos X5 e os maiores picos de força negativa foram alcançados pelos instrumentos X1 e X2. A utilização de PTN a 350 rotações por minuto e quatro movimentos de inserção resultou em menores níveis de torque bem como das forças positiva e negativa.

ABSTRACT

Effect of heat treatment on mechanical and physical properties of NiTi wires and endodontic instruments.

The aim of this work was to compare chemical, physical, and mechanical properties of conventional NiTi wire (CW) and thermomechanically treated (MW) used to the manufacture of endodontic instruments from two different companies (Dentsply Tulsa Dental Specialties, Tulsa - OK, USA and Dentsply Malleifer - Ballaigues, Switzerland). Torque and apical force required for novel ProTaper Next™ endodontic instruments were also evaluated. Chemical composition was determined by energy-dispersive X-ray spectroscopy, phase constitution by X-ray diffraction and transformation temperatures by differential scanning calorimetry. Tensile loading/ unloading, three-point bending, rotating-bending fatigue tests and Vickers microhardness measurements were performed to assess the wires mechanical behavior. Fracture surfaces were observed by scanning electron microscopy and the non-deformed microstructures by transmission electron microscopy. Analysis of variance with 95% of confidence level was applied to the results. The two wires showed approximately the same chemical composition, close to the 1:1 atomic ratio. The phases austenite (β phase), B19' martensite and the R-phase were found in MW in agreement with the higher transformation temperatures found in this specimen. On the other hand, CW wire showed only austenite and transformation temperatures below room temperature. Average Vickers microhardness values were similar for MW and CW ($p = 0.91$) of the first manufacturer. However, MW wires from Dentsply Maillefer presented higher Vickers microhardness values than CW wire. The analysis of the tensile load-unload curves showed lower stress and greater uniformity in the transformation plateau for MW wire, which also showed the smallest stress hysteresis and apparent elastic modulus. The tensile tests to failure resulted in higher ultimate tensile stress and total elongation for MW wire. The wire MW presented flexibility and fatigue resistance higher than CW, In general, it was observed that the termomechanical treatment to which MW was subjected to resulted in improved physical and mechanical properties. The evaluation of torque and apical force during the shaping of artificial canals for ProTaper Next™ instruments (PTN) manufactured with MW showed that X2 instrument required higher torque values. The highest positive forces were obtained with X5 instruments and the highest

peaks of negative force were reached by the instruments X1 and X2. The use of PTN under 350 revolutions per minute and four insertion movements resulted in lower levels of torque as well as apical positive force and negative force.

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. A geometria de seção transversal e conicidades diferentes, assim como, o modo de ação dos instrumentos de NiTi acionados a motor, criaram uma nova categoria de instrumentos endodônticos. Estes 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 da aplicação de tensão. Tais 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, ou seja, assim que o instrumento é removido do interior do canal, a transformação reversa ocorre restaurando a forma original do instrumento (THOMPSON, 2000).

Vários campos de aplicações utilizam a liga NiTi como indústria naval, aeronáutica, nuclear, eletrônica, além das áreas médica e odontológica. No entanto, no que se refere à sua produção, é uma liga difícil de ser fabricada e processada tanto do ponto de vista de fusão, como também da conformação mecânica, sendo as propriedades de EMF e SE altamente dependentes da composição e dos tratamentos termomecânicos recebidos pelo material (DUERIG et al., 1999).

É possível variar a composição 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 da 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). Verifica-se 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 se formar 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 termomecânicos 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 envelhecimento 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; KHALIL-ALLAFI *et al*., 2002).

Nas pesquisas odontológicas da área de Endodontia, ZINELIS *et al*. (2007) mostraram que o tratamento termomecânico por 30 minutos nas temperaturas de 430 e 440°C promoveu aumento da resistência à fadiga em instrumentos rotatórios de NiTi. JOHNSON *et al*. (2008) foram os primeiros a estudar, por comparação, instrumentos endodônticos com mesma geometria, mas produzidos por três diferentes tipos de liga NiTi: Nitinol SE 508 (Nitinol devices and componets Inc); M-Wire NiTi (Dentsply Tulsa Dental Specialties) e Nitinol SE 508 (EuroFlex Gmbh). A nova variante era composta de 508 Nitinol submetida a tratamentos termomecânicos a várias temperaturas, os quais resultariam em um material contendo porções de martensita B19’ e fase R, enquanto a superelasticidade seria mantida. Foi constatado que instrumentos fabricados com o fio MW apresentaram maior resistência à fadiga, enquanto a resistência à torção foi mantida semelhante entre os grupos. Outros autores encontraram maior resistência tanto à torção quanto à fadiga para o fio MW quando comparado ao NiTi convencional (ALAPATI *et al*., 2009). Posteriormente, estudos adicionais verificaram maior resistência à fadiga em instrumentos GT-X, fabricados com M-Wire, comparados aos instrumentos GT, fabricados a partir de fios de NiTi convencional (GAO *et al*., 2010; PEIXOTO *et al*., 2010; AL-HADLAQ *et al*., 2010). Recentemente, os resultados da avaliação da vida em fadiga de diferentes instrumentos mostraram resistência à fadiga e flexibilidade significativamente superiores para Vortex Blue, seguidos pelos fabricados com M-Wire, NiTi convencional e, finalmente, aço inoxidável (GAO *et al*., 2012). YE & GAO (2012) caracterizaram mudanças microestruturais do fio MW, estudando o processo de fadiga sob amplitude de deformação de 4%. Os resultados sugeriram que os instrumentos endodônticos fabricados com o M-Wire, provavelmente, apresentarão maior resistência à fadiga que aqueles fabricados com o fio de NiTi superelástico convencional, devido à sua microestrutura martensítica nano-cristalina. Por outro lado, LOPES *et al*. (2013) estudando as propriedades mecânicas de instrumentos fabricados com NiTi convencional (K3 e Revo-S SU), fio MW (ProFile Vortex) e liga NiTi em fase R (K3XF), mostraram que os instrumentos K3XF apresentaram o melhor desempenho em termos de flexibilidade, deflexão angular até fratura e resistência à fadiga.

Portanto, muitas investigações têm sido realizadas em fios utilizados na fabricação de instrumentos endodônticos de NiTi que receberam tratamentos termomecânicos. O desenvolvimento desses instrumentos representa a tentativa dos fabricantes em melhorar as propriedades mecânicas e resistência à fratura dos mesmos. Entretanto, a maioria das empresas não especifica ou define o tratamento termomecânico exato empregado durante a fabricação de tais fios. Assim, novos estudos são necessários para analisar as características metalúrgicas, bem como as propriedades mecânicas de fios de NiTi que receberam tratamento termomecânico diferenciado comparados aos fios de NiTi convencional. Neste estudo, pretendeu-se avaliar, por meio de análise das características químicas e físicas, bem como das propriedades mecânicas, o efeito do tratamento termomecânico realizado no fio MW, em comparação ao fio de NiTi convencional, utilizados na fabricação de instrumentos endodônticos.

OBJETIVOS

2. OBJETIVOS

2.1. Objetivo geral

Avaliar as características químicas, físicas e o comportamento mecânico de fios de NiTi convencional comparados a fios que receberam tratamento termomecânico diferenciado, empregados na fabricação de instrumentos endodônticos de NiTi, além de analisar o comportamento relativo a torque e força apical de instrumentos endodônticos ProTaper Next™.

2.2. Objetivos específicos

- Analisar as características químicas e físicas, através da composição química, fases presentes e temperaturas de transformação de fios de NiTi convencional (CW) e tratados termomecanicamente, M-Wire, (MW), utilizados na fabricação de instrumentos endodônticos;

- Avaliar o comportamento de fios de NiTi (CW e MW), em ensaios de tração até 6% de alongamento e até a ruptura;

- Estudar a flexibilidade dos fios de NiTi (CW e MW), por meio de ensaios de dobramento em três pontos, determinação da microdureza Vickers e da resistência à fadiga, através de ensaios de flexão rotativa;

- Examinar por meio de microscopia eletrônica de varredura as superfícies de fratura e por microscopia eletrônica de transmissão as microestruturas não deformadas de espécimes de CW e MW utilizados neste trabalho;

- Estudar o comportamento dos novos instrumentos endodônticos ProTaper Next™ relacionando torque e força apical, positiva e negativa, à velocidade e número de inserções do instrumento para se atingir o comprimento de trabalho durante a instrumentação de canais artificiais.

ARTIGOS

3. ARTIGOS CIENTÍFICOS

3.1 Artigo 1

PHYSICAL AND MECHANICAL PROPERTIES OF A THERMOMECHANICALLY TREATED NITI WIRE USED IN THE MANUFACTURE OF ROTARY ENDODONTIC INSTRUMENTS

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Keywords: endodontic instruments, fatigue, mechanical properties, M-Wire, nickel–titanium, thermomechanical treatment, transformation temperatures.

Running title: **MECHANICAL PROPERTIES OF A THERMOMECHANICALLY TREATED NITI WIRE**

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Physical and mechanical properties of a thermomechanically treated NiTi wire used in the manufacture of rotary endodontic instruments

E. S. J. Pereira[1], I. F. C. Peixoto[1], A. C. D. Viana[1], I. I. Oliveira[2], B. M. Gonzalez[2], V. T. L. Buono[2] & M. G. A. Bahia[1]

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Abstract

Pereira ESJ, Peixoto IFC, Viana ACD, Oliveira II, Gonzalez BM, Buono VTL, Bahia MGA. Physical and mechanical properties of a thermomechanically treated NiTi wire used in the manufacture of rotary endodontic instruments. *International Endodontic Journal*, **45**, 469–474, 2012.

Aim To compare physical and mechanical properties of one conventional and one thermomechanically treated nickel–titanium (NiTi) wire used to manufacture rotary endodontic instruments.

Methodology Two NiTi wires 1.0 mm in diameter were characterized; one of them, C-wire (CW), was processed in the conventional manner, and the other, termed M-Wire (MW), received an additional heat treatment according to the manufacturer. Chemical composition was determined by energy-dispersive X-ray spectroscopy, phase constitution by XRD and the transformation temperatures by DSC. Tensile loading/unloading tests and Vickers microhardness measurements were performed to assess the mechanical behaviour. Data were analysed using analysis of variance ($\alpha = 0.05$).

Results The two wires showed approximately the same chemical composition, close to the 1 : 1 atomic ratio, and the β-phase was the predominant phase present. B19′ martensite and the R-phase were found in MW, in agreement with the higher transformation temperatures found in this wire compared with CW, whose transformation temperatures were below room temperature. Average Vickers microhardness values were similar for MW and CW ($P = 0.91$). The stress at the transformation plateau in the tensile load–unload curves was lower and more uniform in the M-Wire, which also showed the smallest stress hysteresis and apparent elastic modulus.

Conclusions The M-Wire had physical and mechanical properties that can render endodontic instruments more flexible and fatigue resistant than those made with conventionally processed NiTi wires.

Keywords: endodontic instruments, fatigue, mechanical properties, M-Wire, nickel–titanium, thermomechanical treatment, transformation temperatures.

