UNIVERSIDADE DE SÃO PAULO
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Murilo Priori Alcalde

Comparison of the mechanical properties of engine-driven Nickel-Titanium instruments manufactured by different thermal treatments

Comparação das propriedades mecânicas de instrumentos mecanizados de Níquel-Titânio fabricados com diferentes tratamentos térmicos

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Orientador: Prof. Dr. Clovis Monteiro Bramante

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FOLHA DE APROVAÇÃO

Murilo Priori Alcalde

DADOS CURRICULARES

14 de janeiro de 1988	Nascimento Jaú - SP
Filiação	Jorge Luiz Alcalde Eliana Alves Priori Alcalde
2008 - 2011	Graduação em Odontologia Universidade de São Paulo (Faculdade de Odontologia de Bauru – FOB - USP)
2012 – 2013	Especialização em Endodontia pela Universidade de São Paulo (FOB – USP)
2013 - 2015	Pós-graduação, mestrado, área Endodontia Faculdade de Odontologia de Bauru Universidade de São Paulo (FOB – USP)
2015 - 2018	Pós-graduação, doutorado, área Endodontia Faculdade de Odontologia de Bauru Universidade de São Paulo (FOB – USP)

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RESUMO

Comparação do comportamento mecânico de instrumentos mecanizados de Níquel-Titânio fabricados com diferentes tratamentos térmicos

O objetivo deste estudo foi avaliar o comportamento mecânico de instrumentos mecanizados de Níquel-Titânio (NiTi) fabricados com diferentes tratamentos térmicos. Na primeira parte do estudo foram utilizados 60 instrumentos reciprocantes para a realização dos teste de fadiga cíclica flexural e torcional (n=20): Reciproc R25 (REC 25.08), Unicone L25 (UNC 25.06) e Prodesign R 25 (PDR 25.06). O teste de fadiga cíclica flexural foi realizado com o objetivo de mensurar o tempo para a fratura dos instrumentos em um canal artificial de aço inoxidável com curvatura com 60º de angulação e 5 mm de raio (n=10). Para o teste de torção, os 3 mm inicias das pontas dos instrumentos foram fixadas em um aparelho de torção (n=10), de acordo com a norma ISO 3630-1. Este teste foi realizado com o objetivo de mensurar o torque máximo e o ângulo de rotação suportado pelos instrumentos até sua fratura. Adicionalmente, todos os fragmentos dos instrumentos fraturados foram examinados em microscopia eletrônica de varredura (MEV) para avaliar as características topográficas da superfície da área da fratura. A análise-estatística foi realizada utilizando o teste de análise de variância com um fator ANOVA e teste de Tukey, o nível de significância foi de 5%. Os resultados de fadiga cíclica flexural demonstraram que o PDR 25.06 apresentou maior tempo para a fratura do que os outros grupos ($P<0.05$). O instrumento REC 25.08 apresentou maior resistência a fadiga cíclica flexural do que o UNC 25.06 ($P<0.05$). O teste de torção demonstrou que o PDR 25.06 apresentou significantemente menor resistência torcional do que o REC 25.08 e UNC 25.06 ($P<0.05$). Além disso, não houve diferença entre REC 25.08 e UNC 25.06. Com relação ao ângulo de rotação, o PDR 25.06 e UNC 25.06 apresentaram diferença significante quando comparado ao REC 25.08. Não houve diferença entre PDR 25.06 e UNC 25.06 ($P>0.05$). Todos os instrumentos apresentaram característica topográficas típicas de fadiga cíclica flexural e torcional. Na segunda parte deste estudo avaliou-se a fadiga cíclica flexural e torcional dos instrumentos reciprocantes Reciproc Blue R25 (RB 25.08), WaveOne Gold Primary (WOG 25.07) e Prodesign R 25 (PDR 25.06) (n=20). O teste de fadiga cíclica

flexural foi realizado com dispositivo descrito anteriormente, utilizando curvaturas de 60º e 5 mm de raio (n=10). O teste de torção foi realizado de acordo com a norma ISO 3630-1 (n=10). Todos os fragmentos dos instrumentos fraturados foram examinados em microscopia eletrônica de varredura (MEV) para avaliar as características topográficas da superfície da área da fratura. A análise-estatística foi realizada utilizando o teste de análise de variância com um fator (ANOVA) e teste de Tukey, o nível de significância foi de 5%. Os resultados de fadiga cíclica flexural demonstraram que o PDR 25.06 apresentou a maior resistência a fadiga cíclica flexural do que dos outros grupos (P<0.05). O RB 25.08 apresentou maior tempo para à fratura do que o WOG 25.07 (P<0.05). O teste de torção, o PDR 25.06 apresentou a menor resistência à torção do que os outros grupos (P<0.05). Não houve diferença significante entre RB 25.08 e WOG 25.07 (P>0.05). Em relação ao ângulo de rotação, o PDR 25.06 apresentou maiores valores quando comparados com RB 25.08 e WOG 25.07. O RB 25.08 apresentou diferença significante quando comparado com WOG 25.07. Todos os instrumentos apresentaram características topográficas típicas de fadiga cíclica flexural e torcional. Na terceira parte deste estudo avaliou-se a resistência torcional de instrumentos empregados para patência do canal radicular, de acordo com a norma ISO 3630-1. Foram utilizados 56 instrumentos rotatórios (n=8): Logic 25.01 (LOG 25.01), Logic CM 25.01 (LOG CM 25.01), Proglider 16.02 (PGD 16.02), Hyflex GPF 15.01, 15.02, 20.02 (HGPF) e Mtwo 10.04. Os fragmentos dos instrumentos fraturados foram examinados em microscopia eletrônica de varredura (MEV) para avaliar as características topográficas da superfície da área da fratura. A análise-estatística foi realizada utilizando o teste de análise de variância com um fator ANOVA e teste de Tukey, o nível de significância foi de 5%. Os resultados demonstraram que o LOG 25.01 apresentou significantemente maior resistência torcional do que os demais grupos (P<0.05). O grupo da PGD 16.02 apresentou diferença significante quando comparado com HGPF 15.01 e 15.02 (P<0.05). O LOG CM 25.01 apresentou maior resistência torcional do que o grupo do HGPF 15.01 e 15.02 (P<0.05). Não houve diferença significante entre os intrumentos Mtwo 10.04 e HGPF 15.01, 15.02 e 20.02. Com relação ao ângulo de rotação, o LOG CM 25.01 e HGPF 15.01 apresentaram os maiores valores (P<0.05). O PGD 16.02 apresentou o menor valor de todos os grupos (P<0.05) seguido pelo Mtwo 10.04. O LOG 25.01 apresentou

maiores ângulos de rotação do que o PGD 16.02 e Mtwo 10.04 ($P<0.05$). Todos os instrumentos apresentaram característica topográficas típicas de fadiga torcional. As características da secção transversal, tipo de núcleo, taper e o tratamento térmico possuem forte influência sobre as propriedades mecânicas dos instrumentos de NiTi. No entanto, o tratamento térmico é um dos fatores primordiais para maior flexibilidade dos instrumentos. O instrumento PDR 25.06 apresentou maior resistência à fadiga cíclica flexural e maior ângulo de rotação no teste de torção do que todos os outros instrumentos reciprocantes avaliados. No entanto, menor resistência torcional. Os intrumentos LOG 25.01 apresentaram maior resistência torcional e o LOG CM 25.01 maiores ângulos de rotação.

Palavras-chave: Níquel-Titânio; Instrumentos rotatórios; Instrumentos reciprocantes; Tratamento térmico; Fadiga cíclica flexural; Fadiga torcional.

ABSTRACT

Comparison of the mechanical properties of engine-driven Nickel-Titanium instruments manufactured by different thermal treatments

The aim of this study was to evaluate the mechanical properties of engine-driven Nickel-Titanium instruments manufactured by different thermal treatments. In the first part of this study, 60 reciprocating instruments were used (n=20): Reciproc R25 (REC 25.08), Unicone L25 (UNC 25.06) e Prodesign R 25 (PDR 25.06). The cyclic flexural fatigue resistance was performed measuring the time to failure in an artificial stainless steel canal with a 60º angle and a 5 mm radius of curvature (n=10). The torsional test was performed according to ISO 3630-1, measuring the torque and angle of rotation at failure in the 3 mm from the tip portion (n=10). Additionally, the fractured surface of each instrument was examined by scanning electron microscopy (SEM) to assess the topographic features of the fractured surface. Data were analyzed using one was analysis of variance ANOVA and Tukey test, the level of significance was set at 5%. The results of the cyclic flexural fatigue showed that PDR 25.06 presented significantly higher values than the other groups ($P<0.05$). REC 25.08 showed higher fatigue resistance than UNC 25.06 ($P<0.05$). In relation to the torsional test, the PDR 25.06 presented the lowest torque load than REC 25.08 and UNC 25.06 ($P<0.05$). In addition, there was no significant difference between REC 25.08 and UNC 25.06 ($P>0.05$). The PDR 25.06 and UNC 25.06 showed higher angular rotation until fracture than REC 25.08 ($P<0.05$). No difference was found between PDR 25.06 and UNC 25.06. All the instruments showed typical topographic features of cyclic flexural and torsional fatigue. The second part of this study evaluated the cyclic flexural and torsional fatigue resistance of reciprocating instruments Reciproc Blue (RB 25.08), WaveOne Gold Primary (WOG 25.07) and Prodesign R 25 (PDR 25.06) (n=20). The cyclic flexural fatigue test was performed with the same previsoly described device, using a root curvature with 60º and a 5 mm radius (n=10) and the torsional test was performed according to ISO 3630-1 (n=10). The fractured surface of each instrument was examined by scanning electron microscopy (SEM). Data were analyzed using one was analysis of variance ANOVA and Tukey test, the level of significance was set at 5%. The results of the cyclic

flexural fatigue test showed that PDR 25.06 presented significantly higher values than the other groups (P<0.05). RB 25.08 showed higher fatigue resistance than WOG 25.07 (P<0.05). The torsional test showed that PDR 25.06 had lowed torsional load (P<0.05). No difference was found between RB 25.08 and WOG 25.07 (P>0.05). In relation the angular rotation, the PDR 25.06 showed higher angular rotation values than RB 25.08 and WOG 25.07 (P<0.05). RB 25.08 presented higher angular values than WOG 25.07 (P<0.05). All the instruments showed typical topographic features of cyclic flexural and torsional fatigue. The third part of this study was to evaluate the torsional fatigue resistance of NiTi rotary glide path instruments. The torsional test was performed according to ISO 3630-1, measuring the torque and angle of rotation at failure in the 3 mm from the tip portion. A total of 56 glide path instruments were used (n=8): Logic 25.01 (LOG 25.01), Logic CM 25.01(LOG CM 25.01), Proglider 16.02 (PGD 16.02), Hyflex GPF 15.01, 15.02, 20.02 (HGPF) and Mtwo 10.04. The fractured surface of each instrument was examined by scanning electron microscopy (SEM). Data were analyzed using one was analysis of variance ANOVA and Tukey test, the level of significance was set at 5%. The results showed that LOG 25.01 had a significantly higher torsional load than the other groups (P<0.05). The PGD 16.02 had significantly difference in comparison with HGPF 15.01 and 15.02 (P<0.05). LOG CM 25.01 had higher torsional load than HGPF 15.01 and 15.02 (P<0.05). No difference was found among Mtwo 10.04, HGPG 15.01, 15.02 and 20.02. In relation the angular rotation, LOG CM 25.01 and HGPF 15.01 presented the highest values (P<0.05). PGD 16.02 had the lowest values (P<0.05) followed by Mtwo 10.04. LOG 25.01 had higher angle of rotation than PGD 16.02 and Mtwo 10.04 (P<0.05). All the instruments showed typical topographic features of torsional fatigue.

Keywords: Nickel-Titanium; Rotary instruments; Reciprocating instruments; Thermal treatments; Cyclic flexural fatigue; Torsional fatigue.

LIST OF ABREVIATIONS AND ACRONYMS

%	percentage
<	less than
>	greater than
#	tip diameter
º	degree
SEM	scanning electron microscopy
mm	millimeter
n	number
NiTi	Nickel-Titanium
P	statistical significance
rpm	rotation per minute
μm^2	square micrometer
CW	clockwise direction
CCW	counterclockwise direction
Ti_3Ni_4	Titanium nitride
M-Wire	Martensitic wire
B2	Austenite phase
B19	Martensite phase
PDR	Prodesign R
RB	Reciproc Blue
WOG	WaveOne Gold
UNC	Unicone
CM-Wire	controlled memory wire
R-Phase	Romboedric Phase
D3	three millimeters from the instrument tip
D5	five millimeters from the instrument tip

SUMMARY

1 INTRODUCTION

1 INTRODUCTION

The stainless steel hand files were the main and the most method used for root canal preparation during many decades (PETERS, 2004; HULSMANN; PETERS; DUMMER, 2005). However, the low flexibility of the stainless steel became the root canal preparation of curved canals a challange (WALIA et al., 1988; PETERS, 2004), increasing the risks of procedure errors such as root canal perforations, working lenght loss or the root canal patency, ledging, etc (BAUMANN, 2004; CHEUNG; LIU, 2009).

The Nickel-Titanium (NiTi) was introduced in endodontics by Walia et al. (1988). The authors showed the NiTi files were two or three time more flexible than stainless steel hand files and greater resistance by torsion. After this publication, there was a revolution on the root canal preparation and many studies were published showing that the NiTi hand files favor a greater canal centrability and safety for root prepararion of curved canals than stainless steel (PORTO CARVALHO; BONETTI; GAGLIARDI BORGES, 1999; PETERS; SCHONENBERGER; LAIB, 2001; PETTEIETE; DELANO; TROPE, 2001; PETERS, 2004; CHEUNG; LIU, 2009).

The NiTi present low stiffness, high springback, shape memory and superelastic properties (BURSTONE; QIN; MORTON, 1985; MIURA et al., 1986; WALIA et al., 1998; THOMPSON, 2000). The shape memory is defined as the ability of the NiTi to deform and recover to the original shape. The superelasticity is the ability of the NiTi alloy to suffer large deformation previously to fracture. These features exist because of the phases of the NiTi alloy, the Austenite (B2) and Martensite (B19) (TORRISI, 1999; THOMPSON, 2000). The austenite is the initial phase of the NiTi where the atoms are in a cubic cristallyne form. This phase is responsable to the shape memory and can be induced by heating or the absence of tension. When the NiTi is submited by tension or cooling, the atoms are rearranged into a monoclinic crystalline strutucre with superelasticity, the Martensite phase (TORRISI, 1999; THOPSON, 2000). The shape memory effect is a direct consequence of reversible transformation between austenite and martensite, differently from stainless steel (THOMPSON, 2000; BAUMANN, 2004).

The NiTi endodontic instruments allowed that curved canals could be mechanically prepared using a continuous rotary motion (THOMPSON, 2000; BAUMAN, 2004; HAAPASSALO; SHEN, 2013). The flexibility is beneficial for maintaining the original shape of root canals especially in severe curvature (THOMPSON, 2000) and favors an adequate torsional and flexural resistance, reducing the risk of the instrument separation (THOPSON, 2000; BAUMAN, 2004; ZHOUG; PENG; ZHENG, 2013).

The first NiTi rotary system was designed by Dr. John McSpadden and came to the market in 1992. In 1994, Dr. Ben Johnson introduced a line of files, which became known as the Profile 0.04 tapered series. Afterwards, was introduced the ProFile 0.06 tapers and several others rotary systems manufactured by conventional NiTi alloy were developed (HAAPASSALO; SHEN, 2013). The NiTi rotary instruments manufactured by conventional NiTi are used clinically in the austenitic phase (body temperature) and when submitted by tension stress, the martensitic transformation occurs, given rise to a more elastic material (TORRISI, 1999). Therefore, the rotary system began to change how the dentists viewed the root preparation of curved canals (HULSMANN; PETERS; DUMMER, 2005; PLOTINO et al., 2009; HAAPASSALO; SHEN, 2013; SHEN et al., 2013).