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Introduction

Nickel–titanium (NiTi) alloys allowed the development of rotary endodontic instruments because of their superelasticity and low elastic modulus, which improved several features of chemo-mechanical root canal preparation. Superelasticity is associated with the occurrence of a martensitic phase transformation of the alloy upon the application of a certain amount of stress to the austenite phase and the spontaneous reversion of the stress-induced martensite when the stress is released, causing the material to recover its original shape (Otsuka & Wayman 1998, Thompson 2000). The superelasticity of NiTi alloys can be improved by

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using special thermomechanical treatments. The mechanism for this improvement is to suppress slip during stress-induced martensitic transformation by raising the critical stress for slip (Miyazaki *et al.* 1982). Two mechanisms are available to raise the critical stress for slip in Ni-rich NiTi alloys: (i) precipitation hardening and (ii) hardening because of a high density of thermally rearranged dislocations (Miyazaki *et al.* 1982, Saburi *et al.* 1982).

Despite the advantages of the rotary NiTi technique, unexpected instrument fracture is not uncommon and represents a major concern in its clinical use. Besides failure by torsional overload (Sattapan *et al.* 2000), metal fatigue has been identified as an important reason for the fracture of endodontic instruments (Pruett *et al.* 1997, Bahia & Buono 2005, Al-Hadlaq *et al.* 2010). Attempting to improve instrument performance, manufacturers concentrated on changing the geometric and dimensional characteristics, such as tip sizing, taper, cross-section design, helical angle and pitch length (Al-Sudani & Al-Shahrani 2006, Schäfer & Oitzinger 2008, Viana *et al.* 2010). More recently, a proprietary thermomechanical procedure has been applied to nitinol with the objective of producing superelastic NiTi wires containing martensite, which can be used to produce endodontic instruments with improved fatigue resistance (Berendt 2007). This wire has been termed M-Wire NiTi (Dentsply Tulsa Dental Specialties), and subsequent studies suggested that commercial instruments made with M-Wire NiTi exhibited enhanced flexibility and fatigue resistance (Johnson *et al.* 2008, Kell *et al.* 2009, Larsen *et al.* 2009, Al-Hadlaq *et al.* 2010, Gao *et al.* 2010, Peixoto *et al.* 2010).

Typical processing of superelastic NiTi-based wires includes vacuum casting of an ingot, hot forging, rolling and cold drawing followed by appropriate heat treating (Thompson 2000). There are reports in the literature suggesting that the transformation temperatures of NiTi instruments can influence their flexibility (Miyai *et al.* 2006, Hayashi *et al.* 2007). Other authors claim that heat treating these instruments can improve their flexibility (Yahata *et al.* 2009, Ebihara *et al.* 2011). Although the exact thermomechanical history of M-Wire is proprietary, the mechanism indicated as responsible for its properties is somewhat different than what could result from a simple heat treatment (Berendt 2007).

In this work, the physical and mechanical properties of M-Wire were assessed and compared with those of a conventional wire used for manufacturing rotary NiTi instruments, in an attempt to provide a better understanding of this new trend in the development of endodontic instruments. The null hypothesis was that M-Wire had the same physical and mechanical properties as a conventional NiTi wire.

Materials and methods

The conventional NiTi wire used for the experiments (CW) was provided by Tulsa (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA) and was processed in a conventional way by drawing and annealing at low temperature. The other wire, M-Wire NiTi (MW), was also provided by Tulsa and is trained by thermally cycling at low temperatures under strain (Berendt 2007). Both types of wires were 1.0 mm in diameter.

X-ray energy-dispersive spectroscopy (EDX) with a Noran TN-M3055 spectrometer (Noran, Middleton, WI, USA) was used to determine the average amounts of the nickel and titanium in the wires. Ten small areas were analysed in each type of wire. To identify the crystallographic phases present, ten segments of each material that were 12 mm in length were glued side by side, forming a specimen of 12 × 12 mm in area. These specimens were analysed by X-ray diffraction (XRD) with a PANalytical PW1710 diffractometer (PANalytical, Almelo, the Netherlands) using Cu-Kα radiation at 40 kV and 30 mA and a graphite monochromator. Before characterization by EDX and XRD, oxide layers in the wire surfaces were removed by etching in an aqueous solution of hydrofluoric, acetic and nitric acids at a ratio of 1 : 5 : 5.

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) with a Shimadzu DSC 60 calorimeter (Shimadzu, Kyoto, Japan). Three tests were performed with different samples of 25 mg of each wire and consisted of heating the sample to 80 °C and then cooling it down to −80 °C at a heating and cooling rate of 10 °C min^{-1}.

Vickers microhardness measurements were performed with a Leica Durimet II tester (Leica, Weltzlar, Germany) using a 100 gf load. Three 15-mm-long specimens of each wire were embedded in acrylic resin, ground to half their diameter and then polished with diamond paste. Twenty indentations were measured for each type of wire. The load/unload behaviour was assessed in tensile tests made with an Instron 5580 testing machine (Instron, Norwood, MA, USA). Specimens 100 mm in length were tensile loaded up to 6%

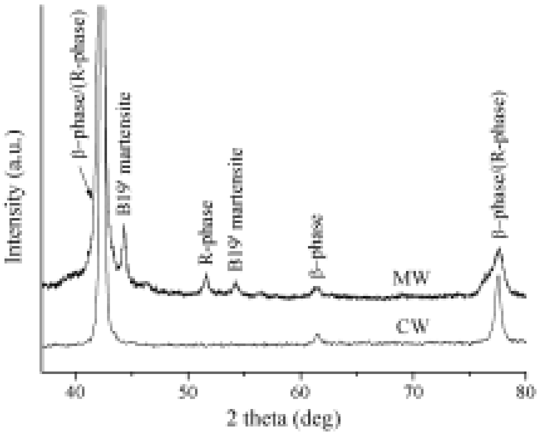

Figure 1 XRD patterns of the wires in the region of the main peaks of austenite, B19′ martensite and the Rphase.

elongation and then unloaded to zero stress. The tests were performed in triplicate.

Results

The data obtained on each wire by EDX were 50.0% Ni-50.0% Ti for MW and 50.3% Ni-49.7% Ti for CW, and the standard deviations were <0.5%. Comparison of these contents using ANOVA ($\alpha = 0.05$) showed no statistical difference, meaning that both type of wires had essentially the same average Ni and Ti contents, close to the equiatomic ratio of 1 : 1. The results of the XRD analysis showed that austenite, which is designated as the β-phase in the NiTi system, was the predominant phase in both wires. Figure 1 shows the main X-ray peaks of austenite at approximately 42.5°, 61.5° and 77.6° in the diffractogram for both wires. For the M-Wire, there were also peaks at 44.4° and 54.2°, which belong to the B19′ martensitic phase, and another peak at 51.6°, indicative of the presence of the R-phase. The presence of the R-phase also caused broadening of the first and the third β-phase peaks in the diffractogram of the M-Wire.

The average transformation temperatures determined by DSC are shown in Table 1. Ms and Mf indicate the beginning and end of the formation of martensite during cooling, whilst As and Af are the corresponding temperatures for the reverse transformation taking place upon heating the wires; the standard deviations were <3 °C. The Af temperature of the CW wire was around room temperature, whilst the same temperature in MW was well above it.

Typical tensile load/unload curves for the two wires are shown in Fig. 2, with dotted lines indicating their initial apparent elastic modulus and the slope of the transformation plateau. For the CW specimen, the initial portion of the loading curve (E_{CW}) was approximately linear until the transformation stress was attained, whilst in MW, it was composed of two linear portions, the first ($E_{1\text{-}MW}$) with a lower inclination and the second ($E_{2\text{-}MW}$) with an inclination similar to that of CW. Both curves showed the transformation plateau, which was lower for MW, with the load level almost constant up to the maximum elongation of 6%. The mean values of the slope of the transformation plateau, calculated between 2% and 6% strains, were $S_{MW} = 8$ MPa and $S_{CW} = 16$ MPa for MW and CW, respectively (maximum standard error smaller than 3%). During the unloading process, the decrease in load corresponding to the reverse transformation was followed by elastic unloading, leaving behind a small permanent residual elongation of approximately 0.5% in both wires. The average values of the stress at the loading plateau (transformation stress) are given in Table 1, which also shows the average values of Vickers microhardness, were similar for the MW and CW wires ($P = 0.91$).

Table 1 Average values of the transformation temperatures, Ms, Mf, As and Af, transformation stress, $\sigma_{A\text{-}M}$, and Vickers microhardness, MHV, of the wires studied

Wires	Transformation temperatures (°C)				$\sigma_{A\text{-}M}$	MHV
	Ms	Mf	As	Af	(MPa)	(kgf mm^{-2})
MW	41	6	17	47	494	428
CW	14	−15	−9	23	567	429

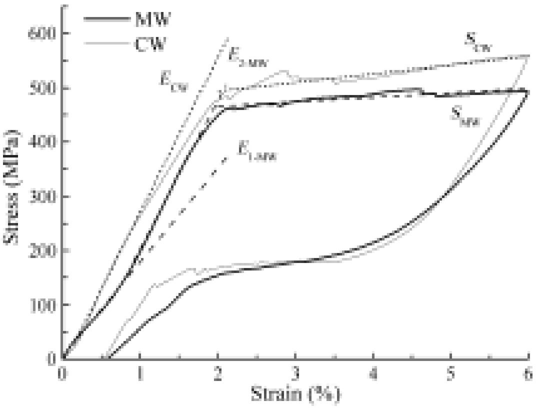

Figure 2 Tensile loading and unloading curves of the wires studied. The difference between the stresses for direct and reverse transformations represents the stress hysteresis.

Discussion

This study evaluated the physical and mechanical properties of two wires used for manufacturing rotary NiTi instruments, one conventional and the other submitted to a proprietary thermomechanical treatment. NiTi alloys with a small excess of Ni in relation to the equiatomic ratio of 1:1 respond well to heat treatment at lower temperatures because coherent, submicroscopic Ti_3Ni_4 precipitates can form in the β-phase matrix (Miyazaki *et al.* 1982). Because of their small size and coherence with the matrix, these precipitates increase the mechanical strength of austenite, helping to prevent plastic deformation upon straining, and thus favouring the occurrence of stress-induced martensitic transformation that is paramount to the superelasticity of the alloy. As the Ti_3Ni_4 precipitates contain more Ni than Ti, their formation decreases the amount of Ni in solid solution and increase the transformation temperatures, which are very sensitive to the Ni content (Tang 1997). The semiquantitative chemical analysis of the wires indicated that they have appropriate amounts of Ni and Ti to be sensitive to the low-temperature heat treatments that improve their superelasticity, and it is reasonable to assume that both the CW and MW wires were submitted to such a kind of treatment.

The results of the XRD analysis (Fig. 1) revealed that austenite, the β-phase, was the predominant phase in the two wires evaluated. Figure 1 also shows peaks of B19′ martensite and of the R-phase in the diffractogram of the M-Wire, whose relatively low intensity indicates that, in addition to the austenite, a small amount of these phases were also present in the MW specimen. These observations are in agreement with those of Johnson *et al.* (2008) and Alapati *et al.* (2009) about M-Wire and are compatible with the higher transformation temperatures found by DSC in MW (Table 1). To understand why such a microstructure would form in MW, it is necessary to examine the description of the proprietary thermomechanical treatment applied to it. First of all, it is said that this treatment is to be performed in conventional nitinol wire after cold drawing in the martensitic phase to the finished diameter and straightening annealed in the temperature interval 400–475 °C (Berendt 2007). The wire is then subjected to thermal cycling between a low temperature bath (below 10 °C) and a high temperature one (up to 180 °C) 3–5 times under a strain in the superelastic regime. Although the effect of this thermomechanical cycling is complex, it is known that thermal cycling under load has the potential to raise the starting temperature for the formation of B19′ martensite, but has little effect on the R-phase starting temperature (Miyazaki *et al.* 1986). This should be the reason why both B19′ and the R-phase coexist in MW at room temperature (Fig. 1).

It has been reported that NiTi instruments with higher transformation temperatures are more flexible (Miyai *et al.* 2006, Hayashi *et al.* 2007) and that heat treating these instruments can increase the transformation temperatures (Yahata *et al.* 2009) and improve this property (Yahata *et al.* 2009, Ebihara *et al.* 2011). However, the greater flexibility of the instruments made with M-Wire should be attributed to their lower initial apparent elastic modulus ($E_{1\text{-}MW}$, Fig. 2), which can be associated with the stress-induced reorientation of the R-phase and B19′ martensite (Miyazaki & Otsuka 1986). Thermal cycling under stress is also a form of 'training' treatment that makes it easy for the same types of martensite variants to form upon loading (Otsuka & Ren 2005). This is responsible for the observation that stress-induced transformation of austenite in MW can be achieved at lower stress levels and has another important consequence. It produces an additional factor contributing to improve the flexibility of the instruments made with this wire. On the other hand, the heat treatments applied to the instruments in the works cited earlier were conducted in the temperature range 400–500 °C, and the observed improvement in flexibility should be the result of softening associated with dislocation annealing and coalescence of precipitates (Reed-Hill *et al.* 2008). In this respect, it is important to observe that the average values of Vickers microhardness obtained here (Table 1) showed that MW was as hard as CW. The importance of this finding lies in the fact that instruments made with M-Wire should possess a torsional resistance comparable with that of the instruments made with conventionally treated NiTi wires (Johnson *et al.* 2008, Kramkowski & Bahcall 2009).

According to the literature (Johnson *et al.* 2008, Kell *et al.* 2009, Larsen *et al.* 2009, Al-Hadlaq *et al.* 2010, Gao *et al.* 2010, Peixoto *et al.* 2010), experimental and commercial instruments made with M-Wire exhibited better fatigue properties than instruments conventionally produced. The results of the load–unload tests presented in Fig. 2 are important to demonstrate this difference in behaviour. As commented before, the superelastic plateau of the MW specimen was achieved under a lower tensile stress (Table 1). Additionally, the stress at the plateau remained relatively constant until the 6% elongation was attained ($S_{MW} = 8$ MPa), whilst this stress in CW tended to increase in the same region (S_{CW} = 16 MPa). This increase is the result of work hardening, which can take place in the superelastic region associated with a certain amount of plastic deformation (Saburi 1998, Otsuka & Ren 2005). This concurrent plastic deformation is observed to take place when superelastic NiTi alloys are strained at temperatures somewhat higher than Af, and the amount of slip deformation increases as the test temperature rises (Saburi *et al.* 1982). This should be the case with CW, which possessed lower transformation temperatures than MW. The occurrence of slip deformation during superelastic straining is also responsible for the higher stress hysteresis exhibited by CW compared with MW (area under the load–unload curve in Fig. 2). This is because more lattice defects were being introduced in CW than in MW when the wires suffered the load/unload cycle. When this reasoning is applied to the rotary endodontic instruments made from these wires, it is easy to understand why those in which MW was used presented higher fatigue resistance (Johnson *et al.* 2008, Kell *et al.* 2009, Larsen *et al.* 2009, Al-Hadlaq *et al.* 2010, Gao *et al.* 2010, Peixoto *et al.* 2010). The reason is that the thermomechanical treatment applied to MW led to a more efficient superelastic behaviour with less generation and accumulation of lattice defects in each load–unload cycle.

Conclusions

Compared with a conventionally treated NiTi wire employed for the manufacture of NiTi instruments, M-Wire showed higher transformation temperatures, contained small amounts of B19′ martensite and R-phase, exhibited the same Vickers hardness, but a lower apparent elastic modulus and smaller transformation stress and mechanical hysteresis. These physical and mechanical properties can render endodontic instruments made with this wire more flexible and fatigue resistant than those made with conventionally processed NiTi wires.

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3.2 Artigo 2

MECHANICAL BEHAVIOR OF M-WIRE AND CONVENTIONAL NITI WIRE USED TO MANUFACTURE ROTARY ENDODONTIC INSTRUMENTS

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Keywords: Endodontic instruments, M-Wire, Nickel-Titanium, thermomechanical treatment

Running title: **COMPARISON BETWEEN M-WIRE AND CONVENTIONAL NITI WIRE**

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Title: Mechanical behavior of M-Wire and conventional NiTi wire used to manufacture rotary endodontic instruments

Article Type: Research Paper

Keywords: Endodontic instruments, M-Wire, Nickel-Titanium, thermomechanical treatment

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Abstract: Introduction: Recently, NiTi wires used in the manufacture of rotary endodontic instruments have received additional heat treatments to improve mechanical properties. Objectives: Comparison of physical and mechanical properties of one conventional and a new NiTi wire, which had received an additional thermomechanical treatment. Methods: Specimens of both conventional (NiTi) and the new type of wire, called M-Wire (MW), were subjected to tensile and three-point bending tests, Vickers microhardness measurements, and to rotating-bending fatigue tests at a strain-controlled level of 6%. Fracture surfaces were observed by scanning electron microscopy and the non-deformed microstructures by transmission electron microscopy. Results: The thermomechanical treatment applied to produce the M-Wire apparently increased the tensile strength and Vickers microhardness of the material, but its apparent Young modulus was smaller than that of conventionally treated NiTi. The three-point bending tests showed a higher flexibility for MW which also exhibited a significantly higher number of cycles to failure. Significance: M-Wire presented mechanical properties that can render endodontic instruments more flexible and fatigue resistant than those made with conventionally processed NiTi wires.

Mechanical behavior of M-Wire and conventional NiTi wire used to manufacture rotary endodontic instruments

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Abstract

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Keywords: Endodontic instruments, M-Wire, Nickel-Titanium, thermomechanical treatment

Introduction

The specific properties of shape memory alloys are related to a reversible solid-to-solid phase transformation, the so-called martensitic transformation [1]. One of these properties is superelasticity, which has allowed for the development of NiTi endodontic instruments. Root canal preparation instruments made of NiTi have been described to promote preparation of curved and narrow root canals whilst maintaining the original anatomy [2,3].

There has been considerable improvement in file design, manufacturing methods, and preparation techniques on rotary endodontic instruments made of NiTi alloys; however, intracanal fracture of instruments caused by flexural fatigue remains a primary concern in the practice of endodontics, especially for canals with severe curvatures [4]. The mechanical performance of NiTi alloys is sensitive to their microstructures and associated thermomechanical history [5]. Therefore, one of many promising solutions to improve fatigue resistance of rotary instruments is to optimize the microstructure of NiTi alloys through novel processing or new manufacturing technologies. Recently, a new NiTi wire termed M-Wire (Sportswire LLC, Langley, OK, USA) has been developed through a proprietary thermomechanical processing procedure. Initial data for this type of NiTi raw material suggests significantly improved fatigue resistance of endodontic rotary instruments in comparison with those made of conventional superelastic NiTi alloys [6-9].

According to Johnson *et al.* [6], M-Wire is composed of 508 Nitinol, which has undergone a processing method comprised of thermomechanically treating the raw wire under specific tensile stresses and temperatures. Compared with a conventionally treated NiTi wire employed for the manufacture of NiTi instruments, M-Wire showed higher transformation temperatures, contained small amounts of B19’ martensite and R-phase with a lower apparent elastic modulus, as well as smaller transformation stress and mechanical hysteresis [10].

In the present work, tensile, three-point bending and rotating-bending fatigue tests were carried out to assess the mechanical properties of M-Wire compared with those of a conventional wire used for manufacturing rotary NiTi instruments. The non-deformed microstructure of the wires was examined by transmission electron microscopy, while the fracture surfaces of failed wires were examined by and scanning electron microscopy. The null hypothesis was that M-Wire had the same physical and mechanical properties as a conventional NiTi wire.

Materials and Methods

The conventional wire (NiTi) and the M-Wire (MW) were both provided by Dentsply Maillefer (Ballaigues, Switzerland). Both wires had 1.2 mm in diameter. Wires with 100 mm in length were tensile tested until rupture in an Instron 5580 testing machine (Instron, Norwood, MA, USA). The tests were performed at room temperature with a strain rate of 1.0 x 10^{-3} s^{-1}. Apparent modulus of elasticity (E), transformation stress from austenite to martensite ($\sigma_{A\text{-}M}$), ultimate tensile stress (σ_{UTS}) and total elongation (e_t) were determined as the average of three tests, using the analysis software Instron Series IX for Windows.

Vickers microhardness measurements were performed with a Leica Durimet II tester (Leica, Wetzlar, Germany) using a 100 gf load. Three 15 mm-long specimens of each wire were embedded in acrylic resin, ground to half their diameter and then polished with diamond paste. Twenty indentations were made for each type of wire.

Three-point bending tests were performed in triplicate in the device shown in Figure 1a. A force F was applied to the wire specimen resting between two supports 26 mm apart. In a three-point bending test, the bending moment (M) in N·cm at L/2 is given by

$$M = FL/4 \qquad (1)$$

where F is the force given in N and L the distance in cm between the two supporting points. When the wire is bent by applying a vertical displacement d, there is tension below the neutral axis and compression above it (Fig. 1b). The maximum vertical displacement was chosen to be 5 mm, so as to reach 4% of maximum tensile strain amplitude at surface of the specimen. This corresponds to approximately the maximum amount of deformation suffered by an endodontic instrument of size #25/0.06 at 3 mm from its tip, when introduced in a standard root canal, according to the equations of kinematics [11].

(Figure 1)

Rotating-bending fatigue tests were carried out in the device shown in Figure 2. One end of the wire specimen was clamped to the axis of a direct current motor controlled by an adjustable power supply. The opposite end of the wire was connected to a tachometer. The average fatigue life was determined from $n = 10$ specimens of each type of wire tested at a tensile-strain amplitude of 6% at the outer surface of curvature. The outer radius of curvature R to give rise to this strain level was 12 mm, as can be calculated using equation 2:

$$\varepsilon = d/(2R + d) \qquad (2)$$

where ε is strain level, R is outer radius of curvature and d is diameter of the wire.

To maintain the specimen temperatures at maximum 10°C above room temperature, a pilot study using a thin thermocouple was performed to choose the appropriate rotation speed, which was set to 32 rpm.

(Figure 2)

All data was found to fit a normal distribution. The data obtained in the tensile tests, Vickers microhardness measurements and rotating bending fatigue tests were analyzed statistically by using one-way analysis of variance, and the comparison of means was conducted by using Tukey multiple comparison tests. The level of confidence was set at $p < 0.05$.

Three samples of the fatigue fractured wires were randomly selected and examined by scanning electron microscopy (SEM) (Jeol JSM 6360, Tokyo, Japan) to assess their surface characteristics. Non-deformed wire specimens were analyzed by transmission electron microscopy using a 200 kV T20 microscope (FEI, Hillsboro, OR, USA). The observed specimens were thin longitudinal and transverse sections of the wires prepared by focused ion beam (FIB) milling in a Helios 600 equipment (FEI, Hillsboro, OR, USA).