Despite the NiTi rotary files had improved the quality and safety of the root canal preparation of curved canals, an unexpected instruments separation can occur, which is caused by flexural and torsional stress (SATTAPAN et al., 2000; PEDULLA et al., 2015). The flexural fatigue occurs by repeated compressive and tensile stresses when the instrument rotates in a curved canal (SATTAPAN et al., 2000). Torsional failure occurs when the tip of the instrument is locked in the canal whilst the shank continues to rotate (SATTAPAN et al., 2000). This can happen in straight or curved canals, especially in the preparation of narrow and constricted canals when the file is susceptible to high torsional loads (SATTAPAN et al., 2000; WYCOOFF; BERZINS, 2012). Torsional resistance is characterized by a maximum torsional load and angle of rotation. This property reveals the ability of the file to twist before fracture (ELNAGHY; ELSAKA, 2015).

During the years, the manufacturers have been proposed changes in the cross-section design, taper, rake angle, diameter of core, helical angle, pitch, tips design and radial land of the NiTi instruments to improve their mechanical properties and clinical performance (PLOTINO et al., 2009; SHEN et al., 2013; ZHOU; PENG; ZHENG, 2013; CAPAR et al., 2015a; ÖZYÜREK, 2016). In addition, it was reported that the surface condition of the NiTi instruments contributes to fatigue resistance because most fatigue failures nucleate from the surface, especially in the presence of high stress amplitude or surface defects (BAHIA; BUONO, 2005; LOPES et al., 2010). Thus, manufacturers began to focus on other methods to increase the resistance to file separation, such as electropolishing of the NiTi (LOPES et al., 2010; GUTMANN; GAO, 2012; HAAPASALO; SHEN, 2013). The BioRaCe was the first rotary system manufactured by grinding and submitted to an electropolishing surface treatment. Some studies showed that this treatment reduces the surface defects and improve the cyclic flexural fatigue resistance (RAPISARDA et al., 2000; TRIPI; BONACCORSO; CONDORELLI, 2006; LOPES et al., 2010; GUTMAN N; GAO et al., 2012; SHEN et al., 2013; KIM et al., 2015).

After the introduction of electropolishing, the manufacturers have been developed several thermal treatments and manufacturing technologies in order to optimize the microstructure of NiTi alloy (HAAPASALO; SHEN, 2013; SHEN et al., 2013). The thermal treatments assist the control of transition temperatures of NiTi alloy and induce a better arrangement of the crystal structure (GAO et al., 2012; SHEN et al., 2013; BRAGA et al., 2014). Previous studies showed that the thermal treatments can increase the percentage of martensite phase (which is known to be more flexible than austenitic NiTi) or R-Phase (an intermediate phase between the austenite and martensite phase), increasing the flexibility and reducing the risk of instrument separation under high stress (SHEN et al., 2012; SHEN et al., 2013; BRAGA et al., 2014; PLOTINO et al., 2014; ELSAKA; ELNAGHY, 2016). In Addition, depending of the thermal treatment the spiral flutes of the endodontic instrument can be manufactured by twisting process instead of grinding due to the presence of R-Phase (LOPES et al., 2013; SHEN et al., 2013).

Currently, there are nearly of six types of thermal treatment used in NiTi instruments: Martensite Wire (M-Wire/ 2007), R Phase NiTi (2008), Controlled Memory Wire (CM-Wire/ 2010), Blue Wire (2013), Gold Wire (2014) and Max-Wire (2015) (SHEN et al., 2013; PLOTINO et al., 2014b; GAGLIARDI et al., 2015; PIRANI et al., 2016; FKG DENTAIRE, 2016). In general, the thermal treatments promote greater flexibility than conventional NiTi due to the specific temperature variation applied during the manufacturing process, inducing a different microstructure arrangement of the NiTi and better mechanical properties (PETERS et a., 2012; SHEN et al., 2013; PLOTINO et al., 2014b; KAVAL et al., 2016; ÖZYÜREK, 2016; KAVAL et al., 2017).

Despite the several improvements on the instruments designs and the metallurgy of the NiTi, the rotary instruments separation continues to be a concernment for the clinicians (SHEN et al., 2013; PEDULLA et al., 2016; KAVAL e al. 2017; DE-DEUS et al., 2017). In 2008, Yared proposed a new concept of the root canal preparation using single instrument and a novel kinematics, called reciprocating motion (DE-DEUS et al., 2010; KIM et al., 2012). The reciprocating motion involves rotation in counterclockwise (CCW) and clockwise (CW) directions with 120° of difference between the two movements and completes 360° in 3 cycles (KIM et al., 2012; PEDULLA et al., 2016; KARATAS et al., 2017). This kinematics reduces the screwing-in effect and the mechanical stress of the instruments. In addition, has been shown to be safer than rotary motion during root preparation of curved and constricted root canals, reducing cyclic flexural and torsional fatigue (VALERA-PATIÑO et al., 2010; KIM et al., 2012; KARATAS et al., 2016).

The two firsts reciprocating instruments was introduced on the market in 2011, the Reciproc (VDW GmbH, Munich, Germany) and Wave-One (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA). The Reciproc system is composed by three instruments with S-Shaped cross-section and present three sizes and tapers: #25, #40, and #50 tip sizes and tapers of 0.08, 0.06, and 0.05, respectively. The Wave-one also consists in three files with triangular cross-section and also three instruments sizes and tapers: #21, #25, and #40 sizes and tapers of 0.06, 0.08 and 0.08 taper. The Reciproc works at 300 rpm speed in 150° CCW and 30° CW direction, while Wave-One operates at 350 rpm in in 170° CCW and 50° CW direction (KIM et al., 2012). These both systems were manufactured by special thermally treated NiTi alloy, called M-Wire. The M-Wire is composed of 508 Nitinol which has undergone a proprietary method of treatment, than submit the raw wire under specific tension and heat treatments at various temperatures resulting in a material that includes some portion in both the martensitic and the premartensitic R phase while maintaining a pseudoelastic state (PLOTINO et al., 2014a). Previous studies showed that M-Wire alloy is more flexible and cyclic flexural fatigue resistant than conventional NiTi (PEREIRA et al., 2012; SHEN et al., 2013).

In 2014, the Unicone (Medin, Nov e Mesto na Morave, Czech Republic) was introduced on the Brazilian market. This reciprocating instruments with convex triangular cross-section; it consists of 3 instruments: #20, #25 and, #40 tip sizes and 0.06 taper (SILVA et al., 2016a). According to the manufacturer, it is manufactured proprietary thermal treatment, which offer highly flexible NiTi. In 2015, a Brazilian reciprocating system was introduced, the Prodesign R (Easy Dental Equipment, Belo Horizonte, MG, Brazil). This system has two instruments presenting an S-shaped cross-section: size 25, 0.06 taper and size 35, 0.05 taper. Additionally, these instruments are made from similar thermal processing of CM-Wire, favoring higher flexibility than conventional NiTi (SILVA et al., 2016b).

Recently, a new generation of the Reciproc and Wave-one instruments was introduced. In 2016, the Wave-One Gold (WOG) was introduced on the Brazilian market. This system uses the same reciprocating motion of the WaveOne file (M-Wire). However, the WOG instruments are manufactured with a new thermal treatment procedure called Gold treatment (WEBBER, 2015; PLOTINO et al., 2017). This system presents different designs and sizes: 20, 25, 35 and 45 tip sizes and tapers of 0.07, 0.07, 0.06 and 0.05, respectively. The cross-sectional design of these instruments is a parallelogram design with 2 cutting edges (WEBBER, 2015). In the Gold thermal process, the NiTi instrument undergoes a slow heating-cooling process that creates Ti_3Ni_4 precipitates dispersed on the NiTi surface (HIEAWY et al., 2015), inducing martensitic transformation to occur in 2 steps and increasing the flexibility (ÖZYÜREK 2016; TOPÇUOĞLU et al., 2017; PLOTINO et al., 2017). The Reciproc Blue is an evolution of Reciproc (M-Wire), which present the same S-Shaped cross-section, instrument tip sizes, tapers and the same reciprocating motion than the former. However, the manufacturer replaced the M-Wire alloy with a new thermal treatment called Blue treatment (DE-DEUS et al., 2017). This thermal treatment is a special heating-cooling method that results in instruments with a blue color due to a titanium oxide layer (PLOTINO et al., 2014a; DE-DEUS et al., 2017). This treatment reduces the shape memory alloy of the NiTi and induces the occurrence of martensitic transformation in 2 phases (PLOTINO et al., 2014b; SHEN et al., 2015), increasing the cyclic flexural fatigue resistance and flexibility compared with Reciproc M-Wire instruments (DE-DEUS et al., 2017).

The advances on the metallurgy and kinematics of the engine-driven NiTi instruments improved the safety during root canal preparation of curved and constricted canals (SHEN et al., 2013; PEDULLA et al., 2016). However, some clinical procedures can also concur for the reduction of the mechanical stress of rotary and reciprocating instruments. Some authors reported that creation of a glide path can favor the reduction of shaping errors (D'AMARIO et al., 2013; ELNAGHY; ELSAKA, 2014), prevent instrument fracture (BERUTTI et al., 2009) and reduce the postoperative pain (PASQUALINI et al., 2012). This procedure can be performed with conventional stainless steel hand files or with engine-drive NiTi instruments with small tip sizes and smaller taper (GAMBARINI et al., 2015; USLU et al., 2017).

The NiTi instruments used for glide path preparation are susceptible to torsional failure in the constricted root canals because they are used at the beginning of the preparation (DE-DEUS et al., 2016; ARIAS et al., 2016). Therefore, to minimize this drawback, the manufacturers have developed new instruments with various cross-sections, designs and thermal treatments (KARATAS et al., 2016; USLU et al., 2017). Several NiTi rotary glide path files are manufactured as single-file or multiple-file systems. Examples of single-file glide path instruments include Proglider (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA), Mtwo (VDW, Munich, Germany) and ProDesign Logic (Easy Equipamentos Odontológicos, Belo Horizonte, Brazil).

The Proglider instrument is made of M-wire alloy (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA) has a square cross section design; It has a 0.16 mm tip size and variable taper of between 2% and 8% along the shaft (DE-DEUS et al., 2016). The Mtwo rotary instrument has a 0.10 tip size and 0.04 taper, manufactured from conventional NiTi-Wire and an S-shaped cross-sectional design with double cutting edeges and noncutting tip (ALVES et al., 2012). The Prodesign Logic 25.01 (Easy Equipamentos Odontológicos, Belo Horizonte, Brazil) is new glide path instrument that has a square cross section design, a 0.25 mm tip size and 0.01 taper. This instrument is made of both conventional NiTi Wire and CM-Wire.

The Hyflex GPF (Coltene-Whaledent, Altstätten, Switzerland) is an example of a multiple-rotary pathfinding system composed of three instruments: size 0.15, .01 taper, size 0.15, .02 taper, and size 0.20, .02 taper. The instrument size 0.15, .01 taper is manufactured of conventional NiTi Wire and a triangular cross section; the others instruments are made of controlled memory wire (CM-Wire) and square cross section (CAPAR et al., 2015a).

Currently, there are several engine-driven NiTi instruments for root canal preparation and glide path. Thus, it is important to know the torsional and cyclic flexural resistance of these instruments to provide a safe and suitable clinical use.

The aim of this study was to evaluate the cyclic flexural and torsional fatigue resistance of reciprocating and the torsional fatigue resistance of pathfinding instruments manufactured by different NiTi alloy. For this purpose, the study was performed in tree steps:

- 1st article: to evaluate the cyclic and torsional fatigue (maximum torque load and angular rotation) of the Prodesign R 25.06, Unicone 25.06 and Reciproc 25.08 instruments.
- 2nd article: to evaluate the cyclic and torsional fatigue (maximum torque load and angular rotation) of Prodesign R 25.06, WaveOne Gold 25.07 and Reciproc Blue 25.08 instruments.
- 3rd article: to evaluate the torsional properties (maximum torque load and angle angular rotation of Proglider 16.02, Hyflex GPF (15.01, 15.02, 20.02), Logic 25.01, Logic 25.01 CM and Mtwo 10.04 instruments.

2 ARTICLES

2 ARTICLES

2.1 Article 1 - **Cyclic fatigue and torsional fatigue resistance of reciprocating single files manufactured by different nickel-titanium alloys.**

The article presented in this thesis was published in the Journal of Endodontics. Annex B contains the permission letter to include a published article from the Journal of Endodontics in this thesis.

ARTICLE IN PRESS

Basic Research—Technology

Cyclic and Torsional Fatigue Resistance of Reciprocating Single Files Manufactured by Different Nickel-titanium Alloys

Murilo P. Alcalde, MSc, Mario Tanomaru-Filho, PhD,† Clovis M. Bramante, PhD,* Marco Antonio H. Duarte, PhD,* Juliane Maria Guerreiro-Tanomaru, PhD,† Jader Camilo-Pinto, DDS,† Marcus Vinicius Reis Só, PhD,‡ and Rodrigo Ricci Vivan, PhD**

Abstract

Introduction: The aim of this study was to evaluate the cyclic and torsional fatigue resistance of the following reciprocating single-file systems: ProDesign R 25.06 (Easy Equipamentos Odontológicos, Belo Horizonte, Brazil), Reciproc R25 (VDW GmbH, Munich, Germany), and Unicone L25 (Medin SA, Nové Město in Moravě, Czech Republic). **Methods:** Sixty instruments of the ProDesign R, Reciproc R25, and Unicone L25 systems ($n = 20$) were used. Cyclic fatigue resistance was tested measuring the time to failure in an artificial stainless steel canal with a 60° angle and a 5-mm radius of curvature ($n = 10$). Torque and angle of rotation at failure of new instruments ($n = 10$) in the 3 mm from the tip portion were measured during torsional testing according to ISO 3630-1. The fractured surface of each fragment was examined by scanning electron microscopy. Data were analyzed using 1-way analysis of variance and Tukey tests, and the level of significance was set at 5%. **Results:** The cyclic fatigue resistance values of ProDesign R 25.06 were significantly higher than the other groups ($P < .05$). Reciproc R25 showed higher fatigue resistance than Unicone L25 ($P < .05$). In relation to the torsional test, the ProDesign R 25.06 and Unicone L25 systems showed higher angular rotation until fracture than Reciproc R25 ($P < .05$). However, Reciproc R25 and Unicone L25 showed higher torque load than ProDesign R 25.06 ($P < .05$). Scanning electron microscopic analysis showed similar and typical features of cyclic and torsional failure for all instruments tested. **Conclusions:** ProDesign R presented the highest cyclic fatigue resistance and angular rotation to failure compared with Reciproc and Unicone. However, Reciproc showed higher torsional strength to failure. (*J Endod 2017;■:1–6*)

Key Words

Cyclic fatigue, nickel-titanium, reciprocating systems, torsional resistance

Significance

New reciprocating systems were introduced with different designs and NiTi alloys. Instrument separation can occur, and the causes are flexural and torsional stress. ProDesign R presented higher cyclic fatigue resistance and Reciproc showed higher torsional strength to failure.

Nickel-titanium instruments (NiTi) show flexibility and elasticity to provide safe root canal preparation in curved canals (1, 2). However, unexpected instrument separation can occur, and many variables may contribute to this occurrence. The most common causes are flexural and torsional stress (3, 4).

Cyclic flexural fatigue occurs by repeated compressive and tensile stresses when the instrument rotates in a curved canal (3), which often happens clinically (3, 5). Torsional failure occurs when the tip of the instrument is locked in the canal while the shank continues to rotate (3). This can happen in straight or curved canals, especially in the preparation of narrow and constricted canals when the file is susceptible to high torsional loads (3, 5). Torsional failure is characterized by a maximum torsional load and angle of rotation. This property reveals the ability of the file to twist before fracture (6). Therefore, to minimize this drawback, the manufacturers developed several strategies such as new cross sections, designs, thermomechanical processes, and kinematics (1, 2, 6–8).

The reciprocating motion used in reciprocating single-file systems has been shown to be safe and effective in the preparation of curved root canals, reducing cyclic fatigue, torsional stress, and working time (9–11). Reciproc (VDW GmbH, Munich, Germany) is fabricated from M-Wire alloy. This NiTi alloy shows more flexibility and mechanical strength than NiTi wire (2, 12). M-Wire instruments are produced by transforming an NiTi wire in the austenite phase into the R-phase, an intermediate phase formed during the transformation from martensite to austenite on heating and reverse transformation on cooling the material (2, 12).

Recently, new reciprocating systems were introduced using different designs and NiTi alloys. ProDesign R (Easy Equipamentos Odontologicos, Belo Horizonte, MG, Brazil) has 2 instruments with #25 and #35 tip sizes and 0.06 and 0.05 tapers, presents an S-shaped cross section, and is manufactured by a special thermomechanical process

From the *Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil; †Department of Restorative Dentistry, Araraquara Dental School, São Paulo State University, São Paulo, São Paulo, Brazil; and ‡Department of Restorative Dentistry, Federal University of Rio Grande do Sul, Porto Alegre, Rio Grande do Sul, Brazil.

Address requests for reprints to Dr Rodrigo Ricci Vivan, Bauru School of Dentistry, University of São Paulo, Al Octávio Pinheiro Brisolla, 9-75, 17012-901 Bauru, SP, Brazil. E-mail address: rodrigo.vivan@fob.usp.br
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that controls the memory of the NiTi. These instruments are mainly in the martensite phase, which provides more flexibility, cyclic fatigue resistance (2, 7, 12, 13), and high deformation capacity during torsional testing (12–14). Unicone (Medin, Nov e Mesto na Morave, Czech Republic) is an NiTi (proprietary treatment not reported by the manufacturer) reciprocating instrument with an inactive tip and a convex triangular cross section; it is composed of 3 instruments with #20, #25, and #40 tip sizes and a 0.06 taper size. Some authors have reported that this instrument has low flexibility and a short lifetime during cyclic fatigue test (15, 16).

There are no studies regarding the cyclic and torsional fatigue resistance of ProDesign R. Furthermore, there is no report of the torsional properties of Unicone. The aim of this study was to evaluate the cyclic and torsional fatigue (maximum torque load and angular rotation) of the ProDesign R 25/.06 and Unicone 25/.06 systems and compare them with the Reciproc R25 instrument. The null hypotheses tested were as follows:

1. There are no differences in the cyclic fatigue resistance among the instruments.
2. There are differences in the torsional resistance among the instruments.

Methods

The sample calculation was performed using G*Power v3.1 for Mac (Heinrich Heine, University of Düsseldorf, Düsseldorf, Germany) by selecting the Wilcoxon-Mann-Whitney test of the *t* test family. An alpha-type error of 0.05, a beta power of 0.95, and a ratio N2/N1 of 1 were also stipulated. A total of 8 samples per group were indicated as the ideal size required for noting significant differences. Ten samples per group were used because an additional 20% was calculated to compensate for possible outlier values that might lead to sample loss.

A sample of 60 NiTi instruments (length = 25 mm) of 3 different reciprocating systems (n = 20 per system) were used in this study as follows: ProDesign R (size #25, 0.06 taper), Reciproc R25 (size #25, 0.08 taper), and Unicone L25 (size #25, 0.06 taper). Every instrument was inspected for defects or deformities before being tested under a stereomicroscope (Carl Zeiss, LLC, Oberkochen, Germany) at 16× magnification; none were discarded. All files used were 25-mm long, with 10 instruments of each brand used for cyclic and torsional fatigue testing.

Cyclic Fatigue Test

The static cyclic fatigue tests were performed using a custom-made device that allowed a reproducible simulation of an instrument confined in an artificial curved canal as previously described (17). The artificial canal was manufactured by reproducing the instrument size and taper, thus providing the instrument with a suitable trajectory with a 60° angle of curvature and a 5-mm radius of curvature (Fig. 1*A* and *B*). The curvature of the stainless steel artificial canal was fitted onto a guide cylinder made of the same material (angle of curvature = 60°, radius = 5 mm). Both the arch and the guide cylinder had a 1-mm-deep groove located 5 mm from the top to match the height of the counterangle. The groove served as a guide path for the instrument, which remained curved and free to rotate between the cylinder and external arch.

Ten instruments of each reciprocating system were activated by using a 6:1 reduction handpiece (Sirona Dental Systems GmbH, Bensheim, Germany) powered by a torque-controlled motor (Silver Reciproc, VDW) using the preset programs "Reciproc ALL" and "WaveOne ALL" to activate Reciproc R25 and ProDesign R 25.06 and Unicone L25, respectively. The preset programs were selected according to the manufacturers' instructions. To reduce the friction of the instrument as it came into contact with the artificial canal walls, a special high-flow synthetic oil prepared for lubrication of mechanical parts (Super Oil; Singer Co Ltd, Elizabethport, NJ) was applied. The time from motor activation was recorded and stopped as soon as a fracture was detected visually and/or audibly on a digital timer. During this step, a video recording was performed simultaneously, and the recordings were observed to ensure the accurate time of instrument fracture.

Torsional Fatigue Test

The torsion tests, based on ISO 3630-1 (1992), were performed by using a torsion machine described in detail elsewhere (18). All files used were 25-mm long, and 10 instruments of each system were used to

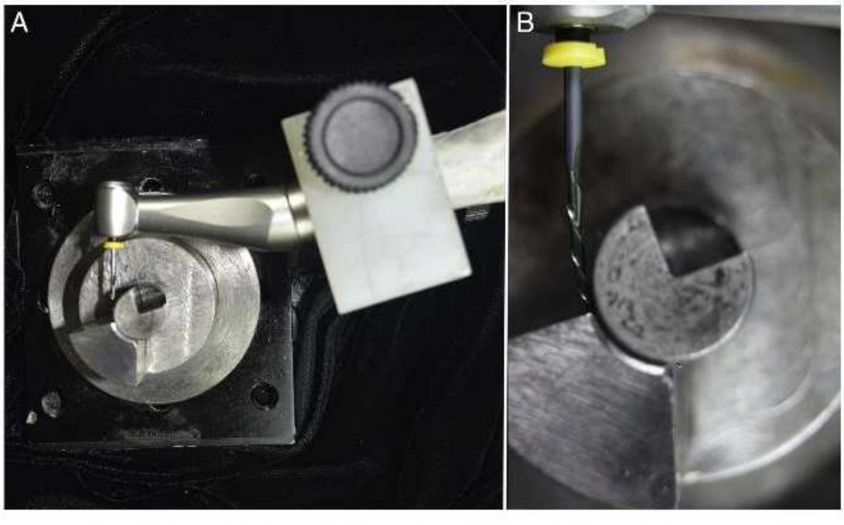

Figure 1. (*A*) The instrument positioned in the cyclic fatigue test device. (*B*) The artificial canal with an angle of curvature of 60° and a radius of 5 mm.

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establish the mean values of torque and maximum angular deflection necessary until rupture.

The torque values were assessed by measuring the force exerted on a small load cell by a lever arm linked to the torsion axis. The rotation angle was measured and controlled by a resistive angular transducer connected to a process controller. Before testing, each instrument handle was removed at the point where the handle was attached to the shaft. The end of the shaft was clamped into a chuck connected to a reversible geared motor. Three millimeters of the instrument tip were clamped in another chuck with brass jaws to prevent sliding. The counterclockwise rotation speed was set to 2 rpm. Continuous recording of torque and angular rotation was monitored, and the ultimate torsional strength and angular rotation (°) were provided by a specifically designed computer program (Analógica, Belo Horizonte, MG, Brazil) and recorded.

Scanning Electron Microscopic Evaluation

The fractured surfaces of 5 instruments of each brand, randomly selected after the cyclic and torsional fatigue test to failure, were examined by scanning electron microscopy (JSM-TLLOA; JEOL, Tokyo, Japan) to look for the topographic features of the fractured instruments. Before scanning electron microscopic (SEM) evaluation, instruments were ultrasonically cleaned to remove debris. The photomicrographs were taken at 250× magnification. Furthermore, additional photomicrographs were taken at 1000× magnification in the center of the fractured surface of the instruments submitted to torsional testing.

Results

The mean and standard deviations of the cyclic and torsional fatigue resistance (torque maximum load and angle of rotation) for each instrument are presented in Table 1. ProDesign R 25.06 had significantly higher cyclic fatigue resistance values than the other groups ($P < .05$). Reciproc R25 showed significantly higher cyclic fatigue resistance than Unicone ($P < .05$).

The maximum torsional strength values of ProDesign R 25.06 were significantly lower than those of Reciproc R25 and Unicone L25 ($P < .05$). Furthermore, there was a significant difference between Reciproc R25 and Unicone L25. In relation to the angular rotation, ProDesign R and Unicone L25 showed a significant difference in comparison with Reciproc R25 ($P < .05$). No difference was found between ProDesign R 25.06 and Unicone L25 ($P > .05$).

SEM Evaluation

Scanning electron microscopy of the fractured surface showed similar and typical features of cyclic fatigue and torsional failure for all brands tested. In the cyclic fatigue test, all the instruments displayed fractured surfaces with microvoids, morphologic characteristics of ductile fracture (Fig. 2A–C). In the torsional test, all the instruments showed concentric abrasion marks and fibrous dimple marks in the center of rotation for torsional failure (Fig. 3A–C and a–c).

Discussion

The cyclic and torsional resistance of NiTi instruments can be affected by instrument size, taper, cross-sectional design, diameter of core, and manufacturing process (1, 12, 19, 20). At present, many changes in instrument design and manufacturing processes have been proposed to improve their mechanical properties and clinical performance (2, 21, 22). Therefore, the aim of this study was to compare the cyclic and torsional resistance of 3 reciprocating files with different designs and manufacturing processes.

The dynamic model approximates a clinical pecking motion accomplished during root canal preparation (23). However, in this study, the static cyclic fatigue model was selected, as used in previous studies (14–16, 24). The static model allows a precise trajectory in the artificial canal and decreases some variables, such as the amplitude of axial movements and speed, which are completely subjective and in a clinical situation their reproduction is unreliable because the axial motion is manually controlled (24). The use of a simulated artificial canal in a stainless steel block was used for cyclic fatigue analysis and has previously been reported (1, 9, 11, 14–17). In this study, the torsional fatigue resistance (maximum torque load and angle of rotation) to fracture was compared. The torsional tests were performed in accordance with ISO 3630-1 as in previous studies (4, 18). After fastening the 3 mm of the tip and shaft of the instruments, a rotational counterclockwise direction rotation was set in a counterclockwise direction for all instruments because of their spiraling flutes (25).

The first result of this study showed that ProDesign R 25.06 had higher cyclic fatigue resistance values than the other groups. Thus, the first null hypothesis was rejected. The ProDesign R, Reciproc, and Unicone instruments have the same tip sizes (#25). However, the tapers differ among them; ProDesign R and Unicone have a taper of 0.06 mm/mm, and Reciproc R25 has a nominal taper of 0.08 mm/mm over the first 3 mm from the tip. The lower taper value of ProDesign R and Unicone when compared with Reciproc R25 should ensure a higher cyclic fatigue time; nevertheless, our results showed that only ProDesign R presented better results than Reciproc and Unicone. In addition, the Reciproc group showed a significantly higher cyclic fatigue resistance value than the Unicone group ($P < .05$). Thus, other variables, such as cross-sectional design, diameter of core, and manufacturing process, should be taken into account for the outcomes of this study.

The results of this study were probably caused by the different cross-sectional designs and types of NiTi alloy of the instruments, which affect the mechanical properties of NiTi instruments (2, 15, 23, 25–28). ProDesign R and Reciproc have S-shaped cross sections with 2 cutting edges, and Unicone has a convex triangular cross section. In a supplementary examination, we captured the cross-sectional configuration of each instrument at 5 mm from the

TABLE 1. Mean Cyclic Fatigue (Time in Seconds), Torque (Ncm), and Angle of Rotation (°) of the Instruments Tested

Instruments	Cyclic fatigue (seconds)		Torque (Ncm)		Angle (°)	
	Mean	SD	Mean	SD	Mean	SD
Reciproc R25	699[b]	111.6	1.401[a]	0.1295	224.4[b]	30.43
ProDesign 25.06	2149.2[a]	403.38	1.011[c]	0.0984	315.5[a]	6.74
Unicone L25	151.2[c]	17.34	1.237[b]	0.1801	286.9[a]	34.93

SD, standard deviation.

Different superscript letters in the same column indicate statistical differences among groups ($P < .05$).

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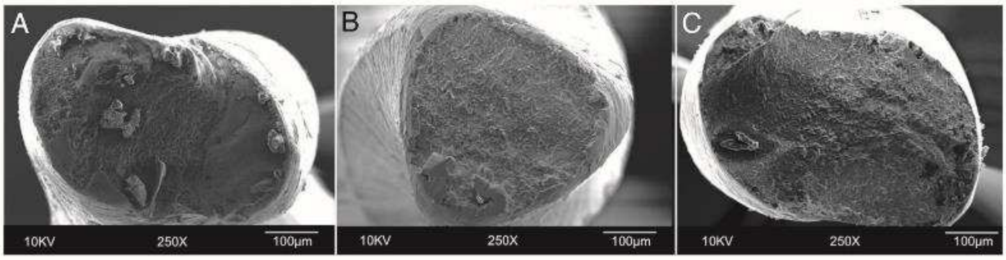

Figure 2. SEM images of fractured surfaces of separated fragments of (*A*) ProDesign R, (*B*) Unicone, and (*C*) Reciproc after cyclic fatigue testing. The images show numerous dimples, a feature of ductile fracture.

tip using scanning electron microscopy and measured the area using software (AutoCAD; Autodesk Inc, San Rafael, CA) (25). ProDesgin R showed the smallest area (239.219 μm^2) followed by Unicone (245.95 μm^2) and Reciproc (274.890 μm^2). NiTi instruments with larger cross-sectional areas present lower cyclic fatigue resistance (19, 23, 25, 26, 28). Therefore, this could contribute to the difference in cyclic fatigue resistance of these instruments.

The mechanical properties of NiTi instruments are affected by the type of alloy used in the manufacturing process (1, 2, 7, 12, 14). All the instruments used in this study were manufactured from different NiTi

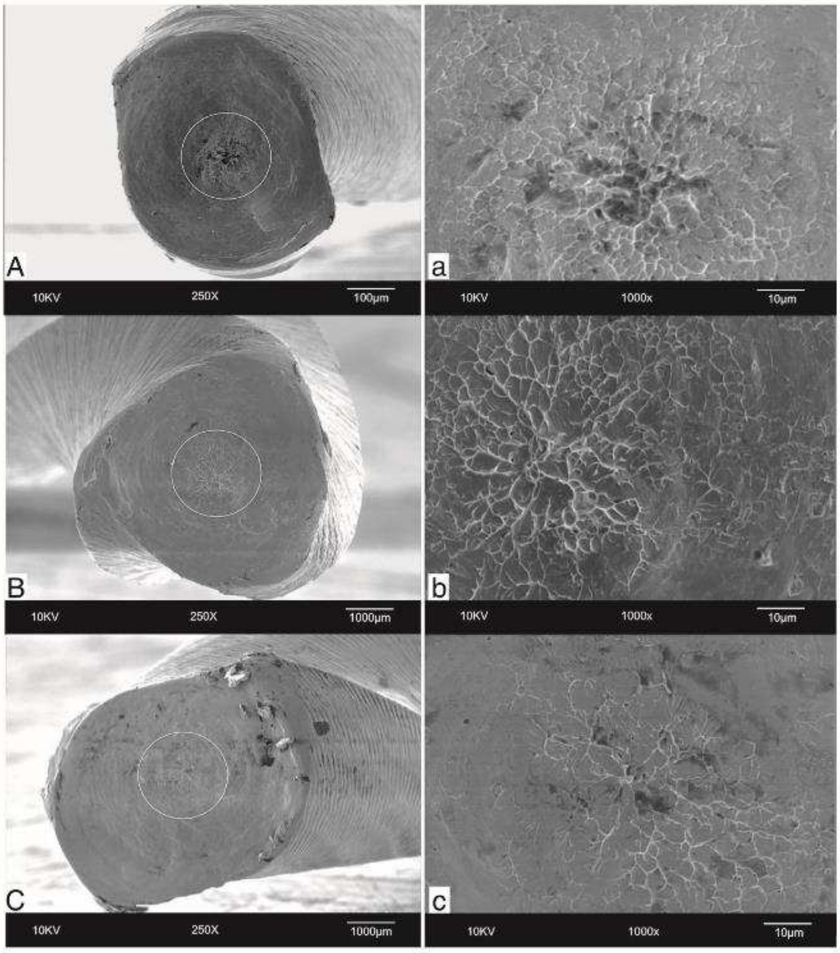

Figure 3. SEM images of the fractured surface of separated fragments (first row: *A* and *a* = ProDesign R; second row: *B* and *b* = Unicone; and bottom row: *C* and *c* = Reciproc R25). The left column shows the images after the torsional test, with the circular box indicating the concentric abrasion mark; the right column shows the concentric abrasion mark at 1000× magnification. The skewed dimples near the center of rotation are typical features of torsional failure.