Results

Typical stress-strain curves for NiTi and MW obtained in the tensile tests are shown in Figure 3, while Table 1 shows the mean values found for the relevant parameters. These results indicate that the thermomechanical treatment received by MW affected its mechanical behavior. It can be observed that ultimate tensile strength and total elongation increased, while the transformation stress remained unchanged. The mean values (and standard deviations) of apparent modulus of elasticity were 24.6 (0.6) GPa for MW and, 43.9 (1.8) GPa for NiTi, i.e., this parameter was significantly lower for MW than for NiTi ($p = 0.003$). Vickers microhardness measurements tended to agree with tensile strength results: mean values found were 425 (18) HV and 356 (9) HV for MW and NiTi, respectively. The higher hardness of MW was also statistically significant ($p = 0.001$).

(Figure 3)

The curves obtained in the three-point bending tests were similar to those presented in Figure 4, showing the increase in the bending moment M as the vertical distance d traveled by the specimen's center increased from zero to 5 mm. The higher flexibility of the MW specimens in relation to the NiTi wires can be appreciated if a vertical distance of 2 mm is taken for reference. For bending the wires by this distance, mean bending moments of 13.5 (0.0) N·cm and 16.0 (1.0) N·cm were found for MW and NiTi, respectively. When larger vertical distances were considered, there was a tendency of the curves to overlap and, for distances larger than approximately 4 mm, MW becomes harder to bend than NiTi.

(Figure 4)

The results of the rotating-bending fatigue tests carried out at a strain amplitude of 6%, expressed as the number of cycles to failure, were 1125 (141) and 436 (86) for MW and NiTi, respectively. The fatigue resistance of MW was also significantly higher ($p = 0.01$) than that of NiTi. The fracture surfaces of the tested specimens observed by SEM exhibited the typical features of this fracture mode. The lower magnification images shown in Figures 5a and c illustrate the presence of small areas of nucleation and slow crack propagation in the cross section's periphery, called smooth regions, and a large fibrous central area associated with the final ductile failure. When observed at higher magnifications (Figs. 5b and d), the smooth regions of the fracture surface have fatigue striations and numerous secondary cracks. These features are similar for MW and NiTi specimens, although the smooth regions appeared to be more numerous in MW than in NiTi, indicating that crack nucleation and propagation occurred along the entire surface of the specimen during fatigue testing.

(Figure 5)

Fine microstructural differences between the two wires can be observed in the TEM images of non-deformed samples shown in Figure 6. While the NiTi wire contained essentially the β-phase austenite, with a dislocation-cell substructure, and precipitates, the MW wire microstructure contained martensite, with cell substructure and precipitates.

(Figure 6)

Discussion

The realization that the mechanical properties of near equiatomic NiTi alloys are strongly influenced by the stress-induced phase transformation taking place in this alloy and that the microstructural changes introduced by thermomechanical treatments can control this phase transformation constitutes the modern trend towards developing new rotary endodontic instruments with improved mechanical properties [6,12]. In the present study, a few methods used in engineering for probing into the microstructure and mechanical behavior of materials were employed to analyze NiTi wires used in the manufacture of endodontic instruments. This approach is justified by the absence of standardized test methods and devices to assess the mechanical behavior of NiTi rotary instruments [13]. By comparing the properties of M-Wire with those of a conventionally treated NiTi wire, it should be possible gain a better understanding of some aspects of the clinical performance of the new instruments made from MW. In addition, the use of wires enables the elimination of any potential effects of file design (size/taper, cross-section geometry, helical angle, etc) on the mechanical properties.

The tensile curves shown in Figure 3 revealed, for both wires, the four stages associated with the uniaxial deformation of superelastic NiTi. The first stage corresponds to the elastic deformation of austenite (the crystalline phase present at room temperature in superelastic NiTi); the second stage is the plateau associated with the stress-induced martensitic transformation; the third stage is the elastic deformation of martensite, while in the fourth stage corresponds to the plastic deformation of martensite until final rupture takes place [1]. As far as the behavior of endodontic instruments made of the two wires is concerned, the main difference in property is the lower apparent elastic modulus of MW (Table 1). The higher strength of MW, confirmed by the microhardness results, would lead to more torsional resistant instruments. These predictions are difficult to confirm due to the effects of file design mentioned above and in fact the results of measurements made on endodontic instruments are controversial [8,14,15].

The results of the three-point bending tests indicated that the force required to bend MW was smaller than the that applied to deform NiTi by the same amount, until larger strains were achieved (Fig. 4). These results are consistent with the tensile-tests data, but their relevance lies in the fact that bending strains, as opposing to uniaxial tensile strains, are the type encountered during the clinical use of endodontic instruments to clean and shape curved root canals. The observation that smaller forces are required to bend MW at intermediate vertical displacements is an indication that the structural damage associated with rotating-bending fatigue of MW should be smaller than in conventional NiTi.

The results of the rotating-bending fatigue tests confirm the prediction just mentioned that the fatigue resistance of MW is greater than that of NiTi. In fact, the mean value of number of cycles to failure of MW, 1125 (141), was almost 3 times greater than that of NiTi, 436 (86). This is in agreement with the high fatigue strength reported in earlier studies performed with endodontic instruments made with MW [6-8, 13].

Studies evaluating the flexural fatigue resistance of nickel-titanium rotary files employed devices with varying testing set-up such as steel grooves, steel slopes or 3-point steel pins to guide the file into the desired curvature [14,16]. In addition, the radii and angles of curvature of the testing devices were different, despite the fact that these two parameters were shown to have a significant effect on the fatigue resistance of rotary nickel-titanium instruments [4,17]. This lack of an internationally accepted standard for a method or device for fatigue testing has resulted in the observed variability of testing results [7]. For the present study, a testing device (Fig. 2) was constructed on the basis of the designs by Tobushi *et al.* [18] and Figueiredo *et al.* [19], which allowed the standardized choice of the tensile-strain amplitude according to Equation 2. The value chosen for the tests, 6% strain, is of the order of magnitude of the maximum strains generally found for rotary NiTi instruments employed in formatting curved root canals [20].

The improved fatigue behavior of MW should be correlated with its fine structure as revealed in the TEM micrographs (Fig. 6), which showed the presence of thin martensite variants in non-deformed specimens. Thus, it is possible that the lower apparent elastic modulus of MW and the smaller force required for bending by the same amount MW in relation to conventional NiTi are the result of the stress-induced reorientation of the R-phase and B19' martensite variants already present in MW [10,21]. The higher fatigue resistance found for MW should also be result of these martensites, which would facilitate crack nucleation but render propagation more difficult because of the large number of interfaces present. These interfaces are known to cause the formation of complex arrays of secondary cracks, dissipating the energy required for crack propagation [22]. In fact, lower fatigue crack propagation rates were observed by McKelvey & Ritchie [23] in martensitic NiTi alloys, in comparison with stable and superelastic austenite. More recently, Figueiredo *et al.* [19] showed that the number of cycles to failure in martensitic NiTi wires can be as much as 100 times larger than in stable and superelastic austenitic NiTi.

Conclusions

The thermomechanically treated NiTi wire termed M-Wire was more flexible but harder than the conventional NiTi superelastic wire. It contained martensite in its non-deformed microstructure and this is probably the reason why M-Wire showed higher fatigue resistance under 6% strain level than the NiTi wire.

Acknowledgements

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Table 1 - Mean values (and standard deviations) of the apparent modulus of elasticity, E, transformation stress, σ_{A-M}, ultimate tensile strength, σ_{UTS}, and total elongation, e_t, of tensile tested NiTi and MW wires.

NiTi wires	E (GPa)	σ_{A-M} (MPa)	σ_{UTS} (MPa)	e_t (%)
NiTi	43.9 (1.8)	506.7 (17.8)	1330.0 (6.6)	19.3 (0.4)
MW	24.6 (0.6)	503.3 (2.3)	1547.3 (1.5)	21.7 (0.5)

Figure Legends

Figure 1. Apparatus (a) and geometry (b) of the three-point bending test.

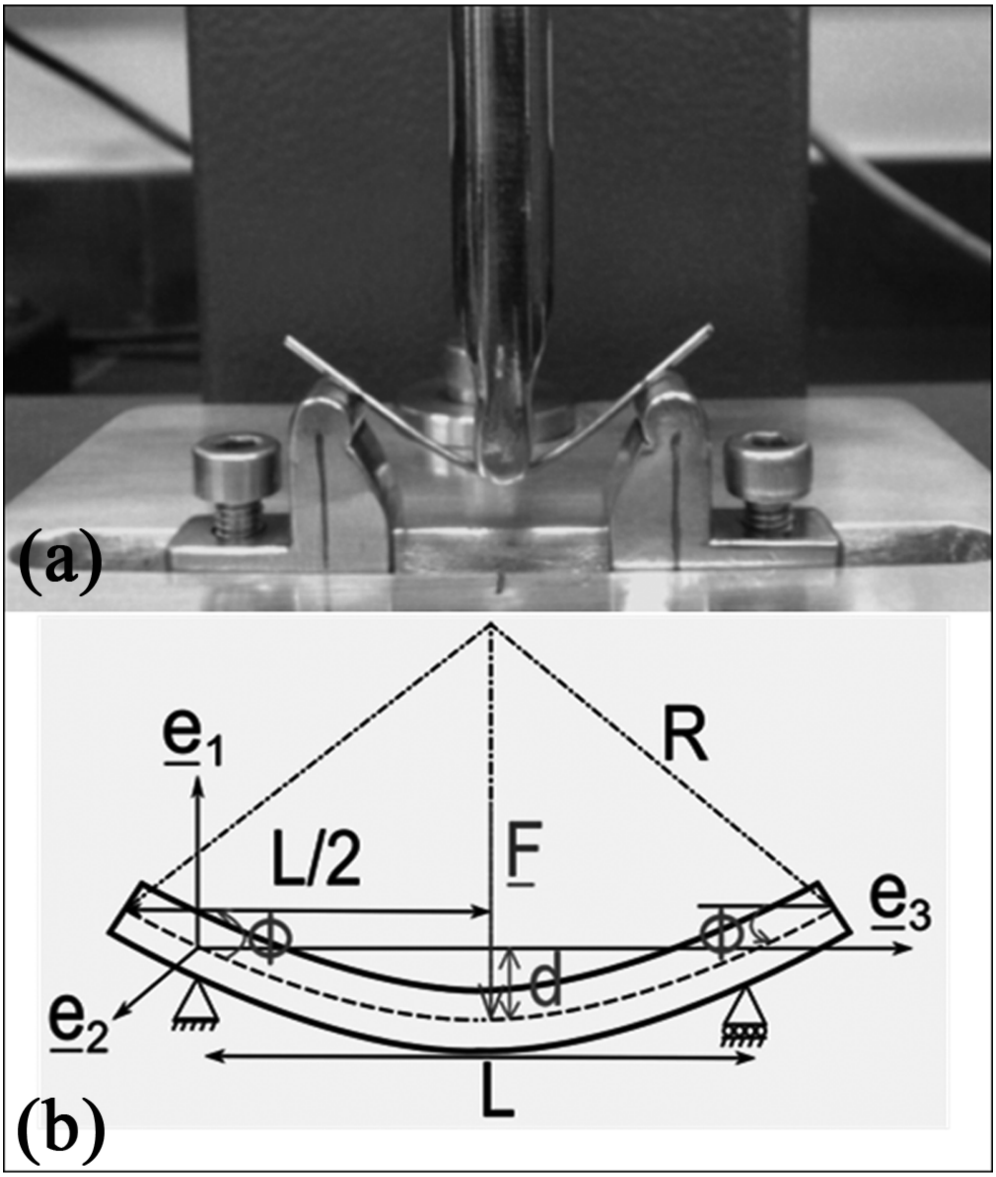
Figure 2. Rotating-bending fatigue bench.