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alloys. ProDesign R had significantly higher cyclic fatigue resistance than the other groups tested, and Reciproc had higher cyclic fatigue resistance than Unicone. ProDesign R was the only instrument made of controlled memory (CM) wire, and this could explain the better results shown in the cyclic fatigue test. It has previously been reported that CM wire instruments have greater flexibility and cyclic fatigue resistance than M-Wire and conventional NiTi instruments (2, 14, 29). There were only 2 previous studies evaluating the mechanical properties of Unicone instruments (15, 16). In agreement with our results, Silva et al (15) showed that Unicone had the lowest cyclic fatigue resistance when compared with Reciproc and WaveOne (Dentsply Maillefer, Ballaigues, Switzerland). These authors reported that the cross-sectional design and the NiTi alloy (proprietary treatment not reported by the manufacturer) of the Unicone instruments could explain these results.

The second result of this study showed that ProDesign R had the lowest maximum torsional strength in comparison with Reciproc and Unicone ($P < .05$). Furthermore, ProDesign R and Unicone supported a greater angular rotation than Reciproc until fracture ($P < .05$). Thus, the second null hypothesis was rejected. The results of this study were probably related to the different cross-sectional designs and NiTi alloy of the instruments, which have a significant influence on the torsional resistance (1, 5, 7, 25, 29–31).

The torsional test was performed by clamping 3 mm of the instrument tip. Thus, in a supplementary examination, we captured the cross-sectional configuration of each instrument in 3 mm from the tip using scanning electron microscopy and measured the area using software (AutoCAD) before the torsional test (14). ProDesign R showed the smallest area (98.521 μm^2) followed by Unicone (110.395 μm^2) and Reciproc (112.686 μm^2). The Reciproc and Unicone groups showed significantly higher torsional strength until fracture than ProDesign R ($P < .05$), but the Unicone group showed lower torsional strength than Reciproc ($P < .05$). It has previously been reported that instruments with larger cross-sectional areas generally present higher torsional stiffness (20, 25, 31). Furthermore, the M-Wire instruments, such as Reciproc, generally had greater torsional stiffness but a smaller angle of rotation to fracture than CM wire instruments, such as ProDesign R (2, 14, 29, 30). Our results were in agreement with the aforementioned studies and could be explained because of the high flexibility of CM wire, providing greater deformation capacity and demanding lower torsional strength values (2, 12–14). There were no previous studies evaluating the torsional fatigue resistance of Unicone instruments. Our results showed that Unicone had greater maximum torsional strength than ProDesign R ($P < .05$). However, it had a similar angle of rotation ($P > .05$). The different alloys and cross-sectional designs of these instruments might also explain the differences in the results obtained.

SEM analysis showed typical fractographic appearance of cyclic fatigue and torsional fractures, with a similar appearance among the 3 brands. After the cyclic fatigue test, the instruments showed crack initiation areas and overload (fast fracture) zones, with numerous dimples spread on the fractured surface. After the torsional test, the fragments demonstrated the typical features of shear failure, including concentric abrasion marks and fibrous microscopic dimples at the center of rotation (1, 6, 14, 25). The CM wire did not prevent but did delay the onset of catastrophic failure (unstable and fast crack growth) of the material.

The higher cyclic fatigue resistance of ProDesign R indicated that these instruments were very flexible and safe for preparing curved root canals and could perhaps cause fewer undesirable changes in the root canal anatomy during instrumentation. Furthermore, the greater angular distortion of ProDesign R could be beneficial and may provide clinicians with an indication that there was plastic/permanent deformation and imminent fracture (29). On the other hand, in constricted and curved canals, the instrument would be submitted to higher torsional load stress, which could induce plastic deformation more easily than the Reciproc and Unicone instruments. Therefore, a glide path would be necessary before the use of the ProDesign R instrument to provide pre-enlargement and reduce the torsional stress. In relation to the Unicone instrument, it should be used with caution for the preparation of curved root canals.

In conclusion, within the limitations of this study, our results showed that ProDesign R had the highest cyclic fatigue resistance values and angular rotation to fracture in comparison with Reciproc and Unicone. However, Reciproc showed higher torsional strength to failure.

Acknowledgments

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2.2 Article 2 - **Cyclic fatigue and torsional strength of three different thermally treated reciprocating nickel-titanium instruments**

The article presented in this Thesis was published in Clinical Oral Investigations. Annex C contains the permission letter to include a published article from the *Clinical Oral Investigations* in this thesis.

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ORIGINAL ARTICLE

Cyclic fatigue and torsional strength of three different thermally treated reciprocating nickel-titanium instruments

Murilo Priori Alcalde[1] · Marco Antonio Hungaro Duarte[1] · Clovis Monteiro Bramante[1] · Bruno Carvalho de Vasconselos[2] · Mario Tanomaru-Filho[3] · Juliane Maria Guerreiro-Tanomaru[3] · Jader Carvalho Pinto[3] · Marcus Vinicius Reis Só[4] · Rodrigo Ricci Vivan[1]

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Abstract

Objectives The aim of this study was to evaluate the cyclic and torsional fatigue resistance of the reciprocating single-file systems Reciproc Blue 25.08 (VDW GmbH, Munich, Germany), Prodesign R 25.06 (Easy Dental Equipment, Belo Horizonte, Brazil), and WaveOne Gold 25.07 (Dentsply/Tulsa Dental Specialties, Tulsa, OK, USA).

Materials and methods Sixty reciprocating instruments of the systems Reciproc Blue R25 (RB #25 .08 taper), Prodesign R (PDR #25 .06 taper), and WaveOne Gold (WOG #25 .07 taper) ($n = 20$) were used. Cyclic fatigue resistance testing was performed by measuring the time to failure in an artificial stainless steel canal with a 60° angle of curvature and a 5-mm radius located 5 mm from the tip ($n = 10$). The torsional test (ISO 3630-1) evaluated the torque and angle of rotation at failure of new instruments ($n = 10$) in the portion 3 mm from the tip. The fractured surface of each fragment was also observed using scanning electron microscopy (SEM). In addition, a supplementary examination was performed to measure the cross-sectional area of each instrument 3 and 5 mm from the tip. The data were analyzed using one-way ANOVA and Tukey's test, and the level of significance was set at 5%.

Results The cyclic fatigue resistance values of PDR 25.06 were significantly higher ($P < 0.05$). RB 25.08 showed higher fatigue resistance than WOG 25.07 ($P < 0.05$). The torsional test showed that PDR 25.06 had lower torsional strength ($P < 0.05$). No differences were observed between RB 25.08 and WOG 25.07 ($P > 0.05$). PDR 25.06 showed higher angular rotation values than RB 25.08 and WOG 25.07 ($P < 0.05$). RB 25.08 presented higher angular rotation than WOG 25.07 ($P < 0.05$). The cross-sectional area analysis showed that PDR 25.06 presented the smallest cross-sectional areas at 3 and 5 mm from the tip ($P < 0.05$).

Conclusion PDR 25.06 presented the highest cyclic fatigue resistance and angular rotation until fracture compared to RB 25.08 and WOG 25.07. In addition, RB 25.08 and WOG 25.07 had higher torsional strength than PDR 25.06.

Clinical relevance In endodontic practice, thermally treated reciprocating instruments have been used for the root canal preparation of curved and constricted canals; therefore, these instruments should present high flexibility and suitable torsional strength to minimize the risk of instrument fracture.

Keywords NiTi alloy · Reciprocating motion · Thermal treatment · Cyclic fatigue

✉ Rodrigo Ricci Vivan
rodrigo.vivan@fob.usp.br

1 Department of Restorative Dentistry, Dental Materials and Endodontics, Bauru Dental School, University of São Paulo, Dr Otávio Pinheiro Brisolla 9-75, Bauru, São Paulo 17012-901, Brazil

2 Department of Endodontics, Federal University of Ceará, Campus Sobral, Cel. Stanislau Frota St, Sobral, Ceará, Brazil

3 Department of Restorative Dentistry, Araraquara Dental School, São Paulo State University, R. Humaitá, 1680, Araraquara, São Paulo, Brazil

4 Department of Restorative Dentistry, Federal University of Rio Grande do Sul, Paulo Gama Av., 110, Porto Alegre, Rio Grande do Sul, Brazil

Introduction

Engine-driven nickel-titanium (NiTi) has been widely used in endodontics due to its high level of flexibility and elasticity, providing safe root canal preparation in curved canals [1, 2]. However, instrument fracture continues to be a problem for clinicians. Therefore, several technological improvements have been developed for NiTi instruments to improve their mechanical properties, such as new designs, manufacturing processes, kinematics, and thermal treatments [1–6].

The reciprocating motion involves rotation in counterclockwise and clockwise directions with 120° of difference between the two movements [3–6]. These kinematics reduce

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the screwing-in effect and the mechanical stress of the instruments, allowing for the use of single instruments for root canal preparation [3, 4, 6]. In addition, this motion has been shown to be safer than rotary motion during root preparation of curved and constricted root canals, reducing cyclic and torsional fatigue [3, 4, 6, 7]. Cyclic fatigue occurs when the instrument rotates in a curved canal, and repeated tension-compression stress occurs at the point of maximum flexure [8, 9]. Torsional fatigue generally occurs during straight root canal preparation when the tip of the instrument is locked into the dentin walls and the instrument continues to rotate, inducing plastic deformation or fracture [9, 10].

Manufacturers have developed several thermally treated NiTi alloys to improve the mechanical properties of endodontic instruments [1, 2, 5]. Controlled memory technology is a special thermal treatment that induces a certain amount of R-phase and B19 martensite phase, maintaining superelasticity [2]. This treatment increases cyclic fatigue resistance [2, 11] and angular deformation capacity [2, 12] compared with martensite wire (M-Wire) and conventional NiTi wire (NiTi-Wire). Thermal treatments have been widely used to improve the mechanical properties of rotary files and have also been used for reciprocating instruments [5, 6].

In 2015, a new reciprocating system—the WaveOne Gold (WOG; Dentsply/Tulsa Dental Specialties, Tulsa, OK, USA) system—was introduced to be used with the same reciprocating motion of the WaveOne file (Dentsply/Tulsa Dental Specialties) (M-Wire). However, the WOG instruments are manufactured with a new thermal treatment procedure called Gold treatment [13, 14]. This system presents different designs and sizes: #20, #25, #35, and #45 tip sizes and tapers of 0.07, 0.07, 0.06, and 0.05, respectively. The cross-sectional design of these instruments is a parallelogram design with two cutting edges [13]. In the Gold thermal process, the NiTi instrument undergoes a slow heating-cooling process that creates Ti_3Ni_4 precipitates dispersed on the NiTi surface [15], inducing martensitic transformation to occur in two steps and increasing the flexibility [13, 16, 17]. According to previous studies [13, 14], WOG 25.07 has higher cyclic fatigue resistance than the Reciproc (VDW GmbH, Munich, Germany) (M-Wire) and Wave One (M-Wire) systems. Furthermore, WOG presents higher torsional strength until fracture than Reciproc (M-Wire) [18].

Recently, a new generation of the Reciproc system—Reciproc Blue—was introduced. This reciprocating system has the same S-shaped cross section, instrument tip sizes, and tapers as the Reciproc (M-Wire) system. However, the manufacturer replaced the M-Wire alloy with a new thermal treatment called Blue treatment [5]. This thermal treatment is a special heating-cooling method that results in instruments with a blue color due to a titanium oxide layer [5, 19]. This treatment reduces the shape memory alloy of the NiTi and induces the occurrence of martensitic transformation in two phases [19, 20], increasing the cyclic fatigue resistance and flexibility compared with Reciproc M-Wire instruments [5].

The Prodesign R (Easy Dental Equipment, Belo Horizonte, MG, Brazil) is a new reciprocating single-file system that uses controlled memory technology. This system has two instruments presenting an S-shaped cross section: one size 25 with a taper of 0.06 and one size 35 with a taper of 0.05. Previous studies have reported that the 25.06 instrument has higher cyclic fatigue resistance than Reciproc (M-Wire) [21, 22] and WaveOne (M-Wire) [21].

Despite the importance of the effects of these thermal processes on the mechanical properties of NiTi instruments, there have been no studies comparing the mechanical properties among these new thermally treated reciprocating instruments. The aim of this study was to evaluate the cyclic and torsional fatigue (maximum torque load and angular rotation) of the Prodesign R 25.06, WaveOne Gold 25.07, and Reciproc Blue 25.08 instruments. The null hypotheses tested were as follows: (1) there is no difference in the cyclic fatigue resistance among the instruments, and (2) there is no difference in the torsional resistance among the instruments.

Materials and methods

Sample size calculation was performed before the mechanical testing using G*Power v. 3.1 for Mac (Heinrich Heine, University of Düsseldorf) and by selecting the Wilcoxon–Mann–Whitney test from the *t* test family. The alpha-type error of 0.05, beta power of 0.95, and N2/N1 ratio of 1 were also stipulated. The test calculated a total of eight samples for each group as the ideal size for noting significant differences. However, we used an additional 20% of the total instruments to compensate for possible atypical values that might lead to sample loss.

A total of 60 NiTi instruments (length, 25 mm) were used for this study. The samples were divided into three groups ($n = 20$ per system) as follows: Reciproc Blue (RB #25, 0.08 taper), Prodesign R (PDR #25, 0.06 taper), and WaveOne Gold (WOG #25, 0.07 taper). All of the instruments were inspected under a stereomicroscope (Carl Zeiss, LLC, USA) at × 16 magnification to detect possible defects or deformities before the mechanical testing; none were discarded.

Cyclic fatigue test

The static cyclic fatigue test was performed in a custom-made device that simulated an artificial canal made of stainless steel, with a 60° angle of curvature and a 5-mm radius of curvature located 5 mm from the tip, as previously described [22]. During activation of the instruments, the artificial canal was lubricated with a synthetic oil (Super Oil; Singer Co. Ltd., Elizabethport, NJ, USA). All of the instruments were activated until fracture occurred, and the time to fracture was recorded using a digital chronometer. Throughout the testing, video recordings were obtained simultaneously, and the videos were observed to ensure the exact time of instrument fracture.

A total of ten instruments coupled to a VDW Silver Motor (VDW GmbH) connected to the cyclic fatigue device for each reciprocating system were used. The preset programs were selected according to the manufacturers' recommendations. RB 25.08 and PDR 25.06 were operated with the "Reciproc All" program, and WOG 25.07 was operated with the "WaveOne All" program. The length of the fractured tip was measured using digital calipers (Digimatic, Mitutoyo Co., Kawasaki, Japan) [10].

Torsional test

The torsional tests were performed, based on the International Organization for Standardization (ISO) standard 3630-1 (1992), using a torsion machine as previously described by other studies [22–24]. A total of ten instruments, 25 mm in length, for each reciprocating system were used. The purpose of this test was to measure the mean values of torque and maximum angular rotation until instrument fracture.

The torque and angular rotation were measured throughout the entire test, and the ultimate torsional load and angular rotation (°) values were provided by a specifically designed machine (Analógica, Belo Horizonte, MG, Brazil) connected to a computer. All of the data were recorded by a specific program of the machine (MicroTorque; Analógica). Before testing, the handles of all of the instruments were removed at the point where they were attached to the torsion shaft. The 3 mm of the instrument tips was clamped into a mandrel connected to a geared motor. The geared motor operated in the counterclockwise direction at a speed set to 2 rpm for all of the groups.

SEM evaluation

A total of 30 fractured instruments ($n = 10$ per group) were selected for SEM evaluation (JEOL, JSM-TLLOA, JSM-TLLOA, Tokyo, Japan) to determine the topographic features of the fragments after the cyclic and torsional fatigue tests. Before SEM evaluation, the instruments were cleaned in an ultrasonic cleaning device (Gnatus, Ribeirão Preto, SP, Brazil) in saline solution for 3 min. All of the fractured surfaces of the instruments were examined at × 250 magnification after cyclic fatigue testing. In addition, the fractured surfaces of the instruments submitted to torsional testing were examined at × 200 and × 1000 magnification in the centers of the surfaces.

The images of the fractured surfaces obtained by SEM were used to measure the areas of the cross-section configurations at 3 and 5 mm from the tip using software (AutoCAD; Autodesk Inc., San Rafael, CA, USA) [6, 23].

Results

The means and standard deviations of the cyclic and torsional fatigue tests (torque maximum load and angle of rotation) are presented in Table 1. PDR 25.06 had the highest cyclic fatigue resistance compared to all of the other groups ($P < 0.05$). RB 25.08 showed a significantly higher lifetime value than WOG 25.07 ($P < 0.05$).

The maximum torsional strength and angular rotation values are also presented in Table 1. PDR 25.06 showed the lowest torsional strength of all the groups ($P < 0.05$). There was no difference between RB 25.08 and WOG 25.07 ($P > 0.05$). In relation to angular rotation, PDR 25.06 showed higher values than RB 25.08 and WOG 25.07. In addition, RB 25.08 had higher values than WOG 25.07 ($P < 0.05$).

The means and standard deviations of the fragment length and cross-sectional area are presented in Table 2. There were no significant differences among the instruments regarding the fragment lengths ($P > 0.05$). The cross-sectional area 3 mm from the tip showed that PDR 25.06 presented the smallest area of the groups ($P < 0.05$). There was a significant difference between RB 25.08 and WOG 25.07 ($P < 0.05$). At 5 mm, WOG 25.07 presented the largest area of all of the instruments ($P < 0.05$). PDR 25.06 showed a significantly smaller cross-sectional area than RB 25.08 ($P < 0.05$).

SEM evaluation

Scanning electron microscopy of the fragment surfaces showed similar and typical features of cyclic fatigue and torsional failure for all of the instruments tested. After the cyclic fatigue test, all of the fractured instrument surfaces showed microvoids, which are morphologic characteristics of ductile fractures (Fig. 1). Following the torsional tests, all of the instruments showed abrasion marks and fibrous dimples near the center of rotation (Fig. 2).

Discussion

Previous studies have shown that reciprocating motion promoted a significant reduction in cyclic and torsional fatigue resistance compared to rotary motion [4, 6]. However, several other factors also affect the mechanical properties of NiTi instruments such as tip size, taper, cross-sectional design, diameter of the core, and type of thermal treatment of the NiTi

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Table 1 Mean cyclic fatigue (time in seconds), torque (N.cm), and angle of rotation (°) of instruments tested

Instruments	Cyclic fatigue (s)		Torque (N.cm)		Angle (°)	
	Mean	SD	Mean	SD	Mean	SD
Reciproc Blue 25.08	876.5[b]	161.30	1.380[b]	0.1395	306.5[b]	8.592
Prodesign R 25.06	2099.8[a]	391.20	1.016[a]	0.0699	318.7[a]	8.396
WaveOne Gold 25.07	409.3[c]	77.24	1.230[b]	0.1859	296.0[c]	8.409

Different superscript letters in the same column indicate statistical differences among groups ($P < .05$)

SD, standard deviation

alloy [1, 2, 25, 26]. Thus, manufacturers have modified the instrument designs and/or thermal treatment of reciprocating instruments [1, 2, 20]. Therefore, the aim of this study was to evaluate the cyclic and torsional fatigue resistance of reciprocating instruments manufactured with different designs and thermal treatments of the NiTi alloy.

The static cyclic fatigue test was performed in simulated artificial canals in stainless steel blocks, as previously reported [5, 21–23]. Although the dynamic model simulates the clinical pecking motion performed during root canal preparation, a static model was used to reduce some variables, such as the amplitude of axial motion and speed, which are subjective, because the manually controlled axial motion could be performed in different forms by clinicians [27, 28]. The torsional test was performed in accordance with the ISO 3630-1 specification, as in previous studies [22, 24]. A 3-mm point from the tip was chosen because it is the point most susceptible to fracture during constricted root canal preparation [28]. In addition, counterclockwise rotation was used for all of the instruments because it is the direction of their spiraling flutes [6].

PDR 25.06 showed the highest cyclic fatigue resistance compared to the other groups ($P < 0.05$), and RB 25.08 showed higher cyclic fatigue than WOG 25.07 ($P < 0.05$). Thus, our first null hypothesis was rejected. Although all of the tested instruments presented the same tip sizes (#25), the taper, cross-sectional design, and thermal treatment of the NiTi instruments differed among them. PDR, WOG, and RB presented tapers of 0.06, 0.07, and 0.08 mm/mm, respectively, over the first 3 mm from the tip. Usually, instruments with lower taper ensure higher cyclic fatigue resistance [29]; however, our results showed that RB 25.08 had significantly higher cyclic fatigue resistance than WOG 25.07 ($P < 0.05$). Thus, other variables, such as cross-sectional design, diameter of the core, and thermal treatment, also played roles in the results of this study.

In this study, the cyclic fatigue test was performed using the preset programs "Reciproc All" to activate RB25.08 and PDR 25.06 and "WaveOne All" to activate WOG 25.07. The mode "Reciproc All" presents 150° counterclockwise (CCW) and 30° clockwise (CW) angles of rotation and a speed of 300 rpm; the mode "WaveOne All" presents 170° CCW and 50° CW (CW) angles of rotation and a speed of 350 rpm [4]. Previous studies have shown that larger angles of rotation during reciprocating motion [4, 30] and higher rotation speeds tend to decrease the cyclic fatigue time resistance of NiTi instruments [27, 31]. However, it was previously reported that the "Reciproc All" and "WaveOne All" modes did not influence the cyclic fatigue resistance of NiTi instruments [6, 31]. It is likely that the different reciprocating modes used among the instruments did not influence our results.

The cross-sectional design and core diameter have significant effects on the cyclic fatigue resistance of NiTi instruments [8, 26, 29]. PDR 25.06 and RB 25.08 have S-shaped cross sections, and WOG 25.07 has a parallelogram-shaped cross section. In a supplementary examination, we captured the cross-sectional configuration of each instrument 5 mm from the tip by SEM and measured the area using software (AutoCAD) [5, 8]. PDR 25.06 showed the smallest area (236.549 μm^2), followed by RB 25.08 (274.780) and WaveOne Gold (309.861 μm^2) ($P < 0.05$). Previous studies have shown that a larger metal mass volume at the maximum stress point of NiTi instruments affected cyclic fatigue resistance [8, 26, 28], which could concur with the difference in the cyclic fatigue lifetimes of the instruments.

Table 2 Mean of the fragment length (mm) and cross-sectional area at 3 and 5 mm from the tip (μm^2)

Instruments	Fragment length (mm)		Cross-sectional area (3 mm)		Cross-sectional area (5 mm)	
	Mean	SD	Mean	SD	Mean	SD
Reciproc Blue 25.08	5.01[a]	0.066	113.282[c]	0.149	274.780[b]	0.328
Prodesign R 25.06	4.98[a]	0.035	98.825[a]	0.501	236.549[a]	0.216
WaveOne Gold 25.07	5.06[a]	0.054	108.301[b]	0.359	309.861[c]	0.739

Different superscript letters in the same column indicate statistical differences among groups ($P < .05$)

SD, standard deviation

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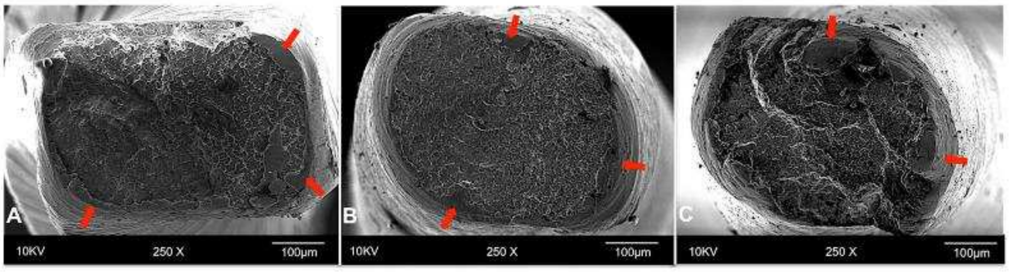

Fig. 1 Scanning electron microscopy images of the fractured surfaces of separated fragments of **a** WaveOne Gold, **b** Reciproc Blue, and **c** Prodesign R after cyclic fatigue testing. The crack origins are identified by red arrows. The images show numerous dimples spread on the fractured surfaces, which constitute a typical feature of ductile fracture

The thermal treatments of the NiTi alloys have strong influences on martensitic/austenitic transformation behavior [15, 19, 20], which could induce a different arrangement of the crystalline structure and a higher percentage of martensite transformation [2]. Previous reports have indicated that a higher percentage of martensitic phase in the NiTi alloy promoted more flexibility and greater fatigue resistance [2, 18, 32]. Our results showed that PDR 25.06 had a higher cyclic fatigue time to fracture values than all of the groups, and RB 25.08 had a higher cyclic fatigue resistance than WOG 25.07. It is likely that the different thermal treatments among them could result in different martensitic phase transformations and could induce different dissipations of the energy required for crack formation and/or propagation during cyclic fatigue testing [2]. Accordingly, Gündoğar and Özyürek [33] showed that RB 25.08 had higher cyclic fatigue resistance than WOG 25.07. In addition, it was previously reported that instruments manufactured with controlled memory technology had higher

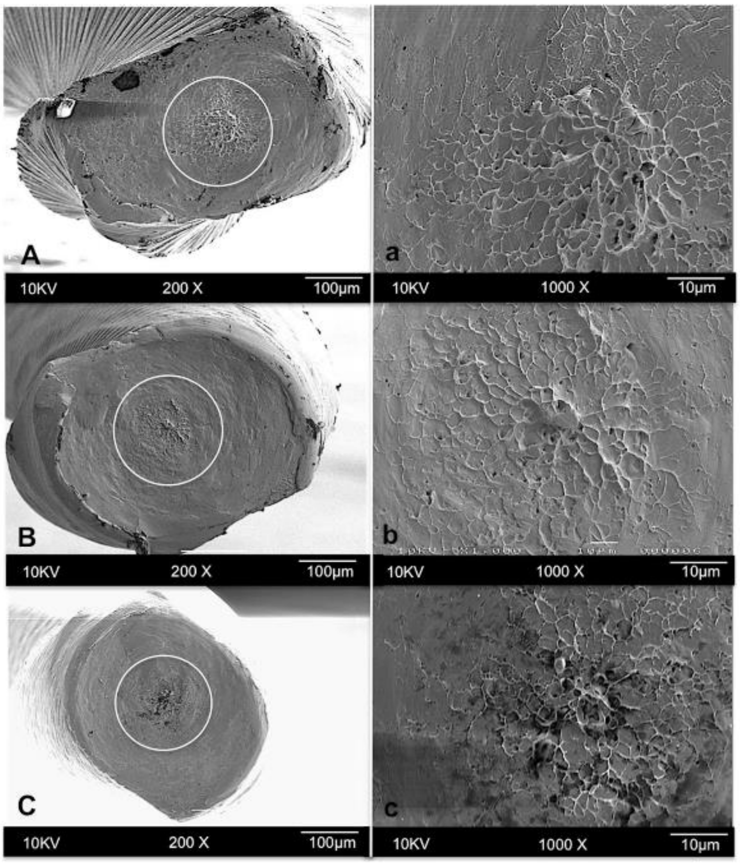

Fig. 2 Scanning electron microscopy images of the fractured surfaces of separate fragments after torsional testing (first row: A, a = WaveOne Gold; second row: B, b = Reciproc Blue; bottom row: C, c = Prodesign R). The left column shows images with the circular boxes indicating concentric abrasion marks at × 200 magnification; the right column shows concentric abrasion marks at × 1000 magnification; and the skewed dimples near the center of rotation are typical features of torsional failure

cyclic fatigue resistance than instruments manufactured by Blue [34] and Gold treatments [26]. The results of this study are in agreement with the aforementioned studies, showing that instruments manufactured with controlled memory technology are likely more fatigue resistant—and more flexible—than those manufactured with Blue and Gold treatments.

In this study, the torsional test evaluated the maximum torsional load and angular rotation to fracture while the instruments were rotating in a counterclockwise direction; however, in clinical situations, the reciprocating motion minimizes the torsional stress when the reverse motion occurs [6]. Thus, this test evaluated the torsional behavior of the instrument when undergoing a high level of torsional stress [32]. PDR 25.06 presented the lowest torsional load, compared with RB 25.08 and WOG 25.07 ($P < 0.05$); no difference was found between RB 25.08 and WOG 25.07. The second null hypothesis was rejected because significant differences were observed among the three tested instruments ($P < 0.05$): PDR 25.06 supported greater angular rotation to fracture, followed by RB 25.08 and WOG 25.07. The results of this study were likely related to the different cross-sectional designs and thermal treatments.

In a supplementary evaluation, the cross-sectional configuration of each instrument was captured in D3 by SEM, and the cross-sectional area was measured by means of software (AutoCAD) before torsional testing [5, 15]. PDR 25.06 showed the smallest area (98.825 μm^2), followed by WOG 25.07 (108.301 μm^2) and RB 25.08 (113.282 μm^2) ($P < 0.05$). Previous studies have shown that instruments with larger cross-sectional areas tend to present higher torsional load [6, 22, 23, 34]. In addition, NiTi instruments manufactured with CM-Wire demanded lower torsional loads and higher angular rotation capacity until fracture than instruments manufactured with Blue [35] and Gold treatments [26]. Our results are in agreement with the aforementioned studies and could explain the results with PDR 25.06, which presented greater deformation capacity and demanded a lower torsional load.

There have been no previous studies comparing the torsional fatigue resistance of RB 25.08 and WOG 25.07. Our results showed that RB 25.08 presented higher angular rotation values than WOG 25.07 ($P < 0.05$), but they presented similar torsional loads. The higher angular rotation values of RB 25.08 might be related to the Blue treatment, which could favor the higher flexibility and greater deformation capacity. Additionally, the different cross-sectional designs and core diameters promoted different torsional stress distribution behaviors, which could affect the susceptibility to fatigue [25, 26, 36].

The SEM analysis showed the typical features of cyclic and torsional fatigue for the three tested reciprocating files. After the cyclic fatigue test, all of the instruments evaluated showed crack initiation areas and overload zones, with numerous dimples spread on the fractured surfaces. After the torsional test, the fragments showed concentric abrasion marks and fibrous dimples at the center of rotation [6, 23, 29].

The reciprocating motion promoted a significant reduction in cyclic and torsional fatigue resistance [4, 6]. However, clinicians should be aware of the differences in mechanical properties of different available NiTi reciprocating systems [1]. According to the present results, the higher cyclic fatigue resistance of PDR 25.06 and RB 25.08 suggested these instruments to be safer than WOG 25.07 for the root canal preparation of curved canals. In contrast, the higher torsional load of RB 25.08 and WOG 25.07 indicated that they could support higher torsional stress during constricted canal preparation. Therefore, the results suggested that PDR 25.06 should be used in association with glide path preparation to decrease torsional stress, thus reducing the risk of fracture.

In conclusion, within the limitations of this study, the instruments f

eatures, such as cross-sectional design, taper, and thermal treatments, had significant influences on the mechanical properties of the NiTi instruments. Our results showed that PDR 25.06 had the highest cyclic fatigue resistance and highest angular rotation values to fracture, compared with RB 25.08 and WOG 25.07. However, RB 25.08 and WOG 25.07 showed higher torsional resistance to fracture than PDR 25.06.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflicts of interest.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent For this type of study, formal consent is not required.

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2.3 Article 3 - **Torsional fatigue resistance of pathfinding instruments manufactured from several NiTi alloys**

The article presented in this Thesis was published in the International Endodontic Journal. Annex C contains the permission letter to include a published article from *the International Endodontic Journal* in this thesis.