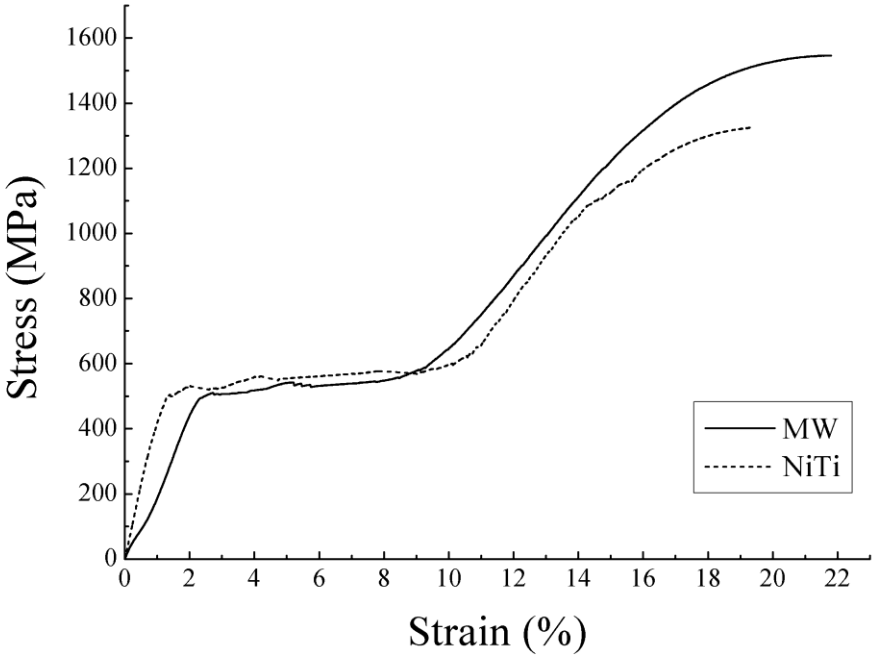
Figure 3. Typical stress-strain curves of the wires obtained in tensile tests.

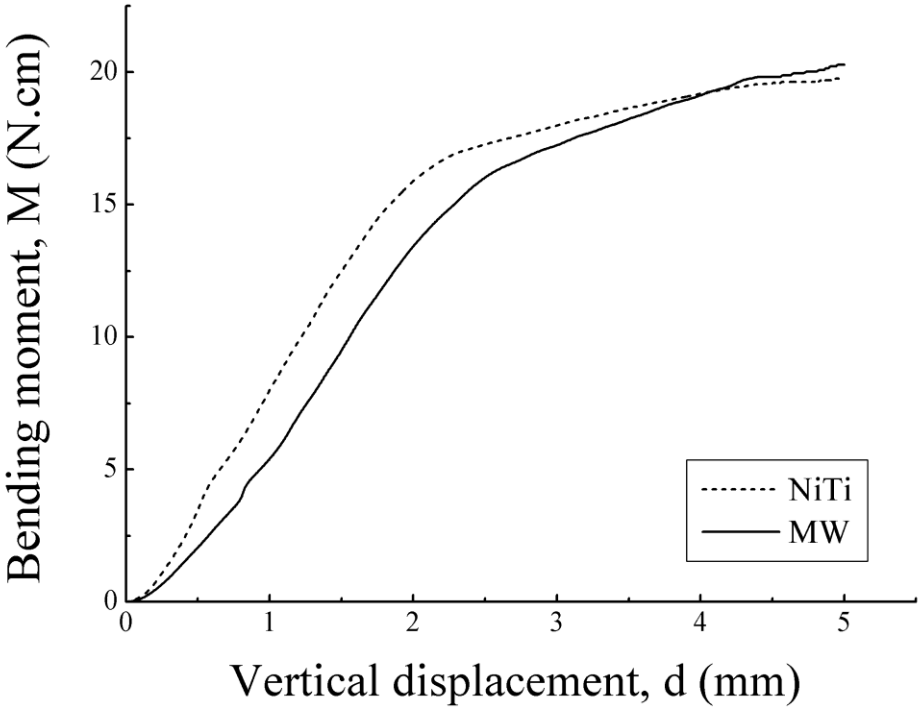
Figure 4. Bending moment versus displacement curves obtained in the three-point bending tests.

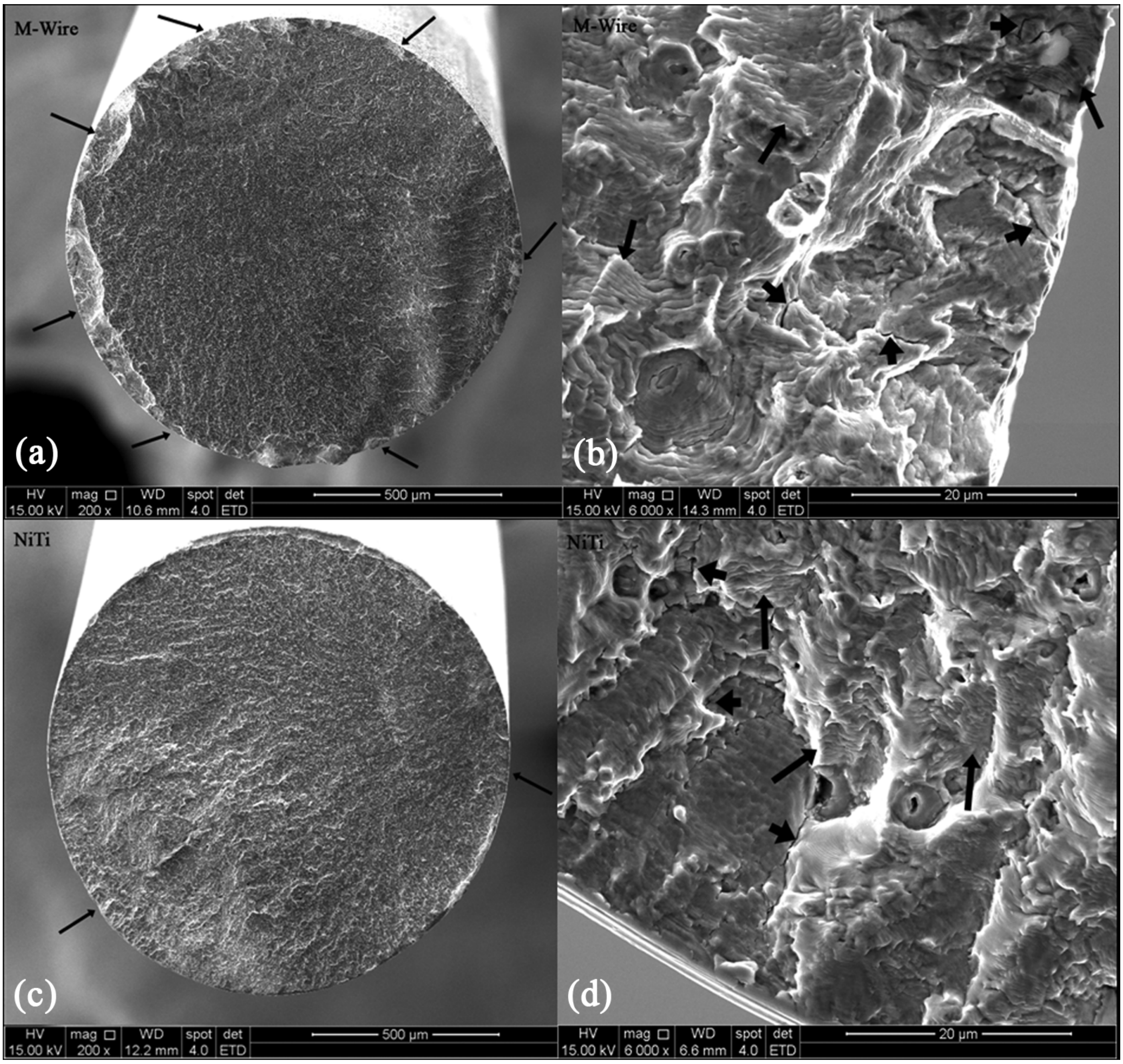
Figure 5. SEM images of fractured MW (a and b) and NiTi (c and d) surfaces after fatigue testes. Left: smooth regions at the edges of the cross-section (arrowed) and fibrous region at the centre; right: detail of the smooth region, showing fatigue striations (dark arrows) and secondary cracks (wider dark arrows).

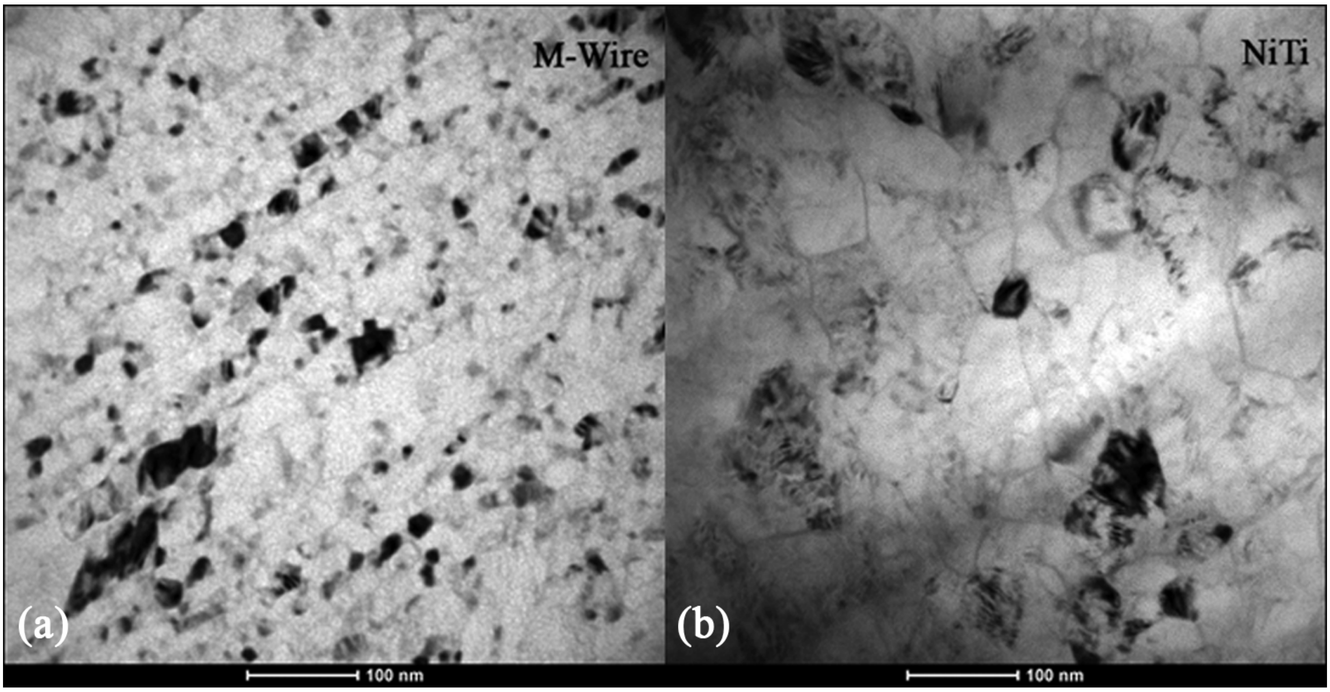
Figure 6. TEM micrographs showing the microstructure of (a) MW with 20-nm wide martensite variants and precipitates; (b) NiTi containing essentially the β-phase austenite, with a dislocation-cell substructure, and precipitates.