Alcalde MP, Duarte MAH, Bramante CM et al. Torsional fatigue resistance of pathfinding instruments manufactured from several nickel-titanium alloys. International Endodontic Journal. 2017. doi: 10.1111/iej.12879.

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Torsional fatigue resistance of pathfinding instruments manufactured from several nickel-titanium alloys

M. P. Alcalde[1], M. A. H. Duarte[1], C. M. Bramante[1], M. Tanomaru-filho[2], B. C. Vasconcelos[3], M. V. R. Só[4] & R. R. Vivan[1]

[1]Department of Dentistry, Endodontics and Dental Materials, Bauru Dental School, University of São Paulo, Bauru, SP; [2]Department of Restorative Dentistry, Araraquara Dental School, São Paulo State University, Araquara, SP; [3]School of Dentistry of Sobral, Federal University of Ceará, Sobral, CE; and [4]Department of Endodontics, Federal University of Rio Grande do Sul, Porto Alegre, RS, Brazil

Abstract

Alcalde MP, Duarte MAH, Bramante CM, Tanomaru-filho M, Vasconcelos BC, Só MVR, Vivan RR. Torsional fatigue resistance of pathfinding instruments manufactured from several nickel-titanium alloys. *International Endodontic Journal.*

Aim To evaluate the torsional properties of pathfinding nickel-titanium (NiTi) rotary instruments manufactured from several NiTi alloys, ProGlider (M-wire), Hyflex GPF (conventional NiTi Wire and controlled memory wire), Logic (conventional NiTi wire and controlled memory wire) and Mtwo (conventional NiTi wire).

Methodology A total of 56 NiTi instruments from Glidepath rotary systems ($n = 8$) were used: Logic (size 25, .01 taper), Logic CM (size 25, .01 taper), ProGlider (size 16, .02 taper), Hyflex GPF (size 15, .01 taper), Hyflex GPF CM (size 15, .02 taper; size 20, .02 taper) and Mtwo (size 10, .04 taper). The torsion tests were performed based on ISO 3630-1 (1992). Three millimetres of each instrument tip was clamped to a small load cell by a lever arm linked to the torsion axis. Data were analysed using a one-way analysis of variance (ANOVA) and Tukey test with a significance level at $a = 5\%$.

Results The Logic size 25, .01 taper had significantly higher torsional strength values ($P < 0.05$). The ProGlider was significantly different when compared with Hyflex GPF size 15, .01 taper and size 15, .02 taper ($P < 0.05$). The Logic CM size 25, .01 taper had significantly higher torsional strength than Hyflex GPF size 15, .01 taper and size 15, .02 taper ($P < 0.05$). No difference was found amongst Mtwo size 10, .04 taper and Hyflex GPF groups (size 15, .01 taper; size 15, .02 taper; size 20, .02 taper). In relation to the angle of rotation, Logic CM size 25, .01 taper and Hyflex GPF size 15, .01 taper had the highest angle values ($P < 0.05$). The ProGlider had the lowest angle values in comparison with all the groups ($P < 0.05$) followed by Mtwo size 10, .04 taper. The Logic size 25, .01 taper had significantly higher angle of rotation values than ProGlider and Mtwo size 10, .04 taper ($P < 0.05$).

Conclusion The Logic size 25, .01 taper instrument made of conventional NiTi alloy had the highest torsional strength of all instruments tested. In addition, the ProGlider instrument manufactured from M-Wire alloy had the lowest angle of rotation to fracture in comparison with the other instruments.

Keywords: Nickel-Titanium, pathfinding instruments, rotary instruments, thermal treatment.

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Introduction

Maintenance of the original root canal morphology is mandatory during root canal preparation (Patiño *et al.* 2005, De-Deus *et al.* 2016). Creation of a glide path is an important clinical procedure with the

Correspondence: Rodrigo Ricci Vivan, Bauru School of Dentistry, University of São Paulo, Al. Octávio Pinheiro Brisolla, 9-75, CEP 17012-901, Bauru, São Paulo, Brazil (Tel.: +55 14 32358344; fax: +55 14 32352788; e-mail: rodrigo.vivan@fob.usp.br).

purpose of preshaping the root canal from its orifice to its apical foramen (West 2010, Ruddle *et al.* 2014) to prevent shaping errors (Patiño *et al.* 2005, Berutti *et al.* 2009, West 2010). This clinical step has been widely recommended to improve the safety of rotary nickel-titanium preparation by decreasing the incidence of instrument fracture (Pasqualini *et al.* 2012, D'Amario *et al.* 2013).

The glide path can be performed with conventional stainless steel hand files or with mechanical NiTi instruments with small tip sizes and smaller taper (Gambarini *et al.* 2015). At present, use of NiTi rotary instruments for glide path preparation has been recommended because it leads to a reduction in postoperative pain (Pasqualini *et al.* 2012); preservation of the original root canal morphology (D'Amario *et al.* 2013, Elnaghy & Elsaka 2014); prevention of instrument fracture (Berutti *et al.* 2009) and is simple to teach (West 2010).

The NiTi instruments used for glide path preparation are susceptible to torsional failure in constricted root canals because they are used at the beginning of the procedure (Arias *et al.* 2016, De-Deus *et al.* 2016). Torsional failure occurs when the tip of the instrument is locked in the canal whilst the shank continues to rotate (Sattapan *et al.* 2000). This can happen especially in the preparation of constricted canals when the file is susceptible to high torsional loads (Wycoff & Berzins 2012). Therefore, to minimize this drawback, the manufacturers have developed new instruments with various cross-sections, designs and thermomechanical treatments (Karataş *et al.* 2016).

Several NiTi rotary glide path files are manufactured as single-file or multiple-file systems. Examples of single-file glide path instruments include ProGlider (Dentsply Sirona, Ballaigues, Switzerland), Mtwo (VDW, Munich, Germany) and ProDesign Logic (Easy Equipamentos Odontológicos, Belo Horizonte, Brazil).

The ProGlider instrument is made of M-wire alloy (Dentsply Sirona) and has a square cross-section design. It has a size 16 tip and variable taper of between 2% and 8% along the shaft (De-Deus *et al.* 2016). The Mtwo rotary instrument has a size 10 tip and .04 taper, manufactured from conventional NiTi Wire and an S-shaped cross-sectional design with double cutting edges and noncutting tip (de Oliveira Alves *et al.* 2012). The Prodesign Logic size 25, .01 taper (Easy Equipamentos Odontológicos) is new glide path instrument that has a square cross-section. This instrument is made of both conventional NiTi Wire and CM-Wire.

The Hyflex GPF (Coltene-Whaledent, Altstätten, Switzerland) is an example of a multiple-rotary pathfinding system composed of three instruments: size 15, .01 taper, size 15, .02 taper, and size 20, .02 taper. The instrument size 15, .01 taper is manufactured of conventional NiTi Wire and a triangular cross-section; the other instruments are made of controlled memory wire (CM-Wire) and have a square cross-section (Capar *et al.* 2015).

The advantages of rotary techniques for glide path preparation are evident; however, unexpected instrument fracture can occur. The torsional properties of pathfinding instruments can vary according to the instrument's taper, tip size, cross-sectional design and the type of NiTi used during the manufacturing process (Elnaghy & Elsaka 2015, Arias *et al.* 2016, De-Deus *et al.* 2016). Thus, knowledge of the best torque recommendations of these instruments is of fundamental importance to provide a safe and effective clinical application. The torsion test provides the maximum torsional strength and angle of rotation supported by an instrument before fracture (Arias *et al.* 2016, Pedullà *et al.* 2016). This test attempts to simulate a standardized and extreme clinical situation with high torsional load (Kim *et al.* 2012, Elnaghy & Elsaka 2015, Arias *et al.* 2016) and has been previously described (Elnaghy & Elsaka 2015, Arias *et al.* 2016, Pedullà *et al.* 2016, Alcalde *et al.* 2017).

There is a lack of information comparing the torsional properties of these instruments, especially of the Proglider, GPF Hyflex, Logic size 25, .01 taper and Logic CM size 25, .01 taper systems. The aim of this study was to evaluate the torsional properties (maximum torsional strength and angle of rotation) of the Proglider, Hyflex GPF, Logic and Mtwo instruments. The null hypothesis was that there would be no difference in the torsional properties amongst the types of instruments.

Materials and methods

The sample calculation was performed using the G*Power v3.1 for Mac (Heinrich Heine University Düsseldorf (HHU) by selecting the Wilcoxon–Mann–Whitney test of the *t*-test family. The alpha-type error of 0.05, a beta power of 0.95, and a ratio N2/N1 of 1 were also stipulated. A total of six samples per group were indicated as the ideal size required for noting significant differences. Eight samples per group were used because an additional 20% was calculated to compensate for possible outlier values that might lead to sample loss.

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A sample of 56 NiTi instruments (length, 25 mm) from seven different Glidepath rotary systems ($n = 8$ per system) were used, as follows: Logic (size 25, .01 taper), Logic CM (size 25, .01 taper), ProGlider (size 16, .02 taper), Hyflex GPF (size 15, .01 taper), Hyflex GPF CM (size 15, .02 taper; size 20, .02 taper) and Mtwo (size 10, .04 taper). Before testing, every instrument was inspected for defects or deformities under a stereomicroscope (Stemi 2000C; Carls Zeiss, Jena, Germany) at 16× magnification; none were discarded.

Torsional test

The torsion tests were performed, based on the International Organization for Standardisation ISO 3630-1 (1992) specification, using a torsion machine described in detail elsewhere (Bahia *et al.* 2006, Alcalde *et al.* 2017).

Before testing, each instrument handle was removed at the point where the handle was attached to the shaft. The shaft of each instrument was clamped in a chuck with brass jaws to prevent sliding and was connected to the reversible geared motor. The rotation speed of the motor was set at 2 rpm in a clockwise direction for all groups. Three millimetres of the instrument's tip was clamped into another chuck with brass jaws. The torque values were assessed by measuring the force exerted on a small load cell by a lever arm linked to the torsion axis. Measurement and control of the rotation angle were performed by a resistive angular transducer connected to a process controller. Continuous recording of torsional strength and angle of rotation were monitored, and the ultimate torque and angle before fracture (°) were provided by a specifically designed computer program (Analógica, Belo Horizonte, MG, Brazil) and were recorded.

SEM evaluation

After the torsional test, four instruments of each group were randomly selected and ultrasonically cleaned to remove debris. The instruments were examined by scanning electron microscopy (JEOL, JSM-TLLOA, Tokyo, Japan) to assess the topographic features of the fractured surface of the instruments. The photomicrographs were taken at 50× and 350× magnification. Furthermore, additional photomicrographs were taken at 1000× magnification in the centre of the fracture surface of the instruments to improve the analysis of the topographic features.

Statistical analysis

Preliminary analysis of data normality was performed with the Shapiro–Wilk test, showing that the data were normally distributed. The One-way analysis of variance (ANOVA) and Tukey tests were used for multiple and individual comparisons. The Prism 6.0 software (GraphPad Software Inc., La Jolla, CA, USA) was used as the analytical tool, and the level of significance was set at 5%.

Results

The mean and standard deviations of torsional fatigue resistance (torque maximum torsional strength and angle of rotation) for each instrument are presented in Table 1. The Logic size 25, .01 taper had significantly higher torsional strength values than all the other groups ($P < 0.05$). The ProGlider was significantly different only when compared with Hyflex GPF size 15, .01 taper and size 15, .02 taper ($P < 0.05$). The Logic CM size 25, .01 taper showed significantly higher torsional strength values than Hyflex GPF size 15, .01 taper and size 15, .02 taper ($P < 0.05$). No difference was found between the Mtwo size 10, .04 taper and Hyflex GPF groups (size 15, .01 taper; size 15, .02 taper; size 20, .02 taper). The Hyflex GPF CM size 20, .02 taper showed higher torsional strength values than GPF size 15, .01 taper and size 15, .02 taper ($P < 0.05$). No difference was found between Hyflex GPF size 15, .01 taper and size 15, .02 taper.

In relation to the angle of rotation, the ProGlider had the lowest values when compared with the other groups ($P < 0.05$) followed by Mtwo size 10, .04 taper. The Logic size 25, .01 taper had significantly higher angle of rotation values than ProGlider and Mtwo size 10, .04 taper ($P < 0.05$). The Logic CM size 25, .01 taper was not significantly different when compared with Hyflex GPF size 15, .01 taper ($P < 0.05$). No difference was found between the Hyflex GPF size 15, .01 taper and size 15, .02 taper. The Hyflex GPF CM size 20, .02 taper had a significantly lower angle of rotation than Hyflex GPF size 15, .01 taper and size 15, .02 taper ($P < 0.05$).

SEM evaluation

Scanning electron microscopy of the fracture surface revealed similar and typical features of torsional failure for all brands. All the instruments had concentric

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Table 1 Torque (N cm) and angle of rotation (°) of instruments tested

	Instruments													
	Logic 25.01		Logic 25.01 (CM)		ProGlider 16.02		Mtwo 10.04		GPF 15.01		GPF 15.02		GPF 20.02	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Torque (N cm)	0.47^{a}	0.057	$0.29^{b,d}$	0.030	0.30^{b}	0.079	$0.25^{b,d,e}$	0.018	$0.19^{c,e,f}$	0.010	$0.19^{c,e,f,g}$	0.016	$0.30^{b,d,e}$	0.010
Angle (°)	793.1^{a}	58.50	1395^{c}	67.41	388.2^{b}	66.54	544.7^{d}	41.07	$1337^{c,e}$	131.0	$1229^{e,f}$	41.24	1088^{g}	34.34

SD, standard deviation. Different superscript letters in the same line indicate significant differences amongst groups ($P < 0.05$).

abrasion marks and fibrous dimple marks at the centre of rotation for torsional failure (Fig. 1). In addition, in the side view, it is possible to note the deformation of the spiral flutes of the instruments, mainly in those that had a higher angle of rotation value (Fig. 1).

Discussion

Glide path preparation reduces the possibility of operational errors (D'Amario *et al.* 2013, Elnaghy & Elsaka 2014) and the risk of instrument fracture (Berutti *et al.* 2009), particularly in constricted root canals, in which the instrument is susceptible to high torsional loads (Sattapan *et al.* 2000). If instrument fracture occurs at this stage and the fragment cannot be removed, the root canal cannot be cleaned, which could compromise the success of the treatment (Capar *et al.* 2015). Thus, it is important to know the torsional fatigue resistance of the pathfinding instruments for suitable clinical use. The results of this study revealed that there was a significant difference in relation to the maximum torsional strength and angle of rotation amongst the instruments tested. Thus, the null hypothesis was rejected.

In this study, the torsional fatigue resistance (maximum torsional strength and angle of rotation) to fracture was evaluated. The torsional tests were performed in accordance with the ISO Standard 3630-1 specification. In this study, 3 mm of the tip was fastened and rotation in a clockwise direction was set for all instruments. The 3 mm of the tip was chosen because at this point, the instrument is more susceptible to fracture than at 5 mm (Capar *et al.* 2015).

Several variables such as instrument tip size, taper, cross-sectional design and manufacturing techniques affect the clinical performance of endodontic files and their resistance to fracture by torsion (Pereira *et al.* 2012, Arias *et al.* 2016, De-Deus *et al.* 2016, Kaval *et al.* 2016, Magalhães *et al.* 2016, Acosta *et al.* 2017). The results revealed that Logic size 25, .01 taper had significantly higher torsional strength values when compared with all the groups ($P < 0.05$). However, the Logic CM size 25, .01 taper had significant differences only when compared with Hyflex GPF size 15, .01 taper and size 15, .02 taper ($P < 0.05$). Although the Logic size 25, .01 taper and Logic CM size 25, .01 taper have the same tip size, taper and cross-sectional design, the results revealed there was a significant difference between the groups

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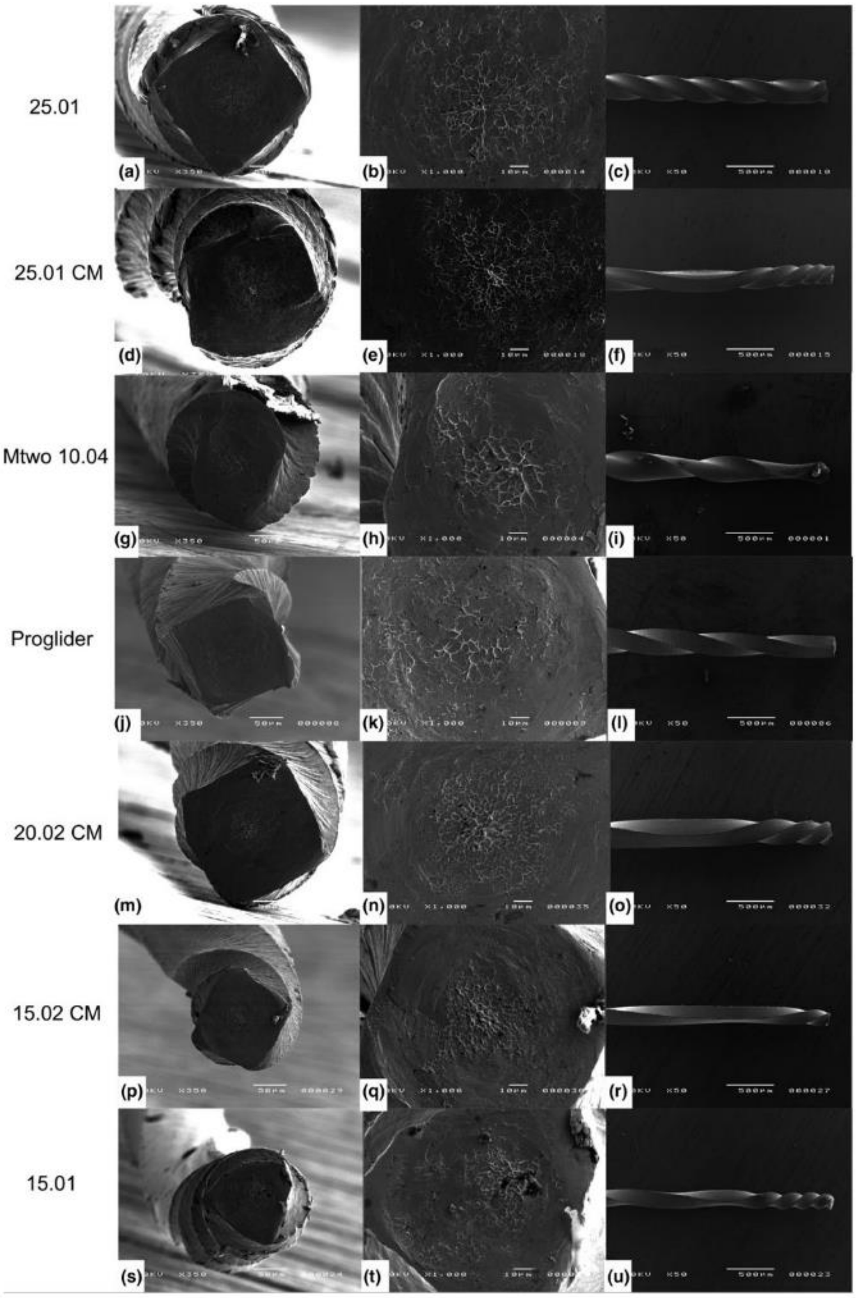

Figure 1 Scanning electron microscopy images of fractured surfaces of separated fragments of (a–c) Logic size 25, .01 taper, (d–f) Logic size 25, .01 taper CM, (g–i) Mtwo size 10, .04 taper, (j–l) ProGlider, (m–o) Hyflex GPF CM size 20, .02 taper, (p–r) Hyflex GPF CM size 15, .02 taper and (s–u) Hyflex GPF size 15, .01 taper after torsional fatigue testing. The first column shows the front-view images of the instruments at 350× magnification; the second column shows the concentric abrasion mark at 1000× magnification; the skewed dimples near the centre of rotation are typical features of torsional failure; the third column represents the side view of the instruments at 50× magnification, showing the plastic deformation of the spiral flutes.

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($P < 0.05$). This could be explained because of the different NiTi alloys used during the manufacturing process. Previous studies have shown that controlled memory wire has greater flexibility and lower torsional load than conventional NiTi Wire (Pereira *et al.* 2012, Shen *et al.* 2013, Kaval *et al.* 2016, Kwak *et al.* 2016, Pedullà *et al.* 2016, Acosta *et al.* 2017).

There was no significant difference ($P < 0.05$) relative to the torsional strength amongst Logic CM size 25, .01 taper, Proglider, Mtwo size 10, .04 taper and GPF CM size 20, .02 taper groups. These results are probably related to their different cross-sectional design, type of NiTi alloy, tip size and taper, which affected the torsional properties of NiTi instruments (Ninan & Berzins 2013, Shen *et al.* 2013, Kaval *et al.* 2016, Pedullà *et al.* 2016, Silva *et al.* 2016, Acosta *et al.* 2017). The Hyflex GPF size 15, .01 taper and size 15, .02 taper had the lowest torsional strength values when compared with the other groups ($P < 0.05$). Previous reports have indicated that instruments with lower metal mass (diameter of core, tip size and taper) generally have lower torsional strength (Baek *et al.* 2011, Zhang *et al.* 2011, Kim *et al.* 2012, Kwak *et al.* 2016, Pedullà *et al.* 2016).

However, it was difficult to make comparisons amongst the different instruments, because of differences in the type of alloy, cross-section, tip size and taper. In a supplementary examination, the cross-sectional configuration of the instruments was captured at D3 by SEM and the area measured by means of software (AutoCAD; Autodesk Inc, San Rafael, CA, USA; Pedullà *et al.* 2016). The Hyflex GPF size 15, .01 taper and Hyflex GPF CM size 15, .02 taper had the smallest area (14.491 and 24.699 μm^2, respectively) followed by ProGlider (30.823 μm^2), Hyflex GPF size 20, .02 taper (33.311 μm^2), Mtwo size 10, .04 taper (36.890 μm^2), Logic size 25, .01 taper and Logic CM size 25, .01 taper (42.469 and 42.472 μm^2). The Hyflex GPF size 15, .01 taper and size 15, .02 taper had lower torsional strength values until fracture and also the smallest cross-sectional areas. Previous reports have stated that instruments with small cross-sectional areas generally have lower torsional strength (Turpin *et al.* 2000, Baek *et al.* 2011, Kim *et al.* 2012, Ninan & Berzins 2013, Pedullà *et al.* 2016). Moreover, the cross-sectional design modified the stress distribution under torsion, which affected the torsional strength and susceptibility to fracture (Turpin *et al.* 2000, Zhang *et al.* 2011, El-Anwar *et al.* 2016).

In relation to the angle of rotation, the Logic CM size 25, .01 taper had the highest deformation capacity when compared with the other groups ($P < 0.05$) followed by GPF size 15, .01 taper, Hyflex GPF CM size 15, .02 taper, Hyflex GPF CM size 20, .02 taper, and Logic size 25, .01 taper. The ProGlider and Mtwo size 10, .04 taper had significantly lower values than the other groups ($P < 0.05$). Additionally, the ProGlider had a significantly lower angle of rotation values than Mtwo size 10, .04 taper. It is important to note that the instruments with smaller taper diameters had the highest angles of rotation to fracture. The results were in agreement with the studies of De-Deus *et al.* (2016) and Arias *et al.* (2016) that revealed that instruments with a larger taper are more susceptible to fracture.

The higher angle of rotation of Logic CM size 25, .01 taper, Hyflex GPF size 15, .02 taper and size 20, .02 taper could be influenced by the special thermal treatment of the NiTi used in manufacturing process, which increased the flexibility and deformation capacity of controlled memory instruments (Pereira *et al.* 2012, Peters *et al.* 2012, Shen *et al.* 2013, Kaval *et al.* 2016, Kwak *et al.* 2016, Pedullà *et al.* 2016, Acosta *et al.* 2017). However, CM of NiTi is not always superior to the conventional NiTi Wire or M-Wire, because other factors, such as cross-sectional design, core mass, taper and tip size (Ninan & Berzins 2013, Shen *et al.* 2013), need to be taken in account, which could explain the high angle of rotation values of Hyflex GPF size 15, .01 taper.

The scanning electron microscopy analysis revealed the typical fractographic appearance of torsional fractures that were similar amongst all brands (Fig. 1). After the torsional test, the fragments demonstrated the typical features of shear failure, including concentric abrasion marks and fibrous microscopic dimples at the centre of rotation (Kim *et al.* 2012, Pedullà *et al.* 2016). In addition, in the side view, it was possible to note the deformation of the spiral flutes of the instruments, particularly in those that had a higher angle of rotation before fracture (Fig. 1).

The torsional test was performed in accordance with the ISO Standard 3630-1 methods for root canal instruments as previously described (Elnaghy & Elsaka 2015, Arias *et al.* 2016, Pedullà *et al.* 2016, Alcalde *et al.* 2017). This test provides accurate information regarding the maximum torsional strength and angle of rotation supported by each instrument before fracture, ensuring precise torque values for their safe and effective clinical use (Bahia *et al.* 2006, Arias *et al.*

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2016). In this study, the test did not simulate the clinical use of instruments. However, it provided precise and standardized conditions of high torsional loads for all groups (Kim *et al.* 2012, Pedullà *et al.* 2016). Furthermore, during the glide path procedure, a pecking motion is recommended to prevent the instrument tip locking in the root canal (Berutti *et al.* 2009, De-Deus *et al.* 2016). However, in this study, the static model was used to allow a precise condition for all instruments with the objective of decreasing several variables, such as, the amplitude motion of the file and the amount of force applied in the axial direction, which are completely subjective in a clinical situation (Kim *et al.* 2012, Lopes *et al.* 2013). In addition, the standardisation of the force and the direction is fundamental to ensure accurate results for the torsional tests (Bahia *et al.* 2006, Lopes *et al.* 2013).

Conclusions

The Logic size 25, .01 taper instrument made of conventional NiTi alloy had the highest torsional strength of all instruments tested. In addition, the ProGlider instrument manufactured from M-Wire alloy had the lowest angle of rotation to fracture in comparison with the other instruments.

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Conflict of interest

The authors have stated explicitly that there are no conflicts of interests in connection with this article.

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3 Discussion

3 DISCUSSION

The introduction of the NiTi engine-drive instruments in endodontics promoted a revolution on the concept of root canal preparation, favoring safety and more efficiency during preparation of curved and constricted canals (PETERS, 2001; HAAPSALO; SHEN, 2013). However, unexpected instrument separation can occur because of flexural and torsional stress (SATTAPAN et al., 2000). If instrument separation occurs and the fragment can not be removed or bypassed, the root canal can not be cleaned, which could compromise the success of the treatment (Capar et al*.,* 2015a). Therefore, it is important to know the mechanical properties of the NiTi instruments for suitable and safe clinical use.

Previous studies reported that the cyclic flexural and torsional resistance of NiTi instruments are affected by instrument size, taper, cross-sectional design, diameter of core and thermal treatment of the NiTi (TURPIN; CHAGNEAU; VULCAIN, 2000; PEREIRA et al., 2012; LOPES et al., 2013; KAVAL et al., 2016; KAVAL et al., 2017; PEDULLA et al., 2016). In addition, the reciprocating motion has been shown to be safe and effective in the preparation of curved canals, reducing cyclic flexural and torsional fatigue in comparison than rotary motion (VALERA-PATIÑO et al., 2010; DE-DEUS et al., 2010; KIM et al., 2012; KARATAS et al., 2016; DE-DEUS et al., 2017). Therefore, the aim of this study was to evaluate the mechanical properties of reciprocating instruments and pathfinding rotary instruments manufactured with different designs and thermal treatments of the NiTi alloy.

In this study, the static cyclic flexural fatigue test was performed to evaluate the time demanded to fracture reciprocating instruments in simulated artificial canals in stainless steel blocks, with 60° angle of curvature and a 5-mm radius of curvature, as previously reported (DE-DEUS et al., 2010; LOPES et al., 2013; DA FROTA et al., 2014; KIM et al., 2012; PEDULLA et al., 2016). Previous studies used the dynamics model to simulate pecking motion accomplished during root canal preparation, which increase the cyclic flexural fatigue resistance in comparison with the static test (HAIKEL et al., 1999; LI et al., 2002). However, the static model has advantage to simulate a situation of higher flexural stress and reduce some variables, such as the amplitude of axial motion and speed, which are subjective, because the manually controlled axial motion could be performed in different forms by the clinicians, difficulting their reproduction (WAN et al., 2011; DA FROTA et al., 2014; PEDULLA et al., 2016).

The torsional test was performed in accordance with the ISO Standard 3630-1 methods for root canal instruments, as previously described (BAHIA; BUONO, 2005; ELNAGHY; ELSAKA, 2015, ARIAS et al. 2016, PEDULLA et al., 2015; PEDULLA et al., 2016). The 3 mm of the instrument's tip was fastened and rotated in a counterclockwise was performed for reciprocating instruments, whereas the rotary pathfinding instruments were rotated clockwise direction until file separation. For all instruments, 3 mm of the tip was chosen because at this point the instrument is more susceptible to fracture than at 5 mm (CAPAR et al., 2015a). This test provides precise and standardised conditions of high torsional loads for the NiTi instruments (KIM et al., 2012, PEDULLA et al., 2015; PEDULLA et al., 2016). Additionally, the test provides accurate information regarding the maximum torsional strength and angle of rotation supported by the instruments before fracture, ensuring precise torque values for their safe clinical use (ARIAS et al., 2016).

The results of cyclic flexural fatigue resistance of reciprocating instruments showed that there was a significant difference among them ($P<0.05$). All the instruments have the same tip sizes (#25). However, the tapers, cross-sectional design and thermal treatments differed among them. The instruments used in this study presented tapers of 0.06, 0.07 and 0.08, over the first 3 mm from the tip. Usually, instruments with lower taper should ensure higher cyclic flexural fatigue resistance (PLOTINO et al., 2009; KAVAL et al., 2017); however, our results showed that UNICONE 25.06 (UNC 25.06) had lower cyclic flexural fatigue resistance than Prodesign R 25.06 (PDR 25.06) and Reciproc 25.08 (REC 25.08) ($P<0.05$). In addition, the WaveOne Gold 25.07 (WOG 25.07) presented lower cyclic flexural fatigue resistance than PDR 25.06 and Reciproc Blue 25.08 (RB 25.08) ($P<0.05$). Therefore, other variables, such as cross-sectional design, diameter of core and thermal treatment of the Nickel-Titanium (NiTi), played roles in the results of this study.

In this study, the cyclic flexural fatigue test was performed using the preset programs "Reciproc All" and "WaveOne All" to activate the reciprocating instruments according the manufacturer's instructions. The mode "Reciproc All" presents 150° counterclockwise (CCW) and 30° clockwise (CW) angles of rotation and a speed of 300 rpm; the mode "WaveOne All" presents 170° CCW and 50° CW (CW) angles of rotation and a speed of 350 rpm (KIM et al., 2012; PEDULLA et al., 2013). Previous studies have shown that larger angles of rotation during reciprocating motion (KARATAS et a., 2016; ARSLAN et al., 2016) and higher rotation speeds tend to decrease the cyclic flexural fatigue time resistance of NiTi instruments (PLOTINO et al., 2009; PEDULLA et al., 2013). However, it was previously reported that the "Reciproc All" and "WaveOne All" modes did not influence the cyclic flexural fatigue resistance of NiTi instruments (KIM et al., 2012; PEDULLA et al., 2013). It is likely that the different reciprocating modes used among the instruments did not influence our results.