(a)
R
$\underline{e}_1$
L/2
$\underline{F}$
$\underline{e}_3$
Φ
Φ
d
$\underline{e}_2$
L
(b)


Stress (MPa)
0
200
400
600
800
1000
1200
1400
1600
Strain (%)
0
2
4
6
8
10
12
14
16
18
20
22
MW
NiTi


Bending moment, M (N.cm)
0
5
10
15
20
Vertical displacement, d (mm)
0
1
2
3
4
5
NiTi
MW


M-Wire
(a)
HV mag ☐ WD spot det
15.00 kV 200 x 10.6 mm 4.0 ETD
500 µm
M-Wire
(b)
HV mag ☐ WD spot det
15.00 kV 6 000 x 14.3 mm 4.0 ETD
20 µm
NiTi
(c)
HV mag ☐ WD spot det
15.00 kV 200 x 12.2 mm 4.0 ETD
500 µm
NiTi
(d)
HV mag ☐ WD spot det
15.00 kV 6 000 x 6.6 mm 4.0 ETD
20 µm


M-Wire
NiTi
(a)
(b)
100 nm
100 nm

3.3 Artigo 3

IN VITRO ASSESSMENT OF TORQUE AND FORCE GENERATED BY NOVEL PROTAPER NEXT™ INSTRUMENTS DURING SIMULATED CANAL PREPARATION

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Keywords: nickel titanium, ProTaper Next, torque, simulated preparation

Running title: **TORQUE AND FORCE GENERATED BY NOVEL PROTAPER NEXT INSTRUMENTS**

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Abstract: Introduction: The purpose of this study was to assess torque and force for simulated canal preparation with a new root canal instrument, ProTaper Next. Methods: Six sets of ProTaper Next Instruments (X1-X5) were used to prepare 36 artificial canals. Files were divided in six groups. Different settings of rotations per minute (250, 300, and 350rpm) and numbers of in-and-out movements to reach WL (3 or 4 insertions) were applied in each group (250rpm/3ins.; 250rpm/4ins.; 300rpm/3ins.; 300rpm/4ins.; 350rpm/3ins. and 350rpm/4ins.) using an automated torque bench. Peak torque (Ncm) as well as positive and negative force (N) was registered. Analysis of Variance and Tukey post hoc tests were applied. Preliminary data for angle and stationary torque at failure were also obtained and compared with peak torque for each instrument. Results: Significant differences in peak torque ($p<0.0001$), positive force ($p<0.002$), and negative force ($p<0.0001$) were found for ProTaper Next instruments overall. X2 showed the highest torque with all settings. X5 showed the highest positive force in all groups. X1 and X2 showed the highest negative forces for all groups except for 350rpm/4ins.. Significantly lower torque ($p<0.0001$) and positive force ($p<0.007$) was measured in the group 350rpm/4ins. for all instruments except for X4. In contrast, X1 showed a significant lower negative force for 350rpm/4ins. Torque at failure according to ADA No. 28/ISO 36030-1 was lower for X1, X2 and X3 than torque during simulated canal preparation ($p<0.0001$). Conclusions: Under the conditions of this study, using ProTaper Next at 350rpm and with four in-and-out movements resulted in lowest levels of peak torque as well as positive and negative forces.

In vitro assessment of torque and force generated by novel ProTaper Next instruments during simulated canal preparation

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Individual contribution to the study and manuscript:
Erika S. J. Pereira - Realization of the experiment. Manuscript.
Rupinderpal Singh - Assistance in the experiment. Manuscript.
Ana Arias - Statistical support. Manuscript.
Ove A. Peters - Design of the study. Manuscript.

Acknowledgement

The authors deny any conflicts of interest.

Abstract

Introduction: The purpose of this study was to assess torque and force for simulated canal preparation with a new root canal instrument, ProTaper Next. **Methods**: Six sets of ProTaper Next Instruments (X1-X5) were used to prepare 36 artificial canals. Files were divided in six groups. Different settings of rotations per minute (250, 300, and 350rpm) and numbers of in-and-out movements to reach WL (3 or 4 insertions) were applied in each group (250rpm/3ins.; 250rpm/4ins.; 300rpm/3ins.; 300rpm/4ins.; 350rpm/3ins. and 350rpm/4ins.) using an automated torque bench. Peak torque (Ncm) as well as positive and negative force (N) was registered. Analysis of Variance and Tukey post hoc tests were applied. Preliminary data for angle and stationary torque at failure were also obtained and compared with peak torque for each instrument. **Results**: Significant differences in peak torque ($p<0.0001$), positive force ($p<0.002$), and negative force ($p<0.0001$) were found for ProTaper Next instruments overall. X2 showed the highest torque with all settings. X5 showed the highest positive force in all groups. X1 and X2 showed the highest negative forces for all groups except for 350rpm/4ins.. Significantly lower torque ($p<0.0001$) and positive force ($p<0.007$) was measured in the group 350rpm/4ins. for all instruments except for X4. In contrast, X1 showed a significant lower negative force for 350rpm/4ins. Torque at failure according to ADA No. 28/ISO 36030-1 was lower for X1, X2 and X3 than torque during simulated canal preparation ($p<0.0001$). **Conclusions**: Under the conditions of this study, using ProTaper Next at 350rpm and with four 'in-and-out" movements resulted in lowest levels of peak torque as well as positive and negative forces.

Introduction

Nickel-titanium (NiTi) rotary instruments are frequently used in contemporary root canal preparation. NiTi rotary files make shaping not only easier and faster but are also more likely to lead to improved outcomes compared to stainless steel hand instruments (1). However and despite increased flexibility and torsional strength, compared with stainless-steel instruments (2), NiTi rotary instruments still seem to have a risk of fracture in the clinical situation (3, 4).

Failure modes of NiTi instruments have been studied extensively (3-9). Flexural fatigue is caused by repetitive compressive and tensile stresses acting on outer fibers of a file rotating in a curved canal; torsional failure occurs when the tip of the instrument binds but the shank of the file continues to rotate (3). Shear fracture of the material then occurs when the maximum strength of the material is exceeded (7).

The torque generated by a rotating instrument during root canal instrumentation depends on the contact area between the file and the canal walls, the applied apical force, instrument diameter, and preoperative canal volume (10-12). In turn mechanical properties of endodontic instruments are affected by a variety of factors, such as size, taper, design, alloy chemical composition, and thermo mechanical processes applied during manufacturing (13-15).

It is held that there is a strong positive correlation between the maximum torque an instrument can withstand and its diameter (12,16). It has also been suggested that the cross-sectional shape of instruments affects the stress distribution pattern and thus their torsional resistance (17,18). Moreover, flexural fatigue developed during curved-root canal shaping may decrease the torsional resistance of endodontic instruments (12, 19-21).

ProTaper Next is a novel set of rotary instruments that are designed with variable tapers and an off-centered rectangular cross-section. The set includes five shaping instruments with overall variable tapers; at the tip, X1 is #17/.04, X2 is #25/.06, X3 is #30/.075, X4 is #40/.06, and X5 is #50/.06. All the instruments are expected passively follow the canal until the working length is achieved (22).

Such a single length technique possibly requires greater torsional strength of a given instrument due to greater contact with the dentin walls resulting in high stresses on its entire length (30). However, the system features an off-centered rectangular cross section associated, which is intended to reduce torsional stress on the instrument (22). These instruments are manufactured from so-called M-Wire raw material, which was shown to possibly extend fatigue life beyond conventional NiTi alloy (24).

Currently there is no data available on torque and force during canal preparation with ProTaper Next used according to the current Directions for Use (DFU). Hence, the aim of this study was to determine baseline torque and force among ProTaper Next instruments during simulated canal preparation. Also, different RPM and insertion settings were compared to suggest the optimum speed and overall handling those instruments should be subjected to during their use in root canal treatments.

Materials and Methods

Six sets of ProTaper Next Instruments (X1, X2, X3, X4 and X5) were used to prepare 36 simulated root canals in plastic blocks (A 0177; Dentsply Maillefer, Ballaigues, Switzerland) in a standardized fashion.

According to rotational speed (250, 300 or 350 rpm) and insertion pattern (3 or 4 ins.), rotaries were divided in six groups (250rpm/3ins.; 250rpm/4ins.; 300rpm/3ins.; 300rpm/4ins.; 350rpm/3ins. and 350rpm/4ins.).

Canals were initially lubricated with liquid soap and instrumented by the same operator throughout the study. Sizes #10 and #15 K-files were used to confirm a glide path and to establish working length (WL), which was set at 16 mm.

Subsequent tests were run in a standardized automated fashion in a torque-testing platform, which has been described in detail earlier (16, 20). In brief, plastic blocks were secured into a rigid holder that was attached to a strain gauge connected to a pre-amplifier (A&D 30; Orientec, Tokyo, Japan). The bench was then configured to determine torque and force during canal preparation. A torque sensor (MTTRA 2; with amplifier Microtest, both Microtec Systems, Villingen, Germany) and a motor (Type ZSS; Phytron, Gröbenzell, Germany) were mounted on a stable metal platform, which moved along a low friction guide rail for a width of approximately 5cm. Following preliminary experiments two sequences for instrument insertion were programmed to allow fully automated canal preparation: reaching working length (WL) with three more aggressive, or four less forceful instrument insertions. These patterns were the same for all 5 ProTaper Next sizes.

The sequence originally recommended by the manufacturer was followed to shape the simulated canals: X1-X5 were used to WL. Prior to each use and upon completion of simulated shaping procedures, instruments were inspected for plastic deformation.

Peak torque (Ncm) as well as positive and negative peak forces (N) was registered using the custom-made ENDOTEST software package (16) and collected for off-line analysis.

For comparison, an initial analysis of torsional limits of ProTaper Next was performed with 6 samples of each instrument. Stationary torque (Ncm) and angle (°) at failure during clockwise rotation were tested according to ANSI/ADA specification No. 28 (ISO3630-1) using the same torque-testing device.

Data for peak torque as well as positive and negative peak force were found to be compatible with normal distribution and standard deviations of subgroups were similar. Results were analyzed with ANOVA and when it showed significant differences, Tukey post hoc tests were used to compare subgroups.

One-sample-t-test was also used to compare peak torque during simulated canal preparation with stationary torque at failure for each individual instrument.

Results

The first set of analyses examined the impact of simulated torque. There were significant differences in peak torque ($p<0.0001$) for the different settings. As illustrated in Fig. 1, ProTaper Next X2 showed the highest torque (statistically significant in all groups), followed by X1 (statistically significant in groups 250rpm/4ins., 300rpm/3ins. and 350rpm/3ins.).

Table 1 shows the torque (Ncm) and angle (°) at failure at 3 mm from the tip. Torque at failure was lower for X1, X2 and X3 than torque during simulated canal preparation ($p<0.0001$).

There were also significant differences in peak force ($p<0.002$) for the different settings. As shown in Fig. 2, X5 showed significantly higher peak force (statistically significant in all groups), followed by X4 (statistically significant in groups 250rpm/4ins., 300rpm/4ins. and 350rpm/4ins.).

In relation to negative force and as illustrated in Fig. 2, the only significant differences that were found were the highest negative force for X1 and X2 when compared to X3, X4 and X5 ($p<0.0001$) for all groups except for 350rpm/4ins. in which the only file with a different higher significant value was X2.

When results of different groups were compared for each file, significantly lower torque ($p<0.0001$) and lower peak force ($p<0.007$) was shown in the group 350rpm/4ins. for all instruments except for X4 that showed significantly lower torque ($p=0.001$) and force after four insertions but when rotated at 300rpm ($p<0.0001$). X1 showed a significant lower negative force when it was rotated at 350rpm and 4 “in-and-out” movements were used to reach WL ($p<0.0001$).

There was no breakage or plastic deformation of any of the rotaries after being used in six artificial root canals each.

Discussion

The aim of this study was to provide *in vitro* data that could guide the clinical use of novel ProTaper Next rotary instruments manufactured from M-Wire NiTi alloy (Sportswire, Langley, OK). Specifically, standard parameters such as peak torque and positive and negative force were measured in simulated clinical conditions. There is, at this moment, no information available for this particular instrument; however, other instruments manufactured from similar alloy have also been investigated recently (15, 23-26).

Plastic blocks with standardized simulated root canals were used in the present experiment, which is similar to previous studies (16, 28). Plastic blocks have been used not only for the assessment of shaping capabilities but also for the cutting behavior of NiTi rotaries (29); however, cutting of dentin varies from cutting plastic material. Nevertheless, torque values obtained during canal preparation in plastic blocks with curved canals were similar to those in mandibular incisors in an earlier study (16).

Another important issue for this type of studies that may vary from real dentin and plastic is the "threading-in" effect of files, which is why peak negative force was also tested. The phenomenon of "threading-in" of a rotary during root canal preparation results in negative force when automatic insertion with a servomechanism such in the current study is used.

A clinician in the same situation would have the sensation that the instrument is pulled into the canal. However, this sensation is exaggerated in simulated canals in plastic blocks. These canals are more homogenously shaped and this results in more overall wall contact compared to the more irregular shape of canals in human teeth (16). Therefore, caution should be exercised in directly taking *in vitro* data describing "threading-in" into the clinic; nevertheless the present data does suggest that the smaller ProTaper Next sizes have a higher potential for "threading-in" behavior compared to the larger ones.

In order to observe the torque and force that these instruments are subjected to while shaping a root canal, six different groups were created in which three levels of rpm and two manners to reach the WL (with three or four depths or advanced movements of insertion) were included. Under the present conditions ProTaper Next X2 showed the highest values of peak torque in all the groups when compared to other instruments, followed by X1 in some of them.

The present results represent a first impression about the system, but despite the limitations of this initial analysis, it could be inferred that X2 may possibly be the most active instrument in cutting dentin walls. One explanation why measured torque values are lower for X1 compared to X2 lies in the fact that the diameter at the apex of the simulated root canal used was approximately 0.20mm and that is a larger size than the diameter that X1 has at the tip (0.17mm). However, this condition regarding canal size is somewhat comparable to what it may occur clinically because the manufacturer suggests the use of Pathfile P1 and P2 before starting with the sequence of ProTaper Next, in order to create a glide path, resulting in an apical diameter of at least 0.16mm (22).

Interestingly, the expected stationary torque to failure for X1 was very low and the mean peak torque generated for the group with the lowest value (350rpm/4 ins) was 80% higher. However, it has to be taken into consideration that this torsional fracture was tested at 3mm from the tip of the instrument and a proper glide path would prevent the file tip to have significant frictional wall contact.

During the tests it appeared that the instruments X1 and X2 had a tendency to cut more dentin as compared to other instruments. X3, X4 and X5 were more likely to smoothen canal walls than actually cutting. This may suggest the possibility that the instruments X1 and X2 are shaping files and X3, X4 and X5 may be described as finishing files.

In relation to the peak force applied, from the data in Fig. 2, it is apparent that instrument X5 showed the highest value compared to all the other instruments in all groups. Again, this result was somewhat expected for an instrument with the largest diameter of the series. According to the study of Peters & Barbakow (16) instruments of larger diameter require higher apical forces to penetrate deeper into the root canals.

An unexpected result was that the instrument X5, despite having greater apical force, showed low peak torque values in all groups. The expected would be the greater the force, the greater the necessary torque to cause the instrument to begin to rotate inside the root canal (16). A possible explanation for this inverse relationship may be the fact that the manufacturer claimed that the off-centered rectangular cross section gives the file a different movement through the root canal, changing the envelope of motion and the compressibility of the file.

All instruments were used in an in-and-out motion and it was apparent that when files were used with three such movements to reach WL significant more torque and more force were generated than with four. It seems possible that these results are due to a more passive shaping technique when using four "in-and-out" movements and a more aggressive one with three.

In conclusion, under the present experimental conditions of simulated root canal preparation, X2 was associated with the highest torque while X5 showed the highest positive force and X1 and X2 the highest peak negative force. This finding suggests that special care should be exercised using X2 because this file may experience significant contact with the canal walls

Bearing in mind the limitations of this *in vitro* study, the results also suggest that ProTaper Next instruments should be used at higher rotational speed than previously suggested for other rotaries and with a gentler movement in order to have the lowest levels of torque. Future experiments should include the use of definite brushing movement as well as dimensional and geometrical characterization of ProTaper Next instruments.

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Figure legends

Figure 1. Distribution of mean and standard deviation of peak torque (Ncm) for each instrument according to rotational speed (250, 300 or 350 rpm) and insertion pattern (3 or 4 in-and-out movements). For comparison, mean stationary torque at failure at 3 mm from the tip for each instrument is represented in the dashed lines.

Figure 2. Distribution of mean and standard deviation of positive and negative peak force (N) for each instrument according to rotational speed (250, 300 or 350 rpm) and insertion pattern (3 or 4 in-and-out movements).

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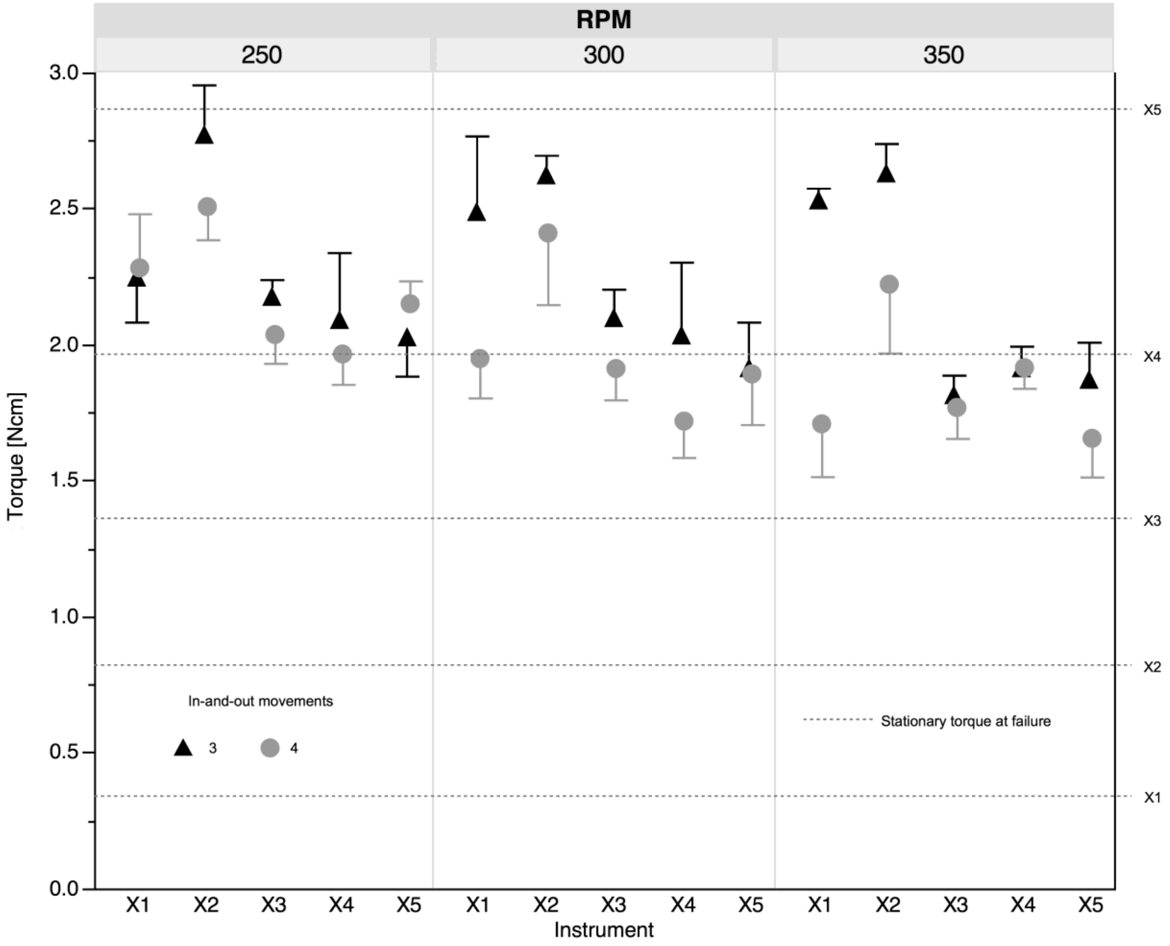
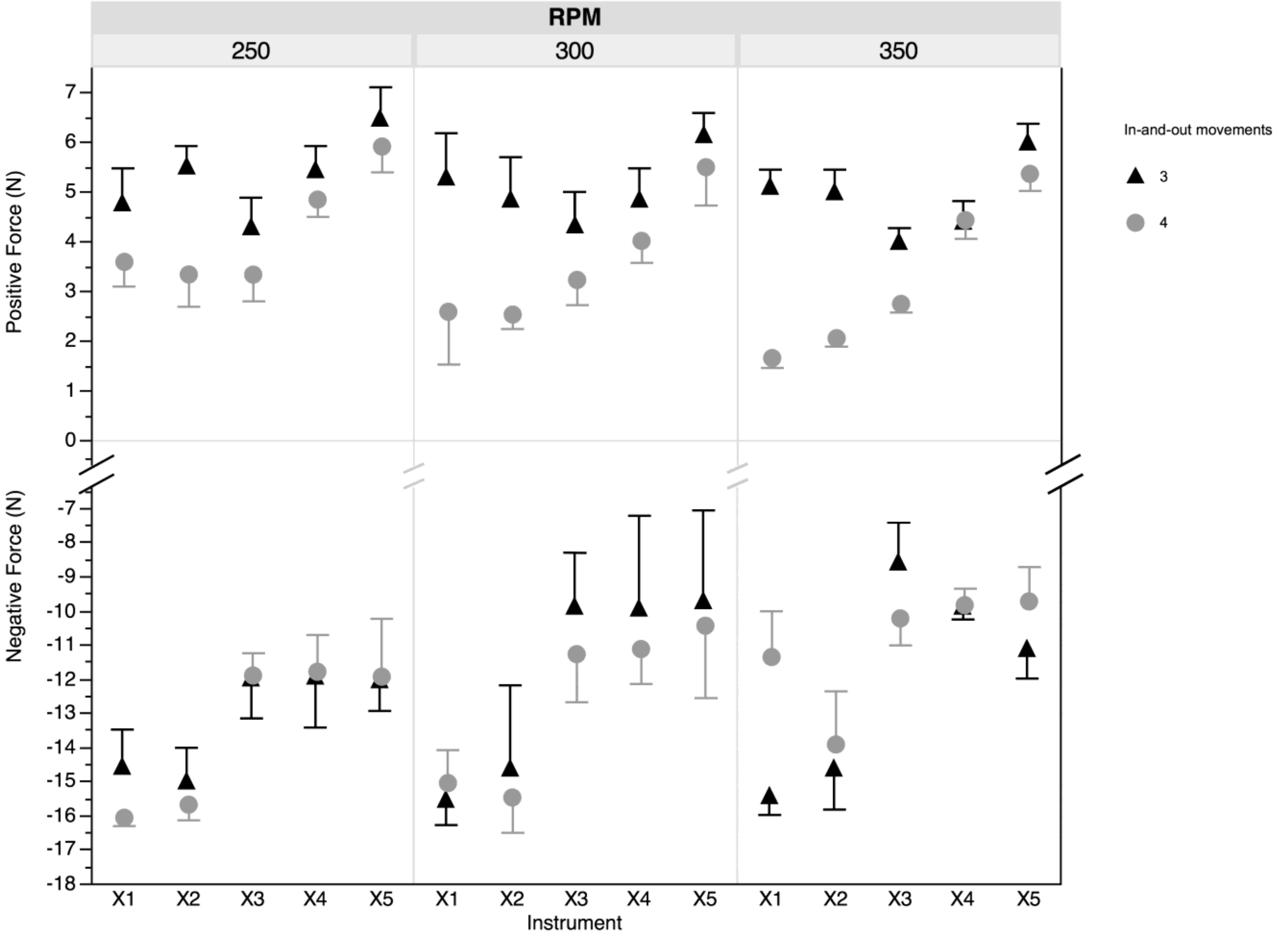
Table 1: Means (±standard deviation) of angular deflection and torque at failure (n=6 per group).