The cross-sectional design and diameter of core have affects the cyclic flexural fatigue resistance of NiTi instruments (GRANDE et al., 2006; SHEN et al., 2013; KIM et al., 2012; SILVA et al., 2016a; PEDULLA et al., 2016; KAVAL et al., 2017). The instruments used in this study presented the following cross-sectional design of the: a convex triangular (UNC 25.06), S-shaped (PDR 25.06; REC 25.08; RB 25.08), and parallelogram (WOG 25.07). A supplementary examination was performed by scanning electron microscopy (SEM) and measured the cross-sectional configuration area of each instrument 5 mm from the tip. This step was performed in the D5 because at this point is the maximum stress of NiTi instruments during cyclic flexural fatigue resistance. The PDR 25.06 showed smallest cross-sectional area (239.219 μm^2) followed by UNC 25.06 (245.95 μm^2) and REC 25.08 (274.890 μm^2). Additonally, PDR 25.06 showed the smallest cross-sectional area (236.549 μm^2), followed by RB 25.08 (274.780 μm^2) and WOG 25.07 (309.861 μm^2) ($P< 0.05$). Previous studies have shown that larger metal mass volume at the maximum stress point of NiTi instruments affect the cyclic flexural fatigue resistance (SATTAPAN et al., 2000; GRANDE et al., 2006; PLOTINO et al., 2009; CAPAR et al., 2015a; KAVAL et al., 2016). However, the UNC 25.06 presented lower cross-sectional area than REC 25.08 and also lower cyclic flexural fatigue resistance. Therefore, the different thermal treatments among them may influenced on their mechanical properties.

All the instruments used in this study are manufactured from different thermal treatments of NiTi alloys. The thermal treatments assist the control of transition temperatures of NiTi alloy and induce a better arrangement of the crystal structure (GAO et al., 2012; SHEN et al., 2013; BRAGA et al., 2014). Previous studies showed that the thermal treatments can increase the percentage of martensite phase or R-Phase (which is known to be more flexible than austenitic NiTi), increasing the flexibility and reducing the risk of instrument separation under high stress (GAO et al., SHEN et al., 2012; SHEN et al., 2013; BRAGA et al., 2014; PLOTINO et al., 2014a; ELSAKA et al., 2016; DE-DEUS et al., 2017; TOPÇUĞOLU et al., 2017). Our results showed that PDR 25.06, which are manufactured by controlled memory technology, had a higher cyclic flexural fatigue resistance than all the instruments tested (P<0.05). The REC 25.08 had higher cyclic flexural fatigue resistance than UNC 25.06 (P<0.05), and the RB 25.08 showed higher time to fracture than WOG 25.07 (P<0.05). It is likely that the different thermal treatments among them could result in different martensitic phase transformations and could induce different dissipations of the energy required for crack formation and/or propagation during cyclic flexural fatigue testing (SHEN et al., 2013). The results of this study are in agreement previous studies that showed that instruments manufactured with controlled memory technology are likely more cyclic flexural fatigue resistant - and more flexible - than those manufactured from Blue (PEREIRA et al., 2015; DE VASCONCELOS et al., 2016), Gold (KAVAL et al., 2016; DE MENEZES et al., 2017; YILMAZ et al., 2017; KAVAL et al., 2017) , M-Wire (PONGIONE et al., 2012; PEDULLA et al., 2016; SILVA et al., 2016b; YILMAZ et al., 2017) and conventional NiTi alloys (CAPAR et al., 2015b; SILVA et al., 2016b; KAVAL et al., 2016; TOPÇUOĞLU et al., 2016).

The torsional test was performed by clamping 3 mm of the instrument tip. In addtion, a sumplementary examination was performed by SEM in D3 of each instrument and measured the area in software (AutoCAD; Autodesk Inc, San Rafael, CA) before the torsional test (KIM et al., 2012; PEDULLA et al., 2016). Our results showed that PDR 25.06 had the lowest torsional strength, greater angular rotation to fracture and lowest cross-sectional area than all the instruments tested (P<0.05). The UNC 25.06 and REC 25.08 presented closer values between them as well as RB 25.08 and WOG 25.07 (P>0.05). NiTi instruments with smaller cross sectional areas generally present lower torsional stiffness and higher angle of rotation to fracture (MELO et al., 2008; ZHANG; CHEUNG; ZHENG, 2011; KIM et al., 2012; NINAN; BERZINS, 2013; SILVA et al., 2016b; PEDULLA et al., 2016). Additionally, the different cross-sectional designs and core diameters promote different torsional stress distribution behaviors, which could affect the susceptibility to fatigue (MELO et al., 2008; ZHANG; CHEUNG; ZHENG, 2011; BAEK et al., 2011; NINAN; BERZINS, 2013; KAVAL et al., 2016). Lastly, the different thermal treatments of the NiTi alloys among the instruments have a strong influence in our results. Probably, the results of PDR 25.06 could be explained because of the controlled memory technology used in manufacturing process. Previous studies that showed that controlled memory technology induce a high flexibility, which favor a lower torsional strength and higher angular rotation until fracture than instruments manufactured from Blue, Gold and M-Wire treatment (SHEN et al., 2013; PEREIRA et al., 2015; PEDULLA et al., 2016; KAVAL et a., 2016; LO SAVIO et al., 2016).

The SEM analysis showed the typical topographic features of cyclic flexural and torsional fatigue for the all the reciprocating files. After the cyclic flexural fatigue test, all of the instruments evaluated showed crack initiation areas and overload zones, with numerous dimples spread on the fractured surfaces. After the torsional test, the fragments showed concentric abrasion marks and fibrous dimples at the center of rotation (KIM et al., 2012; PEDULLA et al., 2016; SILVA et al., 2016a; SILVA et al., 2016b).

Despite the advances on the metallurgy and kinematics of the engine-drive NiTi instruments, some authors has been recommended the glide path preparation before use rotary and/or reciprocating instruments for the reduction of the mechanical stress of instruments, which reduces the incidence of instruments fractures (BERUTTI et al., 2009; USLU et al., 2017). The pathfinding instruments are susceptible mainly to torsional failure in the constricted root canals

they are used at the beginning of the preparation (DE-DEUS et al., 2016; ARIAS et al., 2016).

In this study, the torsional fatigue resistance (maximum torsional strength and angle of rotation) to fracture was evaluated of seven rotary pathfinding instruments. The results revealed that Logic 25.01 had significantly higher torsional strength values when compared with all the groups ($P<0.05$). However, the Logic CM size 25.01 taper had significant differences only when compared with Hyflex GPF 15.01 and 15.02

(P<0.05). There was no significant difference (P<0.05) relative to the torsional strength among Logic CM 25.01, Proglider 16.02, Mtwo 10.04 and GPF CM 20.02 groups.

It was difficult to make comparisons among the different instruments, because of differences in the type of alloy, cross section, tip size and taper which affected the torsional properties of NiTi instruments (NINAN; BERZINS, 2013; SHEN et al., 2013; PEDULLA et al., 2016; SILVA et al., 2016; KAVAL et al., 2016; ACOSTA et al., 2017). Therefore, a supplementary examination was performed, the cross-sectional configuration of the instruments were captured at D3 by SEM and the area measured by means of software (AutoCAD; Autodesk Inc, San Rafael, CA, USA) (KIM et al., 2012; PEDULLA et al., 2016). The cross-sectional design and diameter of core affect the torsional fatigue resistance of NiTi instruments (BAEK et al., 2011; ZHANG et al., 2011; KIM et al., 2012; NINAN; BERZINS, 2013; PEDULLA et al., 2016; KWAK et al., 2016). The instruments used in this study presented the following cross-sectional design of the: a convex triangular (Hyflex GPF 15.01), S-shaped (Mtwo 10.04), and quadrangular (Logic 25.01, Logic 25.01 CM, Proglider 16.02; Hyflex GPF CM 15.02 and 20.02). The Hyflex GPF 15.01 and Hyflex GPF CM 15.02 had the smallest area (14.491 μm^2 and 24.699 μm^2, respectively) followed by Proglider (30.823 μm^2), Hyflex GPF 20.02 (33.311 μm^2), Mtwo 10.04 (36.890μm^2), Logic 25.01 and Logic CM 25.01 (42.469 μm^2 and 42.472 μm^2).

Although the Logic 25.01 and Logic CM 25.01 taper have the same tip size, taper, cross-sectional design and similar cross-sectional area, the results revealed there was a significant difference regarding the torsional strength between them (P<0.05). This could be explained because of the different NiTi alloys used during the manufacturing process. Previous studies have shown that controlled memory wire has greater flexibility and lower torsional load than conventional NiTi Wire (PEREIRA et al., 2012; SHEN et al., 2013; PEDULLA et al., 2016; KAVAL et al., 2016; KWAK et al., 2016; ACOSTA et al., 2017). In addition, the Hyflex GPF 15.01 and 15.02 had the lowest torsional strength values when compared with the other groups (P<0.05) and also the smallest cross-sectional areas. Previous reports have indicated that instruments with smaller cross-sectional area tend to present lower metal mass (diameter of core, tip size and taper), providing lower torsional strength (BAEK et al., 2011; ZHANG et al., 2011; KIM et al., 2012; PEDULLA et al., 2016; KWAK et al., 2016). Moreover, the cross-sectional design modified the stress distribution under torsion, which affected the torsional strength and susceptibility to fracture (ZHANG et al., 2011; EL-ANWAR et al., 2016).

In relation the angle of rotation, the Logic CM 25.01 had the highest deformation capacity when compared with the other groups (P<0.05) followed by GPF 15.01, Hyflex GPF CM 15.02, Hyflex GPF CM 20.02, and Logic 25.01. The Proglider and Mtwo 10.04 had significantly lower values than the other groups (P<0.05). Additionally, the Proglider had a significantly lower angle of rotation values than Mtwo 10.04. It is important to note that the instruments with smaller taper diameters had the highest angles of rotation to fracture. The results were in agreement with the studies of De-Deus et al. (2016) and Arias et al. (2016) that revealed that instruments with a larger taper are more susceptible to fracture.

The higher angle of rotation of Logic CM 25.01, Hyflex GPF 15.02 and 20.02 could be influenced by the special thermal treatment of the NiTi used in manufacturing process, which increased the flexibility and deformation capacity of controlled memory instruments (PEREIRA et al., 2012; PETERS et al., 2012; SHEN et al., 2013; KWAK et al., 2016; KAVAL et al., 2016; PEDULLA et al., 2016; ACOSTA et al., 2017). However, controlled memory instruments are not always more flexible than instruments manufactured from conventional NiTi or M-Wire, because other factors, such as cross-sectional design, core mass, taper and tip size (SHEN et al., 2013; NINAN; BERZINS, 2013; KWAK et al., 2016) need to be taken in account, which could explain the high angle of rotation values of Hyflex GPF 15.01.

The SEM analysis revealed the typical topographic features of torsional fatigue for all instruments. After the torsional test, the fragments demonstrated the typical features of shear failure, including concentric abrasion marks and fibrous microscopic dimples at the centre of rotation (KIM et al., 2012; PEDULLA et al., 2016). In addition, in the side view, it was possible to note the deformation of the spiral flutes of the instruments, particularly in those that had a higher angle of rotation to fracture.

The instruments features (cross-sectional design, taper, tip size and thermal treatment) should be considered to choose the engine-driven NiTi instruments for root preparation (GAGLIARDI et al., 2015; MARCELIANO-ALVES et al., 2015; PINHEIRO et al., 2017; SILVA et al., 2017). Some authors have been recommended to use less tapered instruments to ensure a conservative root canal preparation (RUNDQUIST; VERSLUIS, 2006; COHENCA et al., 2013; GERGI et al., 2015), preserving more remaining dentin thickness, which can favor less risk of vertical tooth fracture or strip perforation (KISHEN, 2006; RUNDQUIST; VERSLUIS, 2006; TANG; WU; SMALES, 2010; ADORNO, 2013). In addition, there are an worldwide tendency to use thermally-treated instruments for root preparation of curved and flattened canals, which reduces the risk of canal transportation and favor to preserve remaining dentin thickness (MARCELIANO-ALVES et al., 2015; PINHEIRO et al., 2017). Probably, the use of less tapered thermally-treated instruments could provide safer root canal preparation, ensuring less risk of instruments fractures and conservative root preparation. However, future studies should be performed to evaluate the impact of conservative preparation for the effectiveness of root canal irrigation techniques and the antimicrobial effect.

Despite the studies have been evaluated the cyclic flexural and torsional fatigue resistance in separated tests, it well known that during root canal preparation the instruments fractures can occur due to a association of both fatigue (SATTAPAN et al., 2000; PEDULLA et al., 2016). Therefore, in future studies, it would be important to develop new methodologies trying to reproduce the real mechanical behaviour of the NiTi instruments during root canal preparation.

The cyclic flexural and torsional fatigue resistance of the NiTi instruments should be considered to choose the suitable instruments for root canal preparation of curved and/or constricted canals. Therefore, the results of this study have an important clinical significance, allowing to speculate the mechanical behavior of the NiTi instruments on the different root canal anatomies. Instruments that present high cyclic flexural fatigue resistance indicate that they are very flexible and safe for preparing curved root canals, and probably cause fewer undesirable changes in the root canal anatomy. On the other hand, in cases of constricted canals, it is suitable to use instruments that present greater torsional resistance because they are submitted to higher torsional stress, which could cause plastic deformation or instrument separation.

The results of this study showed that the cyclic flexural and torsional fatigue resistance of engine-driven NiTi instruments are affected by cross-sectional design, tip size, taper and thermal treatment. However, the controlled memory technology seems to be a key factor to provide greater flexibility for NiTi instruments.

4 CONCLUSION

4 CONCLUSION

- The PDR 25.06 had the highest cyclic flexural fatigue resistance values and angular rotation to fracture in comparison with REC 25.08 and UNC 25.07. However, REC 25.08 showed higher torsional strength to fracture.

- The cross-sectional design, taper and thermal treatments, had a significant influence on the mechanical properties of the NiTi instruments. Our results showed that PDR 25.06 had highest cyclic flexural fatigue resistance and highest angular rotation values to fracture, compared with RB 25.08 and WOG 25.07. However, RB 25.08 and WOG 25.07 showed higher torsional resistance to fracture than PDR 25.06.

- The Logic 25.01 instrument made of conventional NiTi alloy had the highest torsional strength of all instruments tested. In addition, the ProGlider instrument manufactured from M-Wire alloy had the lowest angle of rotation to fracture in comparison with the other instruments.

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APPENDIXES

APENDIX A - DECLARATION OF EXCLUSIVE USE OF THE ARTICLE IN THESIS

We hereby declare that we are aware of the article *Cyclic and torsional fatigue resistance of reciprocating single files manufactured by different Nickel-titanium alloys* will be included in the Thesis of the student (Murilo Priori Alcalde) and may not be used in other works of Graduate Programs at the Bauru School of Dentistry, University of São Paulo.

Bauru, January 1st 2018

Murilo Priori Alcalde
Author
signature

Mario Tanomaru-Filho
Author
signature

Clovis Monteiro Bramante
Author
signature

Marco Antonio Hungaro Duarte
Author
signature

Juliane Maria Guerreiro-Tanomaru
Author
signature

Jader Camilo-Pinto
Author
signature

Marcus Vinicius Reis Só
Author
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Rodrigo Ricci Vivan
Author
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APENDIX B - DECLARATION OF EXCLUSIVE USE OF THE ARTICLE IN THESIS

We hereby declare that we are aware of the article *Cyclic fatigue and torsional strength of three different thermally treated reciprocating nickel-titanium instruments* will be included in the Thesis of the student (Murilo Priori Alcalde) and may not be used in other works of Graduate Programs at the Bauru School of Dentistry, University of São Paulo.

Bauru, January 1st 2018

Murilo Priori Alcalde Author	signature
Bruno Carvalho de Vasconcelos Author	signature
Mario Tanomaru-Filho Author	signature
Clovis Monteiro Bramante Author	signature
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Juliane Maria Guerreiro-Tanomaru Author	signature
Jader Camilo-Pinto Author	signature
Rodrigo Ricci Vivan Author	signature
Marcus Vinicius Reis Só Author	signature

APENDIX C - DECLARATION OF EXCLUSIVE USE OF THE ARTICLE IN THESIS

We hereby declare that we are aware of the article *Torsional fatigue resistance of pathfinding instruments manufactured from several NiTi alloys* will be included in the Thesis of the student (Murilo Priori Alcalde) and may not be used in other works of Graduate Programs at the Bauru School of Dentistry, University of São Paulo.

Bauru, January 1st 2018

Murilo Priori Alcalde Author	signature
Bruno Carvalho de Vasconcelos Author	signature
Mario Tanomaru-Filho Author	signature
Clovis Monteiro Bramante Author	signature
Marco Antonio Hungaro Duarte Author	signature
Rodrigo Ricci Vivan Author	signature
Marcus Vinicius Reis Só Author	signature

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