	Instruments				
	X1	**X2**	**X3**	**X4**	**X5**
Torque at failure (Ncm)	0.35 (±0.04)^{a}	0.82 (±0.11)^{a}	1.37 (±0.06)^{a}	1.96 (±0.26)^{b}	2.87 (±0.22)^{c}
Angular deflection (°)	335.33 (±47.12)	290.67 (±18.41)	330.17 (±20.80)	338.50 (±34.66)	424.5 (±48.33)

Differences compared to torque during simulated canal preparation: a=significantly higher; b=no significance; c=significantly lower.




RPM
250
300
350
Positive Force (N)
Negative Force (N)
7
6
5
4
3
2
1
0
-7
-8
-9
-10
-11
-12
-13
-14
-15
-16
-17
-18
X1
X2
X3
X4
X5
Instrument
In-and-out movements
▲ 3
● 4

CONSIDERAÇÕES FINAIS

4. **CONSIDERAÇÕES FINAIS**

Os resultados obtidos neste trabalho dão suporte às seguintes considerações:

A análise química semiquantitativa dos fios estudados indicou que os mesmos possuem a mesma média de conteúdo de Ni e Ti, próxima à proporção equiatômica de 1:1. Ambos os fios apresentaram um pequeno excesso de Ni, podendo responder bem a tratamentos termomecânicos em baixas temperaturas, com possibilidade de melhora das propriedades de superelasticidade e efeito memória de forma.

Os resultados da análise por espectroscopia de energia dispersiva de raios-X, no primeiro estudo, revelaram que a austenita, fase β, foi a fase predominante nos dois fios avaliados. Entretanto, picos de martensita B19’ e fase R foram identificados no difratograma do fio MW, indicando que, em adição à austenita, pequenas quantidades destas fases estavam presentes neste espécime. Estas informações foram compatíveis com as mais altas temperaturas de transformação encontradas pela calorimetria exploratória diferencial no fio MW.

É importante observar que a média dos valores de microdureza Vickers obtidos no primeiro estudo mostrou que MW é tão resistente quanto CW. A importância deste resultado reside no fato de que os instrumentos produzidos com o fio MW devem possuir resistência comparável àqueles produzidos a partir do fio convencional.

A análise das curvas de carga e descarga demonstra que o patamar superelástico do espécime MW foi alcançado sob menores valores de tensão. Além disso, a tensão no patamar manteve-se relativamente constante até que 6% de alongamento fossem atingidos. Já no espécime CW, esta tensão tendeu a um aumento nesta mesma região da curva. Este aumento pode estar relacionado a certa quantidade de deformação plástica no material. Tal deformação ocorre quando as ligas NiTi superelásticas são alongadas a temperaturas mais altas que a temperatura Af e a quantidade de deformação por escorregamento aumenta com o aumento da temperatura de teste. Este deve ser o caso de CW, que apresentou menores temperaturas de transformação em relação ao MW. A ocorrência de deformação por escorregamento durante o alongamento superelástico também é responsável pela maior histerese de tensão exibida pelo CW comparado ao MW. Isto ocorre porque mais defeitos de rede são introduzidos no CW que no MW, durante os ciclos de carga e descarga a que são submetidos. Quando esta explicação é aplicada aos instrumentos endodônticos fabricados a partir destes fios é possível entender por que aqueles em que o MW foi utilizado apresentaram maior resistência à fadiga. O tratamento termomecânico aplicado ao MW leva a uma maior eficiência no comportamento superelástico com menor geração e acúmulo de defeitos de rede em cada ciclo de carga e descarga.

No segundo estudo, a partir dos ensaios de tração até ruptura, foi possível verificar para ambos os fios, os quatro estágios associados com a deformação uniaxial da liga NiTi superelástica. O primeiro estágio correspondendo à deformação elástica da austenita; o segundo relativo ao patamar associado à transformação martensítica induzida por tensão; o terceiro estágio refletindo a deformação elástica da martensita, enquanto o quarto estágio correspondeu à deformação plástica da martensita até a ruptura final. Verificou-se que a principal diferença nas propriedades de ambos os fios se refere ao menor módulo de elasticidade aparente do MW em relação ao CW. A maior resistência do MW confirmada pelos resultados de microdureza Vickers poderia levar a instrumentos com maior resistência torcional, todavia neste estudo, não foi possível a realização de ensaios de torção até a ruptura.

Os resultados dos ensaios de flexão em três pontos indicaram que a força necessária para flexionar o MW foi menor que aquela aplicada na flexão do CW, até que maiores deslocamentos fossem alcançados. Estes resultados foram consistentes com os resultados dos ensaios de tração até ruptura, com relevância para o fato de que as deformações por flexão, em oposição às deformações por tração uniaxial, são o tipo carregamento mais prevalente durante o uso clínico dos instrumentos endodônticos, durante o processo de limpeza e formatação de canais radiculares curvos. A observação de que forças menores são requeridas para flexionar MW, em níveis intermediários de deslocamentos verticais, é uma indicação de que as mudanças estruturais, associadas com a fadiga rotativa do MW, devem ser menores que aquelas do fio de NiTi convencional. Esta hipótese pode ser confirmada pelos ensaios de flexão rotativa, onde a resistência à fadiga do MW foi significativamente maior que a do fio CW. De fato, a média do número de ciclos até a fratura do MW foi aproximadamente três vezes maior que a do CW.

A maior flexibilidade dos instrumentos produzidos a partir do fio MW deve ser atribuída ao seu baixo módulo de elasticidade, o qual pode estar associado a uma reorientação induzida por tensão nas fases R e martensita B19’, já presentes no MW. A ciclagem térmica sob tensão também é uma forma de “treinamento” da liga a qual facilita a formação das mesmas variantes de martensita em carregamento. Este “treinamento” faz com que a transformação da martensita induzida por tensão no fio MW seja alcançada sob baixos níveis de tensão, introduzindo um fator adicional que contribui na melhora da flexibilidade dos instrumentos produzidos a partir deste fio.

A análise por microscopia eletrônica de transmissão evidenciou finas variantes de martensita nos espécimes não deformados. Assim, é possível que o baixo módulo de elasticidade aparente do MW e a menor força requerida para flexioná-lo em uma mesma amplitude, em relação ao CW, sejam o resultado de reorientação induzida por tensão nas variantes de fase R e martensita B19’, presentes no MW, refletindo o melhor comportamento em fadiga observado. Da mesma forma, a presença de grande número de contornos de variantes e de maclas na martensita no MW, facilita a nucleação de trincas, mas retarda a propagação das mesmas devido ao grande número de interfaces presentes. Estas interfaces proporcionam a formação de trincas secundárias na matriz, dissipando assim a energia necessária para a propagação das mesmas, em um sistema de trincas altamente ramificado. Este pode ser um dos mecanismos responsáveis pela vida útil dos instrumentos rotatórios de NiTi na prática clínica.

Em relação aos instrumentos endodônticos de NiTi ProTaper Next™, produzidos a partir do fio MW, no geral, apresentaram valores mais altos de torque dinâmico em relação ao torque à fratura (ADA 28 / ISO3630-1). Tal fato sinaliza a importância de se realizar um cuidadoso "*glide path*", ou seja, acesso direto com trajetória livre do canal até o limite apical a fim de se prevenir fratura por torção.

Considerando as condições experimentais deste estudo, o instrumento X2 foi associado a maiores valores de torque dinâmico, enquanto o instrumento X5 apresentou maiores valores de força apical positiva. Os instrumentos X1 e X2 demonstraram, por sua vez, os maiores picos de força negativa indicando que sofrem um efeito de apreensão ("*threadin-in" effect*) no momento em que são removidos do interior do canal.

A relação destes resultados com o uso clínico encontra-se no fato de que um cuidado diferenciado deve ser tomado quando da utilização dos instrumentos X2, pois estes instrumentos experimentam um contato maior com as paredes do canal radicular uma vez que apresentam maiores valores de torque em trabalho. Além disso, velocidades mais altas e inserções suaves mostraram-se vantajosas durante o teste de torção dinâmico o que suporta as seguintes direções de uso: realização de um cuidadoso acesso direto com alcance da patência e alargamento do canal radicular ("*glide path"*), utilização de uma velocidade mínima de 300 rpm, inserções suaves e em maior número durante a obtenção do comprimento de trabalho.

Pesquisas futuras devem incluir o estudo dos novos tratamentos termomecânicos a que têm sido submetidas as ligas NiTi. Quanto aos instrumentos ProTaper Next™, seria interessante a realização de estudos adicionais com a utilização do movimento de pressão lateral como também a análise de suas características geométricas e dimensionais.

